

# OPERATIVE DENTISTRY

Mar/Apr 2020

Volume 45

Number 2

113–228



Downloaded from <https://prime-pdf-watermark.prime-prod.pubfactory.com/> at 2025-08-29 via free access

OPERATIVE  
DENTISTRY

# OPERATIVE DENTISTRY

Volume 45 / Number 2  
March/April 2020

[www.jopdent.org](http://www.jopdent.org)

## Aim and Scope

Operative Dentistry publishes articles that advance the practice of operative dentistry. The scope of the journal includes conservation and restoration of teeth; the scientific foundation of operative dental therapy; dental materials; dental education; and the social, political, and economic aspects of dental practice. Review papers, book reviews, letters and classified ads for faculty positions are also published.

## Subscriptions: Fax 317-852-3162

Current pricing for individual, institutional, and dental student subscriptions (both USA and all other countries) can be found at our website: [www.jopdent.org](http://www.jopdent.org), or by contacting our subscription manager via email at [editor@jopdent.org](mailto:editor@jopdent.org). Payment must be in USD and accompany orders. Online payment by credit card (American Express, Discover, Mastercard, and Visa) is available on our website.

Operative Dentistry (ISSN 0361-7734) is published bimonthly by Operative Dentistry, Inc, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186. Periodicals postage paid at Indianapolis, IN and additional mailing offices. Postmaster: Send address changes to: Operative Dentistry, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186.

## Author Instructions

Please refer to author instructions at [www.jopdent.org](http://www.jopdent.org) in the preparation of manuscript submissions and for journal policies.

## Journal Policies

The Operative Dentistry Policy Manual which details journal policies, including late fees and claims, is available online at:

<https://www.jopdent.com/journal/policies.pdf>

## Permissions

For permission to reproduce material from Operative Dentistry please apply to Operative Dentistry at the Editorial Office address or via email at [editor@jopdent.org](mailto:editor@jopdent.org).

## Online Access

Register for online access, manage subscriptions, save favorite articles and searches, get email alerts, and more at:

<http://www.jopdentonline.org/action/registration>

## Editorial Board

Reviewer names available at: [www.jopdent.com/journal/editorial\\_board.html](http://www.jopdent.com/journal/editorial_board.html)

We thank all our reviewers for their time and dedication to Operative Dentistry.

## On The Cover

"Mexican Petunia", Casa Tropicana, Kota Damansara, Selangor, Malaysia. Photo provided by Sylvia Western J of Petaling Jaya, Selangor, Malaysia. Photo taken with a Samsung Galaxy S8, 4.2mm f/1.7 1/250 sec. ISO-40. © Operative Dentistry, Inc."

We welcome the submission of pictures for consideration for use on the cover of Operative Dentistry! All photographs should be submitted via the forms at: <https://www.jopdent.com/journal/journal.html>

## Editorial Office

The views expressed in Operative Dentistry do not necessarily represent those of the academies or the editors.

Operative Dentistry  
Indiana University School of Dentistry, Room S411  
1121 West Michigan Street, Indianapolis, IN 46202-5186  
Phone 317-350-4371, Fax: 317-852-3162  
<http://www.jopdent.org>

## Editorial Staff

**Editor:** Jeffrey A Platt

**Office Manager:** Erin Cody

**Editorial Assistant/CDE Director:** Kevin B Matis

**Associate Editors:** N Blaine Cook, Kim E Diefenderfer, So Ran Kwon, Camila Sabatini

**Managing Editor:** Timothy J Carlson

**Asst Managing Editors:** Paul Hasagawa, Barry O Evans, Lawrence Vanzella

**Statistical Consultant:** George J Eckert



# Conservative Tooth Preparation, Minimal Intervention, and/or Narrow-parallel Preparations: A Narrative

John W Osborne, DDS, MSD

## INTRODUCTION

During my senior year in dental school (1963), I heard a lecture by Dr Miles Markley and a few months later another by Dr Wilmer Eames. Both touched on the subject of smaller posterior preparations that did not use GV Black's standard one-third intercuspal width for posterior teeth. I had done a little reading in GV Black's *Operative Dentistry* during the preclinical operative course. However, I learned early that in the first year of dental school one does not ask questions. Fortunately, I was able to connect with Dr Eames and we had a discussion on cavity preparation.

Black's original ideas on class I and II cavity preparations were to have the occlusal preparation at least one-third isthmus width and the depth at the dentin–enamel junction.<sup>1</sup> In the class II preparation, the margin of the proximal box was carried out just past the adjacent tooth.<sup>2</sup> This was called “extension for prevention.”<sup>3</sup> As a fourth-year student, I was aware that patient factors, areas of decalcification of the enamel, and interception of the caries process changed where margin could be placed.

After graduation, I was in the postgraduate program at Indiana. I was enrolled in this program with majors in operative dentistry and dental materials. I also maintained a part-time private practice. A few months into the graduate program, I found an article by Vale<sup>4</sup> that indicated one-quarter

isthmus prepared teeth were almost twice as strong as teeth with a one-third isthmus width.

I was HOOKED!

## I HAD TO FIND A WAY TO HAVE STRONGER TEETH AFTER CAVITY PREPARATION

I began preparing extracted teeth with different burs looking for a better way to achieve narrower preparations than the one-third isthmus I had been taught. I had discussions with faculty and read the literature constantly. My research on narrower preparations had many start/stop periods in those two years. I prepared teeth, both typodont and extracted, multiple times. I sectioned teeth and photographed the results.

I concentrated on the use of divergent burs in the second year. I was able to have a manufacturer make special divergent burs. I had a difficult time with preparations using divergent burs. If I lifted the bur out of the preparation, I had a parallel preparation. Following the undulating fissures of a molar enamel surface with the neck of the bur as a guide was exceedingly difficult. Even with experience, this did not get easier. I tried to start at the deepest portion of the tooth normally used in cavity preparation and go straight out the grooves. That was not a good solution with different widths of the preparation at the occlusal surface. I found I needed smaller and less divergent burs for lower premolars, especially lower first premolars. Also, if the opposing wall was touched with a bur you had a flared wall that was

almost impossible to correct. After a couple months, I finally abandoned divergent burs.

Then, I tried cylindrical burs. How easy they were. The #56 (1.0 mm diameter) seemed to be a perfect fit. Go to your depth just above the dentin–enamel junction and cut out the grooves and fissures. Where caries were found, remove the decay and widen the preparation as necessary. This narrow-parallel preparation was so easy that I almost could not believe it. If I could do this with so little difficulty, I knew a student could learn it readily. I conducted limited trials on strength and was satisfied these narrow preparations did not substantially weaken the tooth.

However, I recognized the problem of having an acute angle of restorative material at the cavosurface margin. Our metallurgical professor had discussed the problem of greater fracture rates of brittle materials like amalgam when they were at an acute angle. By using the narrow-parallel walled preparation, I was in a bind. I finally gave up on resolving this acute angle dilemma and, when indicated, continued preparing patients' teeth with the narrow-parallel preparation.

In 1966, I had been on faculty at SUNY Buffalo for one year and was asked to be the director of the preclinical operative course. A new preclinical laboratory was to be finished in the fall and my only request was to install high-speed units because these had not been in the plans.

Several faculty members and I prepared a detailed manual and made plaster and typodont models for the students. We began teaching the narrow-parallel width preparations described above. The first three typodont preparations the students learned involved class I and II variations of the narrow-parallel preparations to open the fissures. We continued that philosophy through to the students' graduation. Interestingly, at year 4, I was asked by the New York State Dental Board to explain our narrow-parallel preparation. After my presentation, their question to me was why don't other schools teach this? I had and continued to share this technique with faculty from other dental schools. I ran the operative program at Buffalo until I left in 1973.

My next position was at my old school, Indiana. I was in the dental materials department in charge of clinical research. I was asked to take over a large portion of the department's lectures to dental, hygiene, and graduate students. It was an exciting time in dental materials. Formulas for dental amalgam had radically changed, and clinical research was needed to find the best amalgam alloys.

Composite resins were under accelerated development, and glass ionomers had just been introduced. The department was able to publish many papers in the next years.

### AS FATE WILL HAVE IT

A coauthor of our clinical trials wanted to do some additional work on the amalgam clinical studies. Dr Elliot Gale, PhD psychologist, felt other factors could skew the data. He designed our clinical studies and calculated the statistics. He also told us how many restorations were needed to be placed in a study. Therefore, when he spoke, we listened. He chose a clinical trial where I had placed amalgams during 1974-1975. I had used standard preparations and the narrow-parallel walled preparation. We evaluated 12 amalgam alloys, tooth position, and width of preparations. Three different preparations were 1) one-quarter or less, 2) one-third width, and 3) greater than one-third. We evaluated the amalgam restorations at two different times. At two years, we evaluated 429 amalgam restorations.<sup>5,6</sup> The data indicated that the narrow-parallel prepared teeth with an amalgam exhibited less marginal fracture than amalgams in wider preparations. At the 13- to 14-year evaluation, we examined 193 amalgam restorations.<sup>5,7</sup> Again, results indicated the narrower amalgams exhibited significantly less marginal fracture than in wider preparations. We noted that gamma-2-containing amalgams, which usually show high rates of marginal breakdown in the narrow-parallel preparation, were rated the same as the best high copper amalgams. We also found that the difference between width in upper bicuspid showed a highly significant difference. Both studies were published in the *Journal of Dental Research*.<sup>6,7</sup>

That "feared" acute angle at the cavosurface margin of tooth played an insignificant role in the fracture of amalgam margins. Narrowness seemed to be the key. We found no recurrent decay in any narrow-parallel preparations. On reviewing the data, Dr Gale said, "See, that narrow prep of yours skewed the data. All the amalgams looked the same."

I became a faculty member at SUNY Stony Brook in 1978 and continued the narrow-parallel preparation saga for nine years.

I have two theories why these narrow-parallel preparations have such superior results. First, the teeth prepared in this manner do not flex as much as when undercut or with wider preparations. Second, in teeth with narrow-parallel preparations, no restoration is touched by the opposing dentition.

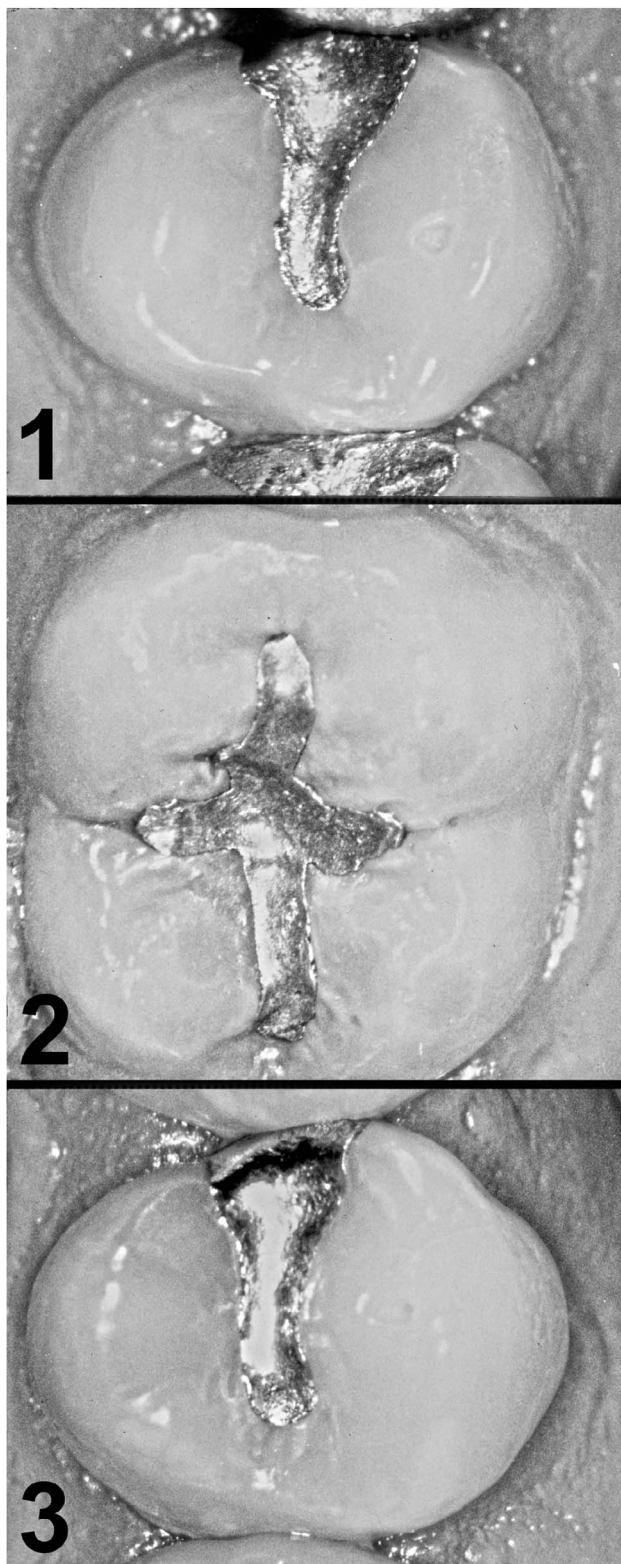


Figure 1. Thirteen-year-old amalgam Distal-Occlusal narrow-parallel occlusal blended into distal portion.  
 Figure 2. Five-year-old Occlusal amalgam narrow-parallel preparation.  
 Figure 3. Thirteen-year-old Mesial-Occlusal amalgam narrow-parallel occlusal blended into mesial portion.

One thing still fascinates me about these amalgam restorations in the narrow-parallel preparations. I have never seen one replaced. Dentists love to replace fillings. Of the hundreds I have placed and the years I have observed them, you would think I would have seen one replaced. But none, that is astonishing.

### SUMMARY

During my postgraduate years after dental school, I wanted to have a better way of preparing teeth than the 60+-year-old preparation I was taught in preclinical labs. I developed a procedure to prepare teeth using small diameter cylindrical burs with the expressed desire to have stronger teeth. I started using this technique of opening fissures with narrow burs in my patients. The early caries situation was the best time to use this procedure. In 1966, I began teaching a narrow-parallel preparation to dental students at SUNY Buffalo. This philosophy was carried through until their graduation. The faculty started using these preparations and an attitude of prevention was more prevalent. Clinical trials demonstrated the significant positive affect of narrow-parallel preparation for amalgam restorations. A prepared cavity for posterior teeth using a small diameter cylindrical bur for the narrow-parallel preparation exhibited significantly less fracture at the margins of amalgams. The positive effect of this narrow-parallel preparation to open fissures was not only longer lasting restorations, but a new excitement that we could provide a better service.

### Acknowledgement

This paper is dedicated to the memory of Dr Elliot N Gale.

### Conflict of Interest Declaration

The Author of this manuscript certifies that he has no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

(Accepted 5 August 2019)

### REFERENCES

1. Black GV (1914) A work on operative dentistry. In: The Technical Procedure on Filling Teeth, volume 2 **2nd edition** Medico-Dental Publishing Company, Chicago, IL 110.
2. Black GV (1914) A work on operative dentistry. In: The Technical Procedure on Filling Teeth, volume 2 **2nd edition** Medico-Dental Publishing Company, Chicago, IL 140.

3. Black GV (1914) A work on operative dentistry. In: The Technical Procedure on Filling Teeth, volume 2 **2nd edition** Medico-Dental Publishing Company, Chicago, IL 142.
4. Vale WA (1956) Cavity preparation *Irish Dental Review* **2** 33-34.
5. Mjor I (2003) Hollenbach Memorial Prize John Osborne *Operative Dentistry* **28(2)** 208.
6. Osborne JW & Gale EN (1981) Failure at the margins of amalgams as affected by cavity width, tooth position, and alloy selection *Journal of Dental Research* **60(3)** 681-685.
7. Osborne JW & Gale EN (1990) Relationship of restoration width, tooth position, and alloy of fracture at the margins of 13 to 14 year-old amalgams *Journal of Dental Research* **69(9)** 1559-1661.

# Multidisciplinary Management and Pulp Vitality Preservation of a Tooth With Extensive Iatrogenic Furcal Root Perforation and Biologic Width Violation

D Angerame • M De Biasi • V Franco • L Generali

## Clinical Relevance

Knowledge is lacking about the possibility of orthodontically moving a root-repaired tooth whose pulp has maintained vitality. The present case report provides an example of the successful management of a molar tooth with severe iatrogenic damage and multiple negative prognostic factors.

## SUMMARY

**This article describes the case of a vital molar tooth with a vast furcal iatrogenic root perforation and biologic width violation, which was successfully managed by a multidisciplinary**

**approach aimed at preserving pulp vitality. The root perforation was cleaned and then sealed with mineral trioxide aggregate, which was positioned onto the pulp at the canal orifices. After one month, the patient was not reporting symptoms, and the tooth was positively responding to the thermal test. The tooth was orthodontically extruded, subjected to minimally invasive crown lengthening, and prepared to receive a full-crown restoration. Radiotransparent composite resin was chosen as a permanent restorative material to better monitor possible endodontic complications at the coronal level. The patient's tooth was followed up for eight years uneventfully. The present case is an example of the possibility to subject a root-repaired tooth with fully formed apices to conservative yet complex multidisciplinary treatment while maintaining pulp vitality.**

\*Daniele Angerame, MD, DDS, University Clinical Department of Medical, Surgical and Health Sciences, University of Trieste, Trieste, Italy

Matteo De Biasi, DDS, MS, PhD, University Clinical Department of Medical, Surgical and Health Sciences, University of Trieste, Trieste, Italy

Vittorio Franco, DDS, private practice, London, United Kingdom

Luigi Generali, DDS, Department of Surgery, Medicine, Dentistry and Morphological Sciences with Transplant Surgery, Oncology and Regenerative Medicine Relevance (CHIMOMO), University of Modena and Reggio Emilia, Modena, Italy

\*Corresponding author: Strada di Fiume 447, Trieste 34149, Italy; e-mail: d.angerame@fmc.units.it

<https://doi.org/10.2341/19-026-T>

## INTRODUCTION

Root perforations are unfortunate mechanical or pathologic complications that involve the direct communication between the endodontic space and the external root surface.<sup>1</sup> The prognosis of a tooth affected by a perforation is related to the size and location of the lesion, the elapsed time from the initial damage, and the possibility that the defect can be effectively cleaned and sealed.<sup>2</sup> The root perforation may cause complications that require extraction of the affected tooth; indeed, iatrogenic perforations and stripping are the reason for extraction in up to 4.2% of endodontically treated teeth.<sup>3</sup>

Though the prognosis for coronally positioned perforations was considered poor in the past,<sup>4</sup> especially in the case of furcal localization, the development of bioactive materials in the past three decades, such as mineral trioxide aggregate (MTA), has improved the management of root perforations.<sup>5</sup> MTA is generally accepted as the root repair material of choice for perforations within bone, thanks to its sealing ability,<sup>6</sup> marginal adaptation,<sup>7</sup> and capacity to promote bone and cementum formation.<sup>8</sup> A recent systematic review reported that the nonsurgical repair of root perforations with MTA cement can reach success rates up to 80.9%.<sup>9</sup>

In addition, the properties and versatility of MTA cement have aroused the interest of clinicians and researchers in the management of vital permanent teeth with cariously exposed pulp.<sup>10</sup> Nowadays, increasing attention is given to minimally invasive endodontic treatments, such as partial and total coronal pulpotomy associated with MTA positioning, as an alternative to conventional root canal treatment, even in teeth with mature apices.<sup>11,12</sup> A systematic review on the topic reported that the two-year success rate of full pulpotomy executed on cariously exposed permanent posterior teeth can exceed 90%.<sup>12</sup>

Extensive caries and other sources of damage that invade the biologic width may cause gingival inflammation, clinical attachment loss, and alveolar bone resorption.<sup>13</sup> Biologic width can be recovered by performing periodontal surgery, orthodontic extrusion, or a combination of both approaches.<sup>14</sup> The coexistence of severe damage and negative prognostic factors can diminish the probability of endodontic success. This case report presents the successful multidisciplinary management and eight-year follow-up of a mandibular molar with extensive iatrogenic perforation, treated with simultaneous perforation repair and full pulpotomy, and violation

of the biologic width recovered by orthodontic extrusion and minimally invasive crown lengthening.

## CLINICAL CASE REPORT

In May 2010, a 26-year-old white man in good general health was referred by a private practitioner to receive root perforation repair and root canal treatment of the lower right first molar (tooth 30), which was originally affected by asymptomatic disto-occlusal decay that apparently did not involve the pulp. During access cavity preparation, the pulp was exposed and vast perforation occurred on the pulp chamber floor while the practitioner attempted to locate the canal orifices. When asked about the preoperative endodontic diagnosis, the dentist declared that he did not detect any clinical evidence of pulpal pathology. Given the aforementioned, the pulp was assumed to be preoperatively healthy. The patient provided radiographic documentation consisting of a preoperative periapical radiograph and an intraoperative radiograph with the perforation already visible (Figure 1A, B).

The tooth was still responding normally to the pulp cold thermal test, which was carried out by applying a cotton pellet soaked with ethyl chloride on the buccal surface of the tooth. The temporary filling was removed, and a composite resin buildup restoration was performed before isolating the tooth with a rubber dam. Even under operating microscope magnification (M525, Leica Microsystems CMS GmbH, Mannheim, Germany), the canal orifices were barely detectable and poorly accessible. Since the pulp at the canal orifices and the periodontium were not bleeding, a second line of treatment was considered that consisted of direct pulp capping and concurrent perforation repair with MTA.

The accessible endodontic space was disinfected with gentle continuous rinses of 2.5% sodium hypochlorite solution for 10 minutes. A considerable amount of MTA cement (ProRoot MTA, Dentsply Tulsa, Tulsa, OK, USA) was placed into the defect with the aim of sealing the perforation and the canal orifices. To achieve immediate coronal seal, a hard-set calcium-hydroxide base (Life, Kerr, Bioggio, Switzerland) was applied to the MTA cement and the coronal access was filled with composite resin (Filtek Flow and Filtek Z250, 3M ESPE, St Paul, MN, USA). The adhesive procedures entailed enamel and dentin etching with 37% phosphoric acid (Total Etch, Ivoclar Vivadent, Schaan, Lichtenstein) for 15 seconds followed by application of the All-Bond 3

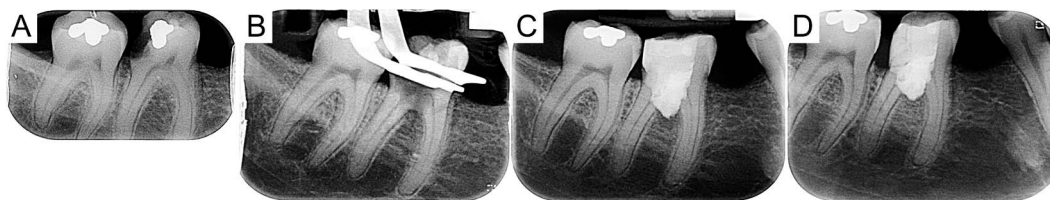


Figure 1. Radiographic documentation provided by the patient at the first visit. (A): Preoperative radiograph and (B) intraoperative periapical radiograph where the iatrogenic furcal perforation of tooth 30 is clearly visible. (C): Immediate postoperative radiograph; after cleaning and disinfection, mineral trioxide aggregate was placed to seal the defect and cover the vital pulp at the canal orifices. (D): One-month control radiograph with no signs of complications.

system (Bisco, Schaumburg, IL, USA) according to the manufacturer's instructions. Leaving the preparation visibly moist, All-Bond parts A and B were mixed in a 1:1 ratio, applied to the entire tooth preparation for 10 seconds, and gently air dried for 15 seconds. Then, All-Bond resin was applied to the preparation, air thinned, and light cured for 10 seconds with a light-emitting diode lamp at 1500 mW/cm<sup>2</sup> (Radii plus, SDI, Melbourne, Australia). The same lamp was used to light-cure the composite resins. A radiographic control revealed extrusion of the MTA cement in the furcation area (Figure 1C).

The tooth was checked weekly for one month and received an occlusodistal patchwork composite restoration to address a partial fracture of the pre-endodontic restoration (Figure 1D). As the patient reported no pain or dysfunction at the recall visits, he was offered the possibility to receive an orthodontic extrusion combined with a surgical biologic width realignment and an indirect restoration. After local anesthesia was administered, a mini-screw (Imtec ORTHO Implant, 3M Unitek, Monrovia, CA, USA) was placed between the first molar and the first premolar of the opposing jaw (Figure 2A), and fibrotomy was performed on tooth 30. Then, an orthodontic buccal tube with hook (Morelli Ortodontia, Sorocaba, Brazil) was bonded to tooth 30 with dual cure resin cement (Heliosit Orthodontic, Ivoclar Vivadent). The extrusion force was exerted by using a rubber band connecting the head of the screw and the buccal tube (Figure 2B). Two months later, the extrusion was deemed satisfactory (Figure 2C, D). A biologic width realignment was performed to reestablish harmonious contour of soft and hard tissue, remodeling only the distal bone while preserving the furcation area (Figure 3A). The initial keratinized gingiva was 2- to 3-mm thick. Under local anesthesia, the distal papilla was incised and displaced; then, a 3-mm-long releasing incision was made mesially (Figure 3A). The interproximal bone crest was remodeled with diamond burs with the aim of removing only what was strictly necessary to restore

the 3-mm distance from the restorative margins and reestablish the biologic width. The bone of the furcation area was fully preserved.

In the same appointment, a bonded retainer was placed on tooth 30 and tooth 31 (Figure 3B, C). The retainer was kept in place for two weeks to stabilize the result obtained with the orthodontic extrusion. In the absence of symptoms and with positive response to the pulp cold thermal test, the retainer was removed. Alginate impressions were taken to obtain a provisional acrylic resin crown. Then, tooth 30 was prepared with a feather-edge margin circumferentially. The crown was relined and finished so as to not interfere with the gingival healing and was cemented with eugenol-free cement (Temp Bond NE, Kerr) (Figure 3D, E). The early placement of the provisional crown served for both soft-tissue conditioning and orthodontic stabilization thanks to the occlusal contacts. After six weeks, the crown was relined again for soft-tissue conditioning and kept in place for six months.

The permanent crown was manufactured with composite resins for indirect restorations (Signum, Heraeus Kulzer, Hanau, Germany) and luted with self-adhesive resin cement (RelyX Unicem, 3M ESPE) (Figure 3F, G, and H) approximately one year after the pulp capping and perforation repair. For eight years, the patient was recalled annually for a clinical and radiographic assessment, and no complications occurred (Figure 4A, B).

## DISCUSSION

Repairing a root perforation can be a simple task with predictable outcome, as in the case of easily accessible small lesions. Conversely, the same procedure may require advanced instruments and skills when the perforation is vast or localized to the deep portions of the endodontic space. Other patient-related factors may contribute to treatment success, primarily compliance and the capability of being subjected to long operative sessions, especially under the operating microscope. When several operative

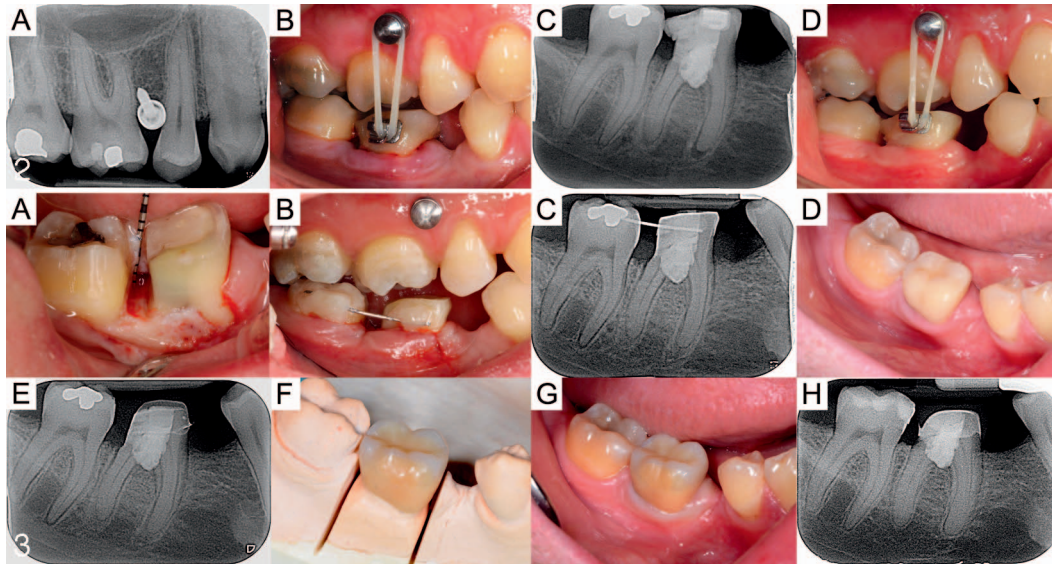


Figure 2. Phases of the orthodontic extrusion of tooth 30. (A): An orthodontic miniscrew was placed between tooth 3 and tooth 5. (B): The extrusion force was applied by a rubber band connected to a bonded hook after fiberotomy. (C and D): After two months, the extrusion was deemed sufficient.

Figure 3. (A): Minimally invasive surgical crown lengthening. (B): A bonded retainer was placed to maintain the achieved position. (C): Postsurgical radiograph. (D and E): The provisional crown of tooth 30 was kept in place for six months to allow soft-tissue stabilization. The composite crown on (F): the stone model and (G and H): immediately after placement.

difficulties coexist, a simplified but effective endodontic treatment may become the preferable option. Although pulpotomy has been advocated as an alternative to full root-canal treatment for limited circumstances,<sup>12</sup> its potential should be explored in a wider array of clinical situations because its success rate can be surprisingly high in selected cases.<sup>12</sup> It is surely known that complete root-canal treatment has predictable outcome on teeth with vital pulp.<sup>15</sup> Nevertheless, endodontically treated teeth are characterized by lower survival rates than vital teeth, with a 7:1 hazard ratio in molars.<sup>16</sup> A possible explanation to this finding may be the deprivation of defensive sensitive mechanisms, such as proprioceptive function,<sup>17</sup> damping property,<sup>18</sup> and tooth sensitivity. Considering the aforementioned aspects, there is an emerging conservative trend to preserve pulp vitality as much as possible.

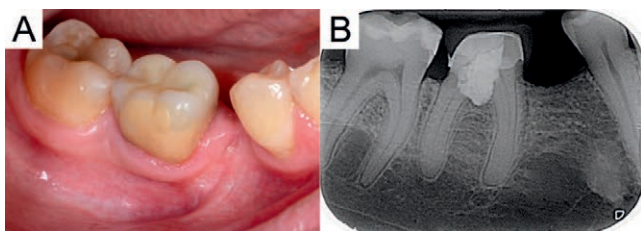


Figure 4. Eight-year clinical (A) and radiographic (B) appearance of tooth 30 after the multidisciplinary treatment.

To prevent an undesirable MTA extrusion in large perforations, the clinician should consider the internal matrix technique, which consists of intracanal placement of an extraradicular barrier.<sup>1</sup> Notwithstanding, the current case report demonstrates that, in the first place, an extensive MTA overfilling does not appear to interfere with bone healing and orthodontic movement and that, secondly, an orthodontic extrusion force may be applied to a tooth subjected to perforation repair and full pulpotomy without jeopardizing the outcome of the endodontic treatment. To the best of the authors' knowledge, even if the performance of MTA in static conditions is well known, no other examples of similar endodontic-orthodontic management attesting to the dynamic properties of MTA are available in the literature. Once set, MTA cement is a firm mass that can effectively adapt to the dentin substrate<sup>19</sup>; this compactness was sufficient to prevent fragmentation of the extruded material during the orthodontic extrusion. When radiographically monitoring the orthodontic extrusion of a tooth in which periapical complications are possible or even likely, as in the case being presented here, the radiolucency left by the root apices may mimic a periapical lesion of endodontic origin (Figure 3C). In these cases, the differential diagnosis should encompass both clinical tests and further periapical radiographs to monitor the radiolucency.

The endodontic management of a tooth with root perforation may be useful in light of eventual orthodontic and/or prosthetic treatments. In the present case, which involved the management of a strategically important tooth in a young adult patient, the endodontic treatment was performed to prepare for orthodontic extrusion, which, in turn, was aimed at the final restoration. In orthodontics, miniscrews can be used for temporary anchorage and removed after the desired movement has been achieved, representing a well-tolerated treatment option that does not require special compliance from the patient. Orthodontic forced tooth eruption is an alternative to crown-lengthening surgery because it exposes sound tooth structure for the placement of restorative margins, thereby preventing marginal bone loss and maintaining esthetics.<sup>20</sup> In the presented case, applying the bracket to the buccal side of the tooth also allowed for the correction of the Wilson curve, thus facilitating the final restoration in occlusion.

The idea of resecting the fibers of marginal periodontium in orthodontic cases is not new; indeed, as early as 1899, Angle<sup>21</sup> proposed gingival surgery in the cervical third of the root to increase tooth mobility and shorten the retention period. Severing the gingival fibers was intended to achieve periodontally accelerated orthodontic tooth movement and enhanced posttreatment stability.<sup>22</sup> We decided to perform fiberotomy with the aim of exposing the restorative margin and minimizing the risk of furcation defect. Since the distal margin was not satisfactorily exposed, a minimally invasive biologic width realignment was carried out to preserve the furcal bone.

Choosing a composite crown for the final restoration in the reported case was justified for several reasons. Generally, there is increasing interest in composite crowns as an alternative to traditional crown restorations.<sup>23–27</sup> In the past, composite crowns were seen as long-term temporary restorations, which are especially suitable for patients with uncertain prognosis because their esthetics and wear resistance are generally inferior to that of traditional ceramic prostheses.<sup>25,28</sup> Eventually, some researchers have started advocating their use as permanent restorations thanks to the evolution of dentinal adhesives, the development of resin composites with improved mechanical properties, the lower costs compared with metal-ceramic crowns, and the possibility of computer-aided design/computer-aided manufacturing production.<sup>28–30</sup> Composite full-coverage indirect restorations of adequate occlusal

thickness may be capable of high fatigue resistance,<sup>31</sup> which may be even better than modern ceramic materials for indirect posterior restoration.<sup>26,27</sup> Unfortunately, the published clinical studies on the performance of composite crowns are sporadic and dated; nonetheless, 96% and 88.5% survival rates have been reported after 3 and 5 years, respectively.<sup>25,32</sup> Moreover, there is no evidence on the clinical behavior of composite crowns fabricated and luted with modern materials and techniques; further research is necessary.

The choice of traditional acrylic resin for long-term provisional restoration and a radiotransparent composite for the permanent restoration material in this specific case allowed for the constant radiographic analysis of the integrity of the MTA mass, the buildup materials, and preparation margins. A radiopaque material would have covered some sites of interest, namely the furcal area and the coronal third of the roots, where pulp canal obliteration might take place and be detected.

## CONCLUSION

The present case report is an example of how a severely compromised tooth can be recovered with a series of multidisciplinary steps, by following the concepts of modern restorative dentistry, to provide the patient with minimally invasive treatments.

## Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Trieste.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 29 May 2019)

## REFERENCES

1. American Association of Endodontists (2016) Glossary of Endodontic Terms **9th edition**. Retrieved online November 8, 2019 from: <http://www.nxtbook.com/nxtbooks/aae/endodonticglossary2016/index.php>
2. Fuss Z & Trope M (1996) Root perforations: Classification and treatment choices based on prognostic factors *Endodontics and Dental Traumatology* **12(6)** 255-264.
3. Toure B, Faye B, Kane AW, Lo CM, Niang B, & Boucher Y (2011) Analysis of reasons for extraction of endodontically treated teeth: A prospective study *Journal of Endodontics* **37(11)** 1512-1515.

4. Alhadainy HA (1994) Root perforations. A review of literature *Oral Surgery, Oral Medicine, Oral Pathology* **78**(3) 368-374.
5. Holland R, Filho JA, de Souza V, Nery MJ, Bernabe PF, & Junior ED (2001) Mineral trioxide aggregate repair of lateral root perforations *Journal of Endodontics* **27**(4) 281-284.
6. Aqrabawi J (2000) Sealing ability of amalgam, super EBA cement, and MTA when used as retrograde filling materials *British Dental Journal* **188**(5) 266-268.
7. Xavier CB, Weismann R, de Oliveira MG, Demarco FF, & Pozza DH (2005) Root-end filling materials: Apical microleakage and marginal adaptation *Journal of Endodontics* **31**(7) 539-542.
8. Zhu Q, Haglund R, Safavi KE, & Spangberg LS (2000) Adhesion of human osteoblasts on root-end filling materials *Journal of Endodontics* **26**(7) 404-406.
9. Siew K, Lee AH, & Cheung GS (2015) Treatment outcome of repaired root perforation: A systematic review and meta-analysis *Journal of Endodontics* **41**(11) 1795-1804.
10. Trope M (2016) The expanding role of vital pulp therapy *Dentistry Today* **35**(6) 82-85.
11. Aguilar P & Linsuwanont P (2011) Vital pulp therapy in vital permanent teeth with cariously exposed pulp: A systematic review *Journal of Endodontics* **37**(5) 581-587.
12. Alqaderi H, Lee CT, Borzangy S, & Pagonis TC (2016) Coronal pulpotomy for cariously exposed permanent posterior teeth with closed apices: A systematic review and meta-analysis *Journal of Dentistry* **44** 1-7.
13. Bader JD, Rozier RG, McFall WT Jr, & Ramsey DL (1991) Effect of crown margins on periodontal conditions in regularly attending patients *Journal of Prosthetic Dentistry* **65**(1) 75-79.
14. Majzoub ZAK, Romanos A, & Cordioli G (2014) Crown lengthening procedures: A literature review *Seminars in Orthodontics* **20** 188-207.
15. Ng YL, Mann V, Rahbaran S, Lewsey J, & Gulabivala K (2008) Outcome of primary root canal treatment: Systematic review of the literature—Part 2. Influence of clinical factors *International Endodontic Journal* **41**(1) 6-31.
16. Caplan DJ, Cai J, Yin G, & White BA (2005) Root canal filled versus non-root canal filled teeth: A retrospective comparison of survival times *Journal of Public Health Dentistry* **65**(2) 90-96.
17. Randow K & Glantz PO (1986) On cantilever loading of vital and non-vital teeth. An experimental clinical study *Acta Odontologica Scandinavica* **44**(5) 271-277.
18. Ou KL, Chang CC, Chang WJ, Lin CT, Chang KJ, & Huang HM (2009) Effect of damping properties on fracture resistance of root filled premolar teeth: A dynamic finite element analysis *International Endodontic Journal* **42**(8) 694-704.
19. Parirokh M & Torabinejad M (2010) Mineral trioxide aggregate: A comprehensive literature review—Part I: Chemical, physical, and antibacterial properties *Journal of Endodontics* **36**(1) 16-27.
20. Camargo PM, Melnick PR, & Camargo LM (2007) Clinical crown lengthening in the esthetic zone *Journal of the California Dental Association* **35**(7) 487-498.
21. Angle EH (1899) Section of peridental membrane and frenum labii *Dental Cosmos* **41** 1143.
22. Young L, Binderman I, Yaffe A, Beni L, & Vardimon AD (2013) Fiberotomy enhances orthodontic tooth movement and diminishes relapse in a rat model *Orthodontics & Craniofacial Research* **16**(3) 161-168.
23. Angerame D, De Biasi M, Del Lupo V, Bevilacqua L, Zarone F, & Sorrentino R (2015) Influence of finish line on the marginal seal of nanohybrid composite crowns after periodontal scaling: A microleakage study *Minerva Stomatologica* **64**(5) 219-230.
24. Angerame D, Sorrentino R, Cettolin D, & Zarone F (2012) The effects of scaling and root planing on the marginal gap and microleakage of indirect composite crowns prepared with different finish lines: An in vitro study *Operative Dentistry* **37**(6) 650-659.
25. Lehmann F, Spiegl K, Eickemeyer G, & Rammelsberg P (2009) Adhesively luted, metal-free composite crowns after five years *Journal of Adhesive Dentistry* **11**(6) 493-498.
26. Magne P & Knezevic A (2009) Simulated fatigue resistance of composite resin versus porcelain CAD/CAM overlay restorations on endodontically treated molars *Quintessence International* **40**(2) 125-133.
27. Magne P, Schlichting LH, Maia HP, & Baratieri LN (2010) In vitro fatigue resistance of CAD/CAM composite resin and ceramic posterior occlusal veneers *Journal of Prosthetic Dentistry* **104**(3) 149-157.
28. Fokkinga WA, Le Bell AM, Kreulen CM, Lassila LV, Vallittu PK, & Creugers NH (2005) Ex vivo fracture resistance of direct resin composite complete crowns with and without posts on maxillary premolars *International Endodontic Journal* **38**(4) 230-237.
29. Ohlmann B, Trame JP, Dreyhaupt J, Gabbert O, Koob A, & Rammelsberg P (2008) Wear of posterior metal-free polymer crowns after 2 years *Journal of Oral Rehabilitation* **35**(10) 782-788.
30. Suzuki S, Nagai E, Taira Y, & Minesaki Y (2002) In vitro wear of indirect composite restoratives *Journal of Prosthetic Dentistry* **88**(4) 431-436.
31. Magne P & Knezevic A (2009) Thickness of CAD-CAM composite resin overlays influences fatigue resistance of endodontically treated premolars *Dental Materials* **25**(10) 1264-1268.
32. Rammelsberg P, Spiegl K, Eickemeyer G, & Schmitter M (2005) Clinical performance of metal-free polymer crowns after 3 years in service *Journal of Dentistry* **33**(6) 517-523.

# 24-Month Clinical Evaluation of Different Bulk-Fill Restorative Resins in Class II Restorations

T Guney • AR Yazici

## Clinical Relevance

Regardless of the presence of a flowable bulk-fill resin or a short-fiber-reinforced resin under a conventional resin composite, restorations in class II cavities restored with bulk-fill resins showed satisfactory and similar clinical performance.

## SUMMARY

The objective of this study was to evaluate the 24-month clinical performance of three different bulk-fill restorative resin materials in class II restorations. Forty patients with at least three approximal lesions in premolar and molar teeth participated in the study. A total of 120 class II cavities were restored using Tetric EvoCeram Bulk Fill (n=40), SureFil SDR flow + Ceram.X mono (n=40), and everX Posterior + G-aenial Posterior (n=40) with their respective adhesives according to the manufacturers' instructions. All restorations were placed by one operator. The restorations were evaluated at baseline and at six, 12, 18, and 24 months using modified US Public Health Ser-

vice criteria by one examiner. The restoration groups for each category were compared using the Pearson chi-square test, while the Cochran Q-test was used to compare the changes across different time points within each restorative material ( $p < 0.05$ ). At the end of 24 months, 94 restorations were evaluated in 33 patients, with a recall rate of 82.5%. There were no statistically significant differences between the groups in terms of retention ( $p > 0.05$ ). At the 24-month recall, two restorations from the SureFil SDR flow + Ceram.X mono group and four from the everX Posterior + G-aenial Posterior group showed slight marginal discoloration and were rated as bravo. No marginal discoloration was observed in any of the Tetric EvoCeram Bulk Fill restorations. Six restorations from the Tetric EvoCeram Bulk Fill group, six from the SureFil SDR flow + Ceram.X mono group, and 12 from the everX Posterior + G-aenial Posterior group received bravo scores in terms of marginal adaptation. No difference was found among the three groups for any of the evaluation criteria tested ( $p > 0.05$ ). There were statistically significant differences between the baseline and 24-month recall in the everX Posterior + G-aenial Posterior group in terms of marginal discoloration

Tugce Guney, DDS, Department of Restorative Dentistry, School of Dentistry, Hacettepe University, Ankara, Turkey; Eskisehir Oral and Dental Health Hospital, Eskisehir, Turkey

\*A Rüya Yazici, DDS, PhD, Department of Restorative Dentistry, School of Dentistry, Hacettepe University, Ankara, Turkey

\*Corresponding author: School of Dentistry, Hacettepe University, Sıhhiye Ankara 06100, Turkey; E-mail: ruyay@hacettepe.edu.tr

DOI: 10.2341/18-144-C

( $p < 0.05$ ). For marginal adaptation, a significant difference was observed between baseline and 24 months for all the restorative resins ( $p < 0.05$ ). All the restorative resins tested performed similarly and showed acceptable clinical performance during the 24-month evaluation.

## INTRODUCTION

Despite the developments in resin composites and their growing use in the posterior region, polymerization shrinkage stress, which is the main cause of adhesive failures, is still a challenge when resin-based restorative materials are used.<sup>1,2</sup> Therefore, numerous approaches have been advocated to decrease shrinkage stress by improving composite formulations, such as increasing filler content or the use of new less shrinking monomers, different curing methods, and manipulation of placement techniques.<sup>3</sup> Although incremental placement has been recommended for applying resin composites in order to reduce polymerization shrinkage, increase depth of cure, and avoid marginal disintegration,<sup>4-7</sup> this technique is time consuming and has a risk of contamination and entrapment of air voids between the layers. Moreover, the adaptation of multiple layers might be difficult, especially in small cavities.

The concept of using bulk placement has recently reemerged and is generating interest due to the introduction of bulk-fill resin restoratives. They are designed for placement in thick layers up to 4 mm, as clinicians wish to perform restorations using minimal chair time. Advancements in resin and filler technology and increased translucency have enhanced the depth of cure of bulk-fill resin composites.<sup>8-11</sup> Moreover, the inclusion of stress-reliever monomers, higher-molecular-weight resins, polymerization modulators, prepolymerized fillers, and more reactive photoinitiators in their composition resulted in reductions in polymerization shrinkage.<sup>12</sup> Bulk-fill resin composites are classified as high-viscosity bulk-fill flowable and low-viscosity bulk-fill nonflowable resins. Flowable bulk-fill resins require capping with a conventional resin composite for inferior mechanical properties, such as strength and wear resistance.<sup>13</sup> They have been generally advocated as dentin replacement materials.

Recent advances by manufacturers have resulted in the launch of a new type of short fiber-reinforced composite (everX Posterior) in order to overcome some of the concerns related to the restoration of large cavities with traditional resin composites.<sup>14</sup> This composite is composed of a resin matrix, short

E-glass fibers, and inorganic particulate fillers. The resin matrix contains cross-linked bisphenol-A glycidyl methacrylate, triethylene glycol dimethacrylate, and linear polymethyl methacrylate, forming a polymer matrix called a semi-interpenetrating polymer network, which contributes to better bonding and flexural strength.<sup>15-18</sup> Glass fibers have been proposed to favor light transmission through the resin composite and to reduce or stop crack propagation.<sup>16-18</sup> Laboratory studies demonstrated good performance of fiber-reinforced materials with respect to fracture resistance, toughness, and polymerization shrinkage.<sup>16,18-21</sup>

To date, only limited data from clinical trials on these topics have been published.<sup>22-27</sup> Even though the *in vitro* data suggest that bulk-fill resins may outperform incrementally placed resins, the limited and thus far controversial clinical data do not fully support that claim. On the other hand, the information from laboratory tests does not always correlate with clinical performance. Therefore, the aim of the present study was to further investigate the clinical performance of bulk-fill flowable, bulk-fill, and fiber-reinforced restorative resins in class II cavities over 24 months. The null hypothesis tested was that the different types of bulk-fill restorative resins would not differ from each other in terms of clinical performance.

## METHODS AND MATERIALS

Approval for the clinical trial was obtained from the Human Ethics Committee of Hacettepe University (#71146310, KA-14043). Prior to the start of the study, the patients were informed about the nature and the objectives of the study, and then their written consent was obtained.

### Inclusion Criteria

Inclusion criteria for patients included 1) 18 years or older, 2) good general health and oral hygiene, 3) at least three similar-sized approximal primary caries lesions in premolar and molar teeth, and 4) available for follow-up visits. Inclusion criteria of the teeth were 1) normal occlusal relationship with natural dentition, 2) adjacent tooth contact, and 3) normal response to a vitality test with no periapical pathology.

### Exclusion Criteria

Exclusion criteria were 1) poor oral hygiene, 2) severe or chronic periodontitis, 3) heavy bruxism, 4) fewer than 20 teeth, 5) history of adverse reaction to

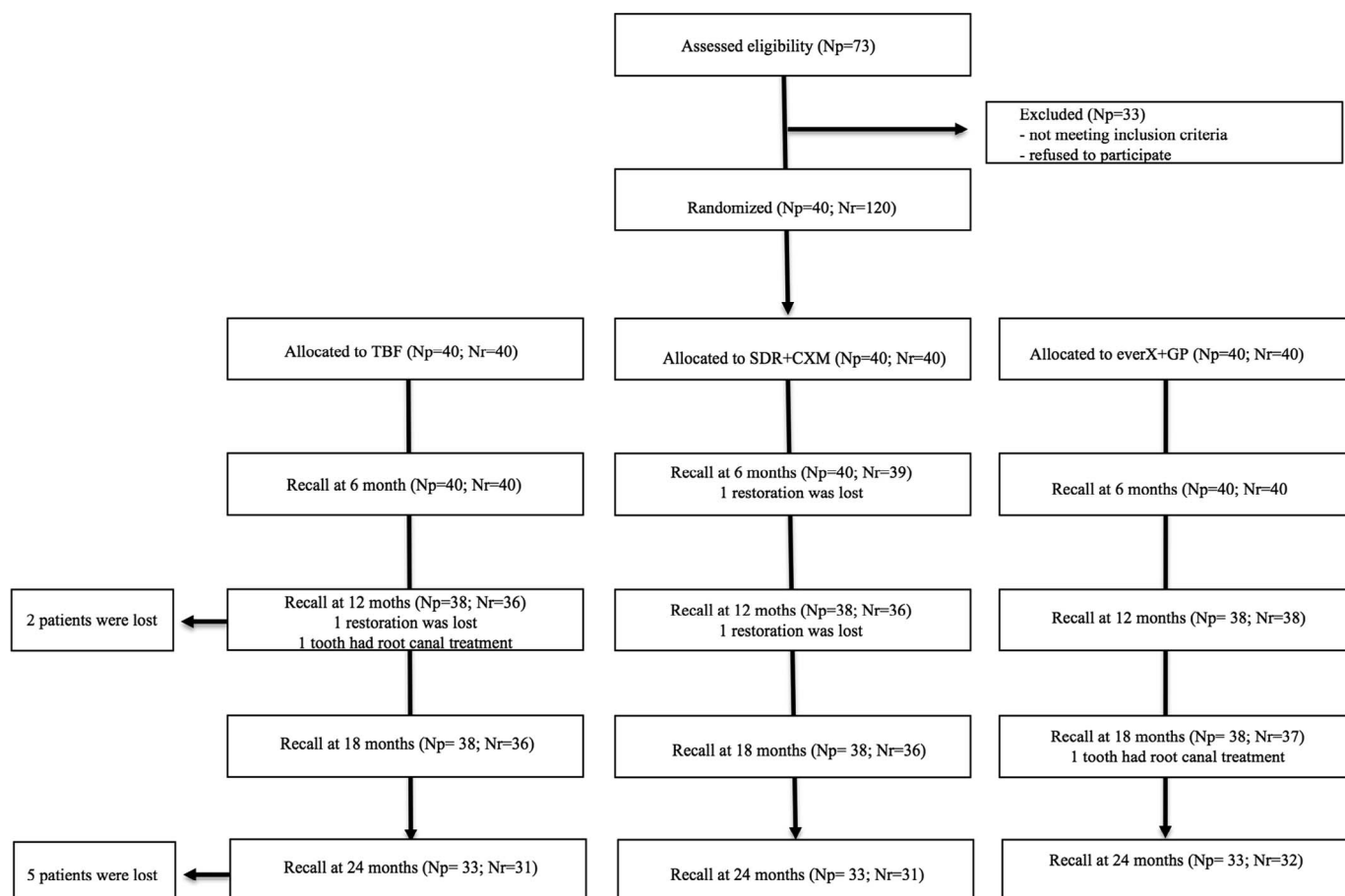


Figure 1. Flowchart of the trial (Np=Number of patients; Nr=number of restorations).

the test materials, 6) pregnancy or lactation period, and 7) potentially unable to attend recall visits.

The sample calculation indicated the need for approximately 35 patients for a confidence level of 85%. In total, 40 subjects (16 men and 24 women) ranging in age from 20 to 41 years were enrolled (Figure 1).

Bitewing radiographs of the teeth to be restored were taken preoperatively. The preparation was made using diamond straight (flat-end) burs (Edenta Ag Dental Products, Au, St.Gallen, Switzerland) in a high-speed hand piece with water cooling. The carious lesion was removed using round steel burs (Edenta). The preparation was limited to the removal of decay, preserving a maximum of sound tooth structure. The average bucco-lingual width of each preparation was equal to or greater than one-third of the distance between the cusp tips. The operative field was isolated using cotton rolls and suction after shade selection.

A total of 120 class II cavities were restored using Tetric EvoCeram Bulk Fill (TBF) (n=40), SureFil SDR flow + Ceram.X mono (SDR+CXM) (n=40), and everX Posterior + G-aenial Posterior (everX+GP) (n=40) with their respective adhesives according to the manufacturers' instructions. The composition and batch numbers are described in Table 1. The randomization process of subjects was performed using a table of random numbers, and the patient was unaware of which tooth received which restoration. All restorations were placed by one operator who was not blinded to group assignment.

For the Tetric EvoCeram Bulk Fill restorations, the preparation was etched with 37% phosphoric acid (Super Etch, SDI, Bayswater, VIC, Australia) applied initially to the enamel for 15 seconds and then to the dentin for 15 seconds. It was then rinsed with an air/water spray and blot dried, leaving the dentin slightly moist. An etch-and-rinse adhesive, ExciTE F (Ivoclar Vivadent, Schaan, Liechtenstein), was applied and agitated on the prepared surfaces

Table 1: *Materials Used in the Study*

| Product Name                                                                                                                                                                                                                                                                                                                                    | Type                                                     | Manufacturer                            | Batch Number | Composition                                                                                                                                              |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------|-----------------------------------------|--------------|----------------------------------------------------------------------------------------------------------------------------------------------------------|
| Tetric EvoCeram Bulk Fill                                                                                                                                                                                                                                                                                                                       | Bulk-fill posterior restorative                          | Ivoclar Vivadent, Schaan, Liechtenstein | R77065       | Bis-GMA, UDMA, bis-EMA, barium alumina silicate glass filler, ytterbium fluoride, spherical mixed oxide                                                  |
| Excite F                                                                                                                                                                                                                                                                                                                                        | Dental adhesive (two-step etch-and-rinse)                | Ivoclar Vivadent                        | P56445       | Phosphonic acid acrylate, HEMA, dimethacrylate, highly dispersed silicone dioxide, initiators, stabilizers and potassium fluoride in an alcohol solution |
| SureFil SDR Flow                                                                                                                                                                                                                                                                                                                                | Bulk-fill flowable resin composite                       | Dentsply Caulk (Milford, DE, USA)       | 1207205      | Barium and strontium alumino-fluoro-silicate glass, TEGDMA, modified UDMA, dimethacrylate, EBPADMA, pigment, photoinitiator                              |
| Ceram.X mono                                                                                                                                                                                                                                                                                                                                    | Nanoceramic resin composite                              | Dentsply Caulk                          | 1203000406   | Methacrylate modified polysiloxane, dimethacrylate resin, barium-aluminum-borosilicate glass, methacrylate functionalized silicon dioxide nanofillers    |
| Prime&Bond NT                                                                                                                                                                                                                                                                                                                                   | Nanotechnology dental adhesive (two-step etch-and-rinse) | Dentsply Caulk                          | 1306000189   | Di- and trimethacrylate resins, functionalized amorphous silica, PENTA, stabilizers, cetylamine hydrofluoride, acetone                                   |
| everX Posterior                                                                                                                                                                                                                                                                                                                                 | Fiber-reinforced resin composite                         | GC Co (Milford, DE, USA)                | 1309121      | Bis-GMA, TEGDMA, PMMA, triethylene glycol dimethacrylate, glass fillers and inorganic granular fillers                                                   |
| G-aenial Posterior                                                                                                                                                                                                                                                                                                                              | Posterior resin composite                                | GC Co                                   | 1211192      | Methacrylate monomers, UDMA, dimethacrylate comonomers, prepolymerized fillers, camphorquinone and amine, fluoroaluminosilicate, fumed silica            |
| G-aenial Bond                                                                                                                                                                                                                                                                                                                                   | One-component self-etch adhesive                         | GC Co                                   | 1401271      | Phosphoric ester monomers, 4-MET, a hydrophilic methacrylate monomer, water, acetone, photoinitiator, nanosilica                                         |
| SDI Super Etch                                                                                                                                                                                                                                                                                                                                  | Etching gel                                              | SDI (Bayswater, VIC, Australia)         | 140554       | 37% phosphoric acid, synthetic amorphous silica (fumed), polyethylene glycol, aluminum oxide, water                                                      |
| Abbreviations: bis-GMA, bisphenol-A glycidyl methacrylate; UDMA, urethane dimethacrylate; bis-EMA, bisphenol-A ethoxylated dimethacrylate; HEMA, hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; EBPADMA, ethoxylated bisphenol-A dimethacrylate; PENTA, phosphonated penta-acrylate ester; PMMA, polymethyl methacrylate |                                                          |                                         |              |                                                                                                                                                          |

for at least 10 seconds. After the adhesive was dispersed into a thin layer with a weak stream of air, light curing was performed for 20 seconds with an LED curing unit (Starlight, Mectron, Carasco, Italy) with an intensity of 1400 mW/cm<sup>2</sup>. The sectional matrix was placed and fixed with wooden wedges. Then the bulk-fill resin composite, TBF, was placed in bulk in about 4-mm thickness and then cured with the same curing unit for 20 seconds.

For SDR + Ceram.X mono restorations, the cavities were etched as described above. A two-step etch-and-rinse adhesive, Prime&Bond NT (Dentsply DeTrey, Konstanz, Germany), was applied to all enamel/dentin surfaces, air-dried for five seconds, and light cured for 10 seconds. After the sectional matrix was placed, the flowable bulk-fill resin composite, SureFil SDR flow (Dentsply Caulk, Milford, DE, USA), was placed in a 4-mm bulk increment in the dentinal part cured for 20 seconds and followed by a covering layer of the nanohybrid resin composite, Ceram.X Mono (Dentsply DeTrey), that was cured for 20 seconds.

For everX + G-aenial Posterior restorations, the enamel was etched with 37% phosphoric acid for 10

seconds, then rinsed and dried with an air/water syringe for at least five seconds. A self-etch adhesive, G-aenial Bond (GC Co, Tokyo, Japan), was applied to all enamel/dentin surfaces and gently air thinned and then light cured for 10 seconds. After the matrix was placed and wedged, mesial or distal walls were built with 1- to 2-mm layers of G-aenial Posterior resin composite (GC Co). The fiber-reinforced composite, everX Posterior (GC Co), was placed into the cavity in approximately 4-mm thickness. The last 2 mm of the cavity were restored using the G-aenial Posterior composite as an overlay layer. Each increment was light cured for 20 seconds using the same LED unit.

The occlusion was checked and adjusted. Finishing was done using diamond finishing burs (Diatech, Coltène/Whaledent AG, Altstätten, Switzerland). Polishing was accomplished with aluminum oxide disks (OptiDisc, Kerr, Bioggio, Switzerland) and rubber cups and points (Kerr).

An experienced dentist, not involved in the placement of the restorations and therefore blinded to the group assignment, performed the evaluation. As an intraexaminer agreement of at least 85% was

Table 2: Distribution of Materials According to Tooth and Arch

| Groups                               | Mandibular |       | Maxillar |       | Total |
|--------------------------------------|------------|-------|----------|-------|-------|
|                                      | Premolar   | Molar | Premolar | Molar |       |
| TetricEvoCeram Bulk Fill             | 24         | 5     | 5        | 6     | 40    |
| SureFil SDR flow + Ceram.X mono      | 12         | 4     | 18       | 6     | 40    |
| everX Posterior + G-aenial Posterior | 11         | 14    | 4        | 11    | 40    |
| Total                                | 47         | 23    | 27       | 23    | 120   |

necessary before the beginning of the evaluation, intraexaminer training was conducted on 40 restorations from approximately 20 patients who were not participating in the present study. Examination was repeated after two weeks to record intraexaminer reproducibility. The examiner was calibrated to 100% intraexaminer agreement for all the evaluated criteria.

The restorations were evaluated at baseline and at six, 12, 18, and 24 months using modified United States Public Health Service criteria.<sup>28</sup> The postoperative sensitivity was evaluated by applying air for 10 seconds from a dental syringe at a distance of 1 cm from the tooth surface and by questioning the patients. During this evaluation, adjacent teeth were isolated with cotton rolls. Clinical photographs were taken pre- and postoperatively at baseline and at each recall. At the end of 24 months, bitewing radiographs were also taken. Table 2 shows the distribution of restorations to teeth, with the majority of restored teeth being premolars. Forty-six restorations (38.3%) were inserted in molars, whereas 74 (61.7%) were inserted in premolars.

The results were analyzed using IBM SPSS 22.0 (SPSS, Chicago, IL, USA). The restoration groups for each category were compared using the Pearson chi-square test, while the Cochran Q test was used to compare the changes across different time points within each restorative material. The level of significance was set at 5%.

## RESULTS

Table 3 presents the results of the clinical evaluation of the restorations.

### Recall and Retention Rate

At the six-month recall, the recall rate was 100%. Two patients did not attend the 12-month recall. At the 12- and 18-month recalls, the recall rates were 95%. At the 24-month recall, five more patients were lost, and the recall rate declined to 82.5%. The reasons were moving to another city for two patients and failed attempts to contact the other three.

The retention rate was 100% for Tetric EvoCeram Bulkfill and everX + G-aenial posterior restorations at six months. For the Surefil SDR Flow + Ceram.X mono group, the retention rate was 97.5% due to the loss of one restoration. At 12 months, one restoration from the TBF group and one from the Surefil SDR Flow + Ceram.X mono group were lost, and one restoration received root canal treatment from the Tetric Bulk Fill group with a retention rate of 97.3%. Within the everX group, one restoration was replaced due to root canal treatment at 18 months. Twenty-four-month retention rates were 100% for all groups, and the number of evaluated restorations was 31 for TB and SDR and 32 for everX. There were no statistically significant differences between the groups after six, 12, 18, or 24 months ( $p > 0.05$ ).

### Marginal Discoloration

One restoration from the SDR group showed slight marginal discoloration at six, 12, and 18 months. At 24 months, two restorations showed discoloration. Two restorations from the everX group showed superficial discoloration at 12 and 18 months. At 24 months, discoloration was observed in four restorations. Figure 2 through 5 show approximal lesions and class II restorations in a patient before the procedure, at baseline, and at the 12- and 24-month recall visits. None of the restorations from the TBF group showed discoloration anywhere along the margin during the evaluation periods.

### Marginal Adaptation

In terms of marginal adaptation, four restorations from both the TBF and the SureFil SDR flow + Ceram.X mono group and seven everX Posterior + G-aenial Posterior restorations were rated as bravo at six months. At 12 months, two more restorations from the everX group showed slight discoloration. At 18 months, five restorations from both the Tetric EvoCeram Bulk Fill and the SureFil SDR flow + Ceram.X mono group and 11 everX Posterior + G-aenial Posterior restorations were rated as bravo. At 24 months, one more restoration from each group was rated as bravo in terms of marginal adaptation.

| Table 3: Results of the Clinical Evaluation of the Restorations                                                                                                              |             |              |              |              |              |              |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|--------------|--------------|--------------|--------------|--------------|
|                                                                                                                                                                              | Baseline    |              |              | 6 Months     |              |              |
|                                                                                                                                                                              | TBF         | SDR + CXM    | everX + GP   | TBF          | SDR + CXM    | everX + GP   |
| Retention                                                                                                                                                                    |             |              |              |              |              |              |
| A                                                                                                                                                                            | 40/40 (100) | 40/40 (100)  | 40/40 (100)  | 40/40 (100)  | 40/39 (97.5) | 40/40 (100)  |
| C                                                                                                                                                                            | 40/0 (0)    | 40/0 (0)     | 40/0 (0)     | 40/0 (0)     | 40/1 (2.5)   | 40/0 (0)     |
| Marginal discoloration                                                                                                                                                       |             |              |              |              |              |              |
| A                                                                                                                                                                            | 40/40 (100) | 40/40 (100)  | 40/40 (100)  | 40/40 (100)  | 39/38 (97.4) | 40/40 (100)  |
| B                                                                                                                                                                            | 40/0 (0)    | 40/0 (0)     | 40/0 (0)     | 40/0 (0)     | 39/1 (2.6)   | 40/0 (0)     |
| Marginal adaptation                                                                                                                                                          |             |              |              |              |              |              |
| A                                                                                                                                                                            | 40/40 (100) | 40/40 (100)  | 40/40 (100)  | 40/36 (90)   | 39/35 (89.7) | 40/33 (82.5) |
| B                                                                                                                                                                            | 40/0 (0)    | 40/0 (0)     | 40/0 (0)     | 40/4 (10)    | 39/4 (10.3)  | 40/7 (17.5)  |
| Color match                                                                                                                                                                  |             |              |              |              |              |              |
| A                                                                                                                                                                            | 40/40 (100) | 40/40 (100)  | 40/39 (97.5) | 40/40 (100)  | 39/39 (100)  | 40/39 (97.5) |
| B                                                                                                                                                                            | 40/0 (0)    | 40/0 (0)     | 40/1 (2.5)   | 40/0 (0)     | 39/0 (0)     | 40/1 (2.5)   |
| Surface texture                                                                                                                                                              |             |              |              |              |              |              |
| A                                                                                                                                                                            | 40/40 (100) | 40/40 (100)  | 40/40 (100)  | 40/40 (100)  | 39/38 (97.4) | 40/39 (97.5) |
| B                                                                                                                                                                            | 40/0 (0)    | 40/0 (0)     | 40/0 (0)     | 40/0 (0)     | 39/1 (2.6)   | 40/1 (2.5)   |
| Anatomic form                                                                                                                                                                |             |              |              |              |              |              |
| A                                                                                                                                                                            | 40/40 (100) | 40/40 (100)  | 40/40 (100)  | 40/40 (100)  | 39/39 (100)  | 40/40 (100)  |
| B                                                                                                                                                                            | 40/0 (0)    | 40/0 (0)     | 40/0 (0)     | 40/0 (0)     | 39/0 (0)     | 40/0 (0)     |
| Postoperative sensitivity                                                                                                                                                    |             |              |              |              |              |              |
| A                                                                                                                                                                            | 40/40 (100) | 40/38 (95.0) | 40/39 (97.5) | 40/37 (92.5) | 39/38 (97.4) | 40/37 (92.5) |
| B                                                                                                                                                                            | 40/0 (0)    | 40/2 (5.0)   | 40/1 (2.5)   | 40/3 (7.5)   | 39/1 (2.6)   | 40/3 (7.5)   |
| Secondary caries                                                                                                                                                             |             |              |              |              |              |              |
| A                                                                                                                                                                            | 40/40 (100) | 40/40 (100)  | 40/40 (100)  | 40/40 (100)  | 39/39 (100)  | 40/40 (100)  |
| Abbreviations: TBF, Tetric EvoCeram Bulk Fill; SDR + CXM, SureFil SDR flow + Ceram X Mono; everX + GP, everX Posterior + G-aenial Posterior; A, alpha; B, bravo; C, charlie. |             |              |              |              |              |              |

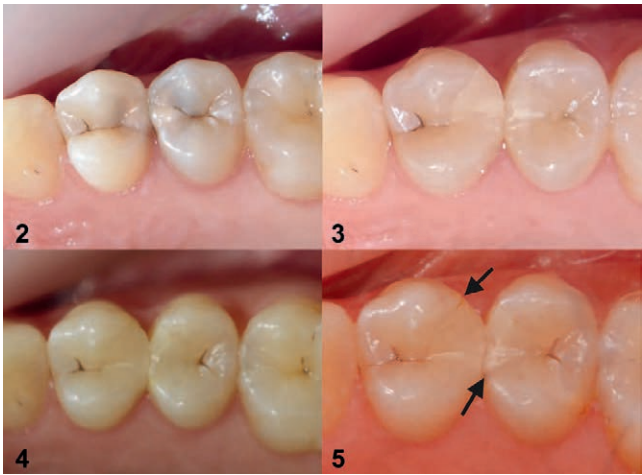


Figure 2. Preoperative approximal lesions on teeth #4 and #5.

Figure 3. Class II restorations on teeth #4 (everX Posterior+G-aenial Posterior) and #5 (SureFil SDR flow+Ceram.X mono).

Figure 4. Restorations at the 12-month follow-up on teeth #4 and #5.

Figure 5. Restorations at the 24-month follow-up on teeth #4 and #5 (arrows show marginal discoloration).

Color Match

One restoration from the everX Posterior + G-aenial Posterior group received a bravo score from the baseline. At 12, 18, and 24 months, two restorations were rated as bravo in the everX group. The rest of the restorations showed no mismatch in color during the study.

Surface Texture

At six months, one restoration from the SureFil SDR flow + Ceram.X mono group and one from the everX Posterior + G-aenial Posterior group were rated as bravo in terms of surface texture, while at 12, 18, and 24 months, two restorations from the same groups received this rating.

Anatomic Form

Only one restoration from the everX Posterior + G-aenial Posterior group received a bravo score at the 12- and 18-month evaluations. However, this restoration could not be evaluated at the 24-month follow-up because this patient did not attend the recall. The

Table 3: Results of the Clinical Evaluation of the Restorations (ext.)

|                           | 12 Months    |              |              | 18 Months    |              |              | 24 Months    |              |              |
|---------------------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|
|                           | TBF          | SDR + CXM    | everX + GP   | TBF          | SDR + CXM    | everX + GP   | TBF          | SDR + CXM    | everX + GP   |
| Retention                 |              |              |              |              |              |              |              |              |              |
| A                         | 37/36 (97.3) | 37/36 (97.3) | 38/38 (100)  | 36/36 (100)  | 36/36 (100)  | 37/37 (100)  | 31/31 (100)  | 31/31 (100)  | 32/32 (100)  |
| C                         | 37/1 (2.7)   | 37/1 (2.7)   | 38/0 (0)     | 36/0 (0)     | 36/0 (0)     | 37/0 (0)     | 31/0 (0)     | 31/0 (0)     | 32/0 (0)     |
| Marginal discoloration    |              |              |              |              |              |              |              |              |              |
| A                         | 36/36 (100)  | 36/35 (97.2) | 38/36 (94.7) | 36/36 (100)  | 36/35 (97.2) | 37/35 (94.6) | 31/31 (100)  | 31/29 (93.5) | 32/28 (87.5) |
| B                         | 36/0 (0)     | 36/1 (2.8)   | 38/2 (5.3)   | 36/0 (0)     | 36/1 (2.8)   | 37/2 (5.4)   | 31/0 (0)     | 31/2 (6.5)   | 32/4 (12.5)  |
| Marginal adaptation       |              |              |              |              |              |              |              |              |              |
| A                         | 36/32 (88.9) | 36/33 (91.7) | 38/29 (76.3) | 36/31 (86.1) | 36/31 (86.1) | 37/26 (70.3) | 31/25 (80.6) | 31/25 (80.6) | 32/20 (62.5) |
| B                         | 36/4 (11.1)  | 36/3 (8.3)   | 38/9 (23.7)  | 36/5 (13.9)  | 36/5 (13.9)  | 37/11 (29.7) | 31/6 (19.4)  | 31/6 (19.4)  | 32/12 (37.5) |
| Color match               |              |              |              |              |              |              |              |              |              |
| A                         | 36/36 (100)  | 36/36 (100)  | 38/36 (94.7) | 36/36 (100)  | 36/36 (100)  | 37/35 (94.6) | 31/31 (100)  | 31/31 (100)  | 32/30 (93.7) |
| B                         | 36/0 (0)     | 36/0 (0)     | 38/2 (5.3)   | 36/0 (0)     | 36/0 (0)     | 37/2 (5.4)   | 31/0 (0)     | 31/0 (0)     | 32/2 (6.3)   |
| Surface texture           |              |              |              |              |              |              |              |              |              |
| A                         | 36/36 (100)  | 36/34 (94.4) | 38/36 (94.7) | 36/36 (100)  | 36/34 (94.4) | 37/35 (94.6) | 31/31 (100)  | 31/29 (93.5) | 32/30 (93.7) |
| B                         | 36/0 (0)     | 36/2 (5.6)   | 38/2 (5.3)   | 36/0 (0)     | 36/2 (5.6)   | 37/2 (5.4)   | 31/0 (0)     | 31/2 (6.5)   | 32/2 (6.3)   |
| Anatomic form             |              |              |              |              |              |              |              |              |              |
| A                         | 36/36 (100)  | 36/36 (100)  | 38/37 (97.4) | 36/36 (100)  | 36/36 (100)  | 37/36 (97.3) | 31/31 (100)  | 31/31 (100)  | 32/32 (100)  |
| B                         | 36/0 (0)     | 36/0 (0)     | 38/1 (2.6)   | 36/0 (0)     | 36/0 (0)     | 37/1 (2.7)   | 31/0 (0)     | 31/0 (0)     | 32/0 (0)     |
| Postoperative sensitivity |              |              |              |              |              |              |              |              |              |
| A                         | 36/34 (94.4) | 36/35 (97.2) | 38/35 (92.1) | 36/36 (100)  | 36/35 (97.2) | 37/36 (97.3) | 31/31 (100)  | 31/31 (100)  | 32/32 (100)  |
| B                         | 36/2 (5.6)   | 36/1 (2.8)   | 38/3 (7.9)   | 36/0 (0)     | 36/1 (2.8)   | 37/1 (2.7)   | 31/0 (0)     | 31/0 (0)     | 32/0 (0)     |
| Secondary caries          |              |              |              |              |              |              |              |              |              |
| A                         | 36/36 (100)  | 36/36 (100)  | 38/38 (100)  | 36/36 (100)  | 36/36 (100)  | 37/37 (100)  | 31/31 (100)  | 31/31 (100)  | 32/32 (100)  |

rest of the evaluated restorations showed no loss of anatomic form and were continuous with existing anatomic form.

### Postoperative Sensitivity

Postoperative sensitivity was observed in three restorations from the TBF group at six months. At 12 months, one of them received root canal treatment; the other two patients were still suffering from mild sensitivity. After 18 months, sensitivity totally disappeared in these two patients. In the SureFil SDR flow + Ceram.X mono group, two patients had postoperative sensitivity at the baseline. In one of them, pain had disappeared by the six-month recall, while the other one still had sensitivity at the 18-month recall. One patient suffered from postoperative sensitivity from baseline up to 24 months in the everX Posterior + G-aenial Posterior group. Two more patients had postoperative sensitivity at the six- and 12-month recalls. At 18 months, one of them received root canal treatment, and pain disappeared in the other one. At 24 months, none of the patients

from any groups suffered from postoperative sensitivity.

Overall, no statistically significant differences were noted among these three groups for marginal discoloration, marginal adaptation, color match, surface texture, or anatomic form ( $p < 0.05$ ). No secondary caries was detected.

When comparing each group at different recall times, a significant difference was observed in all groups for marginal adaptation between baseline and 24 months ( $p < 0.05$ ). In terms of marginal discoloration, there was a statistically significant difference between baseline and 24 months only in the everX Posterior + G-aenial Posterior group ( $p < 0.05$ ).

### DISCUSSION

A recent innovation in posterior resin restoratives is the introduction of bulk-fill restoratives. Although many *in vitro* studies have been conducted to evaluate the performance of these newly introduced restorative materials, their findings do not always

reflect the behavior of these materials, and so the best way to predict their performance is to conduct clinical studies. In previous clinical studies,<sup>23-27,29,30</sup> bulk-fill resins were compared mostly with incrementally placed conventional resins. To the best of the authors' knowledge, no study has so far compared the performance of bulk-fill, bulk-fill flowable, and fiber-reinforced restorative resins in class II cavities over 24 months. In the present study, the tested resin restoratives performed similarly in every aspect over the 24-month evaluation period, leading to acceptance of the hypothesis.

It is important to find out whether application of a flowable bulk-fill resin or a short-fiber-reinforced resin could improve the internal marginal adaptation of restorations. In an *in vitro* study, the marginal adaptation of two flowable bulk-fill and two bulk-fill resin-based composites and one incrementally placed composite was compared, and it was concluded that the bulk-fill resin composites showed better adaptation than the flowable bulk resins.<sup>31</sup> Contrary to this finding, in another study, the marginal and internal integrity of posterior resin composites with or without a 4-mm SDR base was compared, and no influence was reported with the presence of a 4-mm layer of SDR.<sup>32</sup> Patnana and others<sup>33</sup> compared the marginal integrity of short-fiber-reinforced restorations with or without a composite superficial layer and with conventional resin composite. They concluded that fiber-reinforced restorations showed improved marginal integrity compared to traditional ones. In a short-term 12-month clinical follow-up, restorations combining a base of short-fiber-reinforced composite resin with hybrid composite resins showed promising performance.<sup>22</sup> However, the study did not compare their results with those of another type of resin composite. A more recent publication compared the clinical durability of a flowable bulk-fill resin composite, SDR, capped with a layer of nanohybrid composite and an incrementally placed conventional resin composite.<sup>29</sup> At the end of six years, the annual failure rate was 1.0% for both groups. It was concluded that the restorations made with a flowable bulk-fill base demonstrated good results that were not significantly different from those of incrementally placed composites.

In our study, although no significant difference was detected, six restorations each from the TBF and SDR + CXM groups and 12 restorations from the everX + GP group showed poor marginal adaptation. In other words, restorations with the combination of a conventional resin composite over flowable bulk-fill

or a short-fiber-reinforced resin showed similar performance in terms of marginal adaptation. Jung and Park<sup>31</sup> stated that polymerization shrinkage and stress are the major factors for imperfect marginal adaptation. Filler content is also an important material property that affects marginal adaptation. When the polymerization shrinkage is similar, the material with the lower viscosity has better marginal adaptation. Although the polymerization shrinkage of SDR (3.5%) was higher than that of the other tested restorative resins, their marginal adaptation was similar. With a lower modulus of elasticity,<sup>34</sup> SDR might act as a stress buffer. On the other hand, the features of the resin composite material that caps the flowable bulk fills are also important. In the present study, Ceram.X mono, with a polymerization shrinkage rate of 2.3, was used to cap SDR flowable bulk-fill composite. Its polymerization shrinkage value was quite similar to that of the other restorative resins tested. This might be another reason for the similarity in marginal adaptation ratings. However, the marginal adaptation scores of all tested restorative resins varied with respect to baseline measurements. The increase in the amount of inorganic fillers and the decrease in monomer content might also contribute to lower polymerization shrinkage. In the present study, the inorganic filler volume was 60% for Tetric EvoCeram Bulk Fill, 57% for Ceram.X mono, and 62% for G-aenial Posterior. Besides the filler load, polymerization shrinkage might also be affected by the size and shape of fillers, monomer composition, and initiators.

The reason that no differences were observed between the restorative materials might be related to the specific properties of the resins. Tetric EvoCeram Bulk Fill has a germanium-based photoinitiator, the initiator booster Ivocerin.<sup>35</sup> Due to its higher absorption in the region between 400 and 450 nm, it has a higher photo-curing activity than camphorquinone (CQ) does.<sup>9,36</sup> This material contains some prepolymerized fillers that cause low modulus of elasticity.<sup>35,37</sup> In addition, the material includes a shrinkage stress reliever.<sup>35</sup> On the other hand, with the high translucency of SDR, the transmission of light through the material is much easier. Moreover, a special group boosting polymerization that interacts with CQ was incorporated in its monomer composition;<sup>8,9</sup> everX Posterior has CQ and N,N-dimethylaminoethyl methacrylate as a photoinitiator and contains E-glass fibers that conduct and scatter the light, enabling adequate polymerization depth. In an *in vitro* study, suffi-

cient polymerization properties in terms of degree of conversion, hardness, polymerization volume shrinkage, and shrinkage stress were obtained at 4-mm depth for bulk-fill resin composites.<sup>38</sup> As poorly polymerized resin can cause gap formation, leading to marginal leakage, discoloration, and recurrent caries; their adequate depth of cure might have caused the lack of difference between the tested resins' marginal discoloration scores. On the other hand, it might have been expected that restorations with everX base, having short-fiber fillers that may absorb polymerization stress and increase stress-relieving capacity, might improve adaptation and thereby reduce marginal discoloration. In an *in vitro* study, the use of a base material, short-fiber-reinforced composite, everX Posterior, and a surface layer of conventional resin showed less leakage than SDR + conventional composite and bulk-fill resin composite.<sup>39</sup> The results contradict the predictions that could be made for everX based on *in vitro* data. Contrary to these expectations, although not significantly different, Tetric EvoCeram Bulk Fill restorations showed a trend toward superior performance in terms of marginal discoloration. None of the restorations in this group showed any discoloration. In a three-year clinical study that compared the clinical performance of Tetric EvoCeram Bulk Fill and an incrementally placed resin composite, no marginal discoloration was detected in any of the bulk-fill restorations at 24-month recall. However, two restorations showed slight discoloration at three-year recall.<sup>27</sup> It is better to conduct long-term clinical studies in order to obtain clear evidence about their performance.

In the present study, one restoration from the TBF group at 12 months and one restoration from the everX + GP group at 18 months had to be endodontically treated. The reason for the root canal treatment was postoperative sensitivity. As these two restorations were in the same patient, this could be related to low pain perception in that patient.

Anatomic form and surface texture could be related to either the patient's habits and eating or the restorative materials' specifications, such as the filler type and content. Most of the restorations received 100% alpha scores throughout the study. These favorable results could be attributed to the mechanical properties of the resin composites tested and the patients' good oral hygiene as well as their frequent dental visits.

## CONCLUSIONS

Within the limitations of the present study, all investigated bulk-fill restorations, regardless of the presence of a flowable bulk-fill resin or a short-fiber-reinforced resin under a conventional resin composite, were found to be clinically acceptable and similar after 24 months of clinical use. However, this is only a short-term clinical study, and the durability of the tested restorative materials might change over time. Observation of a significant deterioration in marginal adaptation for all the restorations is another important outcome of the present study. Therefore, further recall evaluations have already been planned.

## Acknowledgements

The authors thank for Ivoclar/Vivadent, Dentsply Caulk, and GC Co for providing materials for this study.

## Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Hacettepe University. The approval code for this study is #71146310, KA-14043.

## Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 1 November 2018)

## REFERENCES

1. Tiba A, Charlton DG, Vanderwalle KS, & Cohen ME (2005) Volumetric polymerization shrinkage under simulated intraoral temperature and humidity conditions *Operative Dentistry* **30**(6) 696-701.
2. Kleverlaan CJ & Feilzer AJ (2005) Polymerization shrinkage and contraction stress of dental resin composites *Dental Materials* **21**(12) 1150-1157.
3. Braga RR & Ferracane JL (2004) Alternatives on polymerization contraction stress management *Critical Reviews in Oral Biology and Medicine* **15**(3) 176-184.
4. Kuijs RH, Fennis WM, Kreulen CM, Barink M, & Verdonschot N (2003) Does layering minimize shrinkage stresses in composite restorations? *Journal of Dental Research* **82**(12) 967-971.
5. Coelho Santos MJ, Santos GC Jr, Nagem Filho H, Mondelli RF, & El-Mowafy O (2004) Effect of light curing method on volumetric polymerization shrinkage of resin composites *Operative Dentistry* **29**(2) 157-161.
6. Braga RR, Ballester RY, & Ferracane JL (2005) Factors involved in the development of polymerization shrinkage stress in resin-composites: A systematic review *Dental Materials* **21**(10) 962-970.

7. Ferracane JL (2011) Resin composite state of the art *Dental Materials* **27**(1) 29-38.
8. Ilie N & Hickel R (2011) Investigations on a methacrylate-based flowable composite based on the SDR technology *Dental Materials* **27**(4) 348-355.
9. Ilie N, Bucuta S, & Draenert M (2013) Bulk-fill resin-based composites: An in vitro assessment of their mechanical performance *Operative Dentistry* **38**(6) 618-625.
10. Bucuta S & Ilie N (2014) Light transmittance and micromechanical properties of bulk fill vs. conventional resin-based composites *Clinical Oral Investigations* **18**(8) 1991-2000.
11. El-Damanhoury HM & Platt JA (2013) Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites *Operative Dentistry* **39**(4) 374-382.
12. Fronza BM, Rueggeberg FA, Braga RR, Mogilevych B, Soares LES, Martin AA, Ambrosano G, & Giannini M (2015) Monomer conversion, microhardness, internal marginal adaptation, and shrinkage stress of bulk-fill resin composites *Dental Materials* **31**(12) 1542-1551.
13. Swift EJ (2015) Bulk-fill composites, part I *Journal of Esthetic and Restorative Dentistry* **27**(3) 176-179.
14. GC everX Posterior (2013) *everX Posterior Technical Manual* GC everX Posterior, Leuven, Belgium.
15. Lastumaki TM, Lassila LV, & Vallittu PK (2003) The semi-interpenetrating polymer network matrix of fiber-reinforced composite and its effect on the surface adhesive properties *Journal of Materials Science: Materials in Medicine* **14**(9) 803-809.
16. Garoushi S, Vallittu PK, & Lassila LV (2007) Short glass fiber reinforced restorative composite resin with semi-interpenetrating polymer network matrix *Dental Materials* **23**(11) 1356-1362.
17. Garoushi S, Vallittu PK, Watts DC, & Lassila LV (2008) Polymerization shrinkage of experimental short glass fiber-reinforced composite with semi-interpenetrating polymer network matrix *Dental Materials* **24**(2) 211-215.
18. Garoushi S, Säilynoja E, Vallittu PK, & Lassila L (2013) Physical properties and depth of cure of a new short fiber reinforced composite *Dental Materials* **29**(8) 835-841.
19. Vallittu PK (2014) High-aspect ratio fillers: Fiber-reinforced composites and their anisotropic properties *Dental Materials* **31**(1) 1-7.
20. Bijelic-Donova J, Garoushi S, Lassila LV, Keulemans F, & Vallittu PK (2016) Mechanical and structural characterization of discontinuous fiber-reinforced dental resin composite *Journal of Dentistry* **52** 70-76.
21. Atalay C, Yazici AR, Horuztepe A, Nagas E, Ertan A, & Ozgunaltay G (2016) Fracture resistance of endodontically treated teeth restored with bulk fill bulk fill flowable, fiber-reinforced and conventional resin composite *Operative Dentistry* **41**(5) E131-E140.
22. Garoushi S, Tanner J, Vallittu PK, & Lassila L (2012) Preliminary clinical evaluation of short fiber-reinforced composite resin in posterior teeth: 12-months report *Open Dentistry Journal* **6** 41-45.
23. Dijken V & Pallesen U (2014) A randomized controlled three-year evaluation of "bulk-filled" posterior resin restorations based on stress decreasing resin technology *Dental Materials* **30**(9) 245-251.
24. Dijken V & Pallesen U (2015) Randomized 3-year clinical resin of class I and class II posterior resin restorations placed with a bulk-fill resin composite and a one-step self-etching adhesive *Journal of Adhesive Dentistry* **17**(1) 81-88.
25. Dijken V & Pallesen U (2016) Posterior bulk-filled resin composite restorations: A 5-year randomized controlled clinical study *Journal of Dentistry* **51** 29-35.
26. Bayraktar Y, Ercan E, Hamidi MM, & Colak H (2017) One-year clinical evaluation of different types of bulk-fill composites *Journal of Investigative and Clinical Dentistry* **8**(2) DOI: 10.1111/jicd.12210.
27. Yazici AR, Antonson SA, ZB Kutuk ZB, & Ergin E (2017) Thirty-six-month clinical comparison of bulk fill and nanofill composite restorations *Operative Dentistry* **42**(5) 478-485.
28. Cvar JF & Ryge G (2005) Reprint of criteria for the clinical evaluation of dental restorative materials, 1971 *Clinical Oral Investigations* **9**(4) 215-232.
29. Dijken V & Pallesen U (2017) Bulk-filled posterior resin restorations based on stress-decreasing resin technology: A randomized controlled 6-year evaluation *European Journal of Oral Science* **125**(4) 303-309.
30. Colak H, Tokay U, Uzgur R, Hamidi MM, & Ercan EE (2017). A prospective randomized, double-blind clinical trial of one nano-hybrid and one high-viscosity bulk fill composite restorative systems in class II cavities: 12 months results *Nigerian Journal of Clinical Practice* **20**(7) 822- 831.
31. Jung JH & Park SH (2017) Comparison of polymerization shrinkage, physical properties, and marginal adaptation of flowable and restorative bulk fill resin-based composites *Operative Dentistry* **42**(4) 375-386.
32. Roggendorf MJ, Krämer N, Appelt A, Naumann M, & Frankenberger R (2011) Marginal quality of flowable 4-mm base vs. conventionally layered resin composite *Journal of Dentistry* **39**(10) 643-647.
33. Patnana AK, Vanga VNR, & Chandrabhatla SK (2017) Evaluating the marginal integrity of bulk fill fibre reinforced composites in bio-mimetically restored tooth *Journal of Clinical and Diagnostic Research* **11**(6) 24-27.
34. Scientific documentation of SDR. Retrieved online March 2016 from <http://www.dentsply.com>
35. Scientific documentation of Tetric EvoCeram Bulk Fill. Retrieved online March 2016 from <http://www.ivoclarvivadent.com>
36. Moszner N, Fischer UK, Ganster B, Liska R, & Rheinberger V (2008) Benzoyl germanium derivatives as novel visible light photoinitiators for dental materials *Dental Materials* **24**(7) 901-907.
37. Blackham JT, Vandewalle KS, & Lien W (2009) Properties of hybrid resin composite systems containing pre-polymerized filler particles *Operative Dentistry* **34**(6) 697-702.

38. Zorzin J, Maier E, Harre S, Fey T, Belli R, Lohbauer U, Petschelt A, & Taschner M (2015) Bulk-fill resin composites: Polymerization properties and extended light curing *Dental Materials* **31**(3) 293-301.
39. Garoushi SK, Hatem M, Lassila LVJ, & Vallittu PK (2015) The effect of short fiber composite base on microleakage and load-bearing capacity of posterior restorations *Acta Biomaterialia Odontologica Scandinavica* **1**(1) 6-12.

# Perceptibility and Acceptability of Surface Gloss Variations in Dentistry

RS Rocha • TC Fagundes • TMF Caneppele • E Bresciani

## Clinical Relevance

The surface gloss of esthetic restorations is a determinant in accurately mimicking dental structures in terms of tooth proportion and visual perception. Restorative protocol choices might be guided by understanding the threshold of perceptibility and acceptability of gloss differences.

## SUMMARY

**Objective:** To assess the visual perception of observers regarding perceptibility and acceptability of surface gloss variations of resin composites and to determine the limit of per-

Rafael Santos Rocha, DDS, MS, PhD student, GAPEC – Academic Group of Clinical Research – Department of Restorative Dentistry, Institute of Science and Technology of São José dos Campos – São Paulo State University (UNESP)

Ticiane Cestari Fagundes, DDS, MS, PhD, assistant professor, Department of Restorative Dentistry, São Paulo State University (UNESP), Araçatuba Dental School, Araçatuba, SP, Brazil

Taciana Marco Ferraz Caneppele, DDS, MS, PhD, associate professor, GAPEC – Academic Group of Clinical Research – Department of Restorative Dentistry, Institute of Science and Technology of São José dos Campos – São Paulo State University (UNESP)

\*Eduardo Bresciani, DDS, MS, PhD, associate professor, GAPEC – Academic Group of Clinical Research – Department of Restorative Dentistry, Institute of Science and Technology of São José dos Campos – São Paulo State University (UNESP)

\*Corresponding author: Av Eng Francisco José Longo, No.777, São José dos Campos - São Paulo, Brazil, 12245-000; e-mail: eduardob@ict.unesp.br

DOI: <https://doi.org/10.2341/18-184-C>

ceptibility and acceptability of gloss variations.

**Methods:** Eight resin composite specimens and one human tooth specimen were fabricated. The resin specimens were polished to reach a surface gloss, in gloss units (GUs), of 10 GU, 20 GU, 30 GU, 40 GU, 50 GU, 60 GU, 70 GU, and 80 GU, and the human tooth specimen had a surface gloss of 80 GU. Sixty observers were selected to compare the surface gloss of the specimens in a light booth. For the perceptibility assessment, specimens were randomly displayed two at a time. Each observer performed a total of 144 observations. Observers answered two specific questions for determining the level and limit of perceptibility of gloss variations. The acceptability limit of gloss was determined by comparing the resin specimens with the tooth specimen. The observations were performed with dental practice scenarios (illuminant conditions, visualization field, and observers' education). Data were submitted to a nonlinear probit model and nonlinear regression estimation probit (5%).

**Results:** Differences in perceptibility and acceptability were observed for surface gloss variations ( $\Delta$ GU) ( $p < 0.001$ ). Perceptibility in-

creased with  $\Delta$ GU ( $10 < 20 < 30 < 40 < 50 = 60 = 70$ ), while acceptability decreased with  $\Delta$ GU ( $0 = 10 > 20 > 30 > 40 > 50 > 60 = 70$ ). Acceptability and perceptibility limits were 6.4 GU and 35.7 GU, respectively.

**Conclusions: Perceptibility and acceptability of surface gloss are influenced by gloss variations. A variation of 6.4 GU was required for 50% of observers to notice gloss variations. Concerning acceptability, observers required a variation of 35.7 GU to consider differences in gloss not acceptable.**

## INTRODUCTION

Esthetic restorative treatments strongly rely on the material's optical properties for accurate tooth mimicking. Besides the color of resin composites, it has also been reported that translucency, fluorescence, and surface gloss strongly affect the final result of esthetic restorations.<sup>1-3</sup> Esthetic issues might be noticed when the resin gloss is different from dental enamel or other restoratives present in the mouth.<sup>4</sup> Still, specularity (gloss) is capable of altering color perception,<sup>5</sup> which may favor restoration optical outcomes. Thus, the study of surface gloss of resin composites is of great relevance in dentistry.<sup>6</sup>

In clinical practice, accurate restoration finishing and polishing might affect the way light reflects on the tooth surface, generating larger areas of reflection. This alters esthetics, especially the perception of tooth size and shape.<sup>7-9</sup> It is also known that restorations with increased roughness, besides causing esthetic problems, might also lead to greater biofilm retention.<sup>10</sup>

Objective gloss (measured by a glossmeter) is the amount of light reflected on an object's surface at a determined angle ( $20^\circ$ ,  $60^\circ$ , or  $85^\circ$ ) and presenting the same angulation toward light incidence, but in the opposite direction,<sup>11</sup> compared with a polished standard. Although the objective gloss information is of extreme relevance to perform *in vitro* studies, in clinical practice, objective gloss eligibility is unfeasible, given that teeth and/or restorations do not possess a perfectly flat surface (an important attribute for objective gloss interpretation). Furthermore, the subjective qualification of this property is routine in the dental field.

Although it is common to find a variety of studies regarding surface gloss in the dental literature, limited data address the visual perception of gloss of dental materials by observers. Some data support

a positive correlation between surface gloss perception and surface roughness.<sup>12</sup> However, most studies in that field are limited to presenting a decrease in gloss value related to artificial aging and/or surface gloss obtained through finishing and polishing equipment, with gloss variations assessed in percentage<sup>13</sup> or in gloss units.<sup>14-17</sup> Other than the studies highlighting this feature, the real impact of gloss variations in clinical practice is poorly explored, considering the fact that the human eye's capability to perceive gloss intensity and optical variations is unknown. This supports the need for studies assessing the perception of surface gloss of dental materials.

Thus, if surface gloss perception is targeted, various aspects should be considered. The visualization field, type of illuminant, surface characteristics of materials, color, and color of background may affect the perception of gloss.<sup>11,18</sup> It is also known that gloss perception is not only influenced by the material's intrinsic characteristics, surface features, and illuminants but also by the observer's educational level.<sup>11</sup>

Within the reported context, considering dental practice peculiarities such as illuminating type, visualization field, and observers' instruction, the present study aims to evaluate the observers' visual perceptions in relation to the perceptibility and acceptability of surface gloss variations of resin composites. The null hypothesis of the present study, considering the included parameters, is that there is no difference in the levels of perceptibility and acceptability according to the studied gloss differences.

## METHODS AND MATERIALS

The study was approved by the local Institutional Review Board under protocol #1.824.169. Methodology will be divided into two sections for facilitated comprehension.

### Perceptibility Analysis

**Resin Specimen Fabrication**—Eight specimens were fabricated with the nanofilled resin composite Filtek z350 XT (3M ESPE, St Paul, MN, USA). Stainless steel matrices with 6-mm-diameter orifices and of varying thicknesses, were used. First, a 1-mm-thick matrix was used for fabricating the dentin shade (A2D) portion of the specimen. Resin was inserted into the cylindrical orifice in a single increment and light activated for 40 seconds, using light-emitting diode light (Radii-Cal, SDI, Victoria,

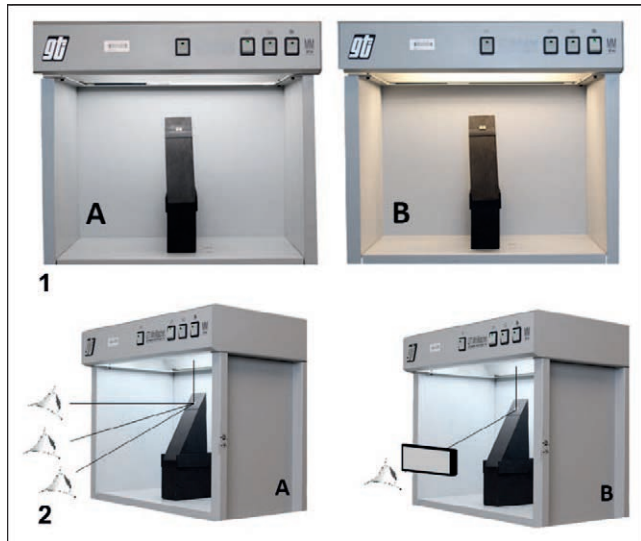


Figure 1. Light booth with different illuminant exposures. (A): D65 light; (B): Fluorescent light.

Figure 2. Representative figure of the visualization field for the light booth. (A): Nonfixed visualization field; (B): Visualization field at 60°.

Australia) at  $900\text{mW}/\text{cm}^2$  and assessed by a radiometer.

Next, a 1.5-mm matrix was used. Dentin resin specimens (A2D) were inserted into the orifices, leaving a 0.5-mm space free of restorative material. A2E resin was then inserted in a single increment, covered with a Mylar strip, and pressed with a glass slide. The resin composite was light activated for 40 seconds. Specimens 1.5 mm thick were obtained with 1 mm in the A2D shade and 0.5 mm in the A2E shade.

### Specimen Polishing Assessment

All specimens received an initial surface polishing (A2E resin surface) with abrasive disc grits #1200 and #2400 (Fepa-P, Exttec, Enfield, CT, USA). The specimens were mounted in a polishing machine (DP-10, Panambra, São Paulo, SP, Brazil) at 300 rpm under distilled water cooling for 30 seconds for each abrasive disc. Then the specimens received different polishing protocols using abrasive disc grits #1200, #2400 and/or #4000 according to the surface gloss to be achieved. Abrasive discs were fixed to a flat surface and used under water irrigation. Polishing was performed with circular movements (resembling a number 8) holding the specimen by the thumb with no pressure, following a previously described protocol.<sup>19</sup> Such movement allowed the surface to be polished leaving no surface scratches.

Following the polishing procedure, the specimens were immersed in an ultrasonic bath to remove residues. Then, the surface gloss was measured. The accepted variation considering each target group was  $\pm 0.9$ . Polishing was repeated in cases where the surface gloss did not match the specimen's group. Gloss was measured using Novo-Curve (Rhopoint Instrumentation, East Sussex, UK) with a  $2 \times 2$  mm square area and 60° of geometry (light incidence), with values expressed in gloss units (GU).<sup>20-22</sup> A metallic positioner was used to block and eliminate possible environment light interference.<sup>23</sup> Three random measurements were performed for each specimen on the A2E resin surface, and the average obtained was used as the final gloss value. The 60° of geometry was chosen considering the targeted gloss values were within the medium gloss range.<sup>24,25</sup>

Specimens were allocated according to surface gloss into 10 GU, 20 GU, 30 GU, 40 GU, 50 GU, 60 GU, 70 GU, or 80 GU groups.

**Selection of Observers**—Sixty observers between 18 and 50 years old were selected: 20 laypeople (no graduation and no relation to dentistry), 20 undergraduate dental students (fifth and sixth semester students who have initiated clinical practice), and 20 dentists (with more than two years of practice).<sup>26</sup> The sample size considered previous studies on the perceptibility and acceptability of color, in which 20 observers per groups were required.<sup>23</sup> Participants were included after written consent was signed.

To participate, observers could not have visual impairment. If they did, then participants were required to have visual correction near 10/10 (or 10/3 on the metric scale) according to the Snellen visual graph.<sup>27</sup> This means participants should be able to visualize at least 80% of letters in line 10 at a distance of 3 m from the graph. Participants were also questioned about stereoscopic vision and asked to report possible differences in vision between the two eyes, which was expected to be normal.<sup>27</sup>

**Assessing Gloss Perception**—Specimens were randomly arranged in the light booth (Elcometer 6300 MM-4E, Gti, New Jersey, USA), two at a time. Specimens were positioned so to allow light incidence of different illuminants (D65 and fluorescent light; Figure 1A,B) on the specimen's surface at a light reflection angle of 60°.

Of the observers, 50% first observed the specimens' surfaces at a nonfixed angle of visualization during gloss qualification, followed by one of the selected illuminants. After those observations, the

booth was closed and observers performed gloss qualifications at a 60° angle (Figure 2A,B).

The other 50% of the observers started with the gloss qualification at a 60° angle, followed by observations in a nonfixed angle of visualization.

All observers performed assessments with a nonfixed angle of visualization and a predetermined angle of visualization at 60° under the two illuminants (D65: daylight and fluorescent light), for a total of 28 combinations of specimen evaluations under each condition. Observers performed random observation of specimens. To avoid eyestrain, observers were given five-minute breaks after each 30 minutes of analysis.<sup>28</sup> Any combination of specimens observed always presented gloss differences. Observers were given no previous explanation about surface gloss.

Perceptibility of surface gloss was collected from the answers of the following questions: (1) Do both specimens present the same surface gloss? and (2) If no, which one presents the greater surface gloss?.

### Acceptability Study

For acceptability, all resin composite specimens (used in the perceptibility study) were randomly compared with a polished human tooth specimen.

*Fabrication of the Human Tooth Standard Specimen*—An anterior human tooth in the A2 shade, assessed with Vita Classical scale (Vita Zahnfabrik, Bad Säckingen, Germany), was selected and observed by three observers at different periods. The tooth was sectioned 2 mm below the cemento-enamel junction. Afterward, an enamel/dentin disc with a 6.0 mm diameter was cut with a trephine diamond bur connected to a drilling bench machine.

Then, the dentin surface was exposed and ground with abrasive disc grit #800 (Fepa-P, Exttec) and mounted in a polishing machine (DP-10, Panambra) at 300 rpm with a load close to 100 g under irrigation until the dentin thickness reached approximately 1.0 mm. Thereafter, the flat part was placed inside a metallic device with a depth of 1.5 mm and ground, resulting in a 1.5 mm specimen (0.5 mm enamel and 1 mm dentin).

*Polishing Human Tooth Specimen*—The human tooth specimen was polished to 80 GU using abrasive disc grits #1200, #2400, and #4000 (Fepa-P, Exttec). The specimen was coupled to a polishing machine (DP-10, Panambra) at 300 rpm with an approximate load of 100 g under distilled water cooling for 30 seconds per disc. After the polishing procedure, the specimen was immersed in an ultrasonic bath to

remove residues. The specimen's gloss was measured, using the Novo-Curve, right after polishing and immediately before the observations took place. Three measurements were performed, and the average obtained was used as the final value (80 GU, variation of  $\pm 0.90$  at most).

*Surface Gloss Assessment in Relation to Acceptability*—For the acceptability study, the resin specimens (10 GU, 20 GU, 30 GU, 40 GU, 50 GU, 60 GU, 70 GU, and 80 GU) were randomly and individually arranged in the light booth (Elcometer 6300 MM-4E, Gti) together with the human tooth specimen (80 GU). Specimens were adapted to a device that allowed light incidence (D65 and fluorescent light) toward the specimen's surface at a 60° angle.

All observers performed their observations with nonfixed and 60° angles of visualization under both illuminants. To avoid eyestrain, observers were given five-minute breaks after each 30 minutes of analysis.<sup>28</sup>

Observers answered two questions. First: Do both specimens present the same surface gloss? In case of a positive answer, there were no further questions and the possible gloss difference was considered acceptable. However, in case of a negative answer, the second question was asked: Would this difference be clinically accepted in an anterior restored tooth?

*Analysis of Variables*—Perceptibility and acceptability data were submitted to a linear/nonlinear regression probit model (5%),<sup>29</sup> which presents the binary response as the dependent variable (right or wrong answer on surface gloss assessment) and the frequency variable (number of right and wrong answers) and gloss variations as the independent variables.

## RESULTS

Each observer assessed 112 perceptibility combinations and 32 acceptability combinations, with two illuminants and two visualization field methods, totaling 144 observations per observer.

Perceptibility and acceptability descriptive analyses are presented in Figures 3 and 4, respectively, by means of the percentage of obtained right answers.

The percentage of gloss perceptibility and acceptability, considering the observers, resulted in differences among gloss variations ( $\Delta$ GU) ( $p < 0.001$ ), according to probit regression analysis. For perceptibility, increased gloss variations ( $\Delta$ GU) resulted in an increased perception by observers, with no differences among  $\Delta$ GUs 50, 60, and 70 ( $\Delta$ GU 10 < 20 < 30 < 40 < 50 = 60 = 70). For acceptability, in-

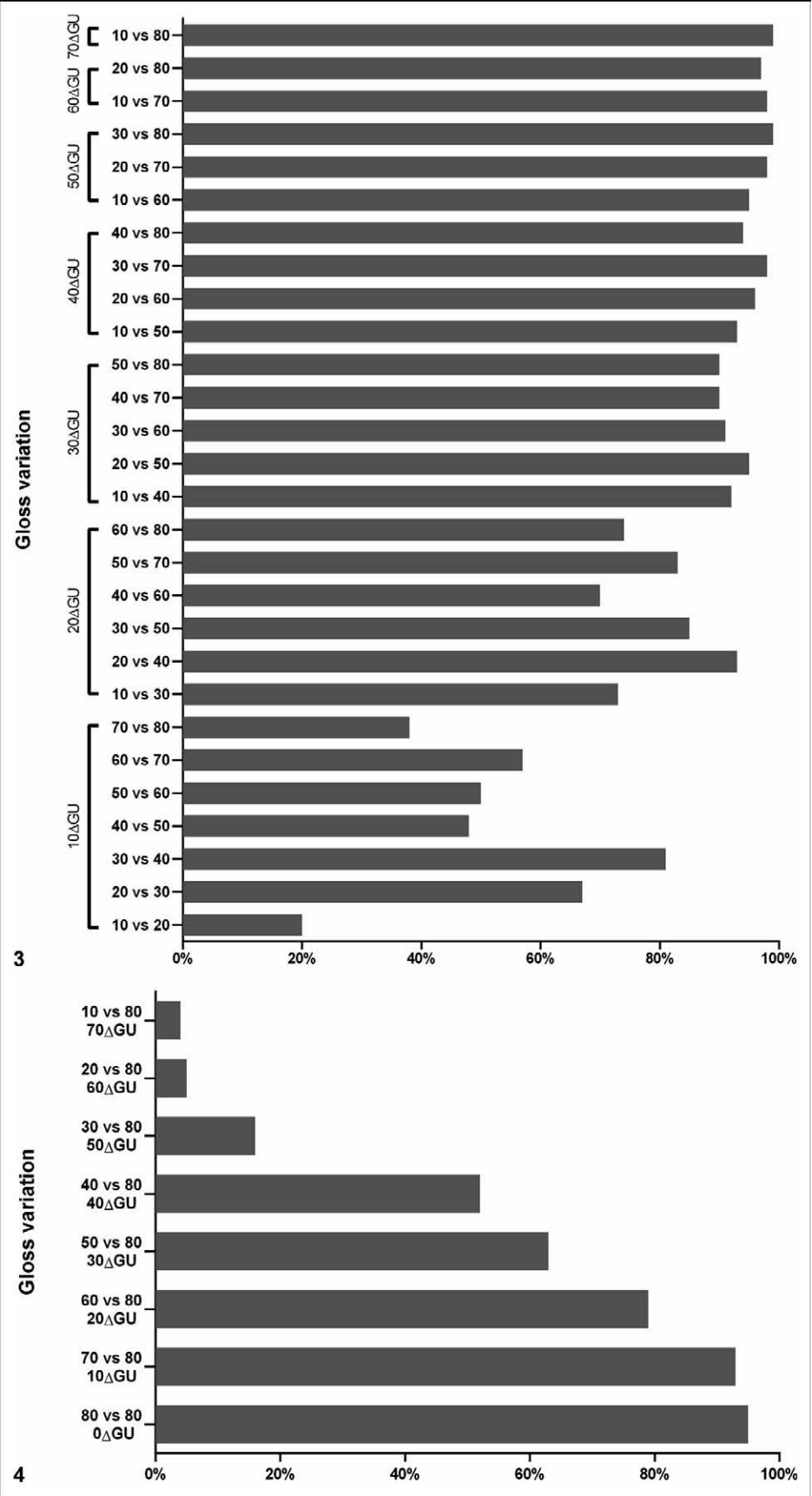


Figure 3. Percentage of perceptibility by observers according to differences in gloss of specimens. The x-axis is arranged according to the percentage (%) of detected differences. The y-axis represents the difference of gloss ( $\Delta$ GU) and the combination of specimens assessed.

Figure 4. Percentage of acceptability by observers according to differences in gloss of specimens. The x-axis is arranged according to the percentage (%) of accepted differences. The y-axis represents the difference of gloss ( $\Delta$ GU) and the combination of specimens assessed.

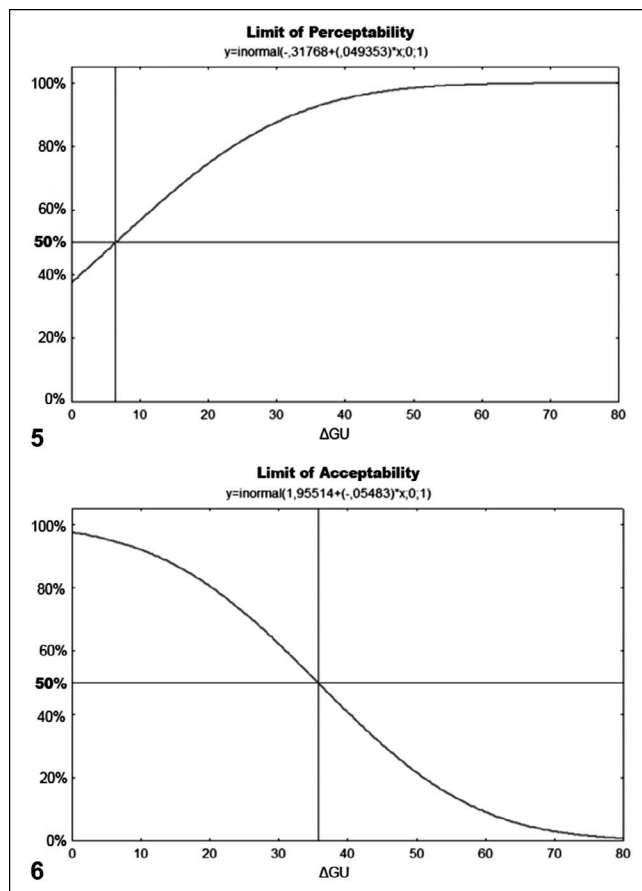


Figure 5. Limit of perceptibility at 6.4  $\Delta$ GU defined by probit nonlinear regression. The x-axis presents the  $\Delta$ GU values. The y-axis shows the estimated percentage of observers perceiving the difference. The limit of perceptibility is defined by the  $\Delta$ GU, in which 50% of observers perceive the gloss difference.  $\Delta$ GU indicates the difference of gloss.

Figure 6. Limit of acceptability at 35.7  $\Delta$ GU defined by probit nonlinear regression. The x-axis presents the  $\Delta$ GU values. The y-axis shows the estimated percentage of observers accepting the difference. The limit of acceptability is defined by the  $\Delta$ GU, in which 50% of observers still accept the gloss difference.  $\Delta$ GU indicates the difference of gloss.

creased gloss variations resulted in decreased acceptability levels, with no acceptance differences between 0 and 10GUs, and between 60 and 70GUs ( $\Delta$ GU 0=10>20>30>40>50>60=70).

The limit of perceptibility for surface gloss, determined by nonlinear estimation probit, taking into account the value in which more than 50% of observers detected differences, was 6.4 GU.

In relation to the limit of acceptability, defined as the value in which half of the observers would still accept gloss differences in a restoration, the value was 35.7 GU.

Figures 5 and 6 correspond to the curves of perceptibility and acceptability, obtained by nonlinear regression, according to gloss variations. The figures also present the detected limits of perceptibility and acceptability of gloss variations.

## DISCUSSION

There is no reported protocol for the methodology proposed in the present study. Thus, considering two main aspects (complexity in gloss observation and possible variables in the observation process), the methodology was designed to include the greatest number of variations in order to obtain an overall scenario for surface gloss perception and acceptability in dentistry. The questions, sample size of observers, and sample arrangements were based on studies determining the limit of perceptibility and acceptability of differences in the color of restorative materials.<sup>26</sup>

In relation to the gloss evaluation, it is known that the subjective assessment of surface gloss seems to be a complex task.<sup>30</sup> It is reported that observers, in general, consider two aspects to classify gloss: (1) the amount of light reflected on a surface and (2) the sharpness of images seen from a reflective surface.<sup>31</sup> No previous information was provided to the volunteers regarding gloss properties and gloss observation; thus, the study considered the previous education of volunteers. In order to obtain a general overview of possible variations in the observers, various volunteers were included in the study (laypeople, dental students, and dentists). The authors do not know if educated observers would perform better, suggesting an area for new studies to assess that concern.

The possible variables affecting the visualization process were exposure to different illuminants (D65 and fluorescent light)<sup>32,33</sup> and the angle of visualization of specimens (nonfixed or at 60°). Two illuminant types, D65 and fluorescent, were used in this study, as D65 (daylight representative) and fluorescent (light representative of houses and business facilities) illuminants are considered the most frequent in the daily routine.<sup>32,33</sup> A material's characteristics are differentiated by light reflection.<sup>34</sup> If a large portion of light is diffusely reflected, one perceives the material as having a rough and spongy surface, like a plaster surface. On the other hand, if spectral reflection is more prevalent, one perceives the material as having a glossy and polished surface, like metals.<sup>34</sup> Besides the specular reflection areas, gloss might be regulated by the

object's sharpness, contrast, and surface curvature.<sup>30</sup>

The angle of visualization can also affect the surface gloss qualification in terms of time, as observed in the present study (data not shown). This assumption is related to the fact that while observing the same object under different angles of light incidence, observers presented a visual compensation toward the incidence of light, leading to no significant differences in gloss assessment.<sup>35</sup> This is named *gloss constancy*, and this compensation might also be influenced by the angle of observation. The two visualization possibilities in the present study were used to reduce the influences of routine exposure esthetic restorations undergo.

The null hypothesis was rejected. As expected, the perceptibility of gloss increased with increasing gloss differences ( $\Delta\text{GU } 10 < 20 < 30 < 40 < 50 = 60 = 70$ ) (Figure 3). No differences were detected after  $\Delta\text{GU}$  of 50, mainly due to the high rates of detected differences in those combinations. Acceptability, on the other hand, increased with decreasing gloss variations ( $\Delta\text{GU } 0 = 10 > 20 > 30 > 40 > 50 > 60 = 70$ ). No differences were detected in the two extremes in gloss differences ( $\Delta\text{GU}$  of 0 and 10 or  $\Delta\text{GU}$  of 60 and 70) (Figure 4). The patterns of perceptibility and acceptability are similar to the findings in studies on color assessment of restorative materials<sup>26</sup> and a preliminary study on gloss assessment.<sup>19</sup> Although the observed pattern was expected (Figures 3 and 4), the importance of the present results is related to the fact they might be used to compare or support studies or to indicate that new studies associating gloss with roughness and biofilm accumulation in clinical scenarios are needed.

The gloss variation used in this study took into account data obtained in a systematic review,<sup>6</sup> which gathered GU values for different finishing and polishing devices in different types of resin composites. In such a revision, the gloss minimum value was 12.5 GU, and the gloss maximum value was 94.7 GU. Based on those values, the variation adopted in the present study was 10 to 80 GU, with an interval of 10 GU.

It is reported that gloss perception presents breakpoints in the observation pattern in areas close to 20 GU and 80 GU.<sup>25</sup> That condition was noticed in the present study, once the perception of gloss variation was hindered, in combinations of 10 vs 20 GU and 70 vs 80 GU (Figure 3).

The results of the present study might not be completely related to clinical conditions, especially

due to the characteristics of specimens, polishing protocols, and assessment conditions. The specimens used during the study were flat and regular and had no mesostructures (bumps on the surface), which might have facilitated the qualification of surface gloss.<sup>32</sup> In clinical practice, other elements should be considered. The human anterior tooth presents depression areas near the mesial and distal line angles, which results in greater light reflection.<sup>7,9</sup> However, this feature cannot be evaluated in laboratory conditions, as a flat surface is required for assessing a specimen's objective gloss. Any surface irregularity would change the direction of light reflection, leading to an incompatible amount of luminosity received by the sensor, therefore compromising the gloss results. Thus, the polishing might not be related to clinical protocols and may be a possible limitation of the study while considering clinical extrapolation.

Still, specimens were placed inside the light box under a black matte background. In a study by Doerschner and others,<sup>36</sup> the authors concluded that spheres were perceived as glossier when analyzed on a black background instead of a white one. According to the authors, dark backgrounds lead to a glossier appearance of objects. Thus, the present data might have been different if a white background had been used. Considering that the background of teeth in clinical conditions is usually dark, the use of a black background may be more realistic for the clinical scenario. Overall, a 6.4 GU was achieved as the limit of perceptibility and 35.7 GU as the limit of acceptability, meaning that 50% of the observers detected differences of 6.4 GU and accepted differences up to 35.7 GU (Figures 5 and 6). These data may strongly support the discussion of future studies assessing different polishing protocols or analyzing resin characteristics in relation to surface gloss toward aging.

Rodrigues-Junior and others<sup>17</sup> assessed the effects of different finishing and polishing protocols, together with different types of resin composites, in relation to the objective gloss. According to that study's data, Z350XT resin surface polished with the Superflif device presented average surface gloss values of 41.32 GU, while the same resin polished with DFL presented values of 52.4 GU. The variation was even larger when the authors compared Enhance and Sof-Lex devices, with surface gloss results at 14 and 59 GU, respectively. In light of the present perceptibility results, different polishing systems might lead to different gloss results that are visible to the human eye. Moreover, knowing the average

values between Enhance and Sof-Lex, if two restorations in the maxillary anterior teeth were polished with each device, it is likely that the esthetic issues would be perceived and that the treatment would not meet patient expectations once the previously defined gloss difference is greater than the acceptability limit, as presently detected.

However, there is an obstacle when exploring this study in clinical practice. In a real situation, several other elements might influence surface gloss qualification. The oral cavity is an area full of shadows, impairing light incidence and reflection. Teeth may present mesostructures that would possibly cause different reflection patterns. In addition, the presence of saliva on the dental surface is likely to completely modify the perception and/or evaluation of this optical property. New studies should be carried out to complement the present results, taking into consideration other aspects that may influence surface gloss in dentistry.

## CONCLUSIONS

Based on the study methodology, it can be concluded that increasing gloss variations resulted in greater perceptibility and reduced acceptability. A variation of 6.4 GU is required so that 50% of observers notice the differences between two composite specimens regarding surface gloss. In relation to acceptability, a variation of 35.7 GU is necessary for observers to reject the gloss differences in the restoration treatment.

## Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the ICT - UNESP. The approval code for this study is: #1.824.169.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 17 April 2019)

## REFERENCES

1. Terry DA, Geller W, Tric O, Anderson MJ, Tourville M, & Kobashigawa A (2002) Anatomical form defines color: function, form, and aesthetics *Practical Procedures & Aesthetic Dentistry* **14**(1) 59-67.
2. Brainard DH & Maloney LT (2004) Perception of color and material properties in complex scenes *Journal of Vision* **4**(9) ii-iv.
3. Manojlovic D, Dramicanin MD, Lezaja M, Pongprueksa P, Van Meerbeek B, & Miletic V (2016) Effect of resin and photoinitiator on color, translucency and color stability of conventional and low-shrinkage model composites *Dental Materials* **32**(2) 183-191.
4. O'Brien WJ, Johnston WM, Fanian F, & Lambert S (1984) The surface roughness and gloss of composites *Journal of Dental Research* **63**(5) 685-688.
5. Xiao B & Brainard DH (2006) Color perception of 3D objects: constancy with Respect to variation of surface gloss *Proceedings APGV : Symposium on Applied Perception in Graphics and Visualization Symposium on Applied Perception in Graphics and Visualization* **2006** 63-68.
6. Kaizer MR, de Oliveira-Ogliari A, Cenci MS, Opdam NJ, & Moraes RR (2014) Do nanofill or submicron composites show improved smoothness and gloss? A systematic review of in vitro studies *Dental Materials* **30**(4) e41-e78.
7. Heymann HO (1987) The artistry of conservative esthetic dentistry *Journal of the American Dental Association Special No* 14E-23E.
8. Brezniak N, Wasserstein A, & Shmuli T (2012) [Light reflection zone on the incisors' surface—A new parameter for smile esthetics evaluation] *Refu'at ha-peh veha-shinayim* **29**(3) 39-43, 57.
9. Fahl Jr N (2011) Step-by-step approaches for anterior direct restorative challenges *Journal of Cosmetic Dentistry* **26**(4) 42.
10. Aykent F, Yondem I, Ozyesil AG, Gunal SK, Avunduk MC, & Ozkan S (2010) Effect of different finishing techniques for restorative materials on surface roughness and bacterial adhesion *Journal of Prosthetic Dentistry* **103**(4) 221-227.
11. Chadwick AC & Kentridge RW (2015) The perception of gloss: a review *Vision Research* **109** 221-235.
12. Barucci-Pfister N & Gohring TN (2009) Subjective and objective perceptions of specular gloss and surface roughness of esthetic resin composites before and after artificial aging *American Journal of Dentistry* **22**(2) 102-110.
13. Rocha RS, Oliveira AC, Caneppele TMF, & Bresciani E (2017) Effect of artificial aging protocols on surface gloss of resin composites *International Journal of Dentistry* **2017** 3483171.
14. Jain V, Platt JA, Moore K, Spohr AM & Borges GA (2013) Color stability, gloss, and surface roughness of indirect composite resins *Journal of Oral Science* **55**(1) 9-15.
15. Salgado VE, Cavalcante LM, Silikas N, & Schneider LF (2013) The influence of nanoscale inorganic content over optical and surface properties of model composites *Journal of Dentistry* **41**(Supplement 5) e45-53.
16. Valente LL, Peralta SL, Ogliari FA, Cavalcante LM, & Moraes RR (2013) Comparative evaluation of dental resin composites based on micron- and submicron-sized monomodal glass filler particles *Dental Materials* **29**(11) 1182-1187.
17. Rodrigues-Junior SA, Chemin P, Piaia PP, & Ferracane JL (2015) Surface roughness and gloss of actual compos-

- ites as polished with different polishing systems *Operative Dentistry* **40(4)** 418-429.
18. Landy MS (2007) Visual perception: A gloss on surface properties *Nature* **447(7141)** 158-159.
  19. Tessarin FBP, Meirelles LCF, Rocha RS, Caneppele TMF, & Bresciani E. (2018) Influence of illuminants and different observers on the perception of surface gloss of resin composite *Brazilian Dental Science* **21(4)** 451-460.
  20. Anagnostou M, Chelioti G, Chioti S, & Kakaboura A (2010) Effect of tooth-bleaching methods on gloss and color of resin composites *Journal of Dentistry* **38(Supplement 2)** e129-e136.
  21. Hosoya Y, Shiraishi T, Odatsu T, Nagafuji J, Kotaku M, Miyazaki M, & Powers JM (2011) Effects of polishing on surface roughness, gloss, and color of resin composites *Journal of Oral Science* **53(3)** 283-291.
  22. Lefever D, Perakis N, Roig M, Krejci I, & Ardu S (2012) The effect of toothbrushing on surface gloss of resin composites *American Journal of Dentistry* **25(1)** 54-58.
  23. Antonson SA, Yazici AR, Kilinc E, Antonson DE, & Hardigan PC (2011) Comparison of different finishing/polishing systems on surface roughness and gloss of resin composites *Journal of Dentistry* **39(Supplement 1)** e9-e17.
  24. International Organization for Standardization (2014). *ISO 2813: Paints and Varnishes: Determination of Gloss Value at 20 Degrees, 60 Degrees and 85 Degrees* International Organization for Standardization, Geneva.
  25. Ji W, Pointer MR, Luo RM, & Dakin J (2006) Gloss as an aspect of the measurement of appearance *Journal of the Optical Society of America A, Optics, Image Science, and Vision* **23(1)** 22-33.
  26. Paravina RD, Ghinea R, Herrera LJ, Bona AD, Igiel C, Linninger M, Sakai M, Takahashi H, Tashkandi E, & Perez Mdel M (2015) Color difference thresholds in dentistry *Journal of Esthetic and Restorative Dentistry* **27(Supplement 1)** S1-S9.
  27. Ged G, Obein G, Silvestri Z, Le Rohellec J, & Vienot F (2010) Recognizing real materials from their glossy appearance *Journal of Vision* **10(9)** 18.
  28. Wee AG LD, Shroyer KM, & Johnston WM. (2007) Use of a porcelain color discrimination test to evaluate color difference formulas *Journal of Prosthetic Dentistry* **98(2)** 101-109.
  29. Douglas RD, Steinhauer TJ, & Wee AG (2007) Intraoral determination of the tolerance of dentists for perceptibility and acceptability of shade mismatch *Journal of Prosthetic Dentistry* **97(4)** 200-208.
  30. Maloney LT & Brainard DH (2010) Color and material perception: achievements and challenges *Journal of Vision* **10(9)** 19.
  31. Harrison VGW & Poulter SRC (1951) Gloss measurement of papers-the effect of luminance factor *British Journal of Applied Physics* **2(4)** 92.
  32. Marlow PJ & Anderson BL (2013) Generative constraints on image cues for perceived gloss *Journal of Vision* **13(14)** 2.
  33. Olkkonen M & Brainard DH (2010) Perceived glossiness and lightness under real-world illumination *Journal of Vision* **10(9)** 5.
  34. Fleming RW, Dror RO, & Adelson EH (2003) Real-world illumination and the perception of surface reflectance properties *Journal of Vision* **3(5)** 347-368.
  35. Obein G, Knoblauch K, & Vienot F (2004) Difference scaling of gloss: nonlinearity, binocularity, and constancy *Journal of Vision* **4(9)** 711-720.
  36. Doerschner K, Maloney LT, & Boyaci H (2010) Perceived glossiness in high dynamic range scenes *Journal of Vision* **10(9)** 11.

# Volumetric Cuspal Deflection of Premolars Restored With Different Paste-like Bulk-fill Resin Composites Evaluated by Microcomputed Tomography

G Demirel • IH Baltacioglu • ME Kolsuz • M Ocak • B Bilecenoglu • K Orhan

## Clinical Relevance

The use of paste-like bulk-fill composites for class II MOD cavities reduces cuspal deflection compared to the paste-like conventional resin composite.

## SUMMARY

**Objectives:** The aim of this study was to measure the volumetric cuspal deflection of premolars restored with different paste-like bulk-fill resin composites using microcomputed tomography (micro-CT).

**Methods and Materials:** A total of 35 freshly extracted human maxillary second premolars were selected for this study. Standardized large MOD cavities were prepared in each

premolar with a bucco-lingual width of 4 mm and a cavity depth of 4 mm measured from the palatal cusp tip. After cavity preparation, all samples were scanned immediately using a micro-CT system. After the initial micro-CT scanning, restorative procedures were performed. Four groups received different paste-like bulk-fill composites—Beautifil-Bulk Restorative (BBR), Admira Fusion x-tra (AFX), x-tra fill, and Sonic Fill—and the control group received a conventional universal composite and Clearfil Majesty Esthetic (CME). Immediately after the restorative procedure, each

Gülbike Demirel, PhD, DDS, Department of Restorative Dentistry, School of Dentistry, Ankara University, Ankara, Turkey

Ismail Hakki Baltacioglu, PhD, DDS, Department of Restorative Dentistry, School of Dentistry, Ankara University, Ankara, Turkey

Mehmet Eray Kolsuz, PhD, DDS, Department of Dentomaxillofacial Radiology, School of Dentistry, Ankara University, Ankara, Turkey

Mert Ocak, PhD, lecturer, Vocational School of Health, Ankara University, Ankara, Turkey

Burak Bilecenoglu, DDS, PhD, associate professor, Department of Anatomy, School of Dentistry, Ankara, Turkey

\*Kaan Orhan, PhD, professor, Department of Dentomaxillofacial Radiology, School of Dentistry, Ankara University, Ankara, Turkey. OMFS IMPATH Research Group, Department of Imaging & Pathology, Faculty of Medicine, University of Leuven Oral & Maxillofacial Surgery, University Hospitals Leuven, Leuven, Belgium

\*Corresponding author: University Hospitals Leuven, Kapucijnenvoer 7 blok a Leuven 3000, Belgium; e-mail: kaan.orhan@uzleuven.be

DOI: <http://doi.org/10.2341/19-019-L>

tooth was scanned by micro-CT in the same manner as the initial scanning. The buccal and palatal regions of each restoration were evaluated separately in terms of cuspal deflection. One-way analysis of variance was used to compare the effect of the composite resin, and multiple comparisons were performed by the Tukey test with a level of significance of  $\alpha = 0.05$ .

**Results and Discussion:** Multiple comparisons showed that teeth restored with the conventional paste-like composite and CME (control) had significantly different cuspal deflection from those filled with paste-like bulk-fill composites ( $p < 0.05$ ). Among the bulk-fill composites, a significant difference was observed between BBR and AFX ( $p < 0.05$ ).

**Conclusions:** Paste-like bulk-fill resin composites had significantly lower cuspal deflection than the conventional paste-like resin composite tested.

## INTRODUCTION

Bulk-fill resin composites have gained popularity among clinicians for posterior restorations due to improvements of materials and enhanced curing,<sup>1</sup> which controls the effect of curing stresses.<sup>2,3</sup> Although the conventional 2-mm incremental layering procedure is needed for conventional composites, advanced bulk-fill composites can be cured as a single bulk layer with a thickness of 4 to 6 mm.<sup>4</sup> According to their viscosity, these materials can be classified into “flowable/base” or “paste-like/full-body” types. Due to their low amount of filler, flowable bulk-fill composites have less wear resistance; therefore, completing the top layer with a composite, which has a high amount of filler, is necessary.<sup>5,6</sup> The paste-like bulk-fill materials contain higher amounts of filler. These composites are particularly viscous, sculptable, and highly wear resistant, and capping of the top layer is not necessary.<sup>6</sup>

Bulk-fill composites also contain monomers, similar to conventional composites. During the polymerization reaction, the monomers approach each other,<sup>7</sup> resulting in a volumetric shrinkage of composite material.<sup>8</sup> Shrinkage can create stress in the composite structure, and the dental tissue can also cause marginal gaps, microleakage, and cuspal deflection.<sup>9,10</sup> Cuspal deflection may lead over time to microcrack propagation, enamel cracks, crazing, reduction in fracture resistance of the restored tooth,

postoperative sensitivity, pulpal problems, and, in extreme cases, cusp fracture.<sup>11,12</sup>

In the literature, several techniques have been reported to calculate cuspal deflection in wide class II cavities with composite restorations, and values of up to 50  $\mu\text{m}$  of mean cusp deformation have been reported.<sup>13-17</sup> These studies used microscopy, strain gauges, flexible ribbons, linear variable differential transformers, and trans-tooth illumination techniques. With these methodologies, cuspal deflection can be measured in one dimension, giving a linear result. Two- and three-dimensional (2D and 3D) microcomputed tomography (micro-CT) allows evaluation of the restorative material itself and adaptation to the dental structures with high spatial resolution.<sup>18</sup> This technique has proved its adequacy to evaluate the polymerization contraction vectors,<sup>19</sup> and it can represent both the pattern and the volume of polymerization shrinkage<sup>20</sup> and calculate the shrinkage volume of the resin composites.<sup>18,21</sup> Although a few studies have examined cuspal deflection using micro-CT, no previous study has analyzed the 3D volumetric cuspal deflection of premolars restored with different paste-like bulk-fill composites using micro-CT.

The aim of this study was to measure the volumetric cuspal deflection of premolars restored with different paste-like bulk-fill resin composites using micro-CT. The null hypothesis was that paste-like bulk-fill resin composites would not generate different amounts of cuspal deflection from conventional resin composites.

## METHODS AND MATERIALS

### Tooth Selection and Cavity Preparation

A total of 35 freshly extracted human maxillary second premolars that were free from caries, hypoplastic defects, or cracks were selected for this study. The maximum buccal-palatal width (BPW) and the maximum mesio-distal width (MDW) of each premolar were measured prior to any work using a digital caliper (Mitutoyo, Kawasaki, Japan) with a tolerance of 10  $\mu\text{m}$ , and the teeth were distributed into five groups ( $n=7$ ) such that the variance of the mean BPW and MDW between groups was less than 5%. Teeth were embedded in a cube-shaped plastic mold 2 cm in diameter using chemically cured acrylic resin, and the mold extended 2 mm cervical to the cemento-enamel junction to simulate the position of the tooth in the alveolar bone and to prevent reinforcement of the crown by the base. Four spheres were placed around

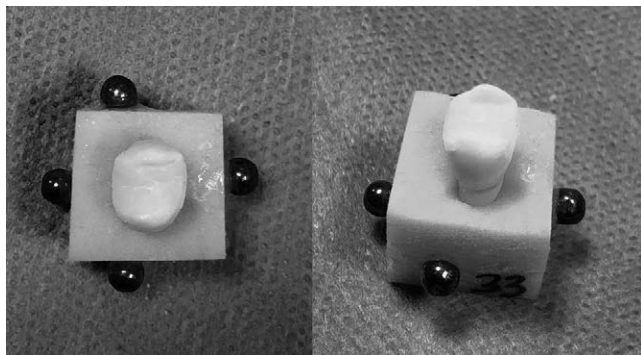


Figure 1. Sample with spheres placed around the acrylic base.

the acrylic base, which functioned as stable reference areas, as shown in Figure 1.

Standardized large MOD cavities were prepared in each premolar with a bucco-lingual width of 4 mm and a cavity depth of 4 mm measured from the palatal cusp tip. The dimensions of each cavity preparation were verified using a digital caliper. The cavities were prepared without proximal boxes to minimize the preparation variation.<sup>22</sup> The buccal and lingual walls of each cavity were prepared parallel to each other.<sup>22,23</sup> Cavosurface margins were prepared without beveling. Cavities were prepared with a straight diamond bur with a rounded end (#881H-012, Hager & Meisinger GmbH, Neuss, Germany) using a high-speed hand piece with copious air-water spray. The bur was changed after each cavity preparation. Teeth with pulp exposure were excluded from the study.

### Initial Micro-CT Scanning

All samples were scanned immediately after cavity preparation using a micro-CT system (Bruker SkyScan 1275, Kontich, Belgium). The teeth were positioned with the buccal surface facing the X-ray tube to ensure standardization. The scanning conditions were as follows: 100 kVp, 120 mA, 0.5-mm Al/Cu filter, 9.1- $\mu$ m pixel size, and rotation at 0.1 steps. Flat field calibration was carried out prior to each scan; moreover, the manufacturer's protocol was followed to minimize ring artifacts. Each specimen was rotated 360° during the five-minute integration time. The mean time of scanning was approximately 70 minutes. The beam hardening correction and other parameters, including optimal contrast, were set according to the manufacturer's instructions.

### Restorative Procedures

After the initial micro-CT scanning, to eliminate the dehydration caused by the dry environment of the

scanning device, the samples were rehydrated for 30 minutes in distilled water, as in the study by Shirani and others,<sup>24</sup> and then restorative procedures were performed. Four groups received different paste-like bulk-fill composites—Beautifil-Bulk Restorative (BBR), Admira Fusion x-tra (AFX), x-tra fill (XTF), and Sonic Fill (SF)—and the control group received a conventional universal composite and Clearfil Majesty Esthetic (CME) (Table 1).

Metal matrix bands (Adapt SuperCap Matrix no 2182, Kerr-Hawe, Bioggio, Switzerland) were seated for each group. The enamel was etched for 15 seconds with 35% orthophosphoric acid (K-ETCH-ANT Syringe, Kuraray Noritake Dental Inc, Okayama, Japan), rinsed for 10 seconds, and then dried with cotton pellets, leaving the surface moist, according to the manufacturer's directions. This procedure was followed by the application of universal adhesive (Single Bond Universal, 3M ESPE, St Paul, MN, USA) to the entire cavity with a rubbing action for 20 seconds. The excess solvent was evaporated with a gentle air spray for five seconds. The bonding agent was cured for 10 seconds with an LED curing light (Elipar DeepCure-S LED Curing Light, 3M ESPE). The output power was periodically monitored with the radiometer of the curing unit to ensure an intensity of at least 1000 mW/cm<sup>2</sup>. The four bulk-fill composites were applied to the prepared cavities in single-bulk increments, while the conventional composite was applied in three horizontal increments. Each layer was cured according to the manufacturers' instructions (Table 1).

### Cuspal Deflection Analysis by Micro-CT

Immediately after the restorative procedure, each tooth was scanned by micro-CT in the same manner as the initial scanning. NRecon software (ver. 1.7.4.2, SkyScan, Brüker, Kontich, Belgium) and CTAn (ver. 1.18.4.0, SkyScan, Brüker) were used for imaging and measurement of the samples. For reconstruction, the algorithm by Feldkamp and others<sup>25</sup> was used to achieve axial, 2D slices. For the reconstruction parameters, ring artifact correction and smoothing were fixed at zero, and the beam artifact correction was fixed at 50%. Cross images were then reconstructed from the whole volume using micro-CT. The reconstructed images were superimposed using DataViewer software (ver. 1.5.6.2, SkyScan, Brüker). Reference points that were distant from the area affected by shrinkage were selected to align the different images of the prepared restored teeth. The spherical balls that were attached before scanning were used for this

Table 1: *Materials Used in the Study (Information as Disclosed by the Manufacturers)*

| Material                         | Shade     | Lot Number | Matrix Composition                                                                                                                                                                                            | Filler % by Weight | Polymerization Shrinkage by Volume (%) | Recommended Thickness | Recommended Curing Time and Light Intensity for Each Layer | Number of Layer of Samples | Manufacturer                  |
|----------------------------------|-----------|------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------|----------------------------------------|-----------------------|------------------------------------------------------------|----------------------------|-------------------------------|
| Beautifil-Bulk Restorative (BBR) | Universal | 071406     | Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, S-PRG filler based on fluoroboroaluminosilicate glass, polymerization initiator, pigments, others                                                                           | 87                 | 1.7                                    | 4 mm                  | 10 s 1000 mW/cm <sup>2</sup>                               | 1 (4 mm)                   | Shofu (Kyoto, Japan)          |
| Admira Fusion x-tra (AFX)        | Universal | 1813298    | Organically modified silicic acid, silicon dioxide nanofillers (20–50 nm), silicon oxide-based hybrid fillers (1 μm)                                                                                          | 84                 | 1.25                                   | 4 mm                  | 20 s ≥1000 mW/cm <sup>2</sup> × 3                          | 1 (4 mm)                   | VOCO GmbH (Cuxhaven, Germany) |
| x-tra fill (XTF)                 | Universal | 1749292    | Inorganic filler in a methacrylate matrix (bis-GMA, UDMA, TEGDMA)                                                                                                                                             | 86                 | 1.7                                    | 4 mm                  | 10 s ≥800 mW/cm <sup>2</sup>                               | 1 (4 mm)                   | VOCO                          |
| Sonic Fill (SF)                  | A2        | 1012336    | Bis-GMA, TEGDMA, EBPDMA, UDMA, SiO <sub>2</sub> , glass, oxide                                                                                                                                                | 83.5               | 1.6                                    | 4 mm                  | 10 s 1000 mW/cm <sup>2</sup>                               | 1 (4 mm)                   | Kerr Corp. (Orange, CA, USA)  |
| Clearfil Majesty Esthetic (CME)  | A2        | 4H0173     | Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, silanated barium glass filler, prepolymerized organic filler, dl-camphorquinone, initiators, accelerators, pigments, others | 78                 | 1.9                                    | 1.5 mm                | 20 s >300 mW/cm <sup>2</sup>                               | 3 (1.5 mm + 1.5 mm + 1 mm) | Kuraray (Okayama, Japan)      |

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; UDMA, urethane dimethacrylate; Bis-MPEPP, 2,2-bis[(4-methacryloxy polyethoxy)phenyl]propane; TEGDMA, triethylene glycol dimethacrylate; S-PRG, surface reaction-type prereleased glass ionomer; EBPDMA, ethoxylated bisphenol A dimethacrylate.

purpose. The prepared tooth image (reference) and the restored tooth image (target) were superimposed, and the difference in volume was determined by image subtraction. The resulting image represented the volume of the cuspal deflection caused by the polymerization shrinkage of the resin composite restoration (Figures 2 and 3).

CTAn software was used for visualization and calculation of this subtracted image. A region of interest (ROI) was drawn to include the entire specimen within the sample using CTAn software, and all specifications of the program were used to analyze the microarchitecture of each sample. Once the appropriate ROI (in volume) was selected, “binary images” were obtained, and an appropriate grayscale threshold was selected to distinguish the enamel, dentin, and composite fillings. A thresholding (binarization) process was used, which entailed processing the range of gray levels to obtain a superimposed image of only black/white pixels. Despeckling was also performed to remove white speckles in the 2D image that were less than 10 voxels. The buccal and palatal regions of each restoration were evaluated separately in terms of

cuspal deflection. The total cuspal deflection volume values are expressed in mm<sup>3</sup>.

Statistical Analysis

The volume of cuspal deflection after restoration was analyzed using the Shapiro-Wilk test for normality and equality of variances (the Levene test), followed by a parametric statistical test. One-way analysis of variance (ANOVA) was used to compare the effect of the composite resin, and multiple comparisons were performed using the Tukey test. All tests used a significance level of  $\alpha = 0.05$ , and all analyses were conducted with the statistical program GraphPad Prism (ver. 6.0).

RESULTS

The cuspal deflection values for the tested composites are shown in Table 2. One-way ANOVA showed that the resin composite type had a significant effect on cuspal deflection ( $p < 0.0001$ ).

Multiple comparisons (Table 2) showed that teeth restored with the conventional paste-like composite and CME (control) had significantly different cuspal

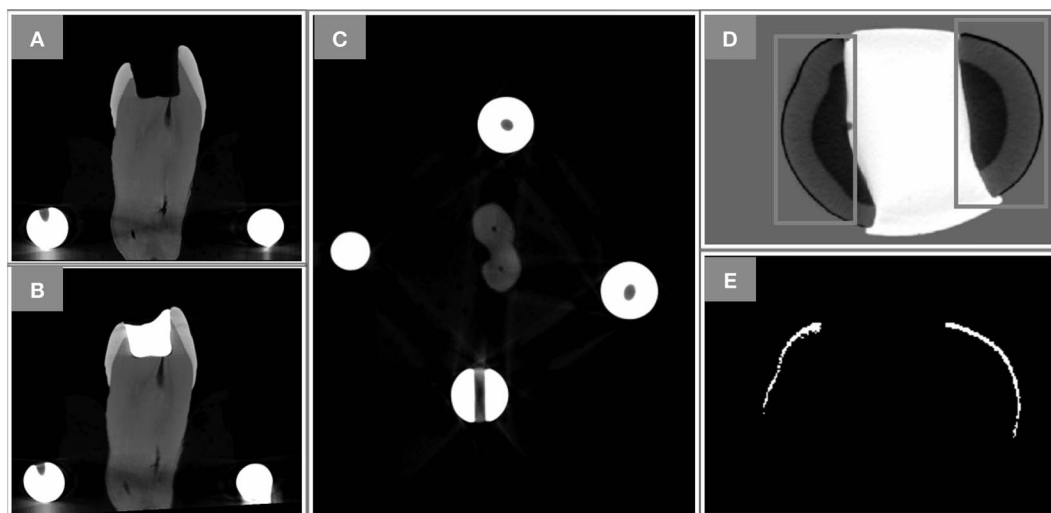


Figure 2. Measurement of the cuspal deflection using micro-CT. (A): Sagittal section of a prepared tooth. (B): Sagittal section of a tooth immediately after restoration. (C): Overlapping of the prepared and restored tooth using spheres. (D): Rectangular regions of interest of the difference image. (E): Binarization of the difference between the prepared and restored tooth (white area).

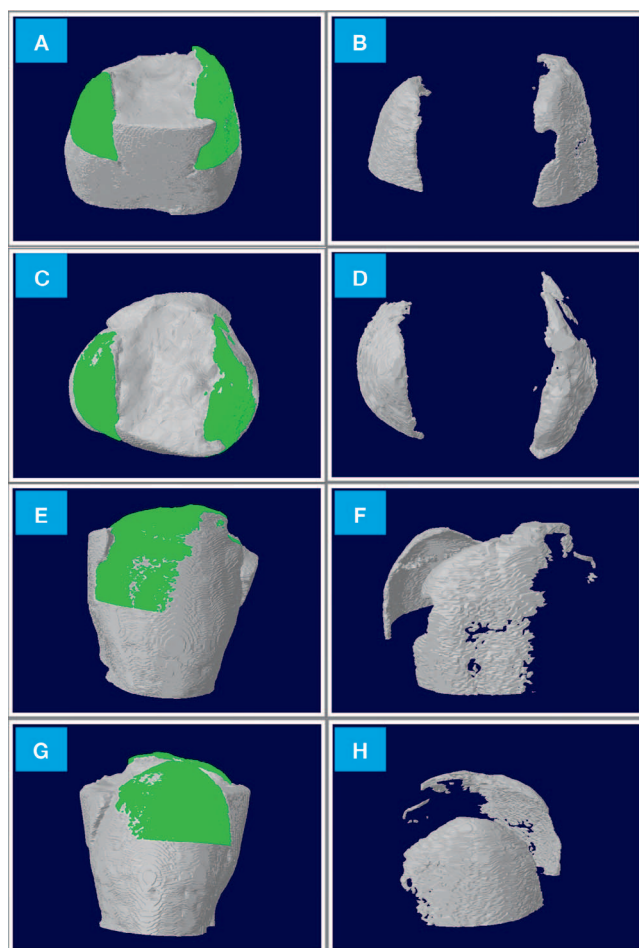


Figure 3. Representative images of the volume of cuspal deflection. Deflected cusp regions in proximal (A, B), occlusal (C, D), buccal (E, F), and palatal (G, H) views.

deflection than those filled with paste-like bulk-fill composites ( $p < 0.05$ ). Among bulk-fill composites, a significant difference was observed between BBR and AFX ( $p < 0.05$ ).

## DISCUSSION

This study investigated the effect of four types of paste-like bulk-fill composites on the volumetric cuspal deflection of maxillary premolar teeth compared to a conventional paste-like composite. Due to polymerization shrinkage, all tested composite groups showed an inward deflection of the cusps, which is consistent with the results of other studies.<sup>14,23,26-30</sup> All paste-like bulk-fill resin composites showed less volumetric cuspal deflection than the conventional paste-like composite group. Thus, the null hypothesis was rejected.

The amount of cuspal deflection is directly related to the amount of tooth structure loss.<sup>31</sup> Therefore, in the present study, large MOD cavity preparations were performed on maxillary premolar teeth to weaken tooth structure and promote cuspal deflection. The dimensional differences of the premolar teeth used in the study were set to a maximum of 5% for standardization and to enable comparison with other cuspal deflection studies.<sup>28</sup>

Although inexpensive and simple methods are available to measure cuspal deflection, these methods measure only linear values between two points on the tooth structure.<sup>31</sup> However, micro-CT is useful to evaluate the volumetric differences.<sup>29</sup> Micro-CT enables the visualization of effects across

| Table 2: Means and Standard Deviations of Cuspal Deflection of Tested Groups |                                                    |                    |                             |
|------------------------------------------------------------------------------|----------------------------------------------------|--------------------|-----------------------------|
| Resin Composite Type                                                         | Total Cusp Deflection<br>(mean – mm <sup>3</sup> ) | Standard Deviation | Difference <i>p</i> < 0.05* |
| Beautifil-Bulk Restorative (BBR)                                             | –5.658                                             | 0.0699             | B                           |
| Admira Fusion x-tra (AFX)                                                    | –5.066                                             | 0.2471             | C                           |
| x-tra fill (XTF)                                                             | –5.258                                             | 0.2235             | BC                          |
| Sonic Fill (SF)                                                              | –5.339                                             | 0.2561             | BC                          |
| Clearfil Majesty Esthetic (CME) (Control)                                    | –7.235                                             | 0.5223             | A                           |
| * No significant difference between groups shown with the same letter.       |                                                    |                    |                             |

the entire end and can analyze the boundary conditions of the exact tooth cavity.<sup>31</sup> In addition, Oliveira and others<sup>29</sup> showed a strong correlation between cuspal deflection values measured using the micro-CT method and those measured using the strain gauge method. Therefore, cuspal deflection was measured by micro-CT in this study.

The difference between the cuspal deflection values of the bulk-fill composites and the conventional composite in the present study could be due to two factors or a combination thereof. One factor was the chemical composition of the different composites, and the other was the incremental (three layers and three sessions of light curing) restorative procedure of the conventional composite. The results of this study are consistent with those of other studies comparing paste-like bulk-fill composites and conventional paste-like composites.<sup>23,29,32</sup> In contrast, a study by Tsujimoto and others<sup>30</sup> reported that paste-like bulk-fill resin composites showed cuspal deflection similar to conventional resin composites. That study used aluminum blocks instead of extracted teeth and investigated the simulated cuspal deflection of paste-like bulk-fill and conventional resin composites with a micrometer and a confocal laser-scanning microscope. The discrepancy between our study and their results might be due to the difference in the methodology or the difference in the type of composite that was compared. Another study investigated the cuspal deflection of premolars restored with three paste-like bulk-fill composite resins and one low-shrinkage silorane-based restorative material.<sup>14</sup> The researchers reported that silorane-based composites caused less cuspal deflection than paste-like bulk-fill composites. Since cuspal deflection is directly caused by polymerization shrinkage, that study clearly demonstrates that the limited shrinkage behavior of the silorane-based composite caused less cusp movement than methacrylate-based composites.

Another finding of the current study was that BBR showed higher volumetric cuspal deflection than

AFX among the bulk-fill composites. This difference might be related to the different chemical compositions of these resins. AFX is anOrmocer-based composite, while BBR contains many different monomers. These composites also contain different types and amounts of fillers.

Although composites are termed bulk fills, these commercially available composites differ in their chemical composition. To provide a 4-mm polymerization depth, every manufacturer organizes its composite structure with different types and quantities of monomers; different types, amounts, shapes, and sizes of fillers; and different photoinitiators,<sup>5,6</sup> and these components differ in each composite. These varied structures also require different amounts of curing light energy for adequate polymerization.<sup>33</sup> Because many variables exist, it is difficult to compare these composites with each other, which limits the studies that can be performed.

This study has some limitations. The major limitation involved the insertion technique and total polymerization energy. Conventional paste-like composite cannot be used for 4-mm bulk polymerization and was applied by an incremental technique. The polymerization time varied for composite groups according to the manufacturer’s instructions. Therefore, the total energy applied to the composite groups also varied. Instead of applying equal energy to all composites, each composite was cured according to the manufacturer’s specified curing time and energy instructions to simulate clinical conditions. These conditions might have affected the results. The second limitation was that composite resins absorb water in the oral environment, which causes hygroscopic expansion and can neutralize the negative effects of polymerization shrinkage and relieve cuspal deflection.<sup>29,34</sup> Since the early effects of the resin materials were examined, no hygroscopic expansion occurred this study. The third limitation of the study was the cavity type and dimensions. Currently, the use of

adhesive restorations results in smaller cavity preparations. Large MOD cavities are usually observed in the replacement of amalgam fillings or teeth undergoing endodontic treatment due to excess caries. Therefore, the type of cavity used in this study is not a common cavity preparation, but to better observe the shrinkage properties of the materials, these large cavities with weak and stretchable walls were used in the study, similar to previous studies.<sup>27,28</sup> The fourth limitation of this study was the microcrack evaluation. Although enamel fractures are one of the most important results of polymerization shrinkage, their detection methods include subjective observations, such as transillumination.

Many studies have examined the cuspal deflection of bulk-fill composites *in vitro*. *In vitro* studies provide information about the general characteristics of the materials, but their interpretations of the intraoral environment are based on speculation. Studies are needed that evaluate cuspal deflection with an intraoral scanner or similar devices in an intraoral environment and that examine the early and long-term effects by evaluating both patient complaints and restoration status using intraoral evaluation criteria.

## CONCLUSIONS

Within the limitations of this study, it can be concluded that paste-like bulk-fill resin composites resulted in significantly less cuspal deflection than the conventional paste-like resin composite tested. Among the bulk-fill resin composites, BBR produced greater volumetric cuspal deflection than AFX.

## Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ankara University Faculty of Dentistry Ethics Committee. The approval code for this study is 2019/01 No:01/05.

## Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 7 March 2019)

## REFERENCES

1. Leprince JG, Palin WM, Hadis MA, Devaux J, & Leloup G (2013) Progress in dimethacrylate-based dental composite technology and curing efficiency *Dental Materials* **29**(2) 139-156. 10.1016/j.dental.2012.11.005
2. Algamaiah H, Sampaio CS, Rigo LC, Janal MN, Giannini M, Bonfante EA, Coelho PG, Reis AF, & Hirata R (2016) Microcomputed tomography evaluation of volumetric shrinkage of bulk-fill composites in class II cavities *Journal of Esthetic and Restorative Dentistry*. 10.1111/jerd.12275
3. El-Damanhoury H & Platt J (2014) Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites *Operative Dentistry* **39**(4) 374-382. 10.2341/13-017-1
4. Furness A, Tadros MY, Looney SW, & Rueggeberg FA (2014) Effect of bulk/incremental fill on internal gap formation of bulk-fill composites *Journal of Dentistry* **42**(4) 439-449. <http://dx.doi.org/10.1016/j.jdent.2014.01.005>
5. Chesterman J, Jowett A, Gallacher A, & Nixon P (2017) Bulk-fill resin-based composite restorative materials: A review *British Dental Journal* **222**(5) 337-344 10.1038/sj.bdj.2017.214.
6. Van Ende A, De Munck J, Lise DP, & Van Meerbeek B (2017) Bulk-fill composites: A review of the current literature *Journal of Adhesive Dentistry* **19**(2) 95-109. 10.3290/jjad.a38141
7. Tsujimoto A, Barkmeier WW, Takamizawa T, Latta MA, & Miyazaki M (2016) Mechanical properties, volumetric shrinkage and depth of cure of short fiber-reinforced resin composite *Dental Materials Journal* **35**(3) 418-424. 10.4012/dmj.2015-280
8. Kaisarly D & Gezawi ME (2016) Polymerization shrinkage assessment of dental resin composites: A literature review *Odontology* **104**(3) 257-270. 10.1007/s10266-016-0264-3
9. Baltacioglu IH, Kamburoglu K, Irmak O, Geneci F, Ocak M, Uzuner MB, & Celik HH (2017) Marginal integrity of self-adhering flowable composites used as liner under class II restorations: a comparative in vitro micro-CT study *Journal of Adhesion Science and Technology* **31**(24) 2719-2729. 10.1080/01694243.2017.1317472
10. Karaman E & Ozgunaltay G (2013) Cuspal deflection in premolar teeth restored using current composite resins with and without resin-modified glass ionomer liner *Operative Dentistry* **38**(3) 282-289. 10.2341/11-400-1
11. Mantri SP & Mantri SS (2013) Management of shrinkage stresses in direct restorative light-cured composites: A review *Journal of Esthetic and Restorative Dentistry* **25**(5) 305-313. 10.1111/jerd.12047
12. Lee MR, Cho BH, Son HH, Um CM, & Lee IB (2007) Influence of cavity dimension and restoration methods on the cusp deflection of premolars in composite restoration *Dental Materials* **23**(3) 288-295. 10.1016/j.dental.2006.01.025
13. Alomari QD, Reinhardt JW, & Boyer DB (2001) Effect of liners on cusp deflection and gap formation in composite restorations *Operative Dentistry* **26**(4) 406-411.
14. Behery H, El-Mowafy O, El-Badrawy W, Saleh B, & Nabih S (2016) Cuspal deflection of premolars restored with bulk-fill composite resins *Journal of Esthetic and Restorative Dentistry* **28**(2) 122-130. 10.1111/jerd.12188

15. Campodonico CE, Tantbirojn D, Olin PS, & Versluis A (2011) Cuspal deflection and depth of cure in resin-based composite restorations filled by using bulk, incremental and transtooth-illumination techniques *Journal of the American Dental Association* **142**(10) 1176-1182. <https://doi.org/10.14219/jada.archive.2011.0087>
16. McCulloch AJ & Smith BG (1986) In vitro studies of cuspal movement produced by adhesive restorative materials *British Dental Journal* **161**(11) 405-409.
17. Meredith N & Setchell DJ (1997) In vitro measurement of cuspal strain and displacement in composite restored teeth *Journal of Dentistry* **25**(3-4) 331-337.
18. Hirata R, Clozza E, Giannini M, Farrokhanesh E, Janal M, Tovar N, Bonfante EA, & Coelho PG (2015) Shrinkage assessment of low shrinkage composites using micro-computed tomography *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **103**(4) 798-806. 10.1002/jbm.b.33258
19. Chiang YC, Rosch P, Dabanoglu A, Lin CP, Hickel R, & Kunzelmann KH (2010) Polymerization composite shrinkage evaluation with 3D deformation analysis from microCT images *Dental Materials* **26**(3) 223-231. 10.1016/j.dental.2009.09.013
20. Sampaio CS, Chiu KJ, Farrokhanesh E, Janal M, Puppini-Rontani RM, Giannini M, Bonfante EA, Coelho PG, & Hirata R (2017) Microcomputed tomography evaluation of polymerization shrinkage of class I flowable resin composite restorations *Operative Dentistry* **42**(1) e16-e23. 10.2341/15-296-1
21. Algamaiah H, Sampaio CS, Rigo LC, Janal MN, Giannini M, Bonfante EA, Coelho PG, Reis AF, & Hirata R (2017) Microcomputed tomography evaluation of volumetric shrinkage of bulk-fill composites in class II cavities *Journal of Esthetic and Restorative Dentistry* **29**(2) 118-127. 10.1111/jerd.12275
22. Jafarpour S, El-Badrawy W, Jazi HS, & McComb D (2012) Effect of composite insertion technique on cuspal deflection using an in vitro simulation model *Operative Dentistry* **37**(3) 299-305. 10.2341/11-086-1
23. Elsharkasi MM, Platt JA, Cook NB, Yassen GH, & Matis BA (2018) Cuspal deflection in premolar teeth restored with bulk-fill resin-based composite materials *Operative Dentistry* **43**(1) e1-e9. 10.2341/16-072-1
24. Shirani F, Malekipour MR, Sakhaei Manesh V, & Aghaei F (2012) Hydration and dehydration periods of crown fragments prior to reattachment *Operative Dentistry* **37**(5) 501-508. 10.2341/10-130-1
25. Feldkamp LA, Goldstein SA, Parfitt AM, Jesion G, & Kleerekoper M (1989) The direct examination of three-dimensional bone architecture in vitro by computed tomography *Journal of Bone and Mineral Research* **4**(1) 3-11. 10.1002/jbmr.5650040103
26. Francis AV, Braxton AD, Ahmad W, Tantbirojn D, Simon JF, & Versluis A (2015) Cuspal flexure and extent of cure of a bulk-fill flowable base composite *Operative Dentistry* **40**(5) 515-523. 10.2341/14-235-1
27. McHugh LEJ, Politi I, Al-Fodeh RS, & Fleming GJP (2017) Implications of resin-based composite (RBC) restoration on cuspal deflection and microleakage score in molar teeth: Placement protocol and restorative material *Dental Materials* **33**(9) e329-e335. 10.1016/j.dental.2017.06.001
28. Moorthy A, Hogg CH, Dowling AH, Grufferty BF, Benetti AR, & Fleming GJ (2012) Cuspal deflection and microleakage in premolar teeth restored with bulk-fill flowable resin-based composite base materials *Journal of Dentistry* **40**(6) 500-505. 10.1016/j.jdent.2012.02.015
29. Oliveira LRS, Braga SSL, Bicalho AA, Ribeiro MTH, Price RB, & Soares CJ (2018) Molar cusp deformation evaluated by micro-CT and enamel crack formation to compare incremental and bulk-filling techniques *Journal of Dentistry* **74** 71-78. 10.1016/j.jdent.2018.04.015
30. Tsujimoto A, Nagura Y, Barkmeier WW, Watanabe H, Johnson WW, Takamizawa T, Latta MA, & Miyazaki M (2018) Simulated cuspal deflection and flexural properties of high viscosity bulk-fill and conventional resin composites *Journal of the Mechanical Behavior of Biomedical Materials* **87** 111-118. <https://doi.org/10.1016/j.jmbbm.2018.07.013>
31. Gonzalez Lopez S, Sanz Chinesta MV, Ceballos Garcia L, de Haro Gasquet F, & Gonzalez Rodriguez MP (2006) Influence of cavity type and size of composite restorations on cuspal flexure *Medicina Oral, Patología Oral y Cirugía Bucal* **11**(6) e536-e540.
32. Politi I, McHugh LEJ, Al-Fodeh RS, & Fleming GJP (2018) Modification of the restoration protocol for resin-based composite (RBC) restoratives (conventional and bulk fill) on cuspal movement and microleakage score in molar teeth *Dental Materials* **34**(9) 1271-1277. 10.1016/j.dental.2018.05.010
33. Zorzin J, Maier E, Harre S, Fey T, Belli R, Lohbauer U, Petschelt A, & Taschner M (2015) Bulk-fill resin composites: Polymerization properties and extended light curing *Dental Materials* **31**(3) 293-301. <https://doi.org/10.1016/j.dental.2014.12.010>
34. Meriwether LA, Blen BJ, Benson JH, Hatch RH, Tantbirojn D, & Versluis A (2013) Shrinkage stress compensation in composite-restored teeth: Relaxation or hygroscopic expansion? *Dental Materials* **29**(5) 573-579. <https://doi.org/10.1016/j.dental.2013.03.006>

# Clinical Evaluation of a Chairside Amalgam Separator to Meet Environmental Protection Agency Dental Wastewater Regulatory Compliance

DS Olivera • MT Morgan • SN Tewolde • EC Botts • FP Horvath • NJ Hamlin

## Clinical Relevance

US dental practices that routinely place and remove amalgam restorations are now required to use an amalgam separator with  $\geq 95\%$  efficiency. This independent study evaluated and characterized the performance of a chairside amalgam separator, demonstrating that minimum standards for compliance can be met.

## SUMMARY

**Objectives:** The aim of this study was to evaluate the performance of a commercially available chairside amalgam separator (CAS) in a clinical setting in which a relatively high number of amalgam restorations are placed. Performance parameters investigated included service life, amalgam collected, mercury concentrations in effluent, and solids retention efficiency.

\*Dorian Scott Olivera, PhD, MS, Naval Medical Research Unit San Antonio, Environmental Surveillance Department, Fort Sam Houston, TX, USA

Matthew T Morgan, BS, Naval Medical Research Unit San Antonio, Environmental Surveillance Department, East Lyme, CT, USA

Senay N Tewolde, PhD, MS, Naval Medical Research Unit San Antonio, Biomedical Systems Engineering and Evaluation Department, Fort Sam Houston, TX, USA

Eric C Botts, MS, Naval Medical Research Unit San Antonio, Environmental Surveillance Department, Fort Sam Houston, TX, USA

**Methods and Materials:** CASs were tested per International Organization of Standardization (ISO) 11143:2008 prior to installation in a military dental treatment facility and after removal from service (n=4) in order to confirm compliance with the recently enacted United States Environmental Protection Agency (EPA) Effluent Limitations Guidelines and Standards for the Dental Category. During the units' time in service, biweekly effluent grab samples were collected from the high-

Francis P Horvath, BS, Naval Medical Research Unit San Antonio, Environmental Surveillance Department, Fort Sam Houston, TX, USA

Nicholas J Hamlin, PhD, DDS, MS, LCDR, DC, USN, Naval Medical Research Unit San Antonio, Environmental Surveillance Department, Fort Sam Houston, TX, USA

\*Corresponding author: 3650 Chambers Pass, Building 3610, Fort Sam Houston, TX 78234, USA; e-mail: dorian.s.olivera.civ@mail.mil

DOI: <http://doi.org/10.2341/18-156-L>

volume evacuation system of each chair (n=6) and analyzed for total mercury concentration by inductively coupled plasma mass spectrometry (ICP-MS). The mean total accumulated solids at the end of service life (n=6) was determined for potential design optimization. The service life expectancy in a military dental treatment facility was determined in terms of calendar and workdays. Procedural data were collected to determine the daily mean number of amalgam surfaces placed during the service life of each chairside amalgam separator (n=9).

**Results:** The CAS evaluated met minimum EPA compliance requirements when used in a military dental treatment facility. The solids removal efficiency at the end of service life was  $99.82\% \pm 0.14\%$  (n=4). The mean service life (n=8) was  $131.6 \pm 45.1$  calendar days ( $67.1 \pm 37.6$  workdays). Effluent mercury concentrations ranged from 0.05 to 11.93 mg/L. Total solids accumulated in each CAS (n=6) at the end of service life was  $195.4 \pm 63.4$  g. The mean number of amalgam surfaces placed per workday during the service life span of each CAS was  $8.4 \pm 1.4$ .

## INTRODUCTION

Dental amalgam has been used as a restorative material for over 150 years, though use has decreased since the 1970s.<sup>1</sup> To this day, however, many dental professionals and patients still prefer amalgam in some cases over other restorative materials because of its durability and relatively low cost. Additionally, many serviceable amalgam restorations are in place today that will likely need replacement sometime in the future.<sup>1</sup> Despite the controversy surrounding amalgam restorations, the American Dental Association, US Food and Drug Administration, and World Health Organization are among many scientific organizations that have released consensus statements, literature reviews, and rulings, affirming the safety and effectiveness of dental amalgam for patient use.<sup>2-4</sup> While the mercury in amalgam is stable under the conditions present within the oral cavity, its release in dental wastewater poses an environmental hazard if improperly managed. The international concern over anthropogenic sources of mercury and mercury-containing compounds released into the environment culminated in the Minamata Convention on Mercury United Nations Treaty, signed by over 100 nations, including the United States, which did so in November 2013. The Minamata Convention went

into effect on August 16, 2017, with the aim to protect human health and the environment by reducing mercury release.<sup>5</sup> In roughly the same time-frame, dental amalgam waste generated by dental offices has become the focus of environmental regulations by the Environmental Protection Agency (EPA).<sup>6</sup> The reduction of anthropogenic mercury released to the environment has recently become a topic of international scope and policy.

According to the EPA,<sup>6</sup> amalgam containing wastewater from dental offices is the main source of Hg loading to publically owned treatment works (POTWs). Concentrations of Hg in dental wastewater can range from 100 to 2000 mg/L.<sup>7,8</sup> The EPA's most recent estimate is that 5.1 tons of Hg and 5.3 tons of other dental amalgam metals (ie, Ag, Cu, Sn, and Zn) are collectively discharged to POTWs annually in the United States.<sup>6</sup> The Hg discharges from dental offices have been attributed to approximately half of total Hg loading to POTWs in the United States.<sup>9</sup>

Advancements in filtration technology, including amalgam chairside traps capable of capturing amalgam scrap and large particles  $>700 \mu\text{m}$ ,<sup>10</sup> as well as amalgam separators capable of capturing smaller particles,<sup>11</sup> have significantly reduced Hg loading to POTWs. Amalgam wastes are typically composed of metal scrap ranging from  $<1 \mu\text{m}$  to 1 mm in diameter.<sup>11</sup> Most amalgam scrap is captured by in-line chair side amalgam traps, which prevent vacuum line blockage by removing the larger tooth fragments and dental material waste particles.<sup>11</sup> The remaining smaller amalgam particles, the majority of which are  $<100 \mu\text{m}$ ,<sup>11</sup> are usually removed from the dental wastewater by an amalgam separator using filtration, sedimentation, centrifugation, or a combination of these methods, before the wastewater is released into POTWs.<sup>6</sup> The remaining amalgam particulate that is too fine to be removed by standard separation methods is passed on to the POTWs where it is extracted as sewage sludge, which may be discarded in landfills, incinerated, or introduced to agricultural land as fertilizer.<sup>6</sup> Incineration releases gaseous Hg into the atmosphere, where it is adsorbed onto the atmospheric aerosol and subsequently precipitated.<sup>12</sup> Whether leached from landfills or crop fields, or precipitated from the atmosphere, mercury eventually finds its way into the water table.

Inorganic Hg, transmitted hydrologically from immediate catchments to wetland and aquatic environments, is gradually converted to organic Hg compounds, such as methylmercury (MeHg).<sup>12</sup> In the

case of MeHg, the organic Hg compound is released into water and biomagnified in algal cells or benthic periphyton, increasing concentration by a factor of  $10^4$  to  $10^6$ .<sup>13,14</sup> As MeHg is transmitted through the food chain to higher-level organisms, it accumulates within the tissue of the consumers. Thus, MeHg concentration is magnified across trophic levels. Biomagnification increases MeHg levels by a factor of two to five from one trophic level to the next and are highest in piscivorous fish, birds, and mammals.<sup>14-16</sup> Environmentally significant concentrations of MeHg have been observed in dental effluent samples collected from two different clinics in which amalgam restoration removals and placements were performed. The reported MeHg levels were 8.26  $\mu\text{g/L}$  for a 30-chair facility and 26.77  $\mu\text{g/L}$  for a 107-chair facility,<sup>17</sup> considerably higher than the recommended freshwater criteria maximum concentration for MeHg in ambient waters (1.4  $\mu\text{g/L}$ ).<sup>18</sup> To minimize toxic effects to wildlife and humans alike, it is of the utmost importance to minimize anthropogenic release of Hg, including that which is introduced through dental waste into POTWs.

In 1991, the US Naval Institute for Dental and Biomedical Research (NIDBR), in collaboration with the American Dental Association, began evaluating the solids removal efficiency (SRE) of commercially available amalgam separators in order to mitigate Hg discharges from Navy dental treatment facilities (DTFs). NIDBR then founded a mercury management program that developed pretreatment systems for amalgam process wastewater. In 2001, NIDBR began a global effort to install and monitor pretreatment systems in Navy DTFs to ensure compliance with local and international discharge limits. The NIDBR Mercury Abatement Program became the Navy's lead agent for testing and evaluating pretreatment systems for Hg mitigation in wastes from DTFs. One pretreatment device, a filtration-based chairside amalgam separator (CAS), was developed by NIDBR in 2008 as a relatively low-cost alternative or addition to centrally located pretreatment systems.<sup>19</sup> NIDBR's original CAS featured a replaceable polypropylene filter element with a nominal pore size of 0.5  $\mu\text{m}$ , which demonstrated 96.8% SRE when tested per International Organization of Standardization (ISO) 11143:2008 protocols.<sup>19,20</sup> Further development of the original NIDBR CAS concept was continued at the Naval Medical Research Unit San Antonio, where the Navy Mercury Abatement Program continues to test heavy metals control devices and provide guidance on CAS installation and maintenance to DTF personnel.

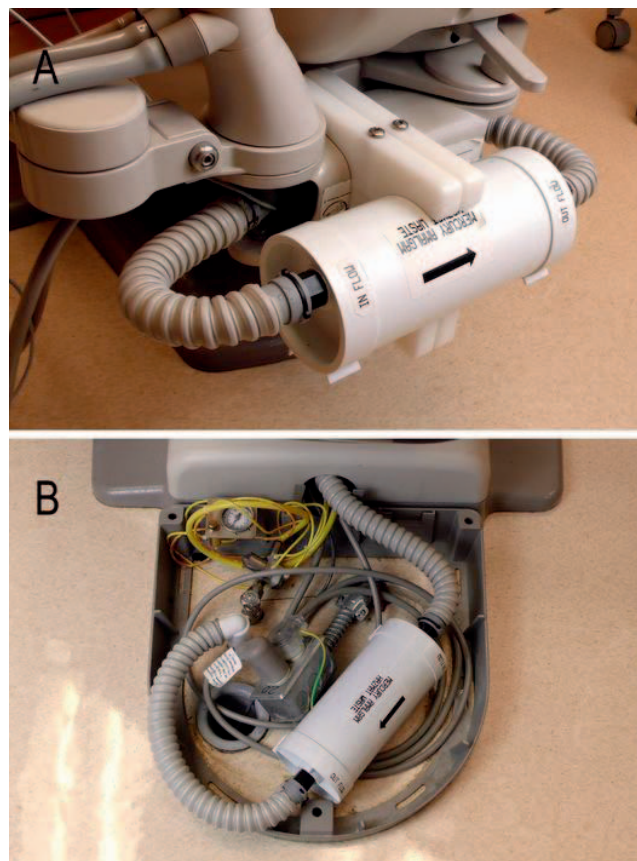


Figure 1. A typical installation of the DD211P chairside amalgam separator (white cylinder) on the rear support link of an A-Dec 500-Series dental chair (A). This particular installation makes use of the optional mounting brackets (white clamps with screws). An alternative installation (B) in which the separator is contained within the floor box under the chair.

The most recent version of the NIDBR CAS concept is commercially available as the DD211P (Dennis J. Duel & Associates, Mundelein, IL, USA).<sup>21</sup> This model has been certified per ISO 11143:2008<sup>20</sup> test methods for 99.6% SRE and is currently in use in several Navy DTFs (Figure 1).

In order to reduce the discharge of Hg-containing dental amalgam to POTWs, the EPA published the final rule of the Effluent Limitations Guidelines and Standards for the Dental Category under 40 Code of Federal Regulations Part 441 with the effective date of July 14, 2017. Dental offices that discharge to POTWs must demonstrate compliance by July 14, 2020. The rule mandates the installation of dental amalgam separators that meet ISO 11143:2008<sup>20</sup> requirements of  $\geq 95\%$  SRE as a pretreatment standard for amalgam process wastewater. Existing sources (ie, dental offices operating before July 14, 2017) must submit a one-time compliance report to their respective control authority no later than

October 12, 2020. New sources (ie, dental offices operating for the first time after July 14, 2017) must submit said compliance report no later than 90 days after the wastewater is first discharged to POTWs. The rule exempts mobile units, as well as practices in which amalgam restoration placement and removal are not typically performed, including the exemption of practices limited to the specialties of oral pathology, oral and maxillofacial radiology, oral and maxillofacial surgery, orthodontics, periodontics, and prosthodontics. With the combination of these new pretreatment standards preceding POTW loading, EPA estimates a reduction in POTW discharge of dental amalgam-sourced Hg to receiving waters from 1003 to 11 lb/y, as POTWs typically remove 90.2% of the Hg from the wastewater. The final rule will cost affected dental offices on average approximately \$800 per year. The EPA projects the total annual cost of the final rule to be \$59-\$61 million.<sup>6</sup>

The primary goal of this study was to characterize the real-world performance of a CAS unit in a large military dental group practice, selectively evaluated on the chairs of providers who place a large number of amalgam restorations. Our secondary aim, after completion of and based on the analysis of chairside separator performance, was to propose possible avenues for future CAS design improvement.

## METHODS AND MATERIALS

### ISO 11143:2008 Solids Removal Efficiency Testing of CASs: Preinstallation

Prior to installation at the DTF (Budge Dental Clinic, Fort Sam Houston DENTAC, JBSA Fort Sam Houston, TX), quality assurance testing per ISO 11143:2008<sup>20</sup> was performed on one DD2011P CAS (Dennis J. Duel and Associates) to verify that the model could attain the minimum requirement of 95% SRE. Further, obtained SRE results were compared with documented certified testing laboratory results to evaluate internal quality assurance. CAS units from the same lot as the tested unit were then installed in the DTF for evaluation.

### Service Life Evaluation of CASs in a Military DTF

CAS units were evaluated in a military DTF to determine service life expectancy under heavy use. Four general dentistry providers at the DTF were identified through review of production numbers in the previous 6 months as those who placed the highest number of amalgam restorations. Each

provider operated on one or two chairs exclusively. In total, six chairs were selected for this study. CAS units were tare weighed, then installed on the six A-dec 500 dental chairs (A-dec. Newberg, OR, USA). The CAS units were left in place until the end of their service lives, while suction data, effluent samples, and amalgam use data were collected and assessed (see Figure 2). At the end of each unit's service life, it was replaced, and data collection for that unit was halted. The end of a CAS unit's service life was defined by its meeting one of three criteria: the high-volume evacuation (HVE) system no longer met the required vacuum of 6-8 inHg (as specified in the Unified Facilities Criteria (UFC) 4-510-01),<sup>22</sup> the DTF staff reported poor HVE suction, or one year passed since installation.

### Clinical Procedural Data Collection

De-identified procedural data from individual providers for the one-year test period were collected at the end of the study. Procedures which utilized amalgam (recorded in the clinical database as 2017 Current Dental Terminology codes D2140, D2150, D2160, and D2161)<sup>23</sup> were tabulated. The collected data were used to calculate the average number of amalgam surfaces placed per workday per chair in the study, beginning with the date the CAS was installed and continuing through to the end of its service life. For providers 1 and 2, the rate was divided by two since those providers each performed the work on two dedicated chairs. Providers 3 and 4, however, each operated on a single chair; therefore, all procedures were attributable to their single respective chair. Providers performed all amalgam restorations with Valiant Ph.D. (Ivoclar Vivadent, Amherst, NY, USA), which contains 40% to 45% Hg per capsule.<sup>24</sup>

### Effluent Sample Collection from HVE System/CASs

Effluent samples were collected from clinically installed CAS units (n=9) every 2 weeks for up to a 12-month period in order to characterize Hg concentrations in the effluent. Prior to effluent sample collection, sample containers, tubing, and connection fittings were cleaned per EPA standard test method guidelines.<sup>25</sup> Two sequential 1 L volumes of Ultrapure, Type 1 water (MilliporeSigma, St Louis, MO, USA) were aspirated through the HVE system of each of the six dental chairs and collected from the outlet of each CAS unit into borosilicate glass sample containers. From each 1 L flush, three 50-mL aliquots were prepared and preserved with Fisher

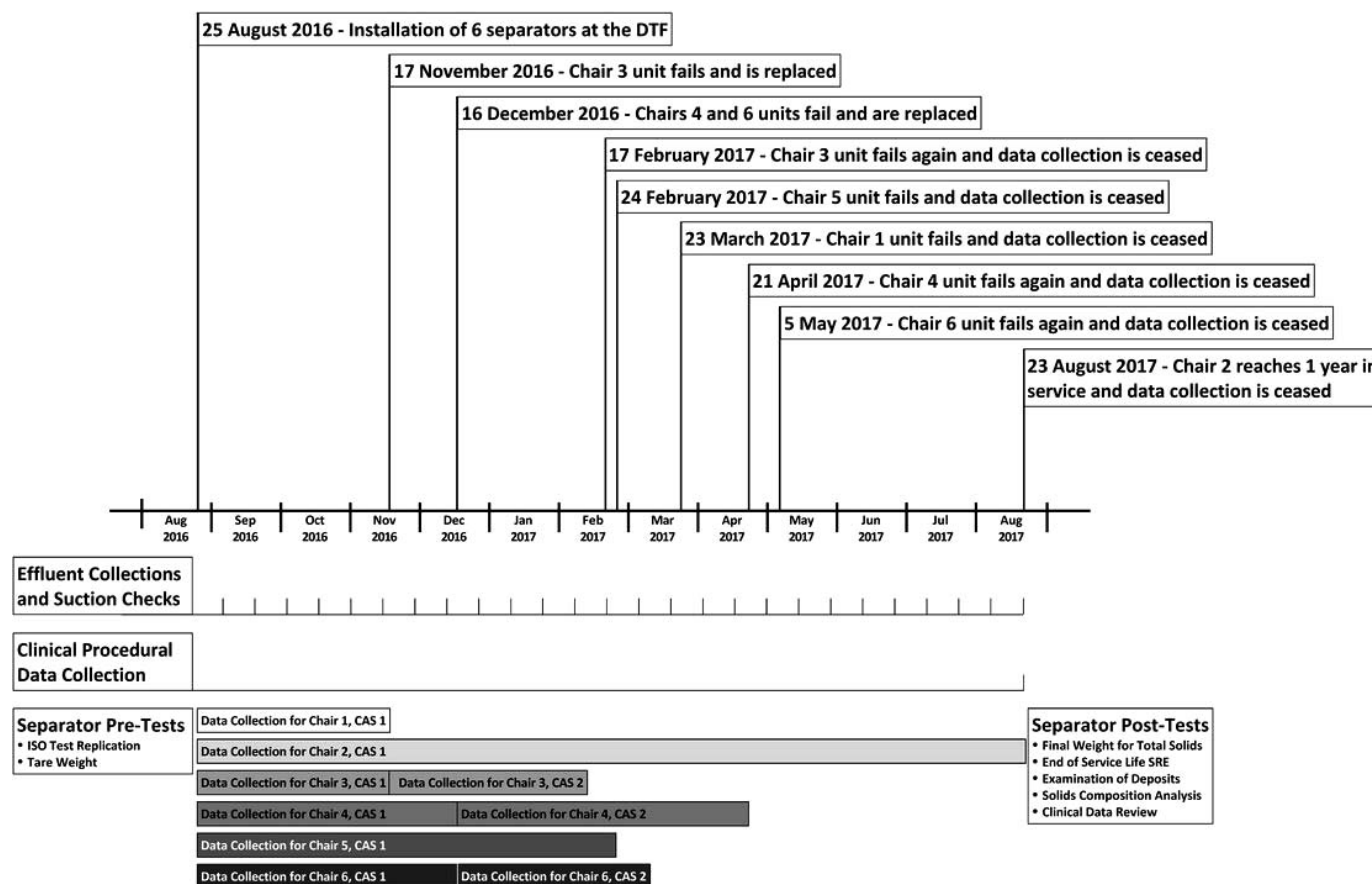


Figure 2. Timeline of the study, beginning August 2016 and running 1 year. Installation of six DD2011P CAS units, including three units replaced during the study (total  $n=9$ ), and the ends of all CAS service lives are indicated with the flags at the top of the figure. Effluent samples were collected and HVE system suction was checked at biweekly intervals from separators remaining on study, per the comb beneath the timeline. Clinical data were collected at the end of the study, after all separator data collection was terminated. Bars at the bottom of the figure indicate pre-study test elements, the data collection periods for each CAS, and post-study test elements.

Chemical TraceMetal grade  $\text{HNO}_3$  (Thermo Fisher Scientific, Waltham, MA, USA) per the EPA standard test methods indicated for aqueous total Hg and aqueous total metals.<sup>26</sup> Effluent sample collections were terminated when end of service life criteria were met as described earlier. To ensure compliance with UFC 4-510-01, vacuum levels through the HVE aspirator were measured with a Flowcheck vacuum meter (RAMVAC, Spearfish, SD, USA) immediately after each effluent sample collection.

### Effluent Sample Preparation, Hg Analysis by Inductively Coupled Plasma Mass Spectrometry, and Statistical Analysis

CAS effluent samples were prepared for trace metals analysis by microwave-assisted acid digestion per EPA Method 3015A.<sup>26</sup> Aliquots were diluted in a digestion matrix comprised of 45 mL of Ultrapure, Type 1 water (MilliporeSigma), 4 mL of concentrated Fisher Chemical TraceMetalGrade  $\text{HNO}_3$ , and 1 mL

of concentrated Fisher Chemical TraceMetalGrade HCl (Thermo Fisher Scientific). The diluted aliquots were then transferred to 75 mL perfluoroalkoxy vessels and digested in a MARS 6 microwave digestion oven (CEM Corporation, Matthews, NC, USA). Each digestion batch of analytic samples included quality control samples in accordance with EPA Methods 3015A and 6020B in order to evaluate laboratory performance and analyte recovery bias and precision.<sup>26,27</sup> Digests were analyzed for Hg concentrations on a Thermo Scientific iCAP Q inductively coupled plasma mass spectrometry (ICP-MS) instrument (Thermo Fisher Scientific) per EPA Method 6020B.<sup>27</sup> The Hg concentration means with standard deviations were calculated from the concentrations of the three aliquots collected from each HVE system effluent collection. Data distribution of the Hg concentration means was assessed for normality using the Shapiro-Wilk test. Statistical difference in the pooled mean Hg concen-

trations between the first and second HVE effluent samples was assessed using a Student *t*-test with the type I error rate (significance level) set at 0.05 ( $\alpha=0.05$ ).

### **ISO 11143:2008 SRE Testing of CASs: Postinstallation**

At the end of service life, four randomly selected CAS units recovered from the clinic were tested using ISO 11143:2008 §9.3.2.2 to §9.3.2.8<sup>20</sup> standards to ensure that the SRE remained above the minimum requirement of 95%.

### **Mass of Total Solids Accumulated by CASs After End of Service Life**

The CAS units were evaluated for mass of total solids captured during each unit's service life using a modified version of Method 2540 B by Rice and others<sup>28</sup>. The tare weight of each CAS unit was measured prior to installation in the DTF by drying at 103-105°C for 1 hour, cooling in a desiccator at 23°C, and then weighing the CAS. This desiccation and weighing cycle was repeated until a consistent weight (<4% difference) was observed. After each separator reached the end of its service life, the desiccation and weighing process was performed again to determine the final weight of each CAS. Total solids accumulated for each CAS were calculated by subtracting the tare weight from the final weight.

We determined the mass of amalgam that could theoretically occupy the fillable space of the CAS unit, defined as the space between the filter element and the housing, as follows: An unused CAS unit was opened by removing the end cap near the inlet with a drywall saw; it was then filled with 1 mm diameter glass beads. The beads were then poured out to measure the volume of the CAS unit and weighed to calculate the density. Finally, the volume of beads was multiplied by the density of amalgam test powder prepared from cured Tytin spherical dental amalgam (Kerr Corporation, Orange, CA, USA), which has a density of 1.694 g/cm<sup>3</sup> for the specific particulate composition described by ISO 11143:2008<sup>20</sup>, in order to estimate the theoretical maximum amalgam mass that could occupy the CAS unit.

### **Composition of Solids Accumulated by CASs at End of Service Life**

Six CAS units collected from the DTF at the conclusions of their service lives were randomly

selected for solids composition analysis. Following removal, the CAS inlet and outlet ports were capped and the CAS units were transported to the Naval Medical Research Unit for solids recovery and analysis. The ISO Method 11143:2008 testing apparatus was assembled with the CAS installed in reverse orientation to generate backflush. Each CAS was flushed from outlet to inlet with 1 L deionized H<sub>2</sub>O and the effluent containing captured sediment was collected. Effluent collected from the six CAS units was combined in a single 6-L container. The polyvinyl chloride housing of each CAS was opened by removing the end caps with a drywall saw, as close to the inlet ends as possible. Sediment that remained inside the CAS housing and on the filter surface after backflush was removed by scraping and irrigating and then pooled with the effluent and sediment collected by backflush. Sediment and effluent were homogenized by orbital shaking, and then the solids were allowed to sediment at the bottom of the container. The supernatant was then transferred to another container and discarded. Sediment was dried overnight using a Thermo Scientific Heratherm laboratory benchtop incubator. Metals composition was determined by homogenizing the sediment to a uniform mixture, then three 250-mg aliquots were taken and digested per EPA Method 3051A: microwave assisted acid digestion of sediments, sludges, soils, and oils. Digested samples were then diluted by serial dilution to concentrations of 1/100, 1/10,000, 1/100,000, and 1/1,000,000. ICP-MS was used as described previously to determine concentration of Hg, Ag, Cu, and Sn.

## **RESULTS**

### **Determination of CAS Service Life and Average Amalgam Surfaces Placed Per Day During Service**

The mean number of amalgam surfaces placed per workday during the service life of each CAS was  $8.4 \pm 1.4$  ( $n=9$ ). The mean service life ( $n=8$ ) for the CAS units was  $131.6 \pm 45.1$  calendar days, or  $67.1 \pm 37.6$  workdays (Table 1). One CAS remained in use for 363 calendar days without loss of performance to the HVE system. However, it was removed from the study after 1 year of use. This particular unit, Chair 2 – CAS 1, proved to be a statistical outlier by Grubbs' test and was excluded from mean service life calculations.

### **Hg Concentrations in Effluent Grab Samples**

No significant difference in mean CAS effluent Hg concentration was observed between the first and second HVE system flush ( $p=0.3345$ ); calculated

Table 1: Service Life in Workdays, Total Amalgam Surfaces Placed, and Amalgam Surfaces Placed Per Day During the Service Life of Each Amalgam Separator<sup>a</sup>

| Provider No. | Chair (CAS) No. | Service Life (Workdays) | Total Amalgam Surfaces Placed | Amalgam Surfaces Placed/Day |
|--------------|-----------------|-------------------------|-------------------------------|-----------------------------|
| Provider 1   | Chair 1 (1)     | 93                      | 528                           | 5.7                         |
|              | Chair 2 (1)     | 157                     | 1200                          | 7.6                         |
| Provider 2   | Chair 3 (1)     | 42                      | 400                           | 9.5                         |
|              | Chair 3 (2)     | 38                      | 336                           | 8.8                         |
|              | Chair 4 (1)     | 57                      | 544                           | 9.5                         |
|              | Chair 4 (2)     | 64                      | 474                           | 7.4                         |
| Provider 3   | Chair 5 (1)     | 42                      | 311                           | 7.4                         |
| Provider 4   | Chair 6 (1)     | 51                      | 510                           | 10.0                        |
|              | Chair 6 (2)     | 60                      | 549                           | 9.2                         |
|              |                 | 67.1±37.6               | 539±263                       | 8.4±1.4                     |

Abbreviation: CAS, chairside amalgam separator.

<sup>a</sup> Selected clinical data were collected for the dates during which each CAS unit was in service. The number of workdays (days on which procedures were recorded) and number of amalgam surfaces placed (procedures logged as codes D2140, D2150, D2160, and D2161) were determined for each chair. The average number of amalgam surfaces placed per day was calculated as Total amalgam surfaces placed/Service life (Workdays). The means±standard deviation are indicated (n=9) for service life (workdays), total amalgam surfaces placed, and amalgam surfaces placed per day.

means did not follow a normal distribution ( $p<0.05$ ). Therefore, mean CAS effluent Hg concentrations were calculated from Hg concentration data measured in both HVE flushes. Mean CAS effluent Hg concentrations ranged from  $0.95 \pm 0.89$  mg/L to  $4.15 \pm 2.51$  mg/L. The minimum and maximum Hg concentrations measured in CAS effluent grab samples were 0.05 mg/L and 11.93 mg/L, respectively, and mean concentration was  $1.99 \pm 2.09$  mg/L (Table 2).

### ISO 11143:2008 Solids Removal Efficiency Testing of CASs

At the end of service life, the mean SRE of the CAS units by ISO 11143:2008<sup>20</sup> was  $99.82 \pm 0.14\%$  (n=4),

Table 2: Mean (SD), Maximum, and Minimum <sup>202</sup>Hg Concentrations Measured in Aliquots of DD2011-P CAS Effluent Grab Samples

| Chair (CAS) No. | Mean (SD), mg/L | Maximum, mg/L | Minimum, mg/L |
|-----------------|-----------------|---------------|---------------|
| Chair 1 (1)     | 2.41 (1.82)     | 6.56          | 0.19          |
| Chair 2 (1)     | 0.93 (0.95)     | 4.70          | 0.09          |
| Chair 3 (1)     | 2.20 (1.12)     | 4.15          | 0.43          |
| Chair 3 (2)     | 1.68 (1.45)     | 4.63          | 0.19          |
| Chair 4 (1)     | 4.07 (3.60)     | 11.93         | 1.17          |
| Chair 4 (2)     | 4.15 (2.51)     | 10.85         | 0.15          |
| Chair 5 (1)     | 0.95 (0.89)     | 3.62          | 0.05          |
| Chair 6 (1)     | 2.75 (2.27)     | 7.62          | 0.43          |
| Chair 6 (2)     | 1.36 (1.20)     | 4.67          | 0.10          |
|                 | 1.99 (2.09)     | 11.93         | 0.05          |

Abbreviations: CAS, chairside amalgam separator; SD, standard deviation.

with a range of 99.65% to 99.95% (Table 3). The accredited testing lab that performed ISO certification tests on the DD2011P reported 99.906% for the empty test condition and 99.626% for the full test condition.<sup>29</sup>

### Mass of Total Solids Accumulated by CASs at End of Service Life

The mean total solids accumulated in each CAS at its respective end of service life was  $195.4 \pm 63.4$  g (n=6) (Table 4). Figure 3 shows that solids accumulation in each unit was mainly due to filtration, as can be seen by the presence of particulate material coating the filter media. Sedimentation within the filter housing only accounted for a small portion of the total accumulated solids, as was evidenced by only a small amount of particulate residing within the space between the filter media and the interior housing wall of the CAS.

The fillable volume of the CAS was determined to be  $500.4 \text{ cm}^3$ , and the corresponding mass of material occupying the fillable volume of the separator was determined to be 634 g of 1 mm glass beads and 2953 g of amalgam powder (of the specific particulate composition described by ISO 11143:2008<sup>20</sup>).

### Composition of Solids Accumulated by CASs at End of Service Life

Composition of the material accumulated in the CASs (an average of 254.933 g) during their service lives was determined to be 57% non-amalgam materials (145.422 g), while the remaining 43%

| Table 3: DD2011P SRE at End of Service Life <sup>a</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |           |               |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|---------------|
| Provider No.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | Chair No. | Final SRE (%) |
| Provider 1                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | Chair 2   | 99.95         |
| Provider 2                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | Chair 3   | 99.76         |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | Chair 4   | 99.92         |
| Provider 3                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | Chair 5   | 99.65         |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |           | 99.82±0.14    |
| Abbreviations: CAS, chairside amalgam separator; SRE, solids retention efficiency.                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |           |               |
| <sup>a</sup> Four DD2011P CAS units were recovered at the ends of their service lives. The CAS units were dried and weighed for total solids analysis and then re-wetted and subjected to a modified efficiency test based on the ISO 11143:2008 method: A defined mixture of dental amalgam particulate was suspended in filtered water and passed through the spent CAS units. The mass of dental amalgam exiting the separators was weighed to calculate the efficiency. The mean efficiency of four CAS units±standard deviation is indicated (n=4). |           |               |

was amalgam (109.511 g). Mercury (20.9%) was the major metal of the recovered solids, with silver (11.7%), tin (6.1%), and copper (4.3%) making up the remaining amalgam constituents (data not shown).

DISCUSSION

The main objective of this study was to perform quality assurance testing per ISO 11143:2008<sup>20</sup> on the CAS to confirm EPA compliance in a clinical setting in which a relatively high volume of amalgam restorations are placed on a routine basis. The results of ISO 11143:2008<sup>20</sup> testing in our lab show that the CAS exceeds the minimum SRE requirements of the EPA dental effluent guidelines. Four randomly selected units that had reached the ends of their service lives in clinical use showed a mean SRE of 99.82 ± 0.14%. The end of service life SRE results

| Table 4: DD2011P Total Solids Captured During Service Life <sup>a</sup>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                 |                    |                     |                 |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------|--------------------|---------------------|-----------------|
| Provider No.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | Chair (CAS) No. | CAS Tare Weight, g | CAS Final Weight, g | Total Solids, g |
| Provider 1                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | Chair 1 (1)     | 409.55             | 563.65              | 154.10          |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | Chair 2 (1)     | 409.33             | 661.55              | 252.22          |
| Provider 2                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | Chair 3 (1)     | 402.74             | 604.04              | 201.30          |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | Chair 4 (1)     | 419.41             | 664.41              | 245.00          |
| Provider 3                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | Chair 5 (1)     | 407.42             | 638.57              | 231.15          |
| Provider 4                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | Chair 6 (1)     | 420.44             | 509.02              | 88.58           |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                 |                    |                     | 195.40±63.40    |
| Abbreviation: CAS, chairside amalgam separator.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                 |                    |                     |                 |
| <sup>a</sup> Tare weights of the first six CAS units prior to installation were recorded after the drying procedure (104±1°C for 2 hours, then stabilized in a desiccator for 24 hrs). At the ends of their service lives, separators were weighed again, following the same drying/stabilization procedure to determine final weight. Total solids are the difference between the tare weight and the final weight. The weights of accumulated solids for six individual CAS units are shown. The mean weight of accumulated solids±standard deviation is indicated (n=6). |                 |                    |                     |                 |

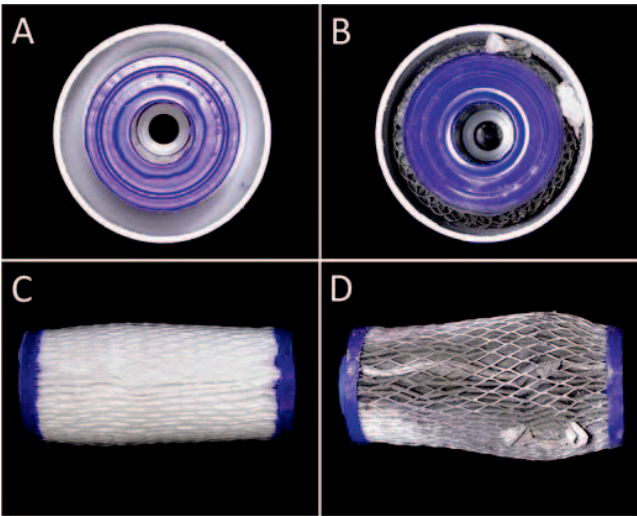


Figure 3. Cross-sectional view of a DD2011P CAS before installation (A). Cross-sectional view of the CAS at the end of service life (B). Note the minimal amount of accumulated sediment (white arrow) within the interior space between the filter element and the plastic filter housing. Panel (C) shows the interior filter medium removed from a new DD2011P CAS unit. Panel (D) shows the same filter.

we obtained were consistent with the SRE values reported in the independent ISO 11143:2008<sup>20</sup> test report provided by the manufacturer (ie, 99.906% on the empty test and 99.626% on the full test). These results suggest that while failure ultimately occurs due to HVE obstruction by trapped particulate matter, SRE remains, through the entire service life, more than adequate to satisfy the EPA rule. This outcome demonstrates that cleaning solutions, human tissues and fluids, and microorganisms, which are present in the clinical application of the amalgam separator but not the ISO test method, have no detrimental effect on long-term separator performance.

CAS effluent grab sampling and subsequent Hg concentration quantification by ICP-MS were used to monitor separator performance throughout the service life of the CAS, as ISO 11143:2008<sup>20</sup> SRE testing was restricted to preclinical deployment and end of service life time points. For context, in terms of Hg concentrations in CAS effluent, few historical data are available, with the exception of a study conducted by Stone and others<sup>19</sup> in which 4.25 ± 6.35 mg Hg/chair/d was observed in effluent composite samples (defined as samples taken over a designated time interval, as opposed to a grab sample) of a CAS with a 0.5-µm filter cartridge. In direct comparison to Stone and others,<sup>19</sup> Hg concentrations observed in the effluent of the CAS evaluated in this study, which also has a nominal pore size of 0.5 µm, appear to be comparable, as the mean Hg concentration

observed in the current study was  $1.99 \pm 2.09$  mg/L. However, Stone and others<sup>19</sup> performed composite sampling, whereas this study utilized biweekly grab sampling. The grab sampling method only reflects the Hg concentrations in the CAS effluent at the point in time during which the samples were collected, whereas composite sampling may better reflect total mass discharge of Hg over time. However, the grab sampling technique used in this study may provide a worst-case estimate of Hg discharge at a given time, as the two consecutive 1 L flushes of the HVE system used for the effluent grab sampling procedure are comparable to routine tap water flushes, which typically occur at the beginning of the workday and between patient seatings. Under normal operating conditions, influent reaches the CAS at a rate on the order of mL/h, rather than in large boluses on the order of mL/s. While the grab sample method used in this study does not account for all effluent, by our estimates, the 2 L collected on a sample day represent the majority of that day's effluent, with perhaps 1 L of other fluids contributing to an estimated daily volumetric total of 3 L of effluent per chair on sample collection day, totaling an approximate 3 mg Hg/chair/d compared with the 4.25 mg Hg/chair/d reported by Stone and others. It is debatable whether effluent arriving in large boluses is more or less effective than the gradual delivery of small fluid volumes at extracting mobile-phase amalgam into the effluent, and thus represents an additional source of variability. Further, the daily mean number of amalgam surfaces placed per chair recorded by Stone and others<sup>19</sup> ( $19.11 \pm 12.31$ ) was greater than in the current study ( $8.4 \pm 1.4$ ). Therefore, CAS influent Hg concentrations observed in the study by Stone and others<sup>19</sup> were likely higher, which may explain why the effluent concentrations were greater than those observed in this study. Since sampling in both studies was collected from the CAS effluent, which goes on to be diluted into the entire facility's effluent, it should be noted that the Hg concentrations reported by both Stone and others<sup>19</sup> and this study do not reflect the final concentration ultimately discharged to POTWs.

A limitation of our approach is that we did not choose the dental practice or providers randomly. Our goal was to evaluate the performance of a specific chairside separator design commonly used in Navy and other military DTFs. We selected a military DTF and selectively installed CAS units to be evaluated on the chairs of providers whose practices were primarily limited to operative den-

tistry, with the additional criteria that they be high users of amalgam. We chose this evaluation design to best reproduce a worst-case scenario of frequent amalgam use in practice to better evaluate how well the CAS would perform under these conditions with respect to EPA compliance. Other providers within the same group practice, other nonmilitary large group practices, and smaller private practice offices may have significantly different usage rates of amalgam. With respect to service life, our evaluation results are limited to the conditions under which the separators were evaluated. However, the range of service lives for the CAS units in our study indicate that there are a number of uncontrolled factors beyond amalgam use rates that contribute to separator filter obstruction and ultimate failure. Therefore, our findings that the CAS units failed due to HVE obstruction and loss of suction rather than failure to maintain EPA-compliant SRE is likely to be a generalizable finding in any practice setting, despite variability in service life.

According to the EPA Dental Effluent Guidelines, dental offices that remove or place dental amalgam only under "unanticipated circumstances" are exempt from any requirements described in the final rule for the exception of the one-time compliance report.<sup>6</sup> However, dental providers who do not place amalgam but know they will periodically see patients that require amalgam removals must install an amalgam separator. These providers may consider installing CAS units as a lower-cost alternative to a centralized amalgam separator in order to meet compliance requirements. Such providers may also consider restricting amalgam removals to a dedicated dental chair or chairs with an installed CAS, rather than placing a CAS on every chair in their office. No correlation was observed between service life and the rate of amalgam use, indicating that other dental particulate and total accumulated solids are the major contributors to failure. Therefore, a CAS installed on a dental chair in which no amalgam placement or removal is performed would similarly become obstructed and require replacement. Additionally, for dental practices that place and/or remove amalgam restorations regularly and have centralized amalgam separators installed in their facilities, CAS units installed upstream of the centralized system may serve as an additional control measure that may increase the efficiency and service life of the centralized amalgam separator. One key advantage of the CAS concept over the centralized amalgam separator is its location at the point of initial discharge, which mitigates amalgam

accumulation in downstream plumbing, especially in low points and longer horizontal sections. The UFC 4-510-01,<sup>22</sup> which governs federal medical and dental facilities, for this very reason, instructs that amalgam separators “shall be incorporated as close to the source point as feasible.”

Examination of the dissected CAS units at the ends of their service lives (Figure 3) revealed that sedimentation accounted for only a small portion of total accumulated solids, as roughly <1% of the interior space between the filter element and the plastic filter housing was occupied. We determined that if the fillable volume were filled entirely with lighter materials (ie, tooth or composite particles), our observed total accumulated solids would occupy approximately 31% of the fillable space, while if the fillable volume were filled entirely with amalgam, our observed total accumulated solids would occupy only about 7% of the fillable space. In reality, the actual composition of the accumulated solids would be somewhere between these two extremes, with the ratio of tooth and composite to amalgam particulate varying with the frequency of amalgam placements and with placement technique. We determined from a pooled random sample of six spent separators that only 43% of the accumulated solids were amalgam. While we did not specifically determine the origin of the other 57%, we can safely surmise it is comprised of tooth structure, dental composite, abrasive particles, and other particulate dental and oral materials. This is not meant to be a robust determination, applicable to all dentists in all facilities, but it does illustrate the point that a filtration-based separator captures more than just amalgam. The ratio of amalgam to other captured material will vary with amalgam placement/removal technique, as well as the size and frequency of these procedures.

The fact that separators reached the end of their service lives with relatively little accumulated material inside them suggests that the housing could be redesigned to be considerably smaller while still accommodating the accumulated solids. The CAS is designated a Type 3 amalgam separator, meaning that it removes dental amalgam particles via filtration alone. The end of service life images also show that the recovered filter cartridges are almost entirely coated with accumulated dental material solids, confirming the intended functional specification. However, the integration of other amalgam separation methods, such as sedimentation, into the CAS design could potentially prolong service life without compromising SRE and HVE suction.

We conducted grab sampling of CAS effluent via a two-step method, whereby two 1 L boluses were sequentially aspirated through the HVE/CAS system and collected separately. This was done to determine the degree to which dissolved mercury contributed to the effluent levels, based on the premise that particulate amalgam trapped within the separator would, over time, leach some amount of Hg into the wet milieu of the separator's interior. A limitation of this method was that the activity of the CAS (or lack thereof) prior to our sample collection was unknown to us, but it was hypothesized that we would see a small difference between the first and second effluent collections, with any observed difference being due to metals that had dissolved into the milieu in the time since the most recent usage. While on occasion this was true for individual data points, including notable outliers that we included in the analysis, overall the difference in effluent Hg concentration between the first and second flushes was insignificant. These results are consistent with the EPA's assertion that only 0.4% of Hg in dental effluent is in the dissolved state.<sup>6</sup>

Based on feedback from providers that used the dental chairs on which the CAS units were installed, it was determined that there is also a need for a more ergonomic chairside design or an alternative installation location. In an attempt to improve ergonomics for the providers, CAS units were successfully installed within the floor box of chairs 5 and 6 (Figure 1). However, if the diameter of the CAS were decreased by 10% to 15%, the unit would be perfectly suited for floor box installation in A-dec 500 dental chairs. In its current form, it precludes full closure of the floor box cover and requires ample creativity to route the tubing appropriately through a tight and crowded space. Further, a smaller CAS design could decrease per-unit purchase price by lowering material costs and, where amalgam waste is managed on a volumetric basis, reduce disposal costs as well.

One fundamental consideration behind the final EPA rule was the current state of best-available amalgam separator technology balanced with economic achievability. Two constraints inform this standard: 1) which technologies are mature enough to implement on a broad, national scale, and 2) which of these technologies can be used in the vast majority of dental practices without imposing undue financial burden. The current state of amalgam separator technology exceeds EPA Dental Effluent Guidelines compliance requirements, as the median

SRE of commercially available, ISO-certified amalgam separators in the United States is 99.3%.<sup>6</sup> Therefore, the CAS we evaluated may be considered a competitive product in terms of SRE, cost, and installation versatility.

It should be noted that certain state and local municipalities have promulgated additional compliance requirements, including, but not limited to, defined Hg discharge limits and compliance monitoring (eg, inspections) by local POTWs.<sup>8,30</sup> Dental providers should consult their respective control authorities to determine their specific compliance requirements, as the final rule of the EPA Dental Effluent Guidelines represents only the federal-level compliance benchmark.<sup>6</sup>

Finally, according to the EPA,<sup>6</sup> dissolved Hg accounts for  $\leq 0.4\%$  of dental amalgam process wastewater. The current EPA guideline limits Hg-containing amalgam separation to particulate and does not account for dissolved Hg. While the CAS design tested is ISO11143:2008-certified for 99.6% SRE, downstream polishing processes (eg, ion-exchange resin) could be added to account for dissolved Hg should future compliance requirements necessitate additional control measures.

## CONCLUSIONS

- 1) The CAS evaluated meets the ISO standard for particulate amalgam removal, and its use on dental chairs that place/remove amalgam will meet the EPA dental wastewater effluent guideline.
- 2) The clinical evaluation of the CAS shows variability in service life due to a variety of clinical factors beyond the amount of amalgam used in practice.

## Acknowledgements

This work was supported by the US Navy Bureau of Medicine and Surgery (BUMED) M41, work unit number G1016. The authors would like to thank Dr G Woollard, clinic director, and Mr D Meyers, practice manager, Budge Dental Clinic, Fort Sam Houston DENTAC, JBSA Fort Sam Houston, Texas, for allowing us access to their DTF and provider procedure data; Drs J Bullard, S Gokhale, R Ruggiero, and CPT R Michalak, U.S. Army, as well as their assistants, Ms K McHazzlett, Ms K Schaefer, Ms Y Perez, Mr Z Poole, Ms F Ibarra, Ms K Sgambelluri, and Ms M Lockhart, for graciously allowing us to evaluate the CAS units on their dental chairs and for providing invaluable feedback; Mr W Purvis, biomedical engineering/equipment technician, and Ms T Dinh, Naval Medical Research Unit San Antonio, for technical assistance; Dr A Mallory, Naval Medical Research Unit San Antonio, for early study development and background; and LT N Dickens, DC, USN, Naval Medical Research Unit San Antonio, for statistical analysis.

## Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Naval Medical Research Institute in San Antonio, Texas.

## Disclosure

The views expressed in this article are those of the authors and do not necessarily reflect the official policy or position of the Department of the Navy, Department of Defense, or the US Government. Dr D Olivera is an employee of the US government, and LCDR N Hamlin is a military service member. This work was prepared as part of their official duties.

Although samples and operative procedure data were obtained from a military dental treatment facility, neither patient personal health information nor personal identifying information was revealed to the authors of this study. Therefore, this study was exempt from Institutional Review Board review. This document is not intended to be a comprehensive summary or review of the EPA Effluent Limitations Guidelines and Standards for the Dental Category, 40 Code of Federal Regulations Part 441. Approved for public release; distribution is unlimited.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 19 January 2019)

## REFERENCES

1. US Department of Health and Human Services Public Health Service (1993) Dental amalgam: a scientific review and recommended Public Health Service strategy for research, education and regulation – amalgam use and benefits. Retrieved online February 12, 2018 from: <https://health.gov/environment/amalgam1/amalgamu.htm>
2. American Dental Association (2009) Statement on dental amalgam; Retrieved online November 16, 2017 from: <http://www.ada.org/en/about-the-ada/ada-positions-policies-and-statements/statement-on-dental-amalgam>
3. FDI World Dental Federation and World Health Organization (1997) FDI policy statement/WHO consensus statement on dental amalgam; Retrieved online November 16, 2017 from: <https://www.fdiworlddental.org/sites/default/files/media/documents/WHO-consensus-statement-on-dental-amalgam-1997.pdf>
4. US Food and Drug Administration (2015b) Appendix I: summary of changes to the classification of dental amalgam and mercury; Retrieved online November 16, 2017 from: <https://www.fda.gov/MedicalDevices/ProductsandMedicalProcedures/DentalProducts/DentalAmalgam/ucm171120.htm>
5. United Nations Environment Programme (2013) Minamata Convention on Mercury *United Nations Environment Programme, Nairobi, Kenya*
6. US Environmental Protection Agency (2017) 40 CFR Part 441: effluent limitations guidelines and standards for the dental category *Federal Register* **82(113)** 27154-27178.

7. Welland C (1991) Dental office waste stream characterization study *Seattle, WA: Municipality of Metropolitan Seattle*.
8. Washington State Department of Ecology (nd) Dentists—amalgam separators; Retrieved online February 13, 2018 from: <https://ecology.wa.gov/Regulations-Permits/Guidance-technical-assistance/Dangerous-waste-guidance/Common-dangerous-waste/Dentists>
9. Vandeven JA & McGinnis SL (2005) An assessment of mercury in the form of amalgam in dental wastewater in the United States *Water, Air, and Soil Pollution* **164**(1-4) 349-366.
10. Drummond JL, Cailas MD, & Croke K (2003) Mercury generation potential from dental waste amalgam *Journal of Dentistry* **31**(7) 493-501.
11. Drummond JL, Liu Y, Wu T-Y, & Cailas MD (2003) Particle versus mercury removal efficiency of amalgam separators *Journal of Dentistry* **31**(1) 51-58.
12. Driscoll CT, Mason RP, Chan HM, Jacob DJ, & Pirrone N (2013) Mercury as a global pollutant: sources, pathways, and effects *Environmental Science & Technology* **47**(10) 4967-4983.
13. Pickhardt PC & Fisher NS (2007) Accumulation of inorganic and methylmercury by freshwater phytoplankton in two contrasting water bodies *Environmental Science & Technology* **41**(1) 125-131.
14. Watras CJ, Back RC, Halvorsen S, Hudson RJ, Morrison KA, & Wente SP (1998) Bioaccumulation of mercury in pelagic freshwater food webs *Science of the Total Environment* **219**(2-3) 183-208.
15. Seixas TG, Moreira I, Siciliano S, Malm O, & Kehrig HA (2014) Differences in methylmercury and inorganic mercury biomagnification in a tropical marine food web *Bulletin of Environmental Contamination and Toxicology* **92**(3) 274-278.
16. Knobeloch LM, Ziarnik M, Anderson HA, & Dodson VN (1995) Imported sea bass as a source of mercury exposure: a Wisconsin case study. *Environmental Health Perspectives* **103**(6) 604-606.
17. Stone ME, Cohen ME, Liang L, & Pang P (2003) Determination of methyl mercury in dental-unit wastewater *Dental Materials* **19**(7) 675-679.
18. US Environmental Protection Agency (2017) National recommended water quality criteria—aquatic life criteria table; Retrieved online November 9, 2017 from: <https://www.epa.gov/wqc/national-recommended-water-quality-criteria-aquatic-life-criteria-table>
19. Stone ME, Cohen ME, Berry DL, & Ragain JC Jr (2008) Design and evaluation of a filter-based chairside amalgam separation system *Science of the Total Environment* **396**(1) 28-33.
20. ISO-Standard (2008) ISO 11143:2008 dentistry – amalgam separators *Geneva: International Organization for Standardization* **2nd edition** 1-25.
21. The Simple One (nd) Chair side amalgam separator; Retrieved online November 17, 2017 from: <http://www.chairsidefilter.com/>
22. Department of Defense (2017) UFC 4-510-01 design: military medical facilities *Unified Facilities Criteria* **Change 2** 89-90.
23. American Dental Association (2016) CDT 2017: Dental Procedure Codes *American Dental Association* **15th edition**. Chicago, IL.
24. Ivoclar Vivadent (2014) Safety data sheet—Valiant, Valiant Snap-Set, Valiant Ph.D., Valiant Ph.D. XT, Valiant Ph.D. EXT; Retrieved online February 12, 2018 from: <http://www.dentalcity.com/MSDS/77-0410.pdf>
25. US Environmental Protection Agency (2014) Inorganic analytes In: *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846 Compendium 3rd edition (Final Update V)* Environmental Protection Agency Washington, DC 1-27.
26. US Environmental Protection Agency (2007) Method 3015A revision 1: microwave assisted acid digestion of aqueous samples and extracts *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846 Compendium 3rd edition (final update iv)* Environmental Protection Agency Washington, DC 1-25.
27. US Environmental Protection Agency (2014) Method 6020B revision 2: inductively coupled plasma-mass spectrometry *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846 Compendium 3rd edition (Final Update V)* Environmental Protection Agency Washington, DC 1-33.
28. Rice EW, Baird RB, Eaton AD, & Clesceri LS (eds) (2012) *Standard Methods for the Examination of Water and Wastewater* **22nd edition** American Public Health Association, American Water Works Association, Water Environment Federation, Washington, DC.
29. NSF International test report FL20151103105324, November 3, 2015, [nsf.org](http://nsf.org)
30. US Environmental Protection Agency (2014) Technical and economic development document for the proposed Effluent Limitation Guidelines and Standards for the Dental Category *EPA-821-R-14-006* 6-5—6-24 Washington, DC: *Environmental Protection Agency*.

# Influence of the Fabrication Technique on the Marginal and Internal Adaptation of Ceramic Onlays

CF Neto • GC Santos • MJMC Santos

## Clinical Relevance

CEREC BlueCam and CEREC OmniCam CAD-CAM systems were able to fabricate onlays with clinically acceptable marginal discrepancies, below 100  $\mu\text{m}$ ; however, the Press system presented superior internal and marginal adaptation.

## SUMMARY

**This study aimed to evaluate the marginal and internal adaptation of partial coverage crowns (ceramic onlays) fabricated with Press, CEREC BlueCam, and CEREC OmniCam systems, using two preparation designs and evaluating the internal discrepancies at different locations. Two phantom maxillary premolars (master teeth) received different preparation designs, with (BX) and without (NB) a modified occlusal box with round internal angles. Sixty IPS e-max ceramic restorations were fabricated with three systems: Press (n=20), CEREC**

**BlueCam (n=20), and CEREC OmniCam (n=20). Both marginal and internal discrepancy width were measured by using a stereomicroscope at  $\times 25$  magnification. The data were evaluated statistically using analysis of variance followed by Tukey's Honestly Significant Difference test ( $\alpha=0.05$ ). The ceramic restorations fabricated with the Press system presented significantly smaller marginal and internal disadaptations than the BlueCam and OmniCam CEREC systems ( $p<0.0001$ ). Regarding the preparation designs, preparation BX presented the smallest marginal discrepancies for all fabrication systems and larger internal discrepancies than for restorations fabricated with the Press system. The occlusal location presented a larger internal discrepancy compared with the axial locations. Although the three systems resulted in the fabrication of restorations within a clinically acceptable adaptation with marginal discrepancies below 100  $\mu\text{m}$ , the Press system presented the smallest marginal and internal discrepancies. An improved marginal adaptation was observed in the preparation design with a modified occlusal box with rounded internal angles.**

Constantino Fernandes Neto, DDS, MSc candidate, Bauru Dental School, Universidade de São Paulo, Bauru SP, Brazil

Gildo Coelho Santos, DDS, MSc, PhD, associate professor, Schulich School of Medicine & Dentistry, University of Western Ontario, London, Ontario, Canada

\*Maria Jacinta MC Santos, DDS, MSc, PhD, associate professor, Schulich School of Medicine & Dentistry, University of Western Ontario, London, Ontario, Canada

\*Corresponding author: DSB room 0149, London, ON N6A 5C1, Canada; e-mail: jacinta.santos@schulich.uwo.ca

DOI: <https://doi.org/10.2341/19-014-L>

## INTRODUCTION

The introduction of computer-aided design/computer-aided manufactured technology (CAD-CAM) has allowed the use of high-performance materials to fabricate dental restorations.<sup>1,2</sup> Among these materials, ceramics have been widely used to attend to the increasing demand for conservative and esthetic treatments.<sup>2-4</sup> In this context, partial coverage crowns (PCCs) or onlays are indicated to restore complex cavity preparations to provide superior esthetics and anatomic contours in occlusal and proximal locations while preserving more tooth structure compared with full coverage crowns.<sup>5</sup> Glass ceramics possess excellent optical properties that result in highly esthetic restorations due to the so-called chameleon effect.<sup>6</sup> The preparation design for ceramic onlays requires divergent walls (15°), round internal line angles, supragingival margins, and avoidance of bevels and sharp angles. These features facilitate the scanning and milling procedures for CAD-CAM restorations.<sup>2,4</sup> Differently from traditional gold onlays, the use of frictional retention on preparations is contraindicated to prevent stress and consequent crack initiation in ceramic materials,<sup>7</sup> which are adhesively bonded to the dental tissues.<sup>7-9</sup>

A range of glassy and polycrystalline ceramic materials are available for laboratory and/or chairside use. They vary in composition and processing techniques, allowing different methods of fabrication, such as powder condensation (conventional powder slurry ceramics), heat-pressed (pressable ceramics), split casting (infiltrated ceramics), and milled (machinable or CAD-CAM ceramics).<sup>10</sup> The CAD-CAM system uses a scanning device, design software, and a milling machine to fabricate restorations.<sup>1</sup> As a result, the professional can take digital scans with a handheld three-dimensional intraoral scanner, using a compact chairside CAD-CAM unit that allows either sending them to a laboratory to fabricate the restoration or milling in-house.<sup>1,11</sup> The CAD-CAM system uses presintered ceramic blocks, which can be composed of feldspathic porcelain-based ceramics, leucite-reinforced glass ceramics, lithium-disilicate glass ceramics, glass infiltrated ceramics (or polycrystalline alumina), or zirconia materials.<sup>1,2</sup> Each material presents different mechanical, physical, and optical properties, which should be taken into consideration according to the type of restoration (partial or total), location (anterior or posterior), and esthetic requirements.

IPS e.max lithium disilicate (Ivoclar Vivadent AG, Schaan, Liechtenstein) restorations can be

fabricated by using either CAD-CAM milling procedures (IPS e.max CAD) or the lost-wax techniques (IPS e.max Press).<sup>11</sup> The use of a chairside CAD-CAM system offers advantages regarding the elimination of conventional impressions taken with polyvinyl siloxane (PVS) materials, need for disinfection, shipment to the laboratory, and use of temporary restorations. Although the three-dimensional intraoral camera can provide a high level of detail of the prepared teeth and the surrounding tissues, there are still questions regarding the accuracy of marginal and internal fit that the CAD-CAM systems can provide. Marginal fit is considered an important criterion for the clinical success of indirect restorations because the presence of gaps can favor biofilm accumulation and secondary caries.<sup>5</sup> Internal gap is related to the accuracy of the internal fit of the restoration and determines the space to be filled by the cement.<sup>12</sup> As a result, factors such as preparation design,<sup>5,13,14</sup> impression technique,<sup>15</sup> luting agent,<sup>8</sup> and system used to fabricate the restoration<sup>2</sup> may influence marginal and internal adaptation.

The survival rates of ceramic onlays range from 93% to 98% at five years and from 64% to 95% after 10 years.<sup>16,17</sup> The high success rate is attributed to the capacity of these glass-ceramic systems to be etched and bonded to the tooth structure with resin-based cements.<sup>4,18</sup> The most common causes of failure are related to fracture of the ceramic material and recurrent caries.<sup>4,19</sup> Previous studies have reported that lack of precision in internal fit could heighten the risk of fractures in ceramic materials.<sup>20,21</sup> For this reason, studies evaluating the internal space to be occupied by the cement are relevant. Furthermore, the internal and marginal adaptation of CAD-CAM restorations may be influenced by the characteristics of preparation design, because the internal features of the restorations are directly related to the diameter of the burs used in the milling machine during manufacturing.<sup>21-23</sup>

CAD-CAM systems have evolved since their introduction in the 1980s. The first Chairside Economical Restoration of Esthetic Ceramics (CEREC) systems, CEREC 1 and 2, presented poor esthetic results, and the average marginal discrepancies were approximately 308 and 207  $\mu\text{m}$ , respectively.<sup>24</sup> Smaller marginal discrepancies have been reported for the CEREC 3 system, ranging from 27 to 162  $\mu\text{m}$ .<sup>25,26</sup> In terms of internal fit, studies have reported internal discrepancies of  $212 \pm 45 \mu\text{m}$  for CEREC 2 and  $146 \pm 15 \mu\text{m}$  for CEREC 3.<sup>25,27</sup> Furthermore, the esthetic features of the CAD-CAM

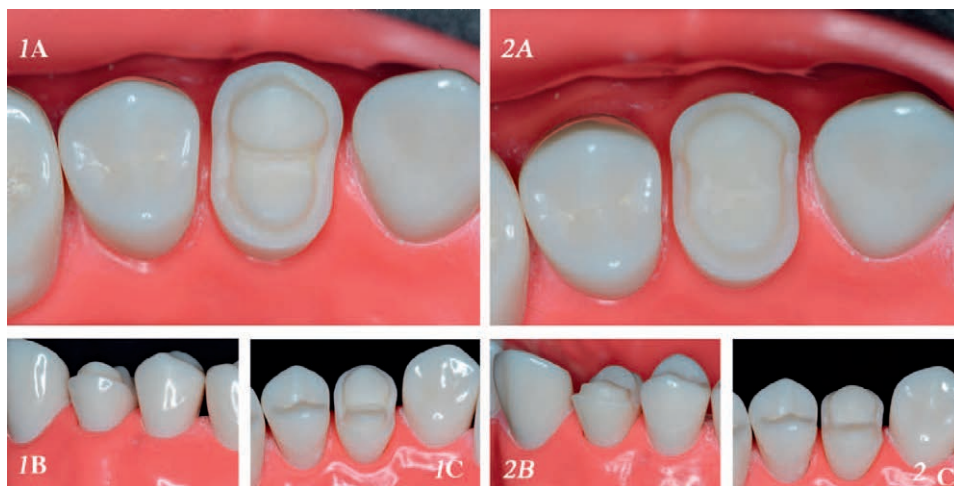


Figure 1. Preparation BX (with occlusal box).

Figure 2. Preparation NB (without occlusal box).

restorations have improved with the introduction of presintered ceramic blocks that are able to create a natural and harmonious transition between a more translucent shade at the occlusal to a more opaque and saturated shade at the cervical area.<sup>28</sup> Additionally, new technologies with improved software versions and three-dimensional intraoral scanners have been released. The CEREC OmniCam system has been introduced in the market, exempting the use of a reflective powder for image capture, and resulting in fewer clinical steps. However, there are still questions regarding its accuracy to generate restorations with superior adaptation compared with other systems. Hence, further investigations are necessary to understand the impact of CAD-CAM technology on the marginal and internal fit of ceramic restorations.

The present study aimed at evaluating the marginal and internal adaptation of PCCs (ceramic onlays) fabricated with heat-pressed (Press), CEREC BlueCam, and CEREC OmniCam systems, using two preparation designs. The three null hypotheses to be tested were that there are no differences on the marginal and internal adaptations: (1) between the three systems used to fabricate the ceramic onlays; (2) between the two preparation designs tested; and (3) between different locations of the internal space of ceramic onlays.

## METHODS AND MATERIALS

### Onlay Preparations

Two plastic teeth from a phantom model (maxillary right first premolars, Typodont, Kilgore International, Coldwater, Michigan, USA) received two ceramic onlay preparation designs (Figures 1 and 2). Both preparations presented divergent walls (15°), round

internal line angles, 2-mm cusp reduction, 1.25-mm shoulder around the margins, and 0.5- to 1-mm supragingival margins. Preparation BX (box) consisted of a modified occlusal box with rounded internal angles and the reduction of both facial and lingual cusps. Preparation NB (no box) presented no occlusal box with the reduction of both facial and lingual cusps. Cavity preparations were performed with flat-end tapered diamond burs with rounded angles #6845 and #837 KR (Brasseler, Savannah, GA, USA) by the same operator using magnifying glasses (2.5×) and headlights.

Sixty indirect restorations were fabricated using three systems: pressable ceramics (n=20); CAD-CAM CEREC BlueCam (n=20); and CAD-CAM CEREC OmniCam (n=20). For the heat-pressed technique, 20 impressions were made using polyvinyl siloxane (Imprint 3, 3M ESPE, Minneapolis, MN, USA) impression material to obtain 10 plaster models from each preparation design, using a die stone of type IV gypsum (Lean Stone, Whip Mix, Louisville, Kentucky, USA). The models were sent to the laboratory to fabricate the restorations using presintered ceramic blocks (IPS e-max Press, Ivoclar-Vivadent AG). The restorations were milled in fine mode, using the milling machine burs (12S Step Bur, 1.4 mm; and 12S Cylinder Pointed Bur, 1.4 mm; Sirona MC XL, Dentsply Sirona, Konstanz, Germany). Burs were replaced after each 10 specimens to avoid variations between groups.

For the CAD-CAM CEREC BlueCam, a thin layer of a reflective powder (IPS Contrast Spray Labside, Ivoclar Vivadent AG) was used to spray the master models, and 20 digital scans were obtained, 10 from each preparation design. To standardize the acquisition process of digital models for the BlueCam group, a total of 10 to 15 snaps were taken from

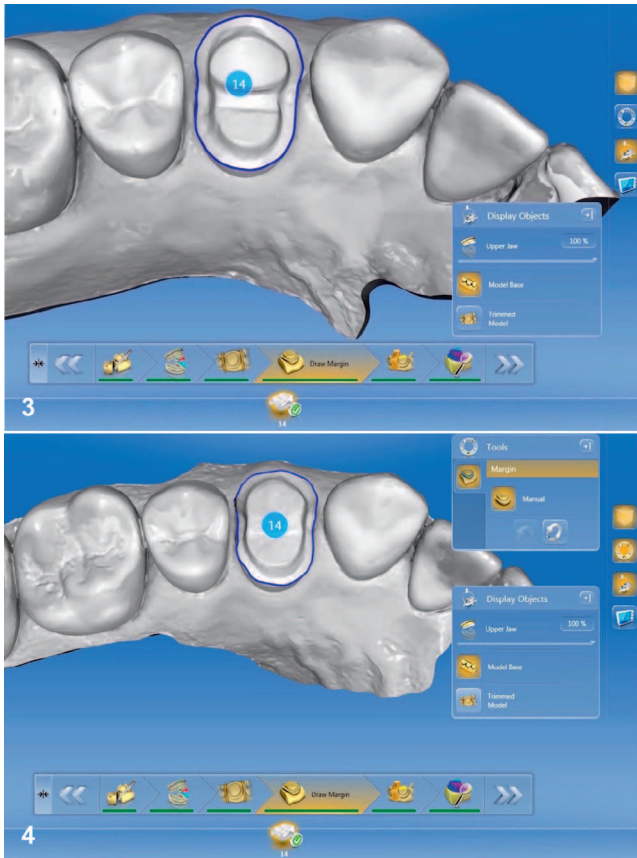


Figure 3. View of preparation BX with occlusal box: Omnicam system.

Figure 4. View of preparation NB without occlusal box: Omnicam system.

predetermined areas of the maxillary right quadrant to allow the acquisition of satisfactory digital models. For the CAD-CAM CEREC OmniCam, 20 digital scans were obtained, 10 from each preparation design (Figures 3 and 4). The image acquisition of the OmniCam group consisted of a sequence of consecutive movements with the handheld scanner without the use of a reflective powder, following the manufacturer's instructions. For both CAD-CAM groups, biogeneric onlay restorations were designed using the CEREC 4.2 software, and restorations were fabricated in the milling cabinet (CEREC MC XL, Dentsply Sirona) using presintered ceramic blocks (IPS e-max CAD, Ivoclar Vivadent AG). The following preset parameters were used, as suggested by the manufacturer's instructions: spacer, 120°; marginal adhesive gap, 60  $\mu\text{m}$ ; occlusal milling offset, 0  $\mu\text{m}$ ; proximal contacts strength, 25  $\mu\text{m}$ ; occlusal contact strength, 25  $\mu\text{m}$ ; dynamic contacts strength, 25  $\mu\text{m}$ ; minimal thickness (radial), 500  $\mu\text{m}$ ; minimal thickness (occlusal), 700  $\mu\text{m}$ .

### Measurement of Marginal Fit

To measure the marginal adaptation, each ceramic restoration was placed on the prepared master tooth and stabilized with composite resin (Filtek Supreme Ultra, 3M ESPE) at two external points away from the margins and connected by a metal wire. The margins of the restorations were measured at four locations (facial, lingual, mesial, and distal) using an optical stereomicroscope (Leica S6 D, Leica Microsystems, Buffalo Grove, IL, USA) at  $\times 25$  magnification and a digital camera (EC3, Leica Microsystems) coupled to the optical stereomicroscope. An imaging software (LAS Application Software Modules, Leica Microsystems) was used to measure the distance between the border of the ceramic restoration and the margin of the preparation at the four locations. For each location, three measurements were taken from four predetermined points, which resulted in a total of 48 measurements from each sample analyzed. The average number ( $\mu\text{m}$ ) obtained from each location was calculated, and the data were submitted for statistical analysis.

### Measurement of Internal Fit

The internal fit was evaluated using the replica technique reported by Souza and others<sup>25</sup> and Bottino and others,<sup>26</sup> which consists of measuring a thin layer of extra-light-body PVS impression material (Imprint 3, 3M ESPE) placed between the master tooth preparation and the restoration. The prepared tooth was filled with a thin layer of extra-light-body PVS, a ceramic onlay was placed over the PVS material, and a load of 750g was applied over the onlay to assure maximum fit. After the impression material was set, the load and onlay were removed, leaving a thin film of silicone adhering to the preparation, representing the space between the onlay and the tooth preparation. For the purpose of stabilization, a light-body PVS (Imprint 3, 3M ESPE) with a different color was placed in the space previously occupied by the onlay, which set over the light-body PVS film. With this procedure, the thin film of extra-light-body PVS could be removed from the preparation without plastic deformation, allowing it to be further cut according to the predetermined areas that would be evaluated.

The replica was then cut mesio-distally at two sites and bucco-lingually at one site (Figure 5). The internal space was measured using an optical stereomicroscope (Leica S6 D, Leica Microsystems) at  $\times 25$  magnification and a digital camera (EC3, Leica Microsystems) coupled to the optical stereomicroscope. Imaging software (LAS Application Soft-

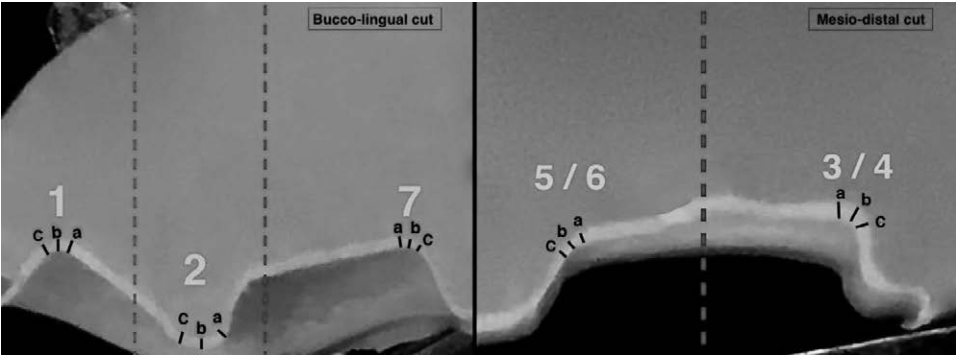


Figure 5. PVS replica. Dotted lines represent cuts made on replica. Numbers represent locations of areas measured: 1, lingual; 2, occlusal; 3/4, mesial; 5/6, distal; 7, facial. Letters indicate the equidistant locations measured in each surface.

ware Modules, Leica Microsystems) was used to measure the extra-light-body PVS layer thickness at the predetermined locations. A total of nine measurements were performed along the bucco-lingual slice and six measurements at the mesio-distal slices, resulting in 21 measurements from each sample. The measurements from each location were grouped as follows: axial (n=18) and occlusal (n=3). The average thickness of each location was calculated and submitted for statistical analysis.

Data Analysis

Marginal adaptation was analyzed using two-way analysis of variance (ANOVA) followed by Tukey’s HSD test ( $\alpha=0.05$ ). Internal fit was analyzed using three-way ANOVA followed by Tukey’s HSD test ( $\alpha=0.05$ ). Data were analyzed using statistical software Statistica 13.3 (StatSoft, TIBCO Software, Palo Alto, California, USA).

RESULTS

The results from marginal fit are shown in Tables 1 and 2. Two-way ANOVA (Table 1) showed that marginal discrepancy varied according to the fabrication system ( $p<0.001$ ) and preparation design ( $p<0.001$ ). No interactions were observed between the three fabrication systems and the two preparation designs ( $p<0.068$ ). Tukey HSD test (Table 2) exhibited a significant difference in marginal discrepancies between the OmniCam, BlueCam, and Press fabrication systems. Restorations fabricated with the Press system ( $32.87\pm28.21\text{ }\mu\text{m}$ ) presented

the smallest average marginal discrepancy compared with restorations fabricated with the other systems. Between the CAD-CAM systems, restorations fabricated with BlueCam ( $41.70\pm40.83\text{ }\mu\text{m}$ ) presented significant smaller marginal discrepancies than restorations fabricated with OmniCam ( $55.26\pm46.85\text{ }\mu\text{m}$ ). A significant difference was also observed between the two preparation designs ( $p<0.0001$ ), with preparation BX yielding smaller marginal discrepancies than preparation NB.

The results from internal fit are shown in Tables 3 and 4. Three-way ANOVA (Table 3) showed that internal discrepancy varied according to the fabrication system ( $p<0.001$ ), preparation design ( $p<0.001$ ), and location of the preparation analyzed ( $p<0.001$ ). Significant interactions were observed between variables fabrication system and preparation design ( $p<0.022$ ). The Tukey HSD test (Table 4) showed statistically significant differences between the OmniCam, BlueCam, and Press systems. The restorations fabricated with the Press ( $118.15\pm64.98\text{ }\mu\text{m}$ ) system presented significantly smaller internal discrepancies compared with the onlays fabricated with the OmniCam system ( $161.13\pm87.86\text{ }\mu\text{m}$ ), which presented smaller internal discrepancies compared with restorations fabricated with the BlueCam system ( $167.47\pm92.04\text{ }\mu\text{m}$ ). Regarding preparation design, a statistically significant difference was observed between preparations BX and NB ( $p<0.001$ ), in which preparation NB ( $139.40\pm78.71\text{ }\mu\text{m}$ ) generated restorations with smaller internal discrepancies than restorations fabricated for preparation BX ( $158.43\pm90.15\text{ }\mu\text{m}$ ). Regarding the

| Table 1: Results From Two-Way ANOVA of Marginal Fit Analysis |                   |                |              |        |        |
|--------------------------------------------------------------|-------------------|----------------|--------------|--------|--------|
| Variables of Interest                                        | Degree of Freedom | Sum of Squares | Mean Squares | F      | p      |
| Fabrication system (FS)                                      | 2                 | 244,185        | 122,092      | 79.237 | <0.001 |
| Preparation design (PD)                                      | 1                 | 31,405         | 31,405       | 20.381 | <0.001 |
| FS × PD                                                      | 2                 | 8,274          | 4,137        | 2.685  | 0.068  |

Table 2: Mean and SD of Marginal Discrepancies ( $\mu\text{m}$ ) for Each Experimental Group<sup>a</sup>

| Group           | Mean $\pm$ SD ( $\mu\text{m}$ ) | <i>p</i> |
|-----------------|---------------------------------|----------|
| Omni Cam system | 55.26 $\pm$ 46.85 A             | <0.0001  |
| Blue Cam system | 41.70 $\pm$ 40.83 B             |          |
| Press system    | 32.87 $\pm$ 28.21 C             |          |
| Preparation BX  | 39.97 $\pm$ 35.54 a             | <0.0001  |
| Preparation NB  | 46.58 $\pm$ 44.60 b             |          |

<sup>a</sup> Upper case letters indicate comparisons between systems and lower case letters indicate comparisons between preparation designs, according to Tukey HSD test ( $p < 0.05$ ).

preparation location, axial and occlusal locations have shown a statistically significant difference ( $p < 0.0001$ ). Axial location ( $140.83 \pm 82.59 \mu\text{m}$ ) presented smaller internal discrepancies compared with the occlusal location ( $197.45 \pm 85.30 \mu\text{m}$ ). Additionally, after analyzing the interactions between fabrication systems and preparation designs (Table 5), the following results were observed: restorations fabricated with the Press system for preparation NB ( $99.36 \pm 42.37 \mu\text{m}$ ) presented the smallest mean internal discrepancy of all groups; restorations fabricated with Press system for preparation BX ( $136.93 \pm 77.20 \mu\text{m}$ ) presented no significant internal discrepancy compared with restorations fabricated with Omni Cam system for preparation NB ( $156.92 \pm 91.52 \mu\text{m}$ ).

## DISCUSSION

The present study verified the marginal and internal adaptation of PCCs fabricated with the Press, CEREC BlueCam, and OmniCam systems using two preparation designs. The first null hypothesis asserting that there would be no differences in the marginal and internal adaptations between the three systems used to fabricate ceramic onlays was denied. The superior marginal fit obtained with the Press system compared with the CAD-CAM systems is in agreement with previous studies.<sup>29-31</sup> In the

Table 4: Mean and SD of Internal Discrepancies ( $\mu\text{m}$ ) for Each Experimental Group<sup>a</sup>

| Group             | Mean $\pm$ SD ( $\mu\text{m}$ ) | <i>p</i> |
|-------------------|---------------------------------|----------|
| Omni Cam system   | 161.13 $\pm$ 87.86 A            | <0.0001  |
| Blue Cam system   | 167.47 $\pm$ 92.04 A            |          |
| Press system      | 118.15 $\pm$ 64.98 B            |          |
| Preparation BX    | 158.43 $\pm$ 90.15 a            | 0.001    |
| Preparation NB    | 139.40 $\pm$ 78.71 a            |          |
| Axial location    | 140.83 $\pm$ 82.59 1            | <0.0001  |
| Occlusal location | 197.45 $\pm$ 85.30 2            |          |

<sup>a</sup> Upper case letters indicate comparisons between systems, lowercase letters indicate comparisons between preparation designs, and numbers indicate comparisons between locations according to Tukey HSD test ( $p < 0.05$ ).

present study, the mean marginal discrepancies of the ceramic onlays were 55  $\mu\text{m}$  for the CEREC OmniCam, 41  $\mu\text{m}$  for the CEREC BlueCam, and 32  $\mu\text{m}$  for the Press system. These results are consistent, although numerically smaller, with the mean marginal discrepancies reported in previous investigations, which are between 80 and 85  $\mu\text{m}$  for ceramic onlays fabricated with the CEREC BlueCam system<sup>32,33</sup> and 42 and 48  $\mu\text{m}$  for the Press system.<sup>34</sup> A different finding was reported by Guess and others,<sup>34</sup> who observed no statistically significant differences in the marginal adaptation between the Press and CAD-CAM systems. Although the present study has identified the presence of larger marginal discrepancies on the CAD-CAM restorations, the average marginal discrepancies were still within a clinically acceptable range (below 100  $\mu\text{m}$ ).<sup>35,36</sup> Furthermore, the slight difference of 10  $\mu\text{m}$  might be clinically irrelevant, and clinical studies are encouraged to evaluate the long-term performance of partial ceramic restorations. The use of a reflective powder spray to allow image capture when using the CEREC BlueCam system did not negatively affect the marginal and internal adaptation of the restorations. This finding is in agreement with da Costa and others,<sup>37</sup> who also demonstrated no

Table 3: Results From Three-Way ANOVA of Internal Fit Analysis

| Variables of Interest     | Degree of Freedom | Sum of Squares | Mean Squares | <i>F</i> | <i>p</i> |
|---------------------------|-------------------|----------------|--------------|----------|----------|
| Fabrication system (FS)   | 2                 | 382,722        | 191,361      | 30.389   | <0.001   |
| Preparation design (PD)   | 1                 | 67,186         | 67,186       | 10.670   | 0.001    |
| Location (L)              | 1                 | 494,592        | 494,592      | 78.545   | <0.001   |
| FS $\times$ PD            | 2                 | 47,715         | 23,858       | 3.7890   | 0.022    |
| FS $\times$ L             | 2                 | 24,395         | 12,198       | 1.937    | 0.144    |
| PD $\times$ L             | 1                 | 1,028          | 1,028        | 0.163    | 0.686    |
| FS $\times$ PD $\times$ L | 2                 | 6,765          | 3,382        | 0.537    | 0.584    |

Table 5: Mean and SD of Internal Discrepancies ( $\mu\text{m}$ ) for the Interaction Between Fabrication System and Preparation Design<sup>a</sup>

| Fabrication System | Preparation Design | Mean $\pm$ SD         | p     |
|--------------------|--------------------|-----------------------|-------|
| Omni Cam           | BX                 | 165.34 $\pm$ 84.05 A  | 0.022 |
|                    | NB                 | 156.92 $\pm$ 91.52 AB |       |
| Blue Cam           | BX                 | 173.01 $\pm$ 104.28 A |       |
|                    | NB                 | 161.93 $\pm$ 77.77 A  |       |
| Press              | BX                 | 136.93 $\pm$ 77.20 B  |       |
|                    | NB                 | 99.36 $\pm$ 42.37 c   |       |

<sup>a</sup> Upper case letters indicate comparisons between the combination of fabrication systems and preparation designs.

influence in the use of a reflective powder on the marginal adaptation of onlays fabricated with the CEREC BlueCam. Regarding the internal fit evaluation, a similar trend could be observed with the smallest mean internal discrepancy exhibited by the ceramic onlays fabricated with the Press system. These findings are in agreement with the results reported by Guess and others,<sup>34</sup> who observed larger internal discrepancies for ceramic onlays fabricated with the CAD-CAM system in comparison to the heat-press system. Previous studies have suggested that the ideal internal space to be occupied by the resin luting cement should be between 20 and 40  $\mu\text{m}$ <sup>38,39</sup>; however, the values reported in the scientific literature are far above this ideal range and may vary from 85 to 308  $\mu\text{m}$ .<sup>39,40</sup> The mean internal discrepancy observed in the present study varied from 99.36 to 167.47  $\mu\text{m}$  and is in a similar range with the previously reported findings.<sup>41,42</sup>

The second null hypothesis asserting that there would be no differences in the marginal and internal adaptations between the two preparation designs was denied. Although the presence of additional vertical walls in the occlusal box, as in preparation BX, might have potentially generated areas of interference, this preparation resulted in superior marginal fit of the ceramic onlays. This result may be related to the features of machinable CAD-CAM restorations, in which only two burs are used to cut and shape the internal features of the restorations and may have resulted in enlarged internal spaces due to the standardized diameter of burs.<sup>21-23</sup> The higher numerical values of internal discrepancies of preparation BX can be visualized in Tables 3 and 4 compared with preparation NB, without the box. Additionally, the features of the preparation design, in which divergent walls and internal round angles were carefully executed and refined to avoid the presence of any sharp angles and undercuts, may

have contributed to proper and easy adaptation. Schaefer and others<sup>43</sup> reported that preparations with sharp internal line angles tend to generate oversized dimensions, as opposed to preparations with round internal line angles, in which reduced marginal and internal discrepancies were observed. Regarding internal fit, the analysis of interaction showed that the presence of an occlusal box in preparation BX did not interfere with the internal adaptation of the CAD-CAM ceramic onlays; however, preparation BX resulted in significantly larger internal discrepancies for the ceramics fabricated with the Press system compared with preparation NB. Seo and others<sup>13</sup> also verified significant differences in the marginal and internal gaps of ceramic onlays depending on the preparation designs.

The third null hypothesis that stated there would be no differences between different locations of the internal space of ceramic onlays was denied. The axial locations presented the smallest average discrepancies, whereas the occlusal location showed the largest. The presence of larger internal spaces in the occlusal location is in agreement with previous studies.<sup>34,41,42,44</sup> Souza and others<sup>44</sup> reported a larger occlusal cement thickness (282  $\mu\text{m}$ ) compared with the axial (117  $\mu\text{m}$ ) for restorations fabricated with CEREC. The results from the present study exhibited an average occlusal cement thickness (197  $\mu\text{m}$ ) far below that reported by Souza and others,<sup>44</sup> whereas axial location (140  $\mu\text{m}$ ) was slightly larger than the findings of Souza and others.<sup>44</sup> Kokubo and others<sup>40</sup> ascribed the differences of internal adaptation to variations of the scanning process, preparation height, convergence angle, and variations between the CAD-CAM systems.

The methodology used in this study employed phantom master teeth that received two different preparation designs, which were subsequently used for the evaluation of the marginal and internal adaptations. The restorations were placed and stabilized on the master teeth without the use of a luting agent for bonding, allowing the possibility of further evaluations if needed. This protocol is in accordance with previous studies that used a similar procedure.<sup>45,46</sup> Regarding the internal fit, an indirect evaluation by means of the silicone replica technique was used. This protocol allowed further evaluation when needed and enabled cutting the silicone replica according to the predetermined areas to be measured.<sup>30,45</sup> Because of the nature of laboratory studies, this investigation had the chance of evaluating a large number of samples

in standardized conditions; however, clinical studies might have posed different challenges that were not simulated in the present experiment. Future longitudinal clinical studies would be relevant to evaluate the long-term performance of ceramic restorations fabricated with pressed and CAD-CAM systems.

## CONCLUSIONS

Based on the findings of this laboratory study, the following conclusions were drawn. 1) The marginal and internal adaptation of ceramic onlays were influenced by the fabrication technique. The Press system generated onlays with significantly smaller marginal and internal disadaptations compared with the BlueCam CEREC system, which presented smaller disadaptations than the OmniCam CEREC system. 2) An improved marginal adaptation was observed in the ceramic onlays with a modified occlusal box with internal rounded angles; however, the presence of an occlusal box resulted in larger internal marginal discrepancies in restorations fabricated with the Press system. 3) The occlusal location presented larger internal discrepancies than the axial locations.

## Acknowledgements

The authors thank laboratory technicians Pavel Belenkov and Hirofumi Shirakawa for excellent work during the construction of the ceramic onlays.

This study was financed in part by the Conselho Nacional de Desenvolvimento Científico e Tecnológico - Brasil (CNPq) - Finance Code 229533/2014-0 and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 28 March 2019)

## References

- Davidowitz G & Kotick PG (2011) The use of CAD-CAM in dentistry *Dental Clinics of North America* **55**(3) 559-570.
- Patel N (2014) Contemporary dental CAD-CAM: modern chairside/lab applications and the future of computerized dentistry *Compendium of Continuing Education in Dentistry* **35**(10) 739-746.
- Pollington S (2011) Novel glass-ceramics for dental restorations *Journal of Contemporary Dental Practice* **12**(1) 60-67.
- Santos MJ, Freitas MC, Azevedo LM, Santos GC Jr, Navarro MF, Francischone CE, & Mondelli RF (2016) Clinical evaluation of ceramic inlays and onlays fabricated with two systems: 12-year follow-up *Clinical Oral Investigations* **20**(7) 1683-1690.
- Contrepolis M, Soenen A, Bartala M, & Laviolle O (2013) Marginal adaptation of ceramic crowns: a systematic review *Journal of Prosthetic Dentistry* **110**(6) 447-454.
- Denry I & Kelly JR (2014) Emerging ceramic-based materials for dentistry *Journal of Dental Research* **93**(12) 1235-1242.
- Zhang Z, Chen J, Li E, Li W, Swain M, & Li Q (2016) Topological design of all-ceramic dental bridges for enhancing fracture resistance *International Journal for Numerical Methods in Biomedical Engineering* **32**(6) e02749.
- Federlin M, Krifka S, Herpich M, Hiller KA, & Schmalz G (2007) Partial ceramic crowns: influence of ceramic thickness, preparation design and luting material on fracture resistance and marginal integrity in vitro *Operative Dentistry* **32**(3) 251-260.
- Federlin M, Schmidt S, Hiller KA, Thonemann B, & Schmalz G (2004) Partial ceramic crowns: influence of preparation design on luting material on internal adaptation *Operative Dentistry* **29**(5) 560-570.
- Santos MJ, Costa MD, Rubo JH, Pegoraro LF, & Santos GC Jr (2015) Current all ceramic systems in dentistry: a review *Compendium of Continuing Education in Dentistry* **36**(1) 31-37.
- Guess PC, Schultheis S, Bonfante EA, Coelho PG, Ferencz JL, & Silva NR (2011) All-ceramic systems: laboratory and clinical performance *Dental Clinics of North America* **55**(2) 333-352.
- Homsy FR, Özcan M, Khoury M, & Majzoub ZAK (2018) Marginal and internal fit of pressed lithium disilicate inlays fabricated with milling, 3D printing, and conventional technologies *Journal of Prosthetic Dentistry* **119**(5) 783-790.
- Seo D, Yi Y, & Roh B (2009) The effect of preparation designs on the marginal and internal gaps in CEREC3 partial ceramic crowns *Journal of Dentistry* **37**(5) 374-382.
- Krifka S, Stangl M, Wiesbauer S, Hiller KA, Schmalz G, & Federlin M (2009) Influence of different cusp coverage methods for the extension of ceramic inlays on marginal integrity and enamel crack formation in vitro *Clinical Oral Investigations* **13**(3) 333-341.
- Almeida e Silva JS, Erdelt K, Edelhoff D, Araújo É, Stimmelmayer M, Vieira LC, & Güth JF (2014) Marginal and internal fit of four-unit zirconia fixed dental prostheses based on digital and conventional impression techniques *Clinical Oral Investigations* **18**(2) 515-523.
- Sjögren G, Molin M, & van Dijken JW (2004) A 10-year prospective evaluation of CAD-CAM-manufactured (Cerec) ceramic inlays cemented with a chemically cured or dual-cured resin composite *International Journal of Prosthodontics* **17**(2) 241-246.
- Arnelund CF, Johansson A, Ericson M, Häger P, & Fyrberg KA (2004) Five-year evaluation of two resin-retained ceramic systems: a retrospective study in a

- general practice setting *International Journal of Prosthodontics* **17**(3) 302-306.
18. Della Bona A, Anusavice KJ, & Hood JA (2002) Effect of ceramic location treatment on tensile bond strength to a resin cement *International Journal of Prosthodontics* **15**(3) 248-253.
  19. Beier US, Kapferer I, Burtscher D, Giesinger JM, & Dumfahrt H (2012) Clinical performance of all-ceramic inlay and onlay restorations in posterior teeth *International Journal of Prosthodontics* **25**(4) 395-402.
  20. Rekow ED, Harsono M, Janal M, Thompson VP, & Zhang G (2006) Factorial analysis of variables influencing stress in all-ceramic crowns *Dental Materials* **22**(2) 125-132.
  21. Yildiz C, Vanliglu BA, Evren B, Uludamar A, & Kulak Ozkan Y (2013) Fracture resistance of manually and CAD/CAM manufactured ceramic onlays *Journal of Prosthodontics* **22**(7) 537-542.
  22. Reich S, Wichmann M, Nkenke E, & Proeschel P (2005) Clinical fit of all ceramic three unit fixed partial dentures, generated with three different CAD/CAM systems *European Journal of Oral Sciences* **113**(2) 174-179.
  23. Renne W, McGill ST, Forshee KV, DeFee MR, & Mennito AS (2012) Predicting marginal fit of CAD/CAM crowns based on the presence or absence of common preparation errors *Journal of Prosthetic Dentistry* **108**(5) 310-315.
  24. Bindl A & Mörmann WH (2003) Clinical and SEM evaluation of allceramic chair-side CAD-CAM-generated partial crowns *European Journal of Oral Sciences* **111**(2) 163-169.
  25. Akbar JH, Petrie CS, Walker MP, Williams K, & Erick JD (2006) Marginal adaptation of CEREC 3 CAD-CAM composite crowns using two different finish line preparation designs *Journal of Prosthodontics* **15**(3) 155-163.
  26. Nakamura T, Dei N, Kojima T, & Wakabayashi K (2003) Marginal and internal fit of CEREC 3 CAD-CAM all-ceramic crowns *International Journal of Prosthodontics* **16**(3) 244-248.
  27. Mou SH, Chai T, Wang JS, & Shiao YY (2002) Influence of different convergence angles and tooth preparation heights on the internal adaptation of CEREC crowns *Journal of Prosthetic Dentistry* **87**(3) 248-255.
  28. Morimoto S, Albanesi RB, Sesma N, Agra CM, & Braga MM (2016) Main clinical outcomes of feldspathic porcelain and glass-ceramic laminate veneers: a systematic review and meta-analysis of survival and complication rates *International Journal of Prosthodontics* **29**(1) 38-49.
  29. Reich S, Gozdowski S, Trentzsch L, Frankenberger R, Lohbauer U (2008) Marginal fit of heat-pressed vs. CAD/CAM processed all-ceramic onlays using a milling unit prototype *Operative Dentistry* **33**(6) 644-650.
  30. Vanliglu BA, Evren B, Yildiz C, Uludamar A, & Ozkan YK (2012) Internal and marginal adaptation of pressable and computer-aided design/computer-assisted manufacture onlay restorations *International Journal of Prosthodontics* **25**(3) 262-264.
  31. Mounajjed R, M Layton D, & Azar B (2016) The marginal fit of E.max Press and E.max CAD lithium disilicate restorations: a critical review *Dental Materials Journal* **35**(6) 835-844.
  32. Denissen H, Dozic A, van der Zel J, & van Waas M (2000) Marginal fit and short-term clinical performance of porcelain-veneered CEREC, CEREC, and Procera onlays *Journal of Prosthetic Dentistry* **84**(5) 506-513.
  33. Stappert CF, Chitmongkolsuk S, Silva NR, Att W, & Strub JR (2008) Effect of mouth-motion fatigue and thermal cycling on the marginal accuracy of partial coverage restorations made of various dental materials *Dental Materials* **24**(9) 1248-1257.
  34. Guess P, Vagkopoulou T, Zhang Y, Wolkewitz M, & Strub J (2014) Marginal and internal fit of heat pressed versus CAD-CAM fabricated all-ceramic onlays after exposure to thermo-mechanical fatigue *Journal of Dentistry* **42**(2) 199-209.
  35. Bindl A & Mörmann WH (2005) Marginal and internal fit of all-ceramic CAD-CAM crown-copings on chamfer preparations *Journal of Oral Rehabilitation* **32**(6) 441-447.
  36. Att W, Komine F, Gerds T, & Strub JR (2009) Marginal adaptation of three different zirconium dioxide three-unit fixed dental prostheses *Journal of Prosthetic Dentistry* **101**(4) 239-247.
  37. da Costa JB, Pelogia F, Hagedorn B, & Ferracane JL (2010) Evaluation of different methods of optical impression making on the marginal gap of onlays created with CEREC 3D *Operative Dentistry* **35**(3) 324-329.
  38. Martins LM, Lorenzoni FC, Melo AO, Silva LM, Oliveira PC, & Bonfante G (2012) Internal fit of two all-ceramic systems and metal-ceramic crowns *Journal of Applied Oral Science* **20**(2) 235-240.
  39. Krejci I, Lutz F, & Reimer M (1993) Marginal adaptation and fit of adhesive ceramic inlays *Journal of Dentistry* **21**(1) 39-46.
  40. Kokubo Y, Ohkubo C, Tsumita M, Miyashita A, Vult von Steyern P, & Fukushima S (2005) Clinical marginal and internal gaps of Procera AllCeram crowns *Journal of Oral Rehabilitation* **32**(7) 526-530.
  41. Bayramoglu E, Ozkan Y, & Yildiz C (2015) Comparison of marginal and internal fit of press-on-metal and conventional ceramic systems for three- and four-unit implant-supported partial fixed dental prostheses: an in vitro study *Journal of Prosthetic Dentistry* **114**(1) 52-58.
  42. Nam SJ, Yoon MJ, Kim WH, Ryu GJ, Bang MK, & Huh JB (2015) Marginal and internal fit of conventional metal-ceramic and lithium disilicate CAD/CAM crowns *Journal of Prosthodontics* **28**(5) 519-521.
  43. Schaefer O, Watts DC, Sigusch BW, Kuepper H, & Guentsch A (2012) Marginal and internal fit of pressed lithium disilicate partial crowns in vitro: a three-dimensional analysis of accuracy and reproducibility *Dental Materials* **28**(3) 320-326.
  44. Souza ROA, Özcan M, Pavanelli CA, Buso L, Lombardo GHL, Michida SMA, Mesquita AM, & Bottino MA (2012) Marginal and internal discrepancies related to margin design of ceramic crowns fabricated by a CAD-CAM system *Journal of Prosthodontics* **21**(2) 94-100.

45. Bottino M, Campos F, Ramos N, Rippe M, Valandro L, & Melo R (2015) Inlays made from a hybrid material: adaptation and bond strengths *Operative Dentistry* **40**(3) 83-91.
46. Hamza TA, Ezzat HA, El-Hossary MM, Katamish HA, Shokry TE, & Rosenstiel SF (2013) Accuracy of ceramic restorations made with two CAD-CAM systems *Journal of Prosthetic Dentistry* **109**(2) 83-87.

# Dynamic Viscoelastic Characterization of Bulk-fill Resin-based Composites and Their Conventional Counterparts

AU Yap • AH Eweis • NA Yahya

## Clinical Relevance

The viscoelastic properties of bulk-fill resin-based composites (RBCs) were affected by aqueous solutions and are often inferior to their conventional counterparts. The clinical use of conventional RBCs are generally preferred over bulk-fill materials for high-stress-bearing situations.

## SUMMARY

**This study compared the viscoelastic properties of restorative and flowable bulk-fill resin-based composites (RBCs) with their conventional counterparts and evaluated the impact of aqueous solutions on viscoelastic properties. The materials examined included three conventional RBCs (Filtek Z350, Tetric N Ceram, and Beautifil II), three restorative bulk-**

**fill RBCs (Filtek Bulk-Fill Restorative, Tetric N Ceram Bulk-Fill, and Beautifil Bulk-Fill Restorative) in addition to three flowable bulk-fill RBCs (Filtek Bulk-Fill Flowable, Tetric N Flow Bulk-Fill, and Beautifil Bulk-Fill Flowable). Beam-shaped specimens (12×2×2 mm) were fabricated using customized stainless-steel molds, finished, and measured. The specimens were randomly divided into four groups and conditioned in air (control), artificial saliva, 0.02 N citric acid, and 50% ethanol-water solution for seven days at 37°C. They were then subjected to dynamic mechanical analysis (n = 10) in flexure mode at 37°C with a frequency of 0.1 to 10 Hz. Storage modulus, loss modulus, and loss tangent data were subjected to statistical analysis using one-way analysis of variance/Tukey *post hoc* test at a significance level of  $\alpha = 0.05$ . Viscoelastic properties of the RBCs were found to be product and conditioning medium dependent. For most RBCs, exposure to aqueous solutions, particularly an ethanol-water solution, degraded viscoelastic properties. With the exception of Filtek Bulk-Fill Restorative, bulk-fill restor-**

Adrian U Yap, PhD, MSc, BDS, adjunct professor, Grad Dip Psychotherapy, Restorative Dentistry, Faculty of Dentistry, University of Malaya, Kuala Lumpur, Malaysia and Department of Dentistry, Ng Teng Fong General Hospital, Singapore

Ahmed H Eweis, MDSc, MFDS RCSEd, BDS, Restorative Dentistry, Faculty of Dentistry, University of Malaya, Kuala Lumpur, Malaysia

\*Noor A Yahya, BDS (Otago), MDentSci (Leeds), DipTrans, Restorative Dentistry, Faculty of Dentistry, University of Malaya, Kuala Lumpur, Malaysia

\*Corresponding author: Restorative Dentistry, Faculty of Dentistry, University of Malaya, Kuala Lumpur, Wilayah Persekutuan 50603, Malaysia; e-mail: nazlin@um.edu.my

DOI: <https://doi.org/10.2341/19-005-L>

**active and flowable RBCs generally had significantly lower storage and loss modulus than their conventional counterparts regardless of conditioning medium. Conventional RBCs are thus favored over their bulk-fill counterparts, particularly for high-stress-bearing areas.**

## INTRODUCTION

Resin-based composites (RBCs) are one of the most widely used materials in contemporary dental practice. They are utilized as direct fillings for all cavities classes as well as indirect restorations, core buildups, and luting cements. Due to depth of cure and polymerization shrinkage issues, conventional RBCs have to be placed in 2-mm increments using a variety of layering techniques for direct restorations.<sup>1</sup> However, these incremental techniques are rather time consuming and technically challenging and may result in voids or bond failure between layers.<sup>2</sup> Bulk-fill RBCs were developed to address the complexities posed by incremental techniques and to streamline restorative processes.<sup>3</sup> These bulk-fill RBCs are said to cure in 4- to 5-mm increments without intensifying polymerization shrinkage stress.<sup>4</sup> Methods employed by manufacturers to create bulk-fill RBCs include reduction of filler content, modification of filler type to increase light transmission, and use of more effective initiators and monomer systems that allow for stress relief during polymerization.<sup>3,4</sup> Based on viscosity, bulk-fill materials can be divided into restorative (high-viscosity) or flowable (low-viscosity) RBCs. Flowable RBCs with their lower filler content are indicated primarily as bases/liners. They generally have lower wear resistance and mechanical properties and require a 2-mm “cap” of conventional hybrid RBCs when used in occlusal areas.<sup>3,5</sup> In contrast, bulk-fill restorative materials are designed to withstand occlusal loading and may not require a “capping” layer of conventional materials.

As bulk-fill RBCs are indicated for restoring large and deep posterior cavities, it is imperative to consider their mechanical properties. To increase curing depths, many commercial bulk-fill products employ the use of larger filler particles to reduce filler content, leading to higher filler weights but lower filler volumes.<sup>6</sup> Light penetration is thought to be enhanced by decreasing the difference in refractive index between the fillers and matrix as well as the quantity of filler-matrix interfaces.<sup>7</sup> Notwithstanding the potential reduction in filler content, the mechanical properties of bulk-fill restoratives remain contentious with studies reporting both lower

and similar mechanical properties when compared to conventional RBCs.<sup>6-10</sup> The variance in findings may be attributed to differences in polymerization initiators, resin composition, filler type, size, and content, all of which influence material viscosity. In addition, food-simulating liquids and aqueous solutions have also been shown to degrade the mechanical properties of RBCs.<sup>9,11</sup>

Although RBCs are viscoelastic in nature, they are usually investigated using destructive static flexure, compression, and tension tests that examine only the elastic element of the materials and provide “single-event” values for strength.<sup>9-12</sup> Dynamic mechanical analysis (DMA) allows for nondestructive characterization of both viscous and elastic components of materials and is particularly useful for evaluating RBCs.<sup>13</sup> DMA is also capable of simulating a wide range of cyclic masticatory and thermal stresses encountered intraorally.<sup>14</sup> The technique involves the application of small oscillatory/sinusoidal stress to materials and measuring the resultant strain response. The stress applied can be divided into viscous and elastic stress, depending on whether it is out of phase or in phase with strain, respectively. Good correlations were observed between static and dynamic testing of RBCs even with smaller flexural specimens.<sup>15,16</sup>

The objectives of this study were to compare the viscoelastic properties of restorative and flowable RBCs with their conventional counterparts and to determine the impact of aqueous solutions on viscoelastic properties. Correlations between the various viscoelastic properties were also performed. The null hypotheses were as follows: 1) viscoelastic properties of restorative/flowable bulk-fill RBCs are no different from their conventional counterparts, 2) aqueous solutions do not influence the viscoelasticity of bulk-fill restorative/flowable and conventional RBCs, and 3) no correlations exist between the various viscoelastic properties of RBCs.

## METHODS AND MATERIALS

### Specimen Preparation and Conditioning

The materials and specimen preparation had been described in our earlier work.<sup>10</sup> RBCs evaluated were comprised of three conventional RBCs (Filtek Z350 [FZ], Tetric N Ceram [TN], and Beautifil II [BT]), three bulk-fill restorative RBCs (Filtek Bulk-Fill Restorative [FB], Tetric N Ceram Bulk-Fill [TB], and Beautifil Bulk-Fill Restorative [BB]) along with three bulk-fill flowable RBCs (Filtek Bulk-Fill Flowable [FF], Tetric N Flow Bulk-Fill [TF], and

Table 1: Technical Profiles and Manufacturers of the Materials Evaluated

| Manufacturer                                            | Material (Abbreviation)           | Type                  | Resin (Photoinitiator)                      | Filler                                                                         | Filler Content % by Weight/<br>% by Volume | Lot No. |
|---------------------------------------------------------|-----------------------------------|-----------------------|---------------------------------------------|--------------------------------------------------------------------------------|--------------------------------------------|---------|
| 3M ESPE (St Paul, MN, USA), manufacturer A              | Filtek Z350 (FZ)                  | Conventional          | Bis-GMA, Bis-EMA, UDMA<br>TEGDMA (CQ)       | Zirconia/silica cluster and silica nanoparticle                                | 78.5/63.3                                  | N771467 |
|                                                         | Filtek Bulk-Fill Restorative (FB) | Bulk-fill restorative | AUDMA, AFM DDDMAA<br>UDMA (CQ)              | Zirconia/silica cluster, ytterbium trifluoride                                 | 76.5/58.4                                  | N693019 |
|                                                         | Filtek Bulk-Fill Flowable (FF)    | Bulk-fill flowable    | Bis-GMA Bis-EMA UDMA<br>(CQ, EDMAB)         | Zirconia/silica, ytterbium trifluoride                                         | 64.5/42.5                                  | N884479 |
| Ivoclar Vivadent Inc (Amherst, NY, USA), manufacturer B | Tetric N Ceram (TN)               | Conventional          | Bis-GMA Bis-EMA UDMA<br>(CQ)                | Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide, prepolymers | 81.2/57                                    | V35260  |
|                                                         | Tetric N Ceram Bulk-Fill (TB)     | Bulk-fill restorative | Bis-GMA Bis-EMA UDMA<br>(CQ, TPO, Ivocerin) | Barium glass filler, ytterbium fluoride and spherical mixed oxide              | 79/60                                      | S38368  |
|                                                         | Tetric N Flow Bulk-Fill (TF)      | Bulk-fill flowable    | Bis-GMA UDMA TEGDMA<br>(CQ, TPO, Ivocerin)  | Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide              | 64.9/NA                                    | V49336  |
| Shofu Inc (Kyoto, Japan), manufacturer C                | Beautifil II (BT)                 | Conventional          | Bis-GMA TEGDMA (CQ)                         | S-PRG based on F-Br-Al-Si glass                                                | 83.3/68.8                                  | 031731  |
|                                                         | Beautifil-Bulk Restorative (BB)   | Bulk-fill restorative | Bis-GMA UDMA Bis-MPEPP TEGDMA (CQ)          | S-PRG based on F-Br-Al-Si glass                                                | 87/74.5                                    | 051623  |
|                                                         | Beautifil-Bulk Flowable (BF)      | Bulk-fill flowable    | Bis-GMA UDMA Bis-MPEPP TEGDMA (CQ)          | S-PRG based on F-Br-Al-Si glass                                                | 73/60                                      | 101615  |

Abbreviations: Bis-GMA, bisphenol-A glycidyl methacrylate; Bis-EMA, ethoxylated bisphenol-A-glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; CQ, camphorquinone; AUDMA, aromatic urethane dimethacrylate; AFM, addition-fragmentation monomers; DDDMA, 1,12-dodecanediol dimethacrylate; EDMAB, ethyl 4-dimethyl aminobenzoate; TPO, 2,4,6-trimethylbenzoyl diphenylphosphine oxide; NA, not available; S-PRG, surface modified prereacted glass; F-Br-Al-Si, fluoroboroaluminosilicate; Bis-MPEPP, bisphenol-A polyethoxy-dimethacrylate.

Beautifil Bulk-Fill Flowable [BF]). The materials and their technical profiles are specified in Table 1. Briefly, 40 beam-shaped specimens (12×2×2 mm) of the different RBCs were fabricated with customized stainless-steel molds. The RBCs were light polymerized using an LED curing light (Demi Plus, Kerr Corp, Orange, CA, USA) with an output irradiance of 1330 mW/cm<sup>2</sup> in two overlapping irradiations of 10 seconds. Specimens were then finished using fine polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA), inspected for defects/parallelism, and measured with a digital caliper (Mitutoyo Corp, Kawasaki, Japan). The RBCs were then randomly divided into four groups (n=10) and stored in sealed containers with the following conditioning mediums for seven days at 37°C in an incubator (IN-460, Memmert, Schwabach, Germany): air, artificial saliva (SAGF),<sup>17</sup> 0.02 N citric acid, and 50% ethanol-water solution. Air served as the control medium, while artificial saliva replicated natural saliva. Citric acid and 50% ethanol mimicked certain fruits, candies, and beverages, including alcohol. The pH of the artificial saliva (6.8) and citric acid (2.6) was verified via a digital pH meter (pH2700, Eutech, Singapore).

### Dynamic Mechanical Analysis

Following conditioning, the RBCs were subjected to dynamic flexural testing (DMA RSAG2, TA Instruments, New Castle, DE, USA) in the various conditioning mediums at 37°C. Details of the testing protocol have been described previously.<sup>16</sup> Specimens were loaded using a three-point bending fixture with a 10-mm support distance and loading frequency of 0.1 to 10 Hz to simulate the range from almost static to the upper limit of chewing rates.<sup>18</sup> However, the upper limit is extremely fast and exceeds the range expected for normal chewing. Loading frequency was progressively increased from 0.1 to 10 Hz in 15 steps with a delay time of one second and a delay cycle of 0.5 Hz. Storage modulus, loss modulus, and loss tangent data were subsequently calculated as follows:<sup>16,19</sup>

Storage modulus:

$$E' = (\sigma^\circ/\epsilon^\circ)\cos\delta = (f_o/bk)\cos\delta$$

Loss modulus:

$$E'' = (\sigma^\circ/\epsilon^\circ)\sin\delta = (f_o/bk)\sin\delta$$

Table 2: Mean Storage Modulus ( $E'$  [GPa]), Loss Modulus ( $E''$  [GPa]), and Loss Tangent ( $\times 10^{-3}$ ) Values for the Various Restorative Materials With Standard Deviations (in Parentheses)

| Medium/Material                   | Storage Modulus (GPa) |                   |             |             | Loss Modulus (GPa) |                   |
|-----------------------------------|-----------------------|-------------------|-------------|-------------|--------------------|-------------------|
|                                   | Air                   | Artificial Saliva | Citric Acid | Ethanol     | Air                | Artificial Saliva |
| Filtek Z350 (FZ)                  | 6.29 (0.29)           | 5.48 (0.56)       | 5.19 (0.42) | 4.49 (0.48) | 0.42 (0.04)        | 0.47 (0.06)       |
| Filtek Bulk-Fill Restorative (FB) | 6.20 (0.72)           | 6.10 (0.49)       | 5.86 (0.50) | 6.04 (0.37) | 0.43 (0.06)        | 0.46 (0.06)       |
| Filtek Bulk-Fill Flowable (FF)    | 4.05 (0.35)           | 4.03 (0.41)       | 3.99 (0.18) | 2.47 (0.21) | 0.38 (0.05)        | 0.41 (0.05)       |
| Tetric N Ceram (TN)               | 6.18 (0.57)           | 5.56 (0.53)       | 5.47 (0.50) | 4.45 (0.49) | 0.58 (0.08)        | 0.59 (0.08)       |
| Tetric N Ceram Bulk-Fill (TB)     | 5.00 (0.37)           | 3.76 (0.40)       | 3.72 (0.48) | 3.68 (0.18) | 0.38 (0.04)        | 0.36 (0.05)       |
| Tetric N Flow Bulk-Fill (TF)      | 5.20 (0.38)           | 4.60 (0.35)       | 4.57 (0.64) | 4.53 (0.53) | 0.45 (0.04)        | 0.44 (0.06)       |
| Beautifil II (BT)                 | 7.02 (0.58)           | 6.63 (0.81)       | 6.02 (0.35) | 5.78 (0.68) | 0.46 (0.07)        | 0.51 (0.08)       |
| Beautifil-Bulk Restorative (BB)   | 5.79 (0.36)           | 5.51 (0.53)       | 5.04 (0.56) | 4.61 (0.47) | 0.38 (0.05)        | 0.43 (0.06)       |
| Beautifil-Bulk Flowable (BF)      | 6.17 (0.55)           | 5.81 (0.57)       | 5.17 (0.65) | 5.14 (0.54) | 0.41 (0.05)        | 0.39 (0.06)       |

Loss tangent:

$$\tan \delta = E''/E'$$

where  $\sigma^\circ$  is the maximum stress at the peak of the sine wave,  $\epsilon^\circ$  is the strain at the maximum stress,  $f_o$  is the force applied at the peak of the sine wave,  $b$  is the sample geometry term, and  $k$  is the sample displacement at the peak.

Sample geometry  $b$  for a three-point bending bar was calculated as follows:

$$4BH^3/L^3$$

where  $B$  is the width of the specimen (in mm),  $H$  is the height of the specimen (in mm), and  $L$  is the distance between the supports (in mm).

### Statistical Analysis

Data were analyzed using the SPSS statistical program (version 12.0.1, SPSS Inc, Chicago, IL, USA). Parametric analysis was performed, as data were normally distributed based on the Shapiro-Wilk test. Two-way analysis of variance (ANOVA) was used to determine interactions between independent variables (materials and conditioning mediums) and dependent variables (storage modulus, loss modulus, and loss tangent). One way ANOVA followed by the *post hoc* Tukey test were used to assess intermedium and intermaterial differences. The latter was grouped by both manufacturer and material type (ie, conventional, bulk-fill restorative, and bulk-fill flowable). Correlation between viscoelastic properties was carried out with the Pearson correlation. All statistical analyses were performed at a significance level of  $\alpha = 0.05$ .

### RESULTS

Mean storage modulus, loss modulus, and loss tangent for the RBCs after conditioning in the various aqueous solutions are shown in Table 2. Intermedium comparisons are shown in Table 3, while intermaterial evaluations are presented in Table 4 (grouped by manufacturer) and Table 5 (grouped by material type). Correlations between the various viscoelastic properties are displayed in Table 6. Mean storage modulus, loss modulus, and loss tangent ranged from 2.47 to 7.02 GPa, 0.33 to 0.64 GPa, and  $66$  to  $143 \times 10^{-3}$ , respectively, for the various material-medium combinations. Viscoelastic properties of the RBCs were found to be material and conditioning medium dependent.

With the exception of FB and FF, storage in air (control) frequently resulted in a significantly higher storage modulus than in aqueous solutions (Table 3). Exposure to the ethanol-water solution usually presented the lowest storage modulus. For the majority of RBCs, conditioning in the ethanol-water solution also resulted in a significantly higher loss modulus and loss tangent. Moreover, the lowest loss tangent was typically observed with the control group (air).

For manufacturer A, FB had a significantly greater storage modulus than FZ, which in turn was stiffer than FF in all mediums except air (Table 4). While no obvious trends were noted for loss modulus, FF had a significantly greater loss tangent than FB and FZ regardless of conditioning medium. For manufacturer B, TN had a significantly greater storage modulus, loss modulus, and loss tangent than TF and TB with the exception of storage in the ethanol-water solution. For manufacturer C, BT showed a significantly higher storage and loss

Table 2: Mean Storage Modulus ( $E'$  [GPa]), Loss Modulus ( $E''$  [GPa]), and Loss Tangent ( $\times 10^{-3}$ ) Values for the Various Restorative Materials With Standard Deviations (in Parentheses) (ext.)

| Medium/Material                   | Loss Modulus (GPa) |             | Loss Tangent ( $\times 10^{-3}$ ) |                   |             |          |
|-----------------------------------|--------------------|-------------|-----------------------------------|-------------------|-------------|----------|
|                                   | Citric Acid        | Ethanol     | Air                               | Artificial Saliva | Citric Acid | Ethanol  |
| Filtek Z350 (FZ)                  | 0.48 (0.06)        | 0.42 (0.05) | 68 (5)                            | 88 (9)            | 91 (10)     | 97 (3)   |
| Filtek Bulk-Fill Restorative (FB) | 0.51 (0.06)        | 0.64 (0.06) | 69 (4)                            | 84 (10)           | 85 (5)      | 106 (5)  |
| Filtek Bulk-Fill Flowable (FF)    | 0.43 (0.04)        | 0.35 (0.02) | 95 (6)                            | 102 (5)           | 107 (7)     | 143 (11) |
| Tetric N Ceram (TN)               | 0.59 (0.07)        | 0.61 (0.08) | 95 (6)                            | 108 (8)           | 109 (5)     | 138 (7)  |
| Tetric N Ceram Bulk-Fill (TB)     | 0.33 (0.04)        | 0.44 (0.05) | 76 (5)                            | 95 (7)            | 94 (10)     | 135 (9)  |
| Tetric N Flow Bulk-Fill (TF)      | 0.41 (0.06)        | 0.55 (0.08) | 88 (5)                            | 97 (10)           | 92 (6)      | 121 (6)  |
| Beautifil II (BT)                 | 0.51 (0.08)        | 0.55 (0.07) | 66 (6)                            | 78 (6)            | 86 (10)     | 96 (5)   |
| Beautifil-Bulk Restorative (BB)   | 0.48 (0.07)        | 0.42 (0.06) | 66 (7)                            | 73 (5)            | 101 (6)     | 93 (3)   |
| Beautifil-Bulk Flowable (BF)      | 0.36 (0.05)        | 0.43 (0.04) | 67 (5)                            | 68 (3)            | 70 (3)      | 85 (4)   |

modulus than BF and BB apart from conditioning in citric acid. However, trends for loss tangent were mixed.

When conventional RBCs were compared (Table 5), BT had a significantly higher storage modulus than FZ and TN regardless of conditioning mediums. The loss modulus and tangent of TN was mostly significantly greater than the other two conventional RBCs. For bulk-fill restoratives, FB and BB had a significantly higher storage modulus than TB for all four mediums. No distinct trends

were noted for loss modulus and tangent. For the flowable RBCs, BF generally had a significantly higher storage modulus than TF and FF. While no obvious patterns were observed for loss modulus, FF and TF had a significantly larger loss tangent than BF.

A significant and moderate correlation was observed between storage and loss modulus ( $r=0.496$ ) as well as storage modulus and loss tangent ( $-0.560$ ) (Table 6). The correlation between loss modulus and loss tangent, though significant, was weak (0.356).

Table 3: Results of Statistical Analysis for Mean Values of Storage Modulus ( $E'$ ), Loss Modulus ( $E''$ ), and Loss Tangent ( $Tan\delta$ ) Based on Materials<sup>a</sup>

| Manufacturer | Material | Differences        |                    |                    |
|--------------|----------|--------------------|--------------------|--------------------|
|              |          | Storage Modulus    | Loss Modulus       | Loss Tangent       |
| A            | FZ       | A > S, C > E       | Nonsignificant     | E > S > A<br>C > A |
|              | FB       | Nonsignificant     | E > C > A<br>E > S | E > C, S > A       |
|              | FF       | A, S, C > E        | C, S > E           | E > C > A<br>E > S |
|              | TN       | A > C > E<br>S > E | Nonsignificant     | E > C, S > A       |
| B            | TB       | A > S, C, E        | E > A, S, C        | E > S, C > A       |
|              | TF       | A > S, C, E        | E > A, S, C        | E > S > A<br>E > C |
|              | BT       | A > C, E<br>S > E  | E > A              | E > C, S > A       |
| C            | BB       | A > C, E<br>S > E  | C > A              | C > E > S > A      |
|              | BF       | A > C, E           | E > C              | E > C, S, A        |

Abbreviations: FZ, Filtek Z350; A, air; S, saliva; C, citric acid; E, ethanol; FB, Filtek Bulk-Fill Restorative; FF, Filtek Bulk-Fill Flowable; TN, Tetric N Ceram; TB, Tetric N Ceram Bulk-Fill; TF, Tetric N Flow Bulk-Fill; BT, Beautifil II; BB, Beautifil-Bulk Restorative; BF, Beautifil-Bulk Flowable.

<sup>a</sup> Results of one-way analysis of variance and post hoc Tukey test; > indicates statistical significance ( $p < 0.05$ ).

Table 4: Results of Statistical Analysis for Mean Values of Storage Modulus ( $E'$ ), Loss Modulus ( $E''$ ), and Loss Tangent ( $\tan\delta$ ) Grouped According to Manufacturers and Based on Conditioning Mediums<sup>a</sup>

| Properties      | Manufacturer/Medium | Differences    |                |                |
|-----------------|---------------------|----------------|----------------|----------------|
|                 |                     | Manufacturer A | Manufacturer B | Manufacturer C |
| Storage modulus | Air                 | FZ, FB > FF    | TN > TF, TB    | BT > BF, BB    |
|                 | Artificial saliva   | FB > FZ > FF   | TN > TF > TB   | BT > BF, BB    |
|                 | Citric acid         | FB > FZ > FF   | TN > TF > TB   | BT > BF, BB    |
|                 | Ethanol 50%         | FB > FZ > FF   | TF, TN > TB    | BT > BF, BB    |
| Loss modulus    | Air                 | Nonsignificant | TN > TF > TB   | BT > BB        |
|                 | Artificial saliva   | Nonsignificant | TN > TF > TB   | BT > BB, BF    |
|                 | Citric acid         | FB > FF        | TN > TF > TB   | BT, BB > BF    |
|                 | Ethanol 50%         | FB > FZ > FF   | TN, TF > TB    | BT > BF, BB    |
| Loss tangent    | Air                 | FF > FB, FZ    | TN > TF > TB   | Nonsignificant |
|                 | Artificial saliva   | FF > FZ, FB    | TN > TF, TB    | BT > BF        |
|                 | Citric acid         | FF > FZ, FB    | TN > TB, TF    | BB > BT > BF   |
|                 | Ethanol 50%         | FF > FB > FZ   | TN, TB > TF    | BT, BB > BF    |

Abbreviations: FZ, Filtek Z350; FB, Filtek Bulk-Fill Restorative; FF, Filtek Bulk-Fill Flowable; TN, Tetric N Ceram; TF, Tetric N Flow Bulk-Fill; TB, Tetric N Ceram Bulk-Fill; BT, Beautifil II; BF, Beautifil-Bulk Flowable; BB, Beautifil-Bulk Restorative.  
<sup>a</sup> Results of one-way analysis of variance and post hoc Tukey test; > indicates statistical significance ( $p < 0.05$ ).

DISCUSSION

This study compared the viscoelastic properties of restorative and flowables RBCs with their conventional counterparts and determined the impact of aqueous solutions on viscoelastic properties. As the viscoelastic properties of the RBCs were found to be material and conditioning medium dependent and significant correlations were observed between viscoelastic properties, all three null hypotheses were duly rejected. The “miniflexural” specimens employed for this study were approximately half the length recommended by the International Organization for Standardization (ISO) (25×2×2 mm).<sup>16</sup> Besides being more efficient to execute and clinically relevant, these miniflexural specimens also correlated well with the ISO flexural tests for both dynamic and static assessments.<sup>16</sup> DMA provided information on storage modulus, loss modulus, and loss tangent. The stiffness/rigidity and flow of the RBCs are characterized by storage and loss modulus, respectively.<sup>14,20</sup> For use in posterior teeth, RBCs with a high storage modulus and low loss modulus are advantageous to resist deformation from occlusal forces. Although a greater flow could possibly decrease or delay wear and fracture, loss modulus values were about 10 times smaller than storage modulus values and may not affect clinical performance greatly. Loss tangent is the ratio of loss modulus to storage modulus.<sup>20</sup> It represents the energy-dissipating capacity of the RBCs, with lower values indicating faster response to load and return to original form.<sup>13</sup>

Comparison Between Conditioning Mediums

In the present study, exposure to aqueous solutions degraded the viscoelastic properties of the RBCs with the exception of FB and FF. Findings corroborated previous studies pertaining to the effect of conditioning mediums/food-simulating liquids on viscoelastic properties of RBCs.<sup>21,22</sup> The lowest storage modulus and highest loss tangent were observed after conditioning in ethanol. This could be attributed to the high propensity of the RBC polymers to absorb ethanol-water solutions, resulting in matrix swelling and decreased mechanical properties.<sup>23,24</sup> The better performance of FB and FF may be attributed partly to the introduction of methacrylates that undergo free-radical addition fragmentation. This might enhance postcuring structural stability in addition to reducing polymerization stress.<sup>25</sup> For manufacturer C, no significant difference in storage modulus was observed between conditioning in citric acid and ethanol for all material types. All the products of manufacturer C were giomers based on prereacted glass ionomer (PRG) technology, where fluoride-containing glass is reacted with polyacid acids, freeze-dried, milled, silanized, and used as fillers.<sup>2</sup> Giomers have been found to be particularly prone to degradation by acids of low pH due to their glass ionomer constituent.<sup>26</sup>

Comparison Between Material Types

When material types were compared, bulk-fill restorative and flowable RBCs generally had a signifi-

Table 5: Results of Statistical Analysis for Mean Values of Storage Modulus ( $E'$ ), Loss Modulus ( $E''$ ), and Loss Tangent ( $\tan\delta$ ) Grouped According to Material Type and Based on Conditioning Mediums<sup>a</sup>

| Properties      | Material Type/Medium | Differences  |                       |                    |
|-----------------|----------------------|--------------|-----------------------|--------------------|
|                 |                      | Conventional | Bulk-Fill Restorative | Bulk-Fill Flowable |
| Storage modulus | Air                  | BT > FZ, TN  | FB, BB > TB           | BF > TF > FF       |
|                 | Artificial saliva    | BT > TN, FZ  | FB > BB > TB          | BF > TF > FF       |
|                 | Citric acid          | BT > TN, FZ  | FB > BB > TB          | BF > FF            |
|                 | Ethanol 50%          | BT > FZ, TN  | FB > BB > TB          | BF > TF > FF       |
| Loss modulus    | Air                  | TN > BT, FZ  | Nonsignificant        | TF > FF            |
|                 | Artificial saliva    | TN > FZ      | FB, BB > TB           | Nonsignificant     |
|                 | Citric acid          | TN > FZ      | FB, BB > TB           | FF, TF > BF        |
|                 | Ethanol 50%          | TN, BT > FZ  | FB > TB, BB           | TF > BF > FF       |
| Loss tangent    | Air                  | TN > FZ, BT  | TB > FB, BB           | FF > TF > BF       |
|                 | Artificial saliva    | TN > FZ > BT | TB > FB > BB          | FF, TF > BF        |
|                 | Citric acid          | TN > FZ, BT  | BB, TB > FB           | FF > TF > BF       |
|                 | Ethanol 50%          | TN > FZ, BT  | TB > FB > BB          | FF > TF > BF       |

Abbreviations: BT, Beautifil II; FZ, Filtek Z350; TN, Tetric N Ceram; FB, Filtek Bulk-Fill Restorative; BB, Beautifil-Bulk Restorative; TB, Tetric N Ceram Bulk-Fill; BF, Beautifil-Bulk Flowable; TF, Tetric N Flow Bulk-Fill; FF, Filtek Bulk-Fill Flowable.

<sup>a</sup> Results of one-way analysis of variance and post hoc Tukey test; > indicates statistical significance ( $p < 0.05$ ).

icantly lower storage modulus than their conventional counterparts with the exception of FB. FB was significantly more rigid than both conventional (FZ) and bulk-fill flowable (FF) materials from manufacturer A. As the filler content by volume of FB was lower than FZ, improvements in mechanical properties can be attributed more to resin modifications. The bulk-fill restorative and flowable RBCs of manufacturers B and C were significantly less rigid than their conventional equivalents. Accordingly, their conventional RBCs should be used preferentially over bulk-fill materials, especially in high-stress-bearing areas. The storage modulus of the bulk-fill restoratives TB and BB were similar or even lower than their flowable equivalents TF and BF. This was unexpected, as the filler content of flowable RBCs is lower than restorative ones to reduce viscosity and enhance cavity adaptation. Previous studies based on static techniques found that bulk-fill RBCs have mechanical properties between conventional and flowable hybrid materials.<sup>6-8</sup> It is plausible that bulk-fill flowable RBCs are formulated similarly to flowable hybrid materials, resulting in

comparable mechanical properties. The depth of cure and degree of conversion of bulk-fill flowable RBCs are also typically greater than bulk-fill restorative materials due to their greater translucency.<sup>27-29</sup> The latter is achieved via decreasing light absorption by pigments and light refraction by matrix-filler interfaces (arising from lower filler content). Degree of conversion governs mechanical properties (with higher degree of conversion being associated with better mechanical properties) and explains in part the similar rigidity of bulk-fill restorative and flowable RBCs in the present study.<sup>30</sup>

Although no obvious trends were observed for manufacturer A, the two conventional RBCs for manufacturers B and C usually had significantly higher loss modulus and flowed more than their bulk-fill counterparts. As filler content of the conventional RBCs was higher or similar to the bulk-fill restoratives, observations can be accounted for only by variance in initiators and resin constituents. While TN is based on camphorquinone, both TF and TB incorporate an adjunct photoinitiator, Ivocerin, giving them a wider absorption range of 370 to 460 nm.<sup>31</sup> As initiator systems influence both the degree of conversion and cross-linking, it is not surprising that flow is reduced in both TF and TB.<sup>32</sup> For BF and BB, the supplementary copolymers added included urethane dimethacrylate (UDMA) and bisphenol-A polyethoxy-dimethacrylate. The lower flow of both giomers can be credited to UDMA, which is a rigid and viscous material, and possibly the use of PRG fillers.<sup>33</sup> As loss tangent values are dependent on both storage and loss modulus, trends

Table 6: Pearson Correlations Between the Various Flexural Properties

| Properties      | Storage Modulus | Loss Modulus | Loss Tangent |
|-----------------|-----------------|--------------|--------------|
| Storage modulus |                 | 0.496*       | -0.560*      |
| Loss modulus    | 0.496*          |              | 0.356*       |
| Loss tangent    | -0.560*         | 0.356*       |              |

\* Correlation significant at the 0.01 level (two-tailed).

are expected to vary, depending on viscoelastic changes after conditioning in the various mediums.

### Comparison Between Products

When conventional materials were compared, BT was found to be significantly stiffer than the other two conventional materials regardless of conditioning medium. This may be due primarily to the higher volume filler loading of BT, including PRG fillers. Likewise, the greater loss modulus and tangent of TN can be accounted for by its relatively lower filler content. Among the bulk-fill restoratives, TB was significantly less rigid than FB and BB. As filler content was similar, the disparity could be due to differences in resin composition and the eclectic mix of fillers used in TB. The lower storage modulus of TB could also be contributed by the nonuse of a polywave curing light that is necessary for the photoactivation of Ivocerin. No obvious patterns were observed for loss modulus and tangent with ranking varying for different conditioning mediums. This reiterates the need for RBCs to be evaluated in different chemical environments instead of just air and/or distilled water, which is often practiced. When comparing the bulk-fill flowable materials, BF, in view of its high filler volume content, was significantly more rigid than TF and FF. As loss tangent is inversely proportion to rigidity, the significantly lower loss tangent of BF was anticipated in view of its higher storage modulus.

### Correlations Between Viscoelastic Properties

As RBCs exhibit both viscous and elastic characteristics under stress, some degree of correlation is anticipated between storage and loss modulus. The correlation was moderately strong, supporting the need for routine viscoelastic characterization of RBCs in conjunction with static testing that evaluates only the elastic component of materials.<sup>10-12</sup> Significant correlations between storage/loss modulus and loss tangent were anticipated, as loss tangent is the ratio of loss modulus to storage modulus. As the correlation between storage modulus and loss tangent was substantially stronger than that between loss modulus and loss tangent, any variance in energy-dissipating capacity is due primarily to changes in rigidity. This corroborated previous work that reported loss tangent increases with increasing filler content.<sup>34</sup>

### Limitations of the Present Study

Some of the limitations of this study had been discussed in our earlier work.<sup>10</sup> They included the

need for longer-term conditioning in the various mediums and independent appraisal of rheological properties and filler content instead of reliance on manufacturers' data, which may not be comparable due to methodological differences. In addition, the evaluation of products from more manufacturers is warranted given the heterogeneity of products under the same material type or classification. With regard to DMA, future work could encompass variation of frequencies, temperatures, and displacement that simulate the range of forces and temperatures encountered intraorally. The glass transition temperature ( $T_g$ ), which is the temperature at which RBCs undergo transition from a glassy to a rubbery state, can also be determined with DMA. This represents the point at which marked changes in physical properties occur and had been linked to curing light energy density and degree of monomer-to-polymer conversion.<sup>35</sup>  $T_g$  values may provide valuable insights toward explaining the observed viscoelastic responses. Viscoelastic properties should also be interrelated with the clinical performance of bulk-fill and conventional RBCs.

### CONCLUSION

The following conclusions can be drawn within the limitations of this study:

- Viscoelastic properties of conventional, bulk-fill restorative, and flowable RBCs were product and conditioning medium dependent.
- For the majority of RBCs evaluated, conditioning in aqueous solutions, particularly an ethanol-water solution, degraded viscoelastic properties.
- Bulk-fill restorative and flowable RBCs generally had lower storage and loss modulus than their conventional equivalents with the exception of FB.
- Conventional and bulk-fill flowable giomers exhibited higher storage modulus and lower loss tangent than the other products.
- Conventional RBCs are generally preferred over their bulk-fill counterparts for high-stress-bearing restorations.

### Acknowledgements

This work was supported by University of Malaya special grant (BKS010-2017). The authors would like to thank 3M EPSE and Shofu Asia for their material support.

### Conflict of Interest

The authors of the article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 20 April 2019)

## REFERENCES

- Chandrasekhar V, Rudrapati L, Badami V, & Tummala M (2017) Incremental techniques in direct composite restoration *Journal of Conservative Dentistry* **20**(6) 386-391.
- Yap AU, Pandya M, & Toh WS (2016) Depth of cure of contemporary bulk-fill resin-based composites *Dental Materials Journal* **35**(3) 503-510.
- Pfeifer CS (2017) Polymer-based direct filling materials *Dental Clinics of North America* **61**(4) 733-750.
- Meereis CTW, Münchow EA, de Oliveira da Rosa WL, da Silva AF, & Piva E (2018) Polymerization shrinkage stress of resin-based dental materials: A systematic review and meta-analyses of composition strategies *Journal of Mechanical Behaviour of Biomedical Materials* **82** 268-281.
- Jung JH & Park SH (2017) Comparison of polymerization shrinkage, physical properties, and marginal adaptation of flowable and restorative bulk fill resin-based composites *Operative Dentistry* **42**(4) 375-386.
- Ilie N, Bucuta S, & Draenert M (2013) Bulk-fill resin-based composites: An in vitro assessment of their mechanical performance *Operative Dentistry* **38**(6) 618-625.
- Czasch P & Ilie N (2013) In vitro comparison of mechanical properties and degree of cure of bulk fill composites *Clinical Oral Investigations* **17**(1) 227-235.
- Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, & Leloup G (2014) Physico-mechanical characteristics of commercially available bulk-fill composites *Journal of Dentistry* **42**(8) 993-1000.
- Eweis AH, Yap AU, & Yahya NA (2018) Impact of dietary solvents on flexural properties of bulk-fill composites *Saudi Dental Journal* **30**(3) 232-239.
- Eweis AH, Yap AU, & Yahya NA (2019) Comparison of flexural properties of bulk-fill restorative/flowable composites and their conventional counterparts *Operative Dentistry* Epub ahead of print April 29.
- Yap AU, Tan DT, Goh BK, Kuah HG, & Goh M (2000) Effect of food-simulating liquids on the flexural strength of composite and polyacid-modified composite restoratives *Operative Dentistry* **25**(3) 202-208.
- Ong J, Yap AU, Hong JY, Eweis AH, & Yahya NA (2018) Viscoelastic properties of contemporary bulk-fill restoratives: A dynamic-mechanical analysis *Operative Dentistry* **43**(3) 307-314.
- Mesquita RV, Axmann D, & Geis-Gerstorfer J (2006) Dynamic visco-elastic properties of dental composite resins *Dental Materials* **22**(3) 258-267.
- Jacobsen PH & Darr AH (1997) Static and dynamic moduli of composite restorative materials *Journal of Oral Rehabilitation* **24**(4) 265-273.
- Sabbagh J, Vreven J, & Leloup G (2002) Dynamic and static moduli of elasticity of resin-based materials *Dental Materials* **18**(1) 64-71.
- Yap AU, Eweis AH, & Yahya NA (2018) Dynamic and static flexural appraisal of resin-based composites: Comparison of the ISO and mini-flexural tests *Operative Dentistry* **43**(5) E223-E231.
- Gal JY, Fovet Y, & Adib-Yadzi M (2001) About a synthetic saliva for in vitro studies *Talanta* **53**(6) 1103-1115.
- Sideridou ID, Karabela MM, & Crysa S (2009) Dynamic mechanical analysis of a hybrid and a nanohybrid light-cured dental resin composite *Journal of Biomaterials Science* **20** 1797-1808.
- Menard KP (2008) *Dynamic Mechanical Analysis: A Practical Introduction* CRC Press, Boca Raton, FL.
- Jones DS, Tian Y, Abu-Diak O, & Andrews GP (2012) Pharmaceutical applications of dynamic mechanical thermal analysis *Advanced Drug Delivery Review* **64**(5) 440-448.
- Eweis AH, Yap AU, & Yahya NA (2017) Dynamic analysis of bulk-fill composites: Effect of food-simulating liquids *Journal of Mechanical Behaviour of Biomedical Materials* **74** 183-188.
- Vouvoudi EC & Sideridou ID (2013) Effect of food/oral-simulating liquids on dynamic mechanical thermal properties of dental nanohybrid light-cured resin composites *Dental Materials* **29**(8) 842-850.
- Sideridou ID & Karabela MM (2011) Sorption of water, ethanol or ethanol/water solutions by light-cured dental dimethacrylate resins *Dental Materials* **27**(10) 1003-1010.
- McKinney JE & Wu W (1985) Chemical softening and wear of dental composites *Journal of Dental Research* **64**(11) 1326-1331.
- Park HY, Kloxin CJ, Abuelyaman AS, Oxman JD, & Bowman CN (2012) Novel dental restorative materials having low polymerization shrinkage stress via stress relaxation by addition-fragmentation chain transfer *Dental Materials* **28**(11) 1113-1119.
- Mohamed-Tahir MA, Tan HY, Woo AA, & Yap AU (2005) Effects of pH on the microhardness of resin-based restorative materials *Operative Dentistry* **30**(5) 661-666.
- Yokesh CA, Hemalatha P, Muthalagu M, & Justin MR (2017) Comparative evaluation of the depth of cure and degree of conversion of two bulk fill flowable composites *Journal of Clinical and Diagnostic Research* **11**(8) ZC86-ZC89.
- Yu P, Yap AU, & Wang XY (2017) Degree of conversion and polymerization shrinkage of bulk-fill resin-based composites *Operative Dentistry* **42**(1) 82-89.
- Bucuta S & Ilie N (2014) Light transmittance and micro-mechanical properties of bulk fill vs. conventional resin based composites *Clinical Oral Investigations* **18**(8) 1991-2000.
- Barszczewska-Rybarek I, & Chladek G (2018) Studies on the curing efficiency and mechanical properties of Bis-GMA and TEGDMA nanocomposites containing silver nanoparticles *International Journal of Molecular Science* **19**(12) E3937.
- Moszner N, Fischer U, Ganster B, Liska R, & Rheinberger V (2008) Benzoyl germanium derivatives as novel

- visible light photoinitiators for dental materials *Dental Materials* **24(7)** 901-907.
32. Randolph LD, Steinhaus J, Möglinger B, Gallez B, Stansbury J, Palin WM, Leloup G, & Leprince JG (2016) Photopolymerization of highly filled dimethacrylate-based composites using type I or type II photoinitiators and varying co-monomer ratios *Dental Materials* **32(2)** 136-148.
  33. Tanoue N, Mikami A, Atsuta M, & Matsumura H (2007) Effects of monomer composition and original filler content on filler loading in the resulting centrifuged composites *Dental Materials Journal* **26(4)** 501-505.
  34. Tanimoto Y, Nishiwaki T, & Nemoto K (2006) Dynamic viscoelastic behavior of dental composites measured by split Hopkinson pressure bar *Dental Materials Journal* **25(2)** 234-240.
  35. Dewaele M, Asmussen E, Peutzfeldt A, Munksgaard EC, Benetti AR, Finné G, Leloup G, Devaux J (2009) Influence of curing protocol on selected properties of light-curing polymers: Degree of conversion, volume contraction, elastic modulus, and glass transition temperature *Dental Materials* **25(12)** 1576-1584.

# Influence of Application Time on Dentin Bond Performance in Different Etching Modes of Universal Adhesives

T Saito • T Takamizawa • R Ishii • A Tsujimoto • E Hirokane  
WW Barkmeier • MA Latta • M Miyazaki

## Clinical Relevance

Although reduced application time of dentin appears to be acceptable for some universal adhesives, care should be taken when using universal adhesives that recommend applying the adhesive with active motion, regardless of the etching mode.

## SUMMARY

**We attempted to determine the effect of universal adhesive application time on dentin**

Tatsuro Saito, DDS, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

\*Toshiki Takamizawa, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Ryo Ishii, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Akimasa Tsujimoto, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Eizo Hirokane, DDS, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Wayne W Barkmeier, DDS, MS, General Dentistry, Creighton University School of Dentistry, Omaha, NE, USA

Mark A Latta, DMD, MS, General Dentistry, Creighton University, Omaha, NE, USA

Masashi Miyazaki, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

\*Corresponding author: 1-8-13, Kanda-Surugadai, Chiyoda-Ku, Tokyo, 101-8310, Japan; e-mail: takamizawa.toshiki@nihon-u.ac.jp

<https://doi.org/10.2341/19-028-L>

**bond performance in different etching modes based on shear bond strength (SBS) tests and surface free energy (SFE) measurements. The five universal adhesives used were Adhese Universal (AU), Clearfil Universal Bond Quick (CQ), G-Premio Bond (GP), Scotchbond Universal (SU), and Tokuyama Universal Bond (TU). Bovine dentin specimens were divided into four groups of 10 for each adhesive. SBS and SFE were determined after applying the following surface treatments: 1) self-etch (SE) mode with immediate air blowing after adhesive application (IA treatment), 2) SE mode with prolonged application time (PA treatment), 3) etch-and-rinse (ER) mode with IA treatment, and 4) ER mode with PA treatment. Bonded specimens were subjected to SBS testing. The SFE of adhesive-treated dentin surfaces was measured after rinsing with acetone and water. Three-way analysis of variance revealed that dentin SBS values ( $p < 0.001$ ) were significantly influenced by the factors of adhesive type and application duration, but the factor of pre-etching was not significant**

( $p=0.985$ ). The manufacturer's instructions require longer application times for AU and SU, which showed significantly lower SBS values in IA than in PA treatment in both etching modes. However, the difference in the other adhesives was not significant between the IA and PA treatments in either etching mode. The total SFE ( $\gamma_s$ ) was dependent on the adhesive and etching mode. The  $\gamma_s$  value of the initial group (SiC paper ground group) at baseline was  $69.5 \text{ (mN}\cdot\text{m}^{-1})$  and that of the pre-etching group at baseline was  $30.6 \text{ (mN}\cdot\text{m}^{-1})$ . For all the adhesives,  $\gamma_s$  in SE mode showed significantly higher values than in ER mode, regardless of the application time. In SE mode, almost all universal adhesives tested showed lower  $\gamma_s$  values in PA treatment than in IA treatment. For ER mode, all the adhesives showed significantly higher  $\gamma_s$  values than those of the pre-etching baseline, regardless of the application time. Most adhesives did not show any significant differences in  $\gamma_s$  values between IA and PA treatments, regardless of etching mode.

## INTRODUCTION

Several years have passed since universal adhesives were introduced as the latest adhesive systems for use in clinical situations.<sup>1-4</sup> Although these adhesive systems were similar to conventional single-step self-etch (SE) adhesives, they may benefit practitioners who utilize multi-etching modes and apply adhesives to different types of indirect restorations.<sup>5,6</sup> Manufacturers continue to develop new universal adhesive products to meet different clinical requirements.

Another trend in the development of universal adhesives is reducing the adhesive's application time.<sup>7-9</sup> Some universal adhesives allow immediate air blowing after adhesive is applied to the tooth surface, which may reduce contamination risk and shorten treatment time.

On the other hand, a previous study investigated how the reduced application time of universal adhesives influenced enamel bonding from the perspective of bond strength and surface free energy (SFE) changes. The findings indicated that the quality and quantity of chemical interaction might be higher with prolonged application time, regardless of the etching mode.<sup>8</sup> However, the influence of these changes on bond strength was unclear.

A recent study examined how the application time of universal adhesives influenced enamel bond

effectiveness in different etching modes through a shear bond strength (SBS) test and SFE measurement.<sup>8</sup> For the SE mode, although all the tested adhesives tended to show increased enamel bond strengths with increased application time, three of five universal adhesives did not show any significant differences between the group with air blown immediately after adhesive application and the group with prolonged application time. Conversely, adhesives with recommendations to apply by rubbing exhibited decreased SBS values with increased application time in etch-and-rinse (ER) mode. However, from the perspective of SFE, chemical bonding tended to increase with increased application time, regardless of the etching mode, suggesting that although prolonged application time of universal adhesives might enhance the chemical reaction with hydroxyapatite (HAp), enamel bond strength values might be influenced by etching mode and adhesive type.

Structurally, enamel is homogeneous in nature and is mainly composed of HAp. On the contrary, dentin is heterogeneous, consisting of collagen and HAp, and the water content is significantly higher than that of enamel. As with conventional single-step SE adhesives, the high levels of water and solvents contained in some universal adhesives allow ionization of the included acidic functional monomers and induce resin monomer infiltration.<sup>10,11</sup> However, residual water inhibits polymerization of resin monomers; thus, evaporating the water is important in order to establish the mechanical properties of the cured adhesive layer.<sup>12,13</sup> Hence, a specific length of application time should allow the residual water and solvents to evaporate, leading to development of a uniform adhesive layer.<sup>7,14</sup> Dissolving the HAp on the dentin surface may reduce chemical bonding while in the ER mode with dentin because HAp has a higher affinity with functional monomers compared with dentin collagen. Therefore, it is important to understand the efficacy of dentin bonding and the characteristics of calcium salt formation in different etching modes using different adhesive application times.

The present study attempted to determine how reduced application time of universal adhesives in different etching modes influenced bonding effectiveness to dentin based on SBS tests, morphologic observations, and SFE characteristics. The null hypotheses proposed that neither reducing application time nor changing etching mode affected dentin SBS or SFE.

Table 1: *Materials Used in the Study*

| Material                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | Main Components                                                                                                                                                      | pH      | Manufacturer                                   |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------|------------------------------------------------|
| Adhesive (Lot No.)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                                                                                                                                                                      |         |                                                |
| AU: Adhese Universal (U49302)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | MDP, bis-GMA, HEMA, MCAP, D3MA, ethanol, water, initiator, stabilizers, silicon dioxide                                                                              | 2.5-3.0 | Ivoclar Vivadent, Schaan, Lichtenstein         |
| CQ: Clearfil Universal Bond Quick (9T0050)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | bis-GMA, MDP, HEMA, hydrophilic amide monomer, filler, ethanol, water, NaF, photoinitiators, chemical polymerization, accelerator, silane coupling agent, others     | 2.3     | Kuraray Noritake Dental, Tokyo, Japan          |
| GP: G-Premio Bond (4G0011)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | MDP, 4-MET, MEPS, BHT, acetone, dimethacrylate resins, initiators, filler, water                                                                                     | 1.5     | GC, Tokyo, Japan                               |
| SU: Scotchbond Universal (41256)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | MDP, HEMA, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane                                                                    | 2.7     | 3M ESPE, St Paul, MN, USA                      |
| TU: Tokuyama Universal Bond (004067)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | Liquid A: phosphate monomer, bis-GMA, TEGDMA, HEMA, MTU-6, others<br>Liquid B: acetone, isopropanol, water, acryl borate catalyst, $\gamma$ -MPTES, peroxide, others | 2.2     | Tokuyama Dental, Tokyo, Japan                  |
| Pre-Etching Agent                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                                                                                                                                      |         |                                                |
| Ultra-Etch (G017)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | 35% phosphoric acid                                                                                                                                                  |         | Ultradent Products, Inc, South Jordan, UT, USA |
| Resin Composite                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |                                                                                                                                                                      |         |                                                |
| Clearfil AP-X (N416713)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | bis-GMA, TEGDMA, silane barium glass filler, silane silica filler, silanated colloidal silica, <i>dl</i> -camphorquinone, pigments, others                           |         | Kuraray Noritake Dental                        |
| <i>Abbreviations: BHT, butylated hydroxytoluene; bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy) phenyl] propane; D3MA, decandiol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; MCAP, methacrylated carboxylic acid polymer; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MEPS, methacryloyloxyalkyl thiophosphate methylmethacrylate; 4-MET, 4-methacryloyloxyethyl trimellitate; <math>\gamma</math>-MPTES, <math>\gamma</math>-methacryloyloxypropyltriethoxysilane; NaF, sodium fluoride; TEGDMA, triethyleneglycol dimethacrylate.</i> |                                                                                                                                                                      |         |                                                |

## METHODS AND MATERIALS

### Study Materials

The materials used in the present study are shown in Table 1. Briefly, we used the following universal adhesives: 1) Adhese Universal (AU; Ivoclar Vivadent, Schaan, Liechtenstein), 2) Clearfil Universal Bond Quick (CQ; Kuraray Noritake Dental, Tokyo, Japan), 3) G-Premio Bond (GP; GC Corp, Tokyo, Japan), 4) Scotchbond Universal (SU; 3M ESPE, St Paul, MN, USA), and 5) Tokuyama Universal Bond (TU; Tokuyama Dental, Tokyo, Japan). Pre-etching with phosphoric acid was performed using Ultra-Etch (Ultradent Products, South Jordan, UT, USA). Clearfil AP-X (Kuraray Noritake Dental) was used as a resin composite to bond to dentin. We used a halogen quartz tungsten curing unit to avoid any influence from the reported nonuniformity of light-emitting diode curing units.<sup>15,16</sup> A visible light curing unit (Optilux 501; SDS Kerr, Danbury, CT, USA) was used, and light irradiance (average 600 mW/cm<sup>2</sup>) was checked during the course of the experiment.

### Specimen Preparation

Extracted mandibular bovine incisors stored frozen for up to 2 weeks were substituted for human teeth. We removed approximately two-thirds of the apical root structure of each tooth using a low-speed diamond saw (IsoMet 1000 Precision Sectioning Saw, Buehler, Lake Bluff, IL, USA). The labial surfaces were ground on wet 240-grit silicon carbide (SiC) paper (Fuji Star Type DDC, Sankyo Rikagaku, Saitama, Japan) to create a flat dentin surface. Next, each tooth was mounted in self-curing acrylic resin (Tray Resin II, Shofu Inc, Kyoto, Japan) to expose the flattened area. A water coolant and a sequence of SiC papers ending with a 320-grit SiC paper were used to polish the dentin surfaces (Fuji Star Type DDC).

### SBS Tests

The SBS to dentin was measured according to ISO 29022.<sup>17</sup> The experimental protocols for the bonding procedures are shown in Table 2 and Figure 1. For each test group, 10 specimens were used to measure

| Table 2: Application Protocol for Pre-etching and Universal Adhesives                                                                                                                                                                                                                                                                                                           |                                                                                                                                                               |                                                                                                                                                                                                                                                                                  |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Method                                                                                                                                                                                                                                                                                                                                                                          | Pre-etching Protocol                                                                                                                                          |                                                                                                                                                                                                                                                                                  |
| ER                                                                                                                                                                                                                                                                                                                                                                              | Dentin surface was etched with phosphoric acid for 15 seconds. Etched surface was rinsed with water for 15 seconds. (three-way dental syringe) and air-dried. |                                                                                                                                                                                                                                                                                  |
| SE                                                                                                                                                                                                                                                                                                                                                                              | Phosphoric acid pre-etching was not performed.                                                                                                                |                                                                                                                                                                                                                                                                                  |
| Adhesive                                                                                                                                                                                                                                                                                                                                                                        | Application Method                                                                                                                                            | Adhesive Application Protocol                                                                                                                                                                                                                                                    |
| AU                                                                                                                                                                                                                                                                                                                                                                              | IA                                                                                                                                                            | Adhesive was applied to the air-dried dentin surface and immediately medium air pressure was applied over the liquid adhesive for 5 seconds. Light irradiation was done for 10 seconds.                                                                                          |
|                                                                                                                                                                                                                                                                                                                                                                                 | PA <sup>a</sup>                                                                                                                                               | Adhesive was applied to the air-dried dentin surface with rubbing motion for 20 seconds, and then medium air pressure was applied to surface for 5 seconds. Light irradiation was done for 10 seconds.                                                                           |
| CQ                                                                                                                                                                                                                                                                                                                                                                              | IA <sup>a</sup>                                                                                                                                               | Adhesive was applied to air-dried dentin surface and immediately medium air pressure was applied over the liquid adhesive for 5 seconds or until the adhesive no longer moved and the solvent had completely evaporated. Light irradiation was done for 10 seconds.              |
|                                                                                                                                                                                                                                                                                                                                                                                 | PA                                                                                                                                                            | Adhesive was applied to air-dried dentin surface for 10 seconds, and then medium air pressure was applied over the liquid adhesive for 5 seconds or until the adhesive was no longer moved and the solvent had completely evaporated. Light irradiation was done for 10 seconds. |
| GP                                                                                                                                                                                                                                                                                                                                                                              | IA <sup>a</sup>                                                                                                                                               | Adhesive was applied to air-dried dentin surface and immediately a strong stream of air was applied over the liquid adhesive for 5 seconds or until the adhesive was no longer moving and the solvent had completely evaporated. Light irradiation was done for 10 seconds.      |
|                                                                                                                                                                                                                                                                                                                                                                                 | PA                                                                                                                                                            | Adhesive was applied to air-dried dentin surface for 10 seconds and then a strong stream of air was applied over the liquid adhesive for 5 seconds or until the adhesive no longer moved and the solvent had completely evaporated. Light irradiation was done for 10 seconds.   |
| SU                                                                                                                                                                                                                                                                                                                                                                              | IA                                                                                                                                                            | Adhesive was applied to air-dried dentin surface and immediately medium air pressure was applied over the liquid adhesive for 5 seconds. Light irradiation was done for 10 seconds.                                                                                              |
|                                                                                                                                                                                                                                                                                                                                                                                 | PA <sup>a</sup>                                                                                                                                               | Adhesive was applied to air-dried dentin surface with rubbing motion for 20 seconds and then medium air pressure was applied to surface for 5 seconds. Light irradiation was done for 10 seconds.                                                                                |
| TU                                                                                                                                                                                                                                                                                                                                                                              | IA <sup>a</sup>                                                                                                                                               | Adhesive was applied to the air-dried dentin surface and immediately medium air pressure was applied over the liquid adhesive for 5 seconds. No light irradiation was done.                                                                                                      |
|                                                                                                                                                                                                                                                                                                                                                                                 | PA                                                                                                                                                            | Adhesive was applied to the air-dried dentin surface for 10 seconds and then medium air pressure was applied over the liquid adhesive for 5 seconds. No light irradiation was done.                                                                                              |
| Abbreviations: ER, Etch-&-rinse; SE, Self-etch; AU, Adhese Universal; CQ, Clearfil Universal Bond Quick; GP, G-Premio Bond; SU, Scotchbond Universal; TU, Tokuyama Universal Bond; IA, immediately air-blow after application of adhesive; PA, application of adhesive according to each manufacturer's instructions (AU and SU) or applied adhesive for 10 seconds (CQ and TU) |                                                                                                                                                               |                                                                                                                                                                                                                                                                                  |
| <sup>a</sup> Manufacturer's instructions                                                                                                                                                                                                                                                                                                                                        |                                                                                                                                                               |                                                                                                                                                                                                                                                                                  |

the dentin SBS in ER mode (phosphoric acid was applied for 15 seconds before applying the adhesive) or SE mode (without phosphoric acid etching). For each different etched dentin surface, the adhesives were applied and immediately subjected to air blowing (IA: immediate air blow), or the GP, SU, and AU adhesives were applied according to the manufacturer's recommended application time, and the CQ and TU adhesives were applied for 10 seconds (PA: prolonged application). Air blowing was always performed as stated in each manufacturer's instructions (Table 2). The experimental groups included four combinations of IA or PA treatment in ER and SE modes for each adhesive, for a total of 20 groups.

A bonding assembly (Ultradent Products, Inc) was used to measure the SBS. After the adhesive was applied to the dentin surface, resin composite cylinders were formed on the surfaces by clamping

plastic molds (2.4-mm internal diameter, approximately 2.5-mm height; Ultradent Products Inc) in a fixture against the adherent surfaces. The resin composite was placed into the mold and light irradiated for 30 seconds. After removing the mold, the specimens were stored in distilled water at 37°C for 24 hours and loaded to failure at 1.0 mm per minute with an Ultradent shearing fixture (Ultradent Products, Inc) using a universal testing machine (Type 5500R, Instron, Canton, MA, USA). The SBS values (MPa) were calculated from the peak load at failure divided by the bonded surface area. After testing, we evaluated the failure mode by viewing the bonding tooth surfaces and debonded resin composite cylinders under an optical microscope (SZH-131, Olympus, Tokyo, Japan) at 10× magnification. On the basis of the percentage of substrate area (adhesive – resin composite – dentin) seen on the debonded cylinders and tooth

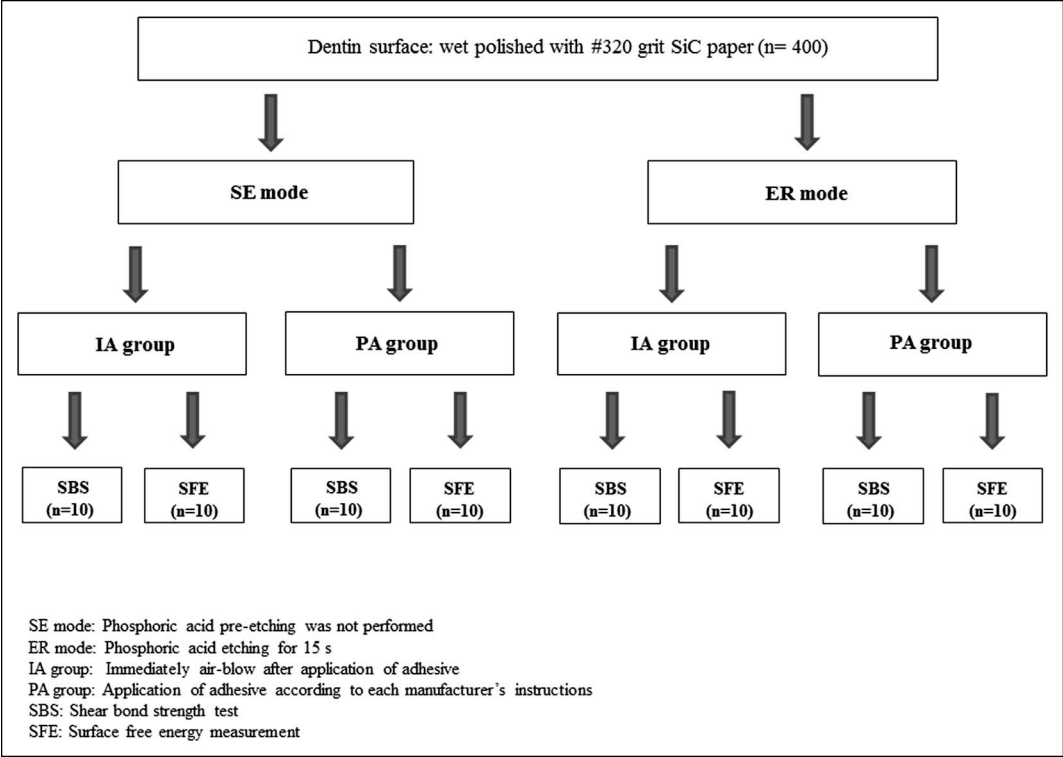


Figure 1. The flow diagram of this study protocol.

surface sites, the types of failure were recorded as 1) adhesive failure, 2) cohesive failure in composite, 3) cohesive failure in dentin, or 4) mixed failure (partially adhesive and partially cohesive).

SFE Measurements

The specimens for measuring SFE were prepared the same as for the SBS test described earlier. After the dentin surface was treated, we removed the uncured

adhesive layer by three alternating rinses with acetone and water. Next, oil-free compressed air was used to dry the dentin surface. Specimens polished with wet 320-grit SiC paper with and without phosphoric acid pre-etching were also measured as a baseline, although they were only rinsed with water. The prepared specimens were used for contact angle measurements, and SFE values were determined by measuring the surface contact angles using the following three test liquids: 1) 1-bromonaphthalene, 2) diiodomethane, and 3) distilled water, each with known SFE parameters as previously reported<sup>8,18</sup> (Table 3). The contact angle meter (Drop Master DM500, Kyowa Interface Science, Saitama, Japan) was connected to a charge-coupled device camera, allowing automatic contact angle measurements.

The equilibrium contact angle ( $\theta$ ) of each test liquid was measured using the sessile drop method at  $23^{\circ}\text{C} \pm 1^{\circ}\text{C}$  in 10 dentin specimens for each condition. Sessile drops of each liquid were dispensed at a volume of  $1.0\ \mu\text{L}$  using a micropipette. The fundamental concepts of wetting were used to determine the SFE parameters of the solids. The Young–Dupré equation describes the work of adhesion for a solid (S) in contact ( $W_{\text{SL}}$ ) with a liquid (L),

| Table 3: Influence of Application Duration on Dentin Bond Strength (MPa) Shown as Mean (Standard Deviation (n=10) <sup>a,b</sup> |                            |                           |                           |                           |
|----------------------------------------------------------------------------------------------------------------------------------|----------------------------|---------------------------|---------------------------|---------------------------|
| Code                                                                                                                             | SE Mode                    |                           | ER Mode                   |                           |
|                                                                                                                                  | IA Group                   | PA Group                  | IA Group                  | PA Group                  |
| AU                                                                                                                               | 26.4 (4.0) <sup>bcB</sup>  | *31.9 (3.7) <sup>bA</sup> | 18.5 (3.5) <sup>cC</sup>  | *34.7 (5.3) <sup>aA</sup> |
| CQ                                                                                                                               | *34.5 (1.9) <sup>aA</sup>  | 34.3 (4.8) <sup>abA</sup> | *34.4 (4.3) <sup>aA</sup> | 34.5 (5.9) <sup>aA</sup>  |
| GP                                                                                                                               | *27.4 (3.4) <sup>bcA</sup> | 29.9 (4.6) <sup>bA</sup>  | *29.1 (4.8) <sup>bA</sup> | 31.6 (3.3) <sup>aA</sup>  |
| SU                                                                                                                               | 25.8 (3.1) <sup>cB</sup>   | *37.7 (4.8) <sup>aA</sup> | 29.6 (2.7) <sup>bB</sup>  | *35.2 (4.5) <sup>aA</sup> |
| TU                                                                                                                               | *30.3 (4.7) <sup>abA</sup> | 29.4 (4.2) <sup>bA</sup>  | *29.5 (3.1) <sup>bA</sup> | 33.9 (4.5) <sup>aA</sup>  |

<sup>a</sup> Asterisk indicates manufacturer's recommended application time.  
<sup>b</sup> Same lowercase letter in vertical columns indicates no difference at 5% significance level. Same capital letter in horizontal rows indicates no difference at 5% significance level.  
Abbreviations: ER, Etch-&-rinse; SE, Self-etch; AU, Adhese Universal; CQ, Clearfil Universal Bond Quick; GP, G-Premio Bond; SU, Scotchbond Universal; TU, Tokuyama Universal Bond;

the interfacial free energy between the solid and the liquid ( $\gamma_{SL}$ ), and the SFE of the liquid and solid ( $\gamma_L$  and  $\gamma_S$ , respectively), as follows:

$$W_{SL} = \gamma_L + \gamma_S - \gamma_{SL} = \gamma_L(1 + \cos\theta).$$

The Fowkes equation was extended as follows, using the Kitazaki-Hata method<sup>19</sup>:

$$\gamma_{SL} = \gamma_L + \gamma_S - 2(\gamma_L^d \gamma_S^d)^{1/2} - 2(\gamma_L^p \gamma_S^p)^{1/2} - 2(\gamma_L^h \gamma_S^h)^{1/2}$$

$$\gamma_L = \gamma_L^d + \gamma_L^p + \gamma_L^h, \gamma_S = \gamma_S^d + \gamma_S^p + \gamma_S^h,$$

where  $\gamma^d$ ,  $\gamma^p$ , and  $\gamma^h$  are SFE ( $\gamma$ ) components arising from the dispersion force, the polar (permanent and induced) force, and the hydrogen-bonding force, respectively. The  $\theta$  values were determined for the three test liquids, and SFE parameters of the treated dentin surfaces were calculated on the basis of the extended Fowkes equation following the Kitazaki-Hata method using add-on software and the included interface measurement and analysis system (FAMAS; Kyowa Interface Science Co). The SFE of dentin was measured on 10 specimens from each group and the mean determined.

### Scanning Electron Microscopy (SEM) Observations

Representative treated dentin surfaces, restorative–dentin interfaces, and debonded fracture sites were observed on field emission SEM (ERA-8800FE, Elionix, Tokyo, Japan). Dentin surfaces were initially treated according to the experimental protocol for bonding procedures and rinsed with acetone and water. For ultrastructural morphologic observations of the restorative–dentin interfaces to determine adhesive penetration, bonded specimens stored in distilled water at 37°C for 24 hours were set in epoxy resin and sectioned lengthwise with a low-speed saw (IsoMet 1000). The sectioned surfaces were polished to a high gloss using SiC papers (Fuji Star Type DDC), followed by diamond pastes, down to a particle size of 0.25  $\mu\text{m}$  (DP-Paste, Struers, Ballerup, Denmark). After ultrasonic cleaning for 3 minutes, the polished surface was etched in hydrogen chloride solution (6 mol/L) for 25 seconds and deproteinized by immersing in 6% sodium hypochlorite solution for 3 minutes. Treated surfaces and debonded fracture sites were prepared directly for the SEM. All SEM specimens were dehydrated in ascending grades of *tert*-butyl alcohol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for 2 hours)

and transferred to a critical-point dryer (Model ID-3, Elionix) for 30 minutes. The resin–dentin interfaces of the specimens were subjected to argon-ion beam etching (EIS-200ER, Elionix) for 20 seconds with an ion beam (accelerating voltage 1.0 kV, ion current density 0.4 mA/cm<sup>2</sup>) directed perpendicular to the polished surfaces. Finally, a vacuum evaporator (Quick Coater, SC-701, Sanyu Electron, Tokyo, Japan) coated all SEM specimens with a thin film of gold. Observations were performed at an operating voltage of 10 kV.

### Statistical Analysis

Before testing, the sample size was determined from the G Power calculator. With an effect size of  $d = 0.5$  (medium),  $\alpha = 0.05$  (two sided), power = 0.95, and number of groups = 20, a total sample size of 210 was needed. And then, we obtained the effect size of more than 0.56 from the F values and df by using three-way ANOVA on the result data. The results indicated that at least 8.8 specimens per group were needed. Therefore, this experiment was initially performed with sample sizes of ten. After gathering the data, post hoc power tests were performed, and these tests indicated that the sample size was adequate.

Three-way analysis of variance (ANOVA) followed by Tukey's honestly significant difference (HSD) test ( $\alpha=0.05$ ) was used to analyze the full data set. Factors included 1) etching mode, 2) application time, and 3) adhesive system. One-way ANOVA followed by Tukey's HSD test ( $\alpha=0.05$ ) was used for making comparisons within subsets of the data, as described later. Statistical analysis was performed using Sigma Plot software, version 11.0 (SPSS Inc, Chicago, IL, USA).

## RESULTS

### Shear Bond Strength

The results for dentin SBS using the different bonding procedures are shown in Table 3. Three-way ANOVA revealed that dentin SBS values ( $p<0.001$ ) were significantly influenced by the factors of adhesive type and application duration, but the factor of pre-etching was not significant ( $p=0.985$ ). The three-way interaction among the factors ( $p<0.001$ ) and all pairwise interactions were significant ( $p<0.05$ ).

When comparing the SBS values between IA and PA treatments in SE mode, the AU and SU values were significantly higher for PA compared with IA treatment. However, no significant difference was

seen between the IA and PA treatments with the other adhesives. Among the tested adhesives in SE mode, SU with IA treatment exhibited the lowest SBS value, and the highest SBS value was observed for SU with PA treatment.

When comparing the SBS values between IA and PA treatments in ER mode, AU and SU showed significantly higher SBS values with PA compared with IA treatment; however, no significant difference in SBS was observed between IA and PA treatments with the other adhesives. Among the tested adhesives in ER mode, AU with IA treatment had a significantly lower SBS value compared with the other adhesives. However, no significant difference was seen among the tested adhesives for PA treatment.

### Failure Mode Analysis of Debonded Specimens

The frequency of different failure modes is shown in Figure 2. The predominant failure mode for all of the adhesives was adhesive failure, regardless of etching mode or application time. However, for all of the adhesives except GP, mixed failure increased in both etching modes with PA treatment.

### Surface Free Energy

The SFE values and components of the different application modes are shown in Figure 3. The total SFE ( $\gamma_S$ ) was dependent on the adhesive and etching mode. The  $\gamma_S$  value of the initial group (320 grit) at baseline was  $69.5 \text{ (mN}\cdot\text{m}^{-1})$  and that of the pre-etching group at baseline was  $30.6 \text{ (mN}\cdot\text{m}^{-1})$ . The pre-etching group demonstrated a significantly lower baseline  $\gamma_S$  value compared with the initial group due to significantly lower values for dispersion ( $\gamma_S^d$ ) and hydrogen-bonding forces ( $\gamma_S^h$ ) in the pre-etching group.

For all the adhesives,  $\gamma_S$  in SE mode showed significantly higher values than in ER mode, regardless of the application time. Further, all the adhesives showed significantly lower  $\gamma_S$  values than the initial baseline. For ER mode, all the adhesives showed significantly higher  $\gamma_S$  values than those of the pre-etching baseline, regardless of the application time. Most adhesives did not show any significant differences in  $\gamma_S$  values between IA and PA treatments, regardless of etching mode.

For all the groups, dispersion force ( $\gamma_S^d$ ) in SE mode showed similar values of approximately  $40 \text{ (mN}\cdot\text{m}^{-1})$  and higher values than in ER mode, irrespective of the application time. Apart from CQ, all the adhesives with IA treatment in SE mode

showed higher polar force ( $\gamma_S^p$ ) values than with PA treatment in SE. On the other hand, none of the adhesives in ER mode showed much difference between IA and PA treatments. Regarding the hydrogen-bonding forces ( $\gamma_S^h$ ), all the adhesives in SE mode showed higher  $\gamma_S^h$  values than in ER mode. In SE mode, most adhesives showed higher  $\gamma_S^h$  values in IA treatment than in PA treatment.

### SEM Observations

Representative SEM images of the treated dentin surfaces are shown in Figures 4 and 5. Remaining scratch marks and smear layer were clearly observed for the specimens with IA treatment in SE mode. Although the specimens with PA treatment in SE mode had a morphologic trend similar to that of IA treatment, part of the smear layer and smear plugs were dissolved. On the other hand, for the specimens in ER mode, the smear layer was completely dissolved and open dentinal tubules were observed, regardless of the application time or type of adhesive.

Representative SEM images of demineralized and deproteinized resin–dentin interfaces are shown in Figures 6 and 7. Clear differences were observed between specimens in the different etching modes in the vicinity of the adhesive–dentin interface. In SE mode, infiltrated resin tags were sparse in both IA and PA treatment; however, the length of resin tags with PA treatment was slightly longer than with IA treatment. This trend was particularly evident in SU and AU compared with the other adhesives. In ER mode, dense resin tags longer than  $50 \text{ }\mu\text{m}$  and approximately  $1 \text{ }\mu\text{m}$  of hybrid layer were detected, regardless of the application time or type of adhesive. In addition, adhesive penetration into the branches of dentinal tubules was more apparent in ER mode. Resin tag density did not vary between IA and PA treatments; however, those in PA treatment appeared to be longer.

Representative SEM images of the failure sites after the SBS test are shown in Figures 8 and 9. The appearance of the failure pattern was dependent on etching mode and adhesive material. The failure pattern of CQ in different etching modes identified similar morphologic etching patterns. However, PA treatment showed more cracks in the adhesives and clearer evidence of resin tags compared with the debonded specimens with IA treatment, regardless of the etching mode (Figure 8). IA treatment of SU in SE mode showed detached areas at the adhesive–resin composite interface. On the other hand, SU in SE mode with PA treatment showed either detached

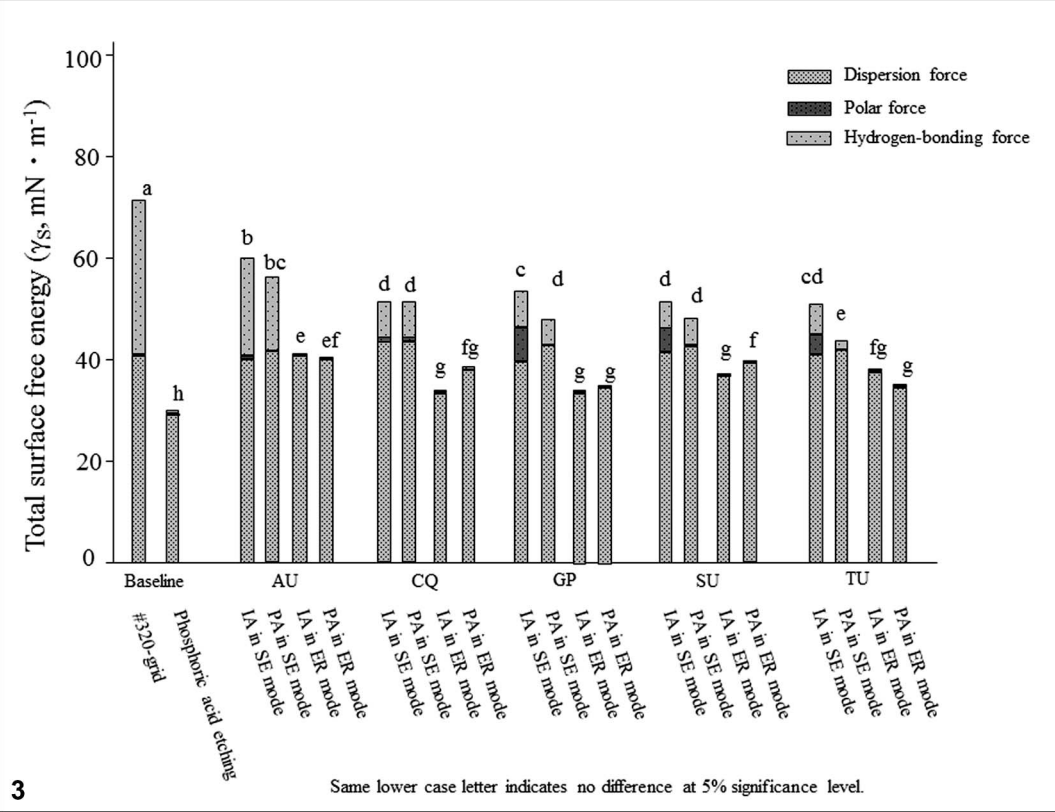
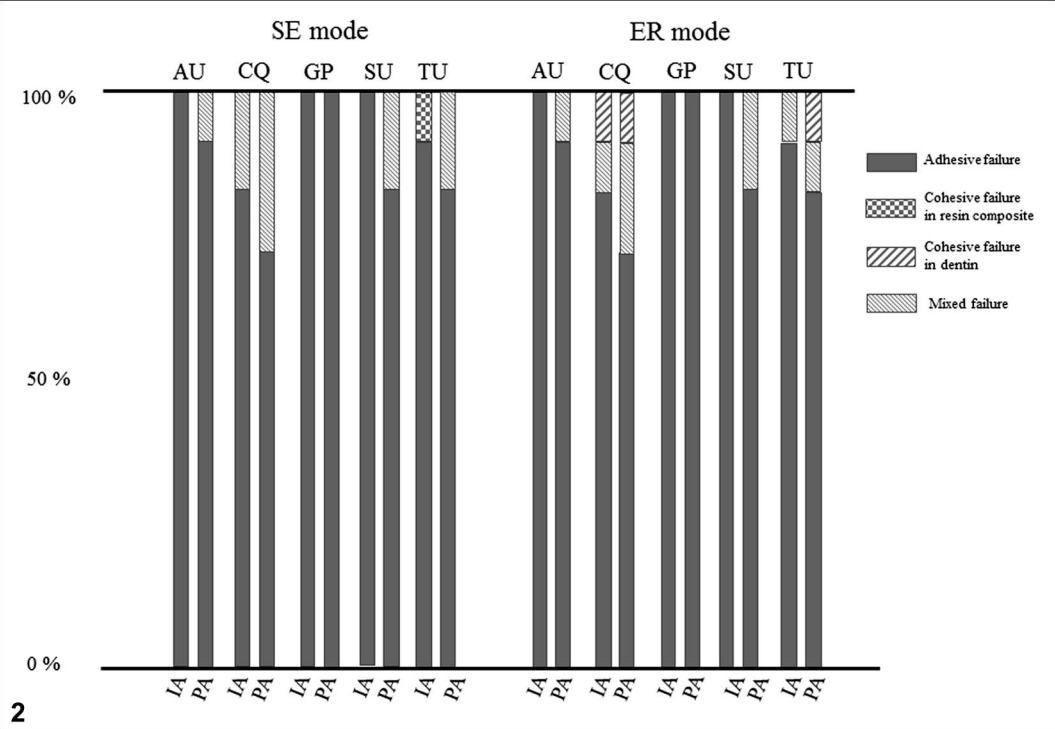
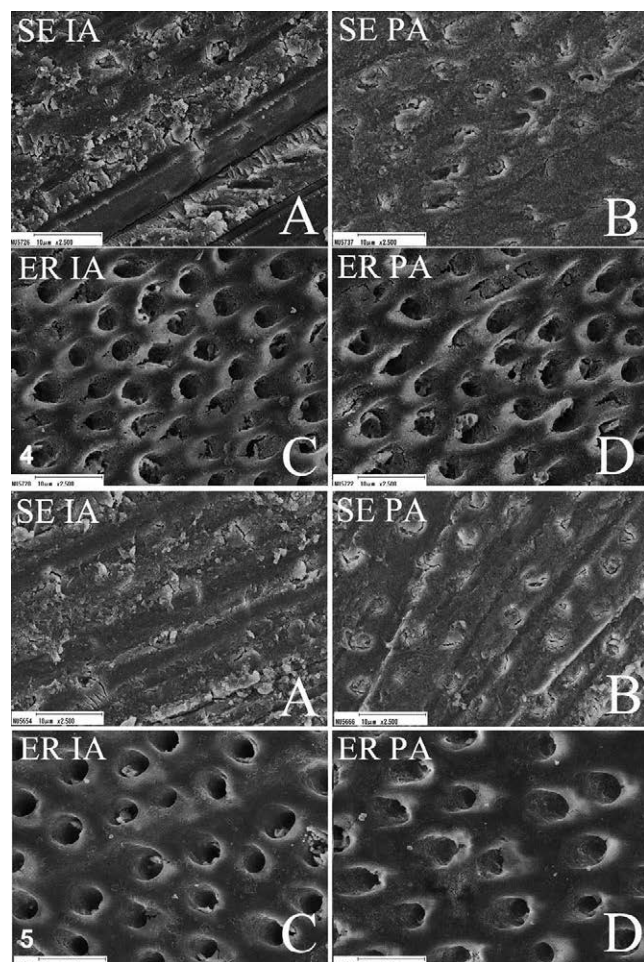


Figure 2. Failure mode analysis of the de-bonded dentin specimens.

Figure 3. Total SFE results from different application times in different etching modes.



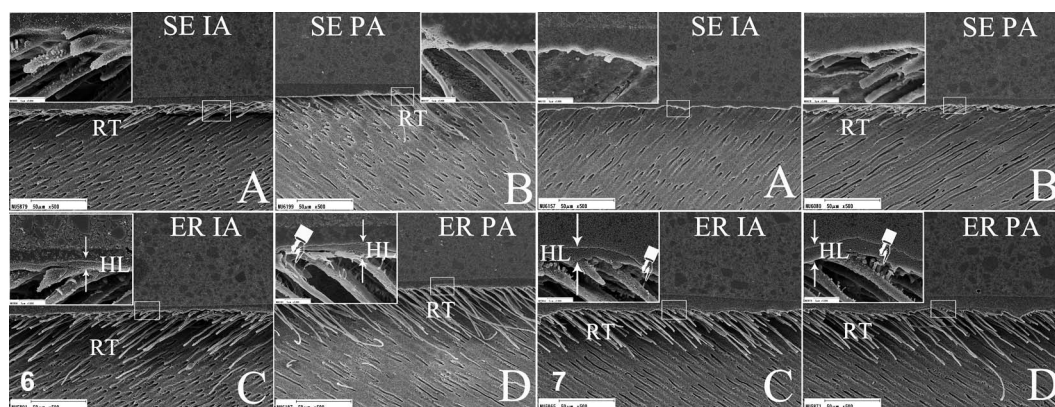
Figures 4 and 5. Representative SEM images of treated dentin surface from different bonding procedures.  $\times 5000$  magnification. (4A): AU with IA treatment in SE mode. (4B): AU with PA treatment in SE mode. (4C): AU with IA treatment in ER mode. (4D): AU with PA treatment in ER mode. (5A): CQ with IA treatment in SE mode. (5B): CQ with PA treatment in SE mode. (5C): CQ with IA treatment in ER mode. (5D): CQ with PA treatment in ER mode.

areas at the adhesive–dentin interface or cohesive failure in dentin. Failure sites for SU in ER mode showed detachment mostly at the adhesive–dentin interface, and evidence of resin tags was observed regardless of the application time (Figure 9).

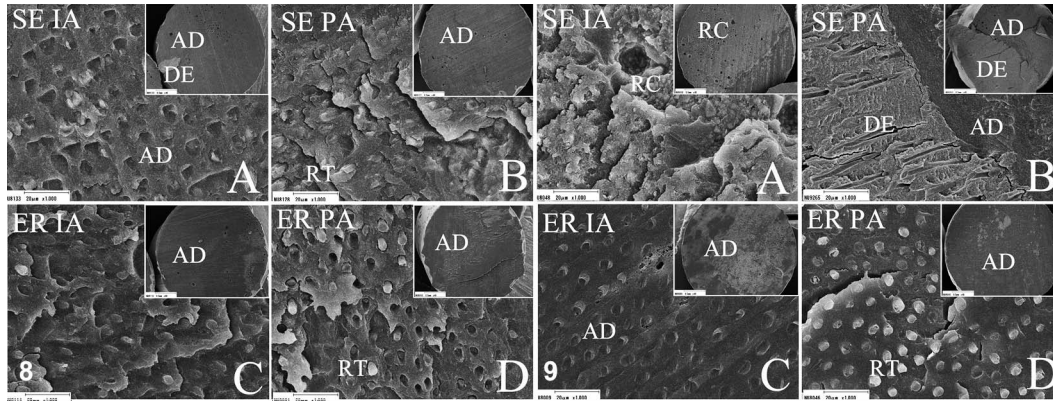
## DISCUSSION

Some of the new universal adhesives feature shortened application times, and this could prove to be a clinically appealing feature for clinicians.<sup>7-9</sup> The present study focused on the influence of different application times for universal adhesives on dentin SBS and SFE values. In the SBS test results, SU and AU, for which the instructions require active motion and longer application times, revealed significantly lower SBS values in IA treatment than in PA treatment. On the other hand, CQ, GP, and TU, which require air blowing immediately after adhesive application, were not significantly different between IA and PA treatments in either etching mode. Therefore, the null hypothesis that reducing application time or changing etching mode did not affect dentin SBS was not rejected for CQ, GP, and TU but was rejected for SU and AU.

The bonding mechanisms in the ER and SE modes are completely different when considering the dentin SBS. For ER systems, phosphoric acid etching performs dentin demineralization with a depth of 5–8  $\mu\text{m}$ , exposing collagen fibrils without Hap.<sup>20,21</sup> To prevent hydrolysis of collagen fibrils, resin monomers should offset and reinforce the spaces formerly occupied by HAp crystals. Micromechanical retention of resin tags within the hybrid layer is considered the primary contribution of phosphoric acid etching to adhesion.<sup>20,21</sup> However, one of the



Figures 6 and 7. Representative SEM micrographs of the resin-dentin interfaces. The main images are at  $\times 5000$  magnification. The smaller white rectangle indicates the location in the main image of the enlarged area, at  $\times 20,000$ , in the upper right or left corner. The visible material is indicated by abbreviations: HL indicates hybrid layer; RT, resin tag. (6A): AU with IA treatment in SE mode. (6B): AU with PA treatment in SE mode. (6C): AU with IA treatment in ER mode. (6D): AU with PA treatment in ER mode. (7A): CQ with IA treatment in SE mode. (7B): CQ with PA treatment in SE mode. (7C): CQ with IA treatment in ER mode. (7D): CQ with PA treatment in ER mode.



Figures 8 and 9. Representative SEM images of the fractured resin surface in SE mode and in ER mode with different application times. The visible material is indicated by abbreviations: AD indicates adhesive; DE, dentin; RC, resin composite; RT, resin tag. (8A): CQ with IA treatment in SE mode (50 $\times$  and 1000 $\times$ ). (8B): CQ with PA treatment in SE mode (50 $\times$  and 1000 $\times$ ). (8C): CQ with IA treatment in ER mode (50 $\times$  and 1000 $\times$ ). (8D): CQ with PA treatment in ER mode (50 $\times$  and 1000 $\times$ ). (9A): SU with IA treatment in SE mode (50 $\times$  and 1000 $\times$ ). (9B): SU with PA treatment in SE mode (50 $\times$  and 1000 $\times$ ). (9C): SU with IA treatment in ER mode (50 $\times$  and 1000 $\times$ ). (9D): SU with PA treatment in ER mode (50 $\times$  and 1000 $\times$ ).

concerns regarding the ER mode is incomplete resin monomer penetration into the full depth of demineralized dentin, resulting in insufficient hybridization and unprotected collagen fibrils.<sup>22,23</sup> For the SE mode, the key components are acidic functional monomers, which partially demineralize the tooth surface and achieve chemical bonding with HAp.<sup>10,11</sup> In particular, chemical bonding is more important for dentin compared with enamel because the smaller crystals and plate-like structure of dentin HAp are considered more accessible to chemical reaction compared with enamel HAp.<sup>24,25</sup>

Although the dentin bonding mechanism of universal adhesives in SE mode is similar to conventional single-step SE adhesives, that of universal adhesives in ER mode may not be exactly the same as those of three-step or two-step systems. On the basis of these results, we can speculate that the role of functional monomers of universal adhesives is important for achieving a chemical interaction not only with HAp but also with exposed collagen fibrils. Hiraishi and others<sup>26</sup> proposed that 10-methacryloyloxydecyl dihydrogen phosphate (MDP) has a relatively stable interaction with collagen due to the hydrophobic interactions between the MDP moieties and the collagen surface, as measured by saturation transfer difference nuclear magnetic resonance spectroscopy. Five of the six tested universal adhesives contained MDP, and functional monomers might penetrate the intact dentin substrate through naked collagen fibrils after pre-etching. In particular, no significant difference was seen between the IA and PA treatments in ER mode using CQ, GP, or TU, suggesting that their resin monomers should have a higher penetration ability. CU contains 2-

hydroxyethyl methacrylate (HEMA) and a newly developed amide monomer, both of which are highly hydrophilic and mobile as monomers, and the amide monomer can penetrate deeper into dentin and polymerize to form a stable bond. Therefore, we could suppose that monomers could penetrate deep into the demineralized dentin and polymerize to develop a stable polymer network producing strong micromechanical interlocking.

In order to better understand the dentin bonding mechanism of universal adhesives, we also examined the surface chemistry. In the SFE measurements, the baseline group demonstrated a significantly lower total free energy ( $\gamma_s$ ) value than did the initial group (SiC paper ground group) in ER mode. In particular, the dispersion force ( $\gamma_s^d$ ) and hydrogen-bonding force ( $\gamma_s^h$ ) values were significantly lower for the demineralized dentin surfaces than for the initial group. The  $\gamma_s$  value was expressed as the sum of three parameters,  $\gamma_s^d$ ,  $\gamma_s^p$ , and  $\gamma_s^h$ ,<sup>19</sup> indicating that dentin wettability after phosphoric acid etching was lower than for dentin surfaces covered by a smear layer. Because it is not easy to standardize dentin moisture conditions, wettability and SFE measurement of demineralized dentin remain controversial.<sup>27-30</sup> Although HAp has high SFE due to the concentration of hydroxyl groups, collagen fibrils composed of insoluble fibrous protein have low SFE.<sup>31</sup> The reason for the decrease in  $\gamma_s$  and  $\gamma_s^h$  values of dentin for phosphoric acid-etched dentin might be related to a decreased mineral/organic ratio due to the loss of HAp.<sup>32</sup> In addition, it can be assumed that changes in surface morphology, including exposed collagen fibrils and dentinal tubules, lead to a decrease in  $\gamma_s^d$ . In contrast to a

smear layer-covered dentin surface, the morphology of demineralized dentin, with a mesh structure of exposed collagen fibrils and opened dentin tubules, is more complex and might trap air, resulting in decreased  $\gamma_S^d$  values.<sup>33</sup>

For ER mode, all the adhesives showed significantly higher  $\gamma_S$  values than the pre-etching baseline. In particular, the  $\gamma_S^d$  in all adhesive treated groups was significantly higher than that of the pre-etching baseline, regardless of the application time. The increased  $\gamma_S$  values in adhesive treated groups after pre-etching may be attributed to the increased  $\gamma_S^d$ . The reason for increased  $\gamma_S^d$  values in the adhesive treated surfaces after phosphoric acid pre-etching is unclear. However, it can be speculated that chemical and physical interaction between surface collagen fibrils and adhesive components influence the  $\gamma_S^d$ . In particular, these interactions may change morphologic features of a demineralized dentin surface in spite of washing with acetone and water.

All of the adhesives in SE mode exhibited significantly lower  $\gamma_S$  values compared with the initial baseline group (320 grit), regardless of the application time. Adhesive-treated surfaces in both IA and PA treatments demonstrated significantly lower  $\gamma_S^h$  values compared with the initial baseline group. The  $\gamma_S^h$  value represents the water and hydroxyl components of the substrate. The  $\gamma_S^p$  value is thought to be associated with electronic and metallic interactions as well as dipolar interactions.<sup>34</sup> Thus,  $\gamma_S^h$  and  $\gamma_S^p$  parameters might be helpful for identifying whether the surface characteristics are hydrophilic or hydrophobic. Substrates with higher  $\gamma_S^h$  and  $\gamma_S^p$  values are typically water soluble, whereas substrates with lower values tend to be soluble in organic solvents. These results indicate that adhesive-treated dentin surfaces lean from hydrophilic to hydrophobic due to chemical interactions and functional monomers forming calcium salts. When comparing different application times in SE mode, lower  $\gamma_S$  values in PA than in IA treatment were seen in almost all of the universal adhesives, although most differences were not statistically significant. Most universal adhesives presented lower  $\gamma_S^p$  and  $\gamma_S^h$  values in PA treatment than in IA treatment; hence, a longer application time may promote chemical interactions between HAp for most universal adhesives in SE mode.

Therefore, the null hypothesis that reduced application time or different etching mode did not affect dentin SFE was rejected for all the adhesives in

terms of etching mode but was not rejected in terms of application time.

Moreover, evidence of a consistent effect of increased application time on SFE was seen in ER mode compared with SE mode. However, AU and SU exhibited significantly lower SBS values in IA treatment than in PA treatment and lower SFE values in PA treatment than in IA treatment in SE mode. In contrast to the enamel smear layer, the gel-like collagen in the dentin smear on sound tissue is thought to interfere with the penetration of resin monomers.<sup>35,36</sup> In addition, from the perspective of pH values of the tested adhesive, AU and SU have relatively higher pH values than the other universal adhesives. Although lower pH of adhesive is thought to be inferior in decalcifying mineralized tissue, longer application time and stirring of adhesive might compensate for lower etching capability due to supplying unreacted  $H^+$  ions.<sup>18</sup> Furthermore, active and longer application time might encourage water and solvent evaporation, creating a hydrophobic layer, promoting the penetration of resin monomers, and inducing a chemical interaction for AU and SU.<sup>37</sup> A trend in SBS values was clearly observed in ER mode, as most tested materials demonstrated increased SBS values with increased application time. Although the same trend was seen for all of the materials, only two of the individual differences were statistically significant.

Saikaew and others<sup>7</sup> examined how reduced application time of three universal adhesives in SE mode influenced dentin bond performance and found that the universal adhesives, which required application times of 10 or 20 seconds, showed significantly lower  $\mu$ -TBS (tensile bond strength) values in the adhesive application groups with immediate air blowing than in the groups following the manufacturer's instructions for bur-cut dentin surfaces. Furthermore, the same research facility investigated the effect of long-term water storage on dentin bond durability, concluding that adhesive application time might compromise bond strength.<sup>9</sup> Although they did not investigate the influence of reduced application time of universal adhesives in ER mode, the present study was in line with their results in terms of immediate bond strength in SE mode. Therefore, based on previous studies as well as the present study, although reduced application time appears to be acceptable for CQ, GP, and TU, at least for immediate dentin bond strength, regardless of etching mode, it has a negative effect on AU and SU.

## CONCLUSION

The results of the present laboratory study did not reveal any significant differences in dentin SBS values between IA and PA treatments in three of five universal adhesives, regardless of etching mode. However, the other universal adhesives, which required active and longer application times, demonstrated significantly lower SBS values in IA than in PA treatment in both etching modes. In the results of SFE measurements, the baseline group demonstrated a significantly lower total free energy ( $\gamma_S$ ) value than did the initial group (SiC paper ground group) in ER mode. The adhesive-treated dentin surfaces exhibited lower  $\gamma_S$  values for all the adhesives in SE mode than did the initial dentin surfaces, and most adhesives showed lower  $\gamma_S$  values in PA compared with IA treatment.

## Acknowledgements

This work was supported in part by Grants-in-Aid for Scientific Research, No. 16K11565, 16K20465, 17K11716, and 17K17141 from the Japan Society for the Promotion of Science. This project was also supported in part by the Sato Fund and by a grant from the Dental Research Center of the Nihon University School of Dentistry, Japan.

## Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Nihon University School of Dentistry.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 29 April 2019)

## REFERENCES

- Wagner A, Wendler M, Petschelt A, Belli R, & Lohbauer U (2014) Bonding performance of universal adhesives in different etching modes *Journal of Dentistry* **42**(7) 800-807. <https://doi.org/10.1016/j.jdent.2014.04.012>
- Muñoz MA, Luque-Martinez I, Malaquias P, Hass V, Reis A, Campanha NH, & Loguercio AD (2015) In vitro longevity of bonding properties of universal adhesives to dentin *Operative Dentistry* **40**(3) 282-292. <https://doi.org/10.2341/14-055-L>
- Da Rosa WL, Piva E, & Da Silva AF (2015) Bond strength of universal adhesives: A systematic review and meta-analysis *Journal of Dentistry* **43**(7) 765-776. <https://doi.org/10.1016/j.jdent.2015.04.003>
- Nagarkar S, Theis-Mahon N, & Perdigão J (2019) Universal dental adhesives: Current status, laboratory testing, and clinical performance *Journal of Biomedical Materials Research. Part B, Applied Biomaterials* (in press). <https://doi.org/10.1002/jbm.b.34305> [Epub ahead of print]
- Suzuki T, Takamizawa T, Barkmeier WW, Tsujimoto A, Endo H, Erickson RL, Latta MA, & Miyazaki M (2016) Influence of etching mode on enamel bond durability of universal adhesive systems *Operative Dentistry* **41**(5) 520-530. <https://doi.org/10.2341/15-347-L>
- Takamizawa T, Barkmeier WW, Tsujimoto A, Berry TP, Watanabe H, Erickson RL, Latta MA, & Miyazaki M (2016) Influence of different etching modes on bond strength and fatigue strength to dentin using universal adhesive systems *Dental Materials* **32**(2) e9-e21. <https://doi.org/10.1016/j.dental.2015.11.005>
- Saikaew P, Chowdhury AFMA, Fukuyama M, Kakuda S, Carvalho RM, & Sano H (2016) The effect of dentine surface preparation and reduced application time of adhesive on bonding strength *Journal of Dentistry* **47** 63-70. <https://doi.org/10.1016/j.jdent.2016.02.001>
- Sai K, Takamizawa T, Imai A, Tsujimoto A, Ishii R, Barkmeier WW, Latta MA, & Miyazaki M (2018) Influence of application time and etching mode of universal adhesives on enamel adhesion *Journal of Adhesive Dentistry* **20**(1) 65-77. <https://doi.org/10.3290/j.jad.a39913>
- Saikaew P, Matsumoto M, Chowdhury AFMA, Carvalho RM, & Sano H (2018) Does shortened application time affect long-term bond strength of universal adhesives to dentin? *Operative Dentistry* **43**(5) 549-558. <https://doi.org/10.2341/17-205-L>
- Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, & De Munck J (2011) State of the art of self-etch adhesives *Dental Materials* **27**(1) 17-28. <https://doi.org/10.1016/j.dental.2010.10.023>
- Miyazaki M, Tsujimoto A, Tsubota K, Takamizawa T, Kurokawa H, & Platt JA (2014) Important compositional characteristics in the clinical use of adhesive systems *Journal of Oral Science* **56**(1) 1-9. <https://doi.org/10.2334/josn.14.056.1>
- Van Landuyt KL, De Munck J, Snauwaert J, Coutinho E, Poitevin A, Yoshida Y, Inoue S, Peumans M, Suzuki K, Lambrechts P, & Van Meerbeek B (2005) Monomer-solvent phase separation in one-step self-etch adhesives *Journal of Dental Research* **84**(2) 183-188. <https://doi.org/10.1177/154405910508400214>
- Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, & De Stefano Dorigo E (2008) Dental adhesion review: Aging and stability of the bonded interface *Dental Materials* **24**(1) 90-101. <https://doi.org/10.1016/j.dental.2007.02.009>
- Cardoso PC, Loguercio AD, Vieira LC, Baratieri LN, & Reis A (2005) Effect of prolonged application times on resin-dentin bond strengths *Journal of Adhesive Dentistry* **7**(2) 143-149.
- Price RBT, Ruggeberg FA, Labrie D, & Felix CM (2010) Irradiance uniformity and distribution from dental light curing units *Journal of Esthetic and Restorative Dentistry* **22**(2) 86-103. <https://doi.org/10.1111/j.1708-8240.2010.00318.x>

16. Michaud PL, Price RBT, Labrie D, Rueggeberg FA, & Sullivan B (2014) Localised irradiance distribution found in dental light curing units *Journal of Dentistry* **42**(2) 129-139. <https://doi.org/10.1016/j.jdent.2013.11.014>
17. ISO (2013) *ISO 29022: 2013 Dentistry-Adhesion-Notched-edge Shear Bond Strength Test* International Organization for Standardization, Geneva, Switzerland, 1-12.
18. Imai A, Takamizawa T, Sai K, Tsujimoto A, Nojiri K, Barkmeier WW, Latta MA, & Miyazaki M (2017) Influence of application method on surface free-energy and bond strength of universal adhesive systems to enamel *European Journal of Oral Science* **125**(5) 385-395. <https://doi.org/10.1111/eos.12361>
19. Hata T, Kitazato Y, & Saito T (1987) Estimation of the surface energy of polymer solids *Journal of Adhesion* **21**(3-4) 177-194.
20. Pashley DH, Tay FR, Breschi L, Tjäderhane L, Carvalho RM, Carrilho M, & Tezvergil-Mutluay A. (2011) State of the art etch-and-rinse adhesives *Dental Materials* **27**(1) 1-16. <https://doi.org/10.1016/j.dental.2010.10.016>
21. Miyazaki M, Tsubota K, Takamizawa T, Kurokawa H, Rikuta A, & Ando S (2012) Factors affecting the *in vitro* performance of dentin-bonding systems *Journal of Dental Science Review* **48**(1) 53-60. <https://doi.org/10.1016/j.jdsr.2011.11.002>
22. Hashimoto M, Ohno H, Kaga M, Sano H, Tay FR, Oguchi H, Araki Y, & Kubota M (2002) Over-etching effects on micro-tensile bond strength and failure patterns for two dentin bonding systems *Journal of Dentistry* **30**(2-3) 99-105. [https://doi.org/10.1016/S0300-5712\(02\)00004-0](https://doi.org/10.1016/S0300-5712(02)00004-0)
23. Hashimoto M, Ohno H, Sano H, Kaga M, & Oguchi H (2003) In vitro degradation of resin-dentin bonds analyzed by microtensile bond test, scanning and transmission electron microscopy *Biomaterials* **24**(21) 3795-3803. [https://doi.org/10.1016/S0142-9612\(03\)00262-X](https://doi.org/10.1016/S0142-9612(03)00262-X)
24. Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Irie M, Ogawa T, Van Landuyt KL, Osaka A, Suzuki K, Minagi S, & Van Meerbeek B (2011) Nanolayering of phosphoric acid ester monomer on enamel and dentin *Acta Biomaterialia* **7**(8) 3187-3195. <https://doi.org/10.1016/j.actbio.2011.04.026>
25. Yaguchi T (2017) Layering mechanism of MDP-Ca salt produced in demineralization of enamel and dentin apatite *Dental Materials* **33**(1) 23-32. <https://doi.org/10.1016/j.dental.2016.09.037>
26. Hiraishi N, Tochio N, Kigawa T, Otsuki M, & Tagami J (2013) Monomer-collagen interactions studied by saturation transfer difference NMR *Journal of Dental Research* **92**(3) 284-288. <https://doi.org/10.1177/0022034512474310>
27. Aguilar-Mendoza JA, Rosales-Leal JI, Rodriguez-Valverde MA, & Cabrerizo-Vilchez MA (2008) Effect of acid etching on dentin wettability and roughness: Self-etching primers versus phosphoric acid *Journal of Biomedical Materials Research. Part B, Applied Biomaterials* **84**(1) 277-285. <https://doi.org/10.1002/jbm.b.30871>
28. Yilmaz Z, Basbag B, Buzoglu HD, & Gümüşderelioglu M (2011) Effect of low-surface-tension EDTA solutions on the wettability of root canal dentin *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics* **111**(1) 109-114. <https://doi.org/10.1016/j.tripleo.2010.08.008>
29. Erickson RL (1992) Surface interactions of dentin adhesive materials *Operative Dentistry* (Supplement 5) 81-94.
30. Pelin IM, Trunfio-Sfarghiu AM, Farge P, Piednoir A, Pirat C, & Ramos SM (2013) Multiscale characterization of partially demineralized superficial and deep dentin surfaces *European Journal of Oral Science* **121**(4) 341-348. <https://doi.org/10.1111/eos.12058>
31. Akinmade AO & Nicholson JW (1993) Glass-ionomer cements as adhesives. Part I. Fundamental aspects and their clinical relevance *Journal of Materials Science: Materials in Medicine* **4**(2) 95-101.
32. Besinis A, Van Noort R, & Martin N (2016) The use of acetone to enhance the infiltration of HA nanoparticles into a demineralized dentin collagen matrix *Dental Materials* **32**(3) 385-393. <https://doi.org/10.1016/j.dental.2015.11.010>
33. Farge P, Alderete L, & Ramos SM (2010) Dentin wetting by three adhesive systems: influence of etching time, temperature and relative humidity *Journal of Dentistry* **38**(9) 698-706. <https://doi.org/10.1016/j.jdent.2010.03.013>
34. Tsujimoto A, Iwasa M, Shiimamura Y, Murayama R, Takamizawa T, & Miyazaki M (2010) Enamel bonding of single-step self-etch adhesives: Influence of surface energy characteristics *Journal of Dentistry* **38**(2) 123-130. <https://doi.org/10.1016/j.jdent.2009.09.011>
35. Thanatvarakorn O, Nakajima M, Prasansuttioporn T, Ichinose S, Foxton RM, & Tagami J (2014) Effect of smear layer deproteinizing on resin-dentine interface with self-etch adhesive *Journal of Dentistry* **42**(3) 298-304. <https://doi.org/10.1016/j.jdent.2013.11.026>
36. Takamizawa T, Barkmeier WW, Sai K, Tsujimoto A, Imai A, Erickson RL, Latta MA, & Miyazaki M (2018) Influence of different smear layers on bond durability of self-etch adhesives *Dental Materials* **34**(2) 246-259. <https://doi.org/10.1016/j.dental.2017.11.002>
37. Moritake N, Takamizawa T, Ishii R, Tsujimoto A, Barkmeier WW, Latta MA, & Miyazaki M (2019) Effect of active application on bond durability of universal adhesives *Operative Dentistry* **44**(2) 188-199. <https://doi.org/10.2341/17-384-L>

# The Effects of Aluminablasting on Bond Durability Between Universal Adhesives and Tooth Substrate

H Ouchi • T Takamizawa • K Tsubota • A Tsujimoto  
A Imai • WW Barkmeier • MA Latta • M Miyazaki

## Clinical Relevance

Aluminablasting may adversely affect the dentin bond strength of universal adhesives in self-etch mode.

## SUMMARY

**The aim of this study was to determine the effect of aluminablasting on the bond durability of universal adhesives and adherent surface characteristics. Adhese Universal (Ivoclar Vivadent), All-Bond Universal (Bisco), Bondmer Lightless (Tokuyama Dental), G-Premio**

Hajime Ouchi, DDS, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

\*Toshiki Takamizawa, DDS, PhD, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

Keishi Tsubota, DDS, PhD, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

Akimasa Tsujimoto, DDS, PhD, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

Arisa Imai, DDS, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

Wayne W Barkmeier, EBM, DDS, MS, Creighton University School of Dentistry, General Dentistry, Omaha, NE, USA

Mark A Latta, DMD, MS, Creighton University, General Dentistry, Omaha, NE, USA

Masashi Miyazaki, DDS, PhD, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

\*Corresponding author: 1-8-13, Kanda-Surugadai, Chiyoda-Ku, Tokyo, 101-8310 Japan; e-mail: takamizawa.toshiki@nihon-u.ac.jp

DOI: 10.2341/18-170-L

**Bond (GC), and Scotchbond Universal (3M ESPE) were used in self-etch mode. The prepared bovine enamel and dentin specimens were divided into two groups based on whether they received an aluminablasting prior to application of the universal adhesives. The resin composite bonded specimens were stored in distilled water at 37°C for 24 hours, following which the shear bond strength (SBS) of half of the specimens was measured (24-hour group). The other half was subjected to 30,000 thermal cycles between 5 and 55°C before SBS measurement (TC group). Surface roughness (Ra) and surface free energy (SFE) of the adherent surfaces were also measured, and scanning electron microscopy observation and scanning electron microscopy/energy-dispersive X-ray analysis were carried out. Most of the adhesives did not show any significant differences in enamel SBS values between the two pretreatment groups, regardless of the storage condition. However, the dentin SBS values were significantly lower in specimens that underwent aluminablasting compared with those that did not, irrespective of their storage conditions. Significantly higher Ra and SFE values were observed in the enamel and dentin of specimens that underwent alu-**

**minablasting. Although aluminablasting increased the Ra and SFE values of enamel and dentin, its effect on the SBS value was dependent on the tooth substrate. In addition to C, O, Na, Mg, P, and Ca, the element Al was detected in the enamel and dentin of samples that had undergone aluminablasting. These results suggest that although aluminablasting of the tooth surface is thought to be effective for modification of the adherent surface, it may not enhance enamel bond performance and may also adversely affect the dentin bond effectiveness of the universal adhesives.**

## INTRODUCTION

It is inevitable that restorations in the oral cavity will partially fracture or develop recurrent caries around their margins over time. In such cases, the surrounding sound tooth structures can be preserved by adopting a minimally invasive approach, which advocates the use of composite resins to repair defective or aged restorations.<sup>1</sup> The tooth cavity is typically made up of more than one material and exhibits a complicated configuration, making it desirable that the resin composites bond effectively to all of these materials. Several procedures, including phosphoric acid etching, airborne particle abrasion, and the use of different primers on the aged restorations prior to application of the adhesive, have been recommended for the achievement of durable bonds between different substrates.<sup>2-4</sup> However, these pretreatments require additional clinical steps, and it may be difficult to apply pretreatments on just the aged restorations or the tooth substrates of the cavity. Moreover, contamination of the tooth substrate by metal primers or silane coupling agents may negatively affect the bond strength between the substrate and resin composite.<sup>5,6</sup>

Universal adhesives have distinctive characteristics and versatility when compared with the previous generations of adhesive systems.<sup>7</sup> Different etching techniques, including self-etch, etch and rinse, or selective etching, can be used in direct composite resin restorations with universal adhesives.<sup>8,9</sup> Furthermore, universal adhesives have also simplified bonding procedures considerably as they contain various functional monomers that allow them to bond to surfaces other than tooth substrates.<sup>10</sup>

Airborne particle abrasion has been used extensively in various dental treatments, including stain removal, cavity preparation, and inner surface modification of indirect restorations.<sup>11,12</sup> The material and size of the abrasion particles depend on the

purpose of application. Aluminablasting of indirect restorations such as metal alloy, zirconia, and cured resin composites has been reported to be a reliable method of pretreatment,<sup>11,13-15</sup> and the resulting surface modifications can increase mechanical interlocking and modify the adhered surface.<sup>16</sup> In contrast, Loomans and others<sup>2</sup> suggested that none of the surface treatments for restoration repair can be recommended as a universally applicable repair technique for the different types of resin composites. Moreover, the effects of aluminablasting on the bond strength and surface characteristics of the restorations are still unknown, and there is a possibility that residual alumina particles and surface modifications of the teeth may negatively affect the chemical bond with the universal adhesives by interfering with the chemical reactions between the functional monomers and the hydroxyapatite (HAp).

The aim of this study was to examine the effects of aluminablasting enamel and dentin on the bonding performance and surface characteristics when using universal adhesives in self-etch mode. This was determined by testing the bond strength and surface free energy measurements of the adherent surfaces before and after thermal cycling. The null hypotheses tested were 1) the bond performance of universal adhesives would not be influenced by aluminablasting, 2) the surface characteristics of the adherent surface would be altered by aluminablasting, and 3) there would be no difference in the effects of aluminablasting between enamel and dentin.

## METHODS AND MATERIALS

### Study Materials

The materials used in this study are shown in Table 1. The five universal adhesives used were 1) All Bond Universal (ABU; Bisco, Schaumburg, IL, USA) 2), Adhese Universal (ADU; Ivoclar Vivadent, Schaan, Liechtenstein), 3) Bondmer Lightless (BML; Tokuyama Dental, Tokyo, Japan), 4) G-Premio Bond (GPB; GC, Tokyo, Japan), and 5) Scotchbond Universal (SBU; 3M ESPE, St. Paul, MN, USA). Clearfil AP-X (Kuraray Noritake Dental, Tokyo, Japan) was used as a restorative material for bonding to enamel and dentin. A halogen quartz tungsten curing unit (Optilux 501, sds Kerr, Danbury, CT, USA) at an average light irradiance of 600 mW/cm<sup>2</sup> was used.

### Specimen Preparation

This study used extracted mandibular bovine incisors as a substitute for human teeth. The labial surfaces of the teeth were ground using wet #240-

Table 1: *Materials Used in This Study*

| Code | Adhesive (Lot No.)                      | Main Components                                                                                                                                                      | Manufacturer                               |
|------|-----------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------|
| ABU  | All-Bond Universal (1300008503)         | MDP phosphate monomer, bis-GMA, HEMA, ethanol, water, initiators                                                                                                     | Bisco Inc., Schaumburg, IL, USA            |
| ADU  | Adhese Universal (U49302)               | MDP, bis-GMA, HEMA, MCAP, D3MA, ethanol, water, initiator stabilizers, silicon dioxide                                                                               | Ivoclar Vivadent Schaan, Lichtenstein      |
| BML  | Bondmer Lightless (004067)              | Liquid A: phosphate monomer, bis-GMA, TEGDMA, HEMA, MTU-6, others<br>Liquid B: acetone, isopropanol, water, acryl borate catalyst, $\gamma$ -MPTES, peroxide, others | Tokuyama Dental Corp., Tokyo, Japan        |
| GPB  | G-Premio Bond (4G0011)                  | MDP, 4-MET, MEPS, BHT, acetone, dimethacrylate resins, initiators, water                                                                                             | GC Corp., Tokyo, Japan                     |
| SBU  | Scotchbond Universal (41256)            | MDP, HEMA, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane                                                                    | 3M ESPE Dental Products, St. Paul, MN, USA |
|      | Resin composite Clearfil AP-X (N416713) | bis-GMA, TEGDMA, silane barium glass filler, silane silica filler, silanated colloidal silica, CQ, pigments, others                                                  | Kuraray Noritake Dental, Tokyo, Japan      |

*Abbreviations: 4-MET, 4-methacryloxyethyl trimellitate; BHT, butylated hydroxytoluene; bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy) phenyl] propane; CQ, di-camphorquinone; D3MA, decandiol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; MCAP, methacrylated carboxylic acid polymer; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MEPS, methacryloyloxyalkyl thiophosphate methylmethacrylate; MTU-6, 6-methacryloyloxyhexyl-2-thiouracil-5-carboxylate; TEGDMA, triethyleneglycol dimethacrylate;  $\gamma$ -MPTES,  $\gamma$ -methacryloyloxypropyltriethoxysilane.*

Table 2: *Protocols for Bonding Procedures*

| Method Code   | Pretreatment for Adherent Surface                                                                                                                                                                                                                                                               |
|---------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| w/o           | Aluminablasting was not performed.                                                                                                                                                                                                                                                              |
| w             | Aluminablasting was performed on the adherent surface with 50 $\mu$ m Al <sub>2</sub> O <sub>3</sub> powder for five seconds at an angle of 45° and a distance of 10 mm with pressure of 0.25 MPa. The treated surface was rinsed with water for 15 s (three-way dental syringe) and air dried. |
| Adhesive Code | Adhesive Application Protocol                                                                                                                                                                                                                                                                   |
| ABU           | Adhesive applied to air-dried enamel or dentin surface (not desiccated for dentin) with rubbing action for 10-15 s per coat. No light cure between coats. Gentle stream of air applied over the liquid for at least 10 s. Light irradiation performed for 10 s.                                 |
| ADU           | Adhesive applied to the air-dried enamel or dentin surface with rubbing action for 20 s and then medium air pressure applied to surface for 5 s. Light irradiate for 10 s.                                                                                                                      |
| BML           | Adhesive applied to the air-dried enamel or dentin surface, and after 10 s, medium air pressure applied over the liquid adhesive for 5 s. No light irradiation.                                                                                                                                 |
| GPB           | Adhesive applied to air-dried enamel or dentin surface, and after 10 s, a strong stream of air applied over the liquid adhesive for 5 s or until the adhesive no longer moved and the solvent had completely evaporated. Light irradiate for 10 s.                                              |
| SBU           | Adhesive applied to air-dried enamel or dentin surface with rubbing action for 20 s medium air pressure applied to surface for 5 s. Adhesive light cured for 10 s.                                                                                                                              |

*Abbreviations: ABU, All-Bond Universal; ADU, Adhese Universal; BML, Bondmer Lightless; GPB, G-Premio Bond; SBU, Scotchbond Universal.*

grit silicon carbide (SiC) paper (Fuji Star Type DDC, Sankyo-Rikagaku, Saitama, Japan) to create flat enamel and dentin surfaces. Each tooth was then mounted in self-curing acrylic resin (Tray Resin II, Shofu, Kyoto, Japan) to expose the flattened area, and the enamel and dentin adherent surfaces were polished using a water coolant and a sequence of SiC polishing papers (up to #320 grit; Fuji Star Type DDC).

### Thermal Cycling and Shear Bond Strength Tests

The experimental protocol for tooth preparation is shown in Table 2. The prepared enamel and dentin specimens were divided into two groups depending on whether they received aluminablasting before the application of the universal adhesive or not. The aluminablasting was performed with a chairside sandblaster (MicroEtcher IIA, Zest Dental Solutions, Carlsbad, CA, USA). For each group, the adhesive agents were applied in accordance with each manufacturer's instructions for self-etch mode. Following application of the adhesive, bonded resin composite cylinders were built on the tooth surfaces by placing the resin composite in plastic molds (Bonding Mold Insert, internal diameter: 2.4 mm, height: 2.4 mm, Ultradent Products, South Jordan, UT, USA) and subjecting it to light irradiation for 30 seconds. The bonded specimens were then stored in distilled water at 37°C for 24 hours, following which the shear bond strength (SBS) was measured in half of the speci-

mens (24-hour group). The remaining half were treated with 30,000 thermal cycles between 5 and 55°C and a dwell time of 30 seconds prior to SBS measurement (thermal cycling [TC] group). The SBSs were measured using the notched edge SBS test, as described by ISO 29022.<sup>17</sup>

There were 40 experimental groups in total, arising from four variables: enamel or dentin, with or without aluminablasting, five different universal adhesives, and 24-hour or thermal cycling. Fifteen specimens were used for each group, as this number gives a statistical power of 0.95 with an  $\alpha = 0.05$  for typical SBS values, as calculated using statistical power analysis. A total of 600 specimens were used.

The bonded specimens were loaded to failure at 1.0 mm/min with an Ultradent shearing fixture (Test Base Clamp, Ultradent Products) using a universal testing machine (Type 5500R, Instron, Canton, MA, USA), and the SBS values (MPa) were obtained. Thereafter, the bonding sites on the tooth surfaces and resin composite cylinders were observed under an optical microscope (SZH-131, Olympus, Tokyo, Japan) at a magnification of 10× to determine the bond failure type. Based on the proportion of the substrate area (adhesive–resin composite–enamel or dentin) observed in the debonded cylinders and tooth-bonding sites, the bond failures were classified into 1) adhesive failure, 2) cohesive failure in composite resin, 3) cohesive failure in tooth, or 4) mixed failure (partially adhesive and partially cohesive).

### Surface Roughness Measurements

The treated surfaces with or without aluminablasting were observed under a three-dimensional laser scanning microscope (LSM; VK-8700; Keyence, Osaka, Japan) with the excitation light at a spectral maximum of 658 nm and the light intensity and amplification of the photomultiplier being kept constant during the observation period. The surface roughness (Ra) values were measured using the software (VK-Analyzer; Keyence) included with the LSM, and each region of measurement was 0.1 mm × 0.1 mm in size. The Ra was measured at three points as close to the center as possible in 10 specimens from each group, and the mean value was calculated for each group thereafter.

### Surface Free Energy Measurements

The surface free energy (SFE) of treated surfaces with or without aluminablasting was determined by measuring the contact angles formed on the surfaces

of three test liquids: 1-bromonaphthalene, diiodomethane, and distilled water. The SFE parameters of these liquids have been reported previously.<sup>18,19</sup> The contact angles were measured automatically using a contact angle meter (Drop Master DM 500, Kyowa Interface Science, Saitama, Japan) that had been connected to a charge-coupled device camera. The equilibrium contact angle ( $\theta$ ) was measured in 10 enamel and dentin specimens for each test liquid. Sessile drops (1.0  $\mu$ L in volume) of each liquid were dispensed at 23°C  $\pm$  1°C using a micropipette, and the SFE parameters of the solids were calculated based on the fundamental concepts of wetting. The Young-Dupré equation describes the adhesion between a solid (S) and liquid (L) that are in contact ( $W_{SL}$ ), the interfacial free energy between the solid and the liquid ( $\gamma_{SL}$ ), and the SFE of the liquid and solid ( $\gamma_L$  and  $\gamma_S$ , respectively), as follows:

$$W_{SL} = \gamma_L + \gamma_S - \gamma_{SL} = \gamma_L(1 + \cos\theta).$$

The Fowkes equation can be extended using the Kitazaki-Hata approach, as follows<sup>20</sup>:

$$\gamma_{SL} = \gamma_L + \gamma_S - 2(\gamma_L^d \gamma_S^d)^{1/2} - 2(\gamma_L^p \gamma_S^p)^{1/2} - 2(\gamma_L^h \gamma_S^h)^{1/2}$$

$$\gamma_L = \gamma_L^d + \gamma_L^p + \gamma_L^h, \quad \gamma_S = \gamma_S^d + \gamma_S^p + \gamma_S^h,$$

where  $\gamma^d$ ,  $\gamma^p$ , and  $\gamma^h$  are the dispersion force, polar (permanent and induced) force, and hydrogen-bonding force, respectively, and are components of the SFE ( $\gamma$ ).  $\theta$  values were determined for all three test liquids, and the surface-energy parameters of the treated surfaces were calculated according to the equations using an add-on software and the interface measurement and analysis system (FAMAS; Kyowa Interface Science). A statistical power analysis indicated that a minimum of nine samples was necessary for effective measurement of the Ra and SFE. Therefore, this experiment included a sample of 10 specimens, and post hoc power tests performed after data collection indicated that this would result in adequate statistical power.

### Scanning Electron Microscopy Observation

The aluminablasted surfaces and tooth/resin interfaces were observed using field-emission scanning electron microscopy (SEM; ERA-8800FE, Elionix, Tokyo, Japan). For ultrastructural morphological observations of the tooth-resin interfaces, the bonded specimens were embedded in epoxy resin and longitudinally sectioned using a diamond saw (Iso-

| Table 3: Influence of Aluminablasting on Enamel Bond Strength <sup>a</sup>                                                                                                                                                                                                                                                                                                                                                                           |                                  |                                   |                                    |                                    |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------|-----------------------------------|------------------------------------|------------------------------------|
|                                                                                                                                                                                                                                                                                                                                                                                                                                                      | 24-h                             |                                   | 30,000 TC                          |                                    |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                      | w/o                              | With                              | w/o                                | With                               |
| ABU                                                                                                                                                                                                                                                                                                                                                                                                                                                  | 27.9 (2.7) <sup>aa</sup> [100%]  | 26.8 (4.2) <sup>aa</sup> [96.1%]  | 27.6 (3.0) <sup>ba</sup> [98.9%]   | 25.8 (2.0) <sup>aa</sup> [92.5%]   |
| ADU                                                                                                                                                                                                                                                                                                                                                                                                                                                  | 26.8 (1.8) <sup>aa</sup> [100%]  | 26.9 (2.0) <sup>aa</sup> [100.3%] | 27.5 (3.5) <sup>ba</sup> [102.6%]  | 28.2 (3.1) <sup>aa</sup> [102.5%]  |
| BML                                                                                                                                                                                                                                                                                                                                                                                                                                                  | 29.0 (3.8) <sup>aa</sup> [100%]  | 30.8 (3.6) <sup>aa</sup> [106.2%] | 30.2 (4.2) <sup>abA</sup> [104.1%] | 28.8 (3.4) <sup>aa</sup> [99.3%]   |
| GPB                                                                                                                                                                                                                                                                                                                                                                                                                                                  | 28.2 (4.6) <sup>aaB</sup> [100%] | 27.0 (4.1) <sup>ab</sup> [95.6%]  | 32.9 (4.5) <sup>aa</sup> [116.7%]  | 29.5 (3.9) <sup>aaB</sup> [104.6%] |
| SBU                                                                                                                                                                                                                                                                                                                                                                                                                                                  | 28.1 (3.3) <sup>aa</sup> [100%]  | 27.2 (4.2) <sup>aa</sup> [96.8%]  | 27.4 (3.2) <sup>ba</sup> [97.5%]   | 26.6 (3.4) <sup>aa</sup> [94.7%]   |
| <sup>a</sup> Abbreviations: ABU, All-Bond Universal; ADU, Adhese Universal; BML, Bondmer Lightless; GPB, G-Premio Bond; SBU, Scotchbond Universal.<br><sup>a</sup> N=15, mean (SD) in MPa. The same lowercase letter in vertical columns indicates no difference at the 5% significance level. The same uppercase letter in horizontal rows indicates no difference at the 5% significance level. Values in parentheses indicate standard deviation. |                                  |                                   |                                    |                                    |

Met 1000, Precision Sectioning Saw, Buehler, Lake Bluff, IL, USA). The sectioned surfaces were polished to a high gloss using SiC papers (Fuji Star Type DDC) followed by diamond pastes up to a particle size of 0.25 µm (DP-Paste, Struers, Ballerup, Denmark). All SEM specimens were dehydrated in ascending grades of *tert*-butyl alcohol and then transferred to a critical-point dryer (Model ID-3, Elionix) for 30 minutes. The tooth-resin interfaces of the specimens were etched (EIS-200ER, Elionix) for 40 seconds using an argon-ion beam (accelerating voltage 1.0 kV, ion current density 0.4 mA/cm<sup>2</sup>) directed perpendicular to the polished surfaces. Finally, all SEM specimens were coated with a thin film of gold in a vacuum evaporator (Quick Coater, Type SC-701, Sanyu Denshi, Tokyo, Japan). All observations were performed under SEM at an operating voltage of 10 kV.

Energy Dispersive X-Ray Microanalysis

The elements present in the tooth surfaces with or without aluminablasting were analyzed using SEM/energy dispersive X-ray (EDX; GENESIS 2000, EDAX, Tokyo, Japan) at 20 kV and 2500× magnification. The measurements were conducted perpendicular to the prepared tooth surface, and the elemental content (wt%) of the carbon-coated (Quick Coater, Type SC-701, Sanyu Denshi) surface was measured in five specimens from each group. The measurements were carried out at three points as close to the center of the specimen as possible, and the mean value was calculated for each group thereafter. The analysis was performed using the ZAF correction method (atomic number, absorption, and fluorescence), based on standard-less correction.<sup>21</sup>

Statistical Analysis

As the data were normally distributed (determined by the Kolmogorov-Smirnov test), the tests used for

statistical analysis included the analysis of variance (ANOVA) followed by Tukey’s honestly significant difference (HSD) test at a significance level of 0.05. A three-way ANOVA along with a Tukey’s HSD test ( $\alpha=0.05$ ) was used for analysis of the SBS data, followed by multiple one-way ANOVA tests to compare the adhesives. A one-way ANOVA followed by Tukey’s HSD test was performed for all other variables. All statistical analyses were performed using the Sigma Plot software version 11.0 (SPSS Inc., Chicago, IL, USA).

RESULTS

Enamel Bond Strength

The effects of aluminablasting on the SBS of enamel are shown in Table 3. The three-way ANOVA test showed that the type of adhesive system significantly affected the SBS values ( $p<0.001$ ), while variations in the storage conditions (24-hour or TC,  $p=0.239$ ) and pretreatment methods (with or without aluminablasting,  $p=0.104$ ) did not. The three-way interaction between the factors was not statistically significant ( $p=0.728$ ), and the interaction between storage condition and adhesive system was the only pairwise interaction that exhibited statistical significance ( $p=0.03$ ; storage condition and pretreatment,  $p=0.277$ ; pretreatment and adhesive system,  $p=0.386$ ).

In the 24-hour group, no significant differences were observed between the five universal adhesives, regardless of their aluminablasting status. In the TC group, no significant differences were observed between the universal adhesives when aluminablasting was performed. However, GPB exhibited a significantly higher SBS value compared with all other adhesives, except BML, in the absence of aluminablasting. When the enamel SBS value (24 hours without aluminablasting) was defined as 100% for each tested adhesive, SBS values in 24 hours with aluminablasting ranged from 95.6% to 106.2%,

Table 4: Influence of Aluminablasting on Dentin Bond Strength<sup>a</sup>

|     | 24-h                             |                                   | 30,000 TC                         |                                   |
|-----|----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|
|     | w/o                              | With                              | w/o                               | With                              |
| ABU | 38.4 (1.3) <sup>aA</sup> [100%]  | 25.5 (3.6) <sup>abB</sup> [66.4%] | 37.5 (3.8) <sup>aA</sup> [97.7%]  | 24.5 (3.8) <sup>abB</sup> [63.8%] |
| ADU | 33.8 (3.2) <sup>bA</sup> [100%]  | 23.3 (2.7) <sup>bB</sup> [68.9%]  | 34.0 (3.1) <sup>aA</sup> [100.5%] | 19.3 (2.9) <sup>cC</sup> [57.1%]  |
| BML | 36.9 (3.9) <sup>abA</sup> [100%] | 27.7 (4.0) <sup>abB</sup> [75.1%] | 34.2 (3.0) <sup>aA</sup> [92.7%]  | 21.1 (2.3) <sup>bcC</sup> [57.2%] |
| GPB | 32.9 (3.0) <sup>bA</sup> [100%]  | 23.1 (2.6) <sup>bB</sup> [70.2%]  | 28.4 (2.3) <sup>bB</sup> [86.3%]  | 26.2 (4.0) <sup>abC</sup> [79.6%] |
| SBU | 36.2 (5.4) <sup>abA</sup> [100%] | 28.8 (3.8) <sup>abB</sup> [79.6%] | 35.2 (3.4) <sup>aA</sup> [97.2%]  | 24.3 (3.5) <sup>abB</sup> [67.1%] |

Abbreviations: ABU, All-Bond Universal; ADU, Adhese Universal; BML, Bondmer Lightless; GPB, G-Premio Bond; SBU, Scotchbond Universal.

<sup>a</sup> N=15, mean (SD) in MPa. The same lowercase letter in vertical columns indicates no difference at the 5% significance level. The same uppercase letter in horizontal rows indicates no difference at the 5% significance level. Values in parentheses indicate standard deviation.

in TC without aluminablasting ranged from 97.5% to 116.7%, and in TC with aluminablasting ranged from 92.5% to 104.6%.

### Dentin Bond Strength

The effects of aluminablasting on the SBS of dentin are shown in Table 4. The three-way ANOVA test showed that all of the factors examined significantly affected the SBS values ( $p < 0.001$ ). In addition, the three-way interaction between the factors was statistically significant ( $p = 0.002$ ). Although the interaction between the aluminablasting status and the adhesive system was significant ( $p < 0.001$ ), none of the other pairwise interactions were statistically significant (storage condition and pretreatment,  $p = 0.396$ ; storage condition and adhesive system,  $p = 0.124$ ).

In the 24-hour group, GPB and ADU tended to exhibit lower SBS values compared with the other adhesives, regardless of their aluminablasting status. In the TC group, the SBS values of the aluminablasting group varied with the adhesive, with ADU exhibiting a significantly lower SBS compared with the other adhesives. In contrast, no significant differences in the SBS values were observed between the adhesives, except for GPB, in the group without aluminablasting. When the dentin SBS value (24 hours without aluminablasting) was defined as 100% for each tested adhesive, 24 hour SBS values with aluminablasting ranged from 66.4% to 79.6%, in TC without aluminablasting ranged from 86.3% to 100.5%, and in TC with aluminablasting ranged from 57.2% to 79.6%. Most of the adhesives exhibited significantly lower SBS values with aluminablasting compared with the absence of aluminablasting, irrespective of their storage conditions.

### Failure Type of Debonded Specimens

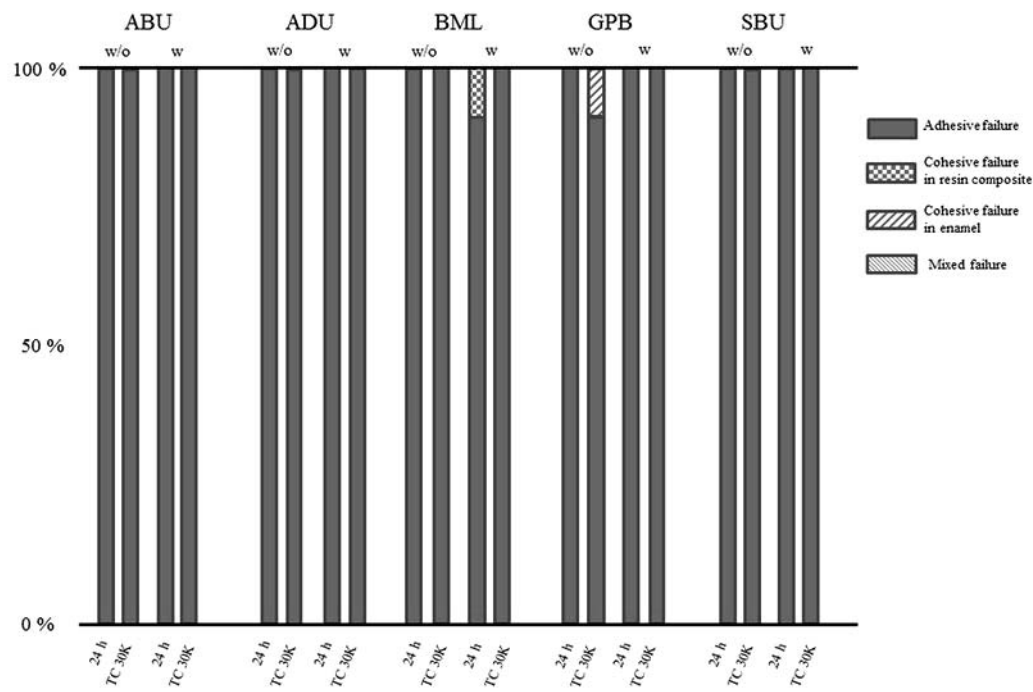
The frequencies of the different failure types are shown in Figures 1 and 2. All enamel debonded

specimens, except for two, exhibited adhesive failure regardless of the storage condition, aluminablasting status, and type of adhesive (Figure 1). However, the dentin debonded specimens exhibited a different trend compared with the enamel (Figure 2), with all debonded specimens that had undergone aluminablasting exhibiting adhesive failure patterns, regardless of the storage condition and type of adhesive. Conversely, adhesives in the specimens that did not undergo aluminablasting tended to mainly exhibit mixed and cohesive failures in the dentin.

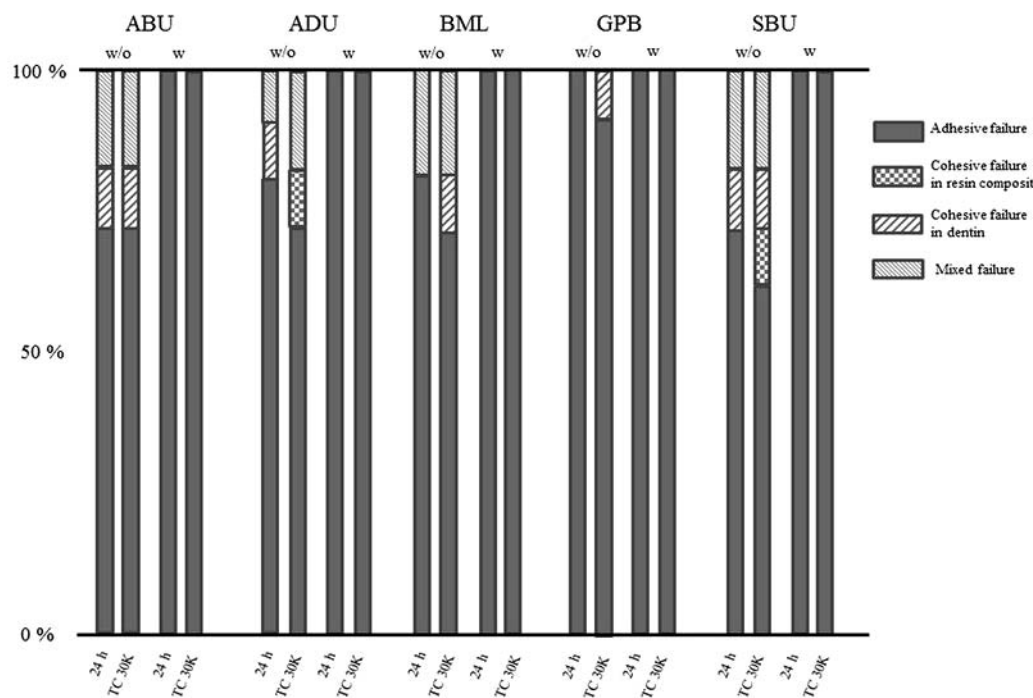
### Ra and SFE

The effects of aluminablasting on the Ra and SFE of the enamel and dentin surfaces are shown in Table 5. No significant differences were observed in the Ra values of the enamel and dentin surfaces that did not undergo aluminablasting. Conversely, the Ra values of specimens with aluminablasting were significantly higher compared with the specimens without aluminablasting, regardless of the tooth substrate. In particular, aluminablasted dentin surfaces exhibited a significantly higher Ra value compared with aluminablasted enamel surfaces, and it was almost four times rougher than enamel and dentin without aluminablasting.

In the aluminablasting group, the enamel specimens exhibited significantly higher total free energy ( $\gamma_s$ ) values compared with the dentin. When comparing groups, the enamel and dentin specimens with aluminablasting exhibited significantly higher  $\gamma_s$  values than those of specimens without aluminablasting. The dispersion force ( $\gamma_s^d$ ) was found to be similar in all groups, and no statistically significant differences were observed between the aluminablasting and no aluminablasting groups. However, the  $\gamma_s^p$  and  $\gamma_s^h$  values were seen to be affected by aluminablasting, with significantly higher values being observed in specimens that had undergone aluminablasting.



1 Failure mode analysis of the de-bonded enamel specimens



2 Failure mode analysis of the de-bonded dentin specimens

Figure 1. Failure mode analysis of the debonded enamel specimens.

Figure 2. Failure mode analysis of the debonded dentin specimens. ABU w, All Bond Universal with aluminablasting; ABU w/o, All Bond Universal without aluminablasting; ADU w, Adhese Universal with aluminablasting; ADU w/o, Adhese Universal without aluminablasting; BML w, Bondmer lightless with aluminablasting; BML w/o, Bondmer lightless without aluminablasting; GPB w, G-Premio with aluminablasting; GPB w/o, G-Premio without aluminablasting; SBU w, Scotchbond Universal with aluminablasting; SBU w/o, Scotchbond Universal without aluminablasting.

| Table 5: Influence of Aluminablasting on Ra and SFE of Adherent Surface <sup>a</sup>                                                                                                                        |                          |                          |                          |                          |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
|                                                                                                                                                                                                             | Enamel                   |                          | Dentin                   |                          |
|                                                                                                                                                                                                             | w/o                      | With                     | w/o                      | With                     |
| Ra                                                                                                                                                                                                          | 0.71 (0.02) <sup>c</sup> | 1.81 (0.05) <sup>b</sup> | 0.70 (0.02) <sup>c</sup> | 2.48 (0.10) <sup>a</sup> |
| $\gamma_s$                                                                                                                                                                                                  | 58.9 (3.9) <sup>c</sup>  | 76.5 (2.8) <sup>a</sup>  | 50.9 (3.8) <sup>d</sup>  | 68.6 (3.9) <sup>b</sup>  |
| $\gamma_s^d$                                                                                                                                                                                                | 41.8 (0.5) <sup>a</sup>  | 42.0 (0.7) <sup>a</sup>  | 41.4 (0.6) <sup>a</sup>  | 42.1 (0.8) <sup>a</sup>  |
| $\gamma_s^p$                                                                                                                                                                                                | 3.7 (1.7) <sup>b</sup>   | 6.7 (2.0) <sup>a</sup>   | 3.5 (1.2) <sup>b</sup>   | 6.2 (1.9) <sup>a</sup>   |
| $\gamma_s^h$                                                                                                                                                                                                | 13.4 (2.2) <sup>c</sup>  | 27.8 (1.2) <sup>a</sup>  | 6.0 (2.4) <sup>d</sup>   | 20.3 (3.5) <sup>b</sup>  |
| Abbreviations: Ra, surface roughness; SFE, surface free energy; $\gamma_s$ , total surface free energy; $\gamma_s^d$ , dispersion force; $\gamma_s^p$ , polar force; $\gamma_s^h$ , hydrogen bonding force. |                          |                          |                          |                          |
| <sup>a</sup> N=10, mean (SD) in MPa. The same uppercase letter in horizontal rows indicates no difference at the 5% significance level. Values in parentheses indicate standard deviation.                  |                          |                          |                          |                          |

blasting compared with those that had not, irrespective of the tooth substrate. The enamel of specimens with aluminablasting exhibited significantly higher  $\gamma_s^h$  values than the dentin.

SEM Observations

SEM images of the tooth surfaces with or without aluminablasting are shown in Figures 3 and 4. Scratches from the SiC papers and smear layers were obvious in the enamel and dentin of all specimens without aluminablasting (Figures 3A and 4A). In addition to the scratches from the SiC paper, the specimens that underwent aluminablasting also exhibited impact marks from the alumina particles, regardless of the substrate, and partial removal of the smear layer, thus exposing the

enamel surface. In addition, the smear layer of the dentin appeared to be compressed by aluminablasting.

Representative SEM images of the tooth-resin interfaces with or without aluminablasting are shown in Figure 5. The interface between the enamel substrate and adhesive in the specimens with aluminablasting appeared to be more irregular compared with those without aluminablasting (Figure 5A,B). A similar trend was observed in the dentin-resin interfaces (Figure 5C,D), although the irregularities in the dentin interfaces of specimens with aluminablasting were deeper than those seen in the enamel substrate. A high-density transitional layer was observed in the vicinity of the adhesive-dentin interface, regardless of the type of adhesive and the aluminablasting status.

EDX Microanalysis

Representative images of the elemental spectra for enamel and dentin surfaces of specimens with or without aluminablasting are shown in Figure 6, and their elemental compositions are shown in Table 6. The elements C, O, Na, Mg, P, and Ca were detected in the enamel and dentin specimens without aluminablasting (Figure 6A,C). In addition to C, O, Na, Mg, P, and Ca, the element Al was also detected in the enamel and dentin of samples with aluminablasting (Figure 6B,D). No significant differences in the wt% of Na, Mg, or Al were observed between the enamel and dentin of specimens with aluminablast-

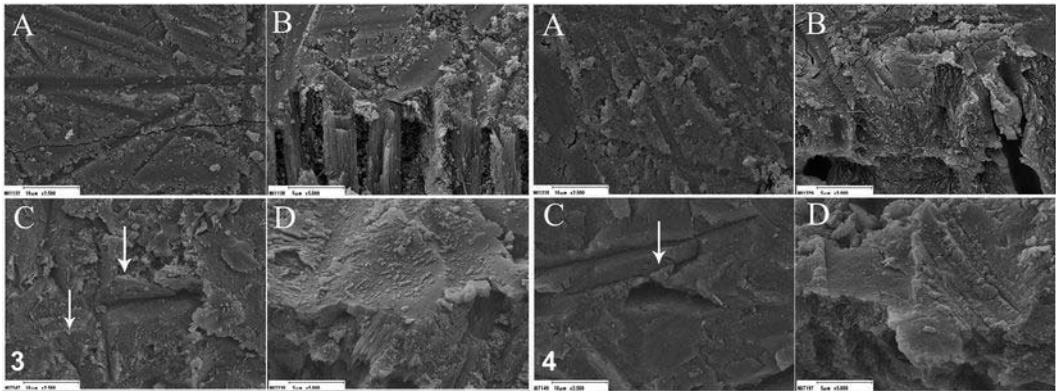


Figure 3. Representative scanning electron micrographs of enamel and dentin adherent surfaces. Arrows indicate evidence of impact marks from alumina particles. (A): Enamel adherent surface without aluminablasting (2500 $\times$ ). (B): A longitudinal section of the enamel adherent surface without aluminablasting (5000 $\times$ ). (C): Enamel adherent surface with aluminablasting (2500 $\times$ ). (D): A longitudinal section of the enamel adherent surface with aluminablasting (5000 $\times$ ).

Figure 4. Representative scanning electron micrographs of enamel and dentin adherent surfaces. Arrows indicate evidence of impact marks from alumina particles. (A): Dentin adherent surface without aluminablasting (2500 $\times$ ). (B): A longitudinal section of the dentin adherent surface without aluminablasting (5000 $\times$ ). (C): Dentin adherent surface with aluminablasting (2500 $\times$ ). (D): A longitudinal section of the dentin adherent surface with aluminablasting (5000 $\times$ ).

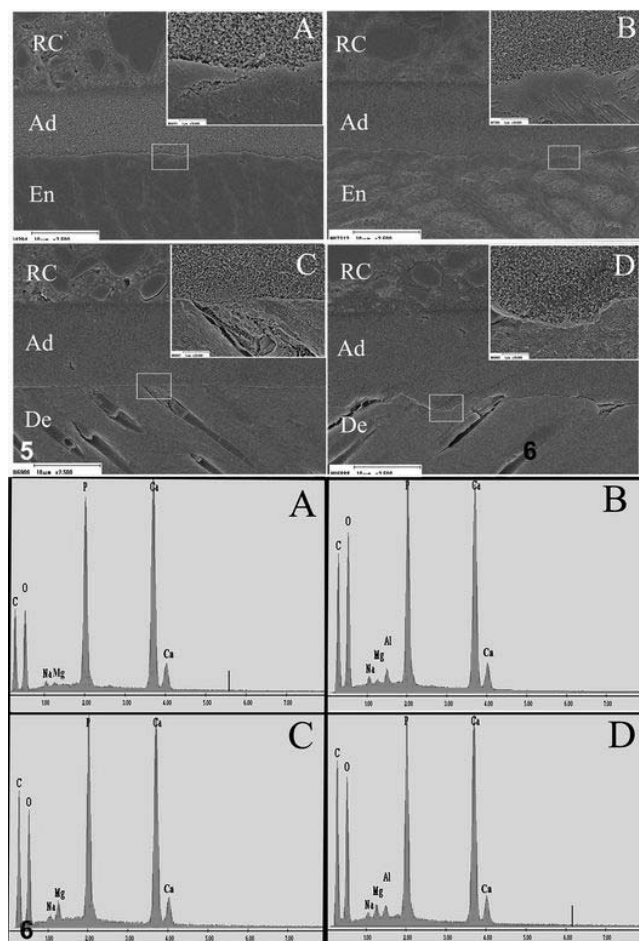


Figure 5. Representative scanning electron micrographs of the resin-tooth interfaces of Scotchbond Universal (SBU). The visible material is indicated by abbreviations: Ad, adhesive; En, enamel; De, dentin; RC, resin composite. (A): SBU and enamel without aluminablasting at magnification (a) 2500 $\times$  and (b) 20,000 $\times$ . (B): SBU and enamel with aluminablasting at magnification (a) 2500 $\times$  and (b) 20,000 $\times$ . (C): SBU and dentin without aluminablasting at magnification (a) 2500 $\times$  and (b) 20,000 $\times$ . (D): SBU and dentin with aluminablasting at magnification (a) 2500 $\times$  and (b) 20,000 $\times$ .

Figure 6. Representative images of element spectra for enamel and dentin surfaces with or without aluminablasting. C, carbon; O, oxygen; Na, sodium; Mg, magnesium; Al, aluminum; P, phosphorus; Ca, calcium. (A): Enamel adherent surface without aluminablasting. (B): Enamel adherent surface with aluminablasting. (C): Dentin adherent surface without aluminablasting. (D): Dentin adherent surface with aluminablasting.

ing. In addition, the wt% of oxygen in samples with aluminablasting was significantly higher than that of samples without aluminablasting.

## DISCUSSION

Many studies have investigated optimal repair techniques with a focus on bonding to aged restorations.<sup>2-4</sup> However, little information about the bonding effectiveness when aluminablasting exposes

sound tooth substrate during repair procedures is available. The purpose of this study was to determine the bonding effectiveness of universal adhesives to different tooth substrates after alumina-blasting from several perspectives.

In the group without aluminablasting, none of the adhesives exhibited a reduction in enamel or dentin bond strength after thermal cycling. This was consistent with previous *in vitro* studies examining the bond durability of universal adhesives.<sup>22,23</sup> In particular, Suzuki and others<sup>23</sup> reported that no reduction of enamel bond strengths in self-etch mode was observed in the degradation condition groups (30,000 thermal cycles and two-year water storage) when compared with the immediate groups (24-hour water storage). These findings suggest that adhesion between the enamel substrate and universal adhesives remains stable once the bond has been established. On the other hand, although aluminablasting had a negligible effect on the bonds to enamel, dentin bond strength tests showed that aluminablasting had a negative impact in both groups (24 hour and TC), regardless of the type of universal adhesive. Therefore, the first null hypothesis that aluminablasting would not affect the bond performance of universal adhesives was rejected only for dentin.

In the group without aluminablasting, none of the adhesives exhibited a reduction in enamel and dentin bond strength after thermal cycling. This was consistent with previous *in vitro* studies examining the bond durability of universal adhesives.<sup>22,23</sup> In particular, Suzuki and others<sup>23</sup> reported that no reduction of enamel bond strengths in self-etch mode was observed in degradation condition groups (30,000 thermal cycles and two-year water storage) when compared with the immediate groups (24-hour water storage). These findings suggest that adhesion between the enamel substrate and universal adhesives remain stable once the qualitative bond has been established.

To obtain a rough and clean surface on restorations, aluminablasting is commonly used in both dental laboratories and clinics.<sup>11,13-16</sup> In addition, aluminablasting is used in orthodontic treatment to create roughness on the enamel surface and increase the mechanical interlocking effect for bracket bonding on occasion.<sup>24</sup> Patcas and others<sup>25</sup> reported that aluminablasting of intact enamel enhanced the surface roughness, and it could produce a rougher surface than phosphoric acid etching to enamel. In the results for changes in surface characteristics after aluminablasting, the aluminablasted enamel

Table 6: Influence of Aluminablasting on Elemental Composition (wt%) of Adherent Surface<sup>a</sup>

|            | Enamel                   |                          | Dentin                   |                           |
|------------|--------------------------|--------------------------|--------------------------|---------------------------|
|            | w/o                      | With                     | w/o                      | With                      |
| Carbon     | 32.8 (0.8) <sup>bb</sup> | 32.4 (1.2) <sup>bb</sup> | 37.0 (1.8) <sup>aA</sup> | 38.2 (1.0) <sup>aA</sup>  |
| Oxygen     | 36.1 (0.9) <sup>aB</sup> | 38.0 (1.1) <sup>aA</sup> | 32.3 (1.8) <sup>bC</sup> | 37.3 (0.5) <sup>aAB</sup> |
| Sodium     | 1.1 (0.3) <sup>eA</sup>  | 1.1 (0.1) <sup>eA</sup>  | 0.8 (0.2) <sup>eB</sup>  | 0.8 (0.1) <sup>dB</sup>   |
| Magnesium  | 0.6 (0.1) <sup>eA</sup>  | 0.6 (0.1) <sup>eA</sup>  | 1.1 (0.2) <sup>eB</sup>  | 1.1 (0.1) <sup>dB</sup>   |
| Aluminum   | NA                       | 0.9 (0.1) <sup>eA</sup>  | NA                       | 1.0 (0.1) <sup>dA</sup>   |
| Phosphorus | 11.6 (0.5) <sup>dA</sup> | 10.9 (0.5) <sup>dA</sup> | 11.3 (0.9) <sup>dA</sup> | 9.0 (0.2) <sup>cB</sup>   |
| Calcium    | 17.7 (1.0) <sup>cA</sup> | 16.3 (1.0) <sup>cA</sup> | 17.4 (2.8) <sup>cA</sup> | 12.7 (0.4b) <sup>bb</sup> |

<sup>a</sup> N=10, mean (SD) in wt%. The same lowercase letter in vertical columns indicates no difference at the 5% significance level. The same uppercase letter in horizontal rows indicates no difference at the 5% significance level. Values in parentheses indicate standard deviation.

and dentin exhibited twofold and threefold increases in Ra values, respectively, when compared with the specimens that did not undergo aluminablasting. In addition, the total SFE of the aluminablasted surfaces of the specimens was 30% higher than the surfaces of specimens that did not undergo aluminablasting. Therefore, the second null hypothesis, that the surface characteristics of the teeth would not differ irrespective of whether they had been aluminablasted or not, was rejected.

Changes in surface roughness are only to be expected, but the changes in SFE may need more explanation. The  $\gamma_s^h$  parameter represents the water and hydroxyl components of the substrate, while  $\gamma_s^p$  is thought to be dependent on electric and metallic interactions in addition to dipolar interactions.<sup>18,19</sup> The increased  $\gamma_s$  values of the enamel and dentin specimens that underwent aluminablasting can be attributed to the enhanced surface cleanliness and the presence of residual alumina particles, which are highly polar. This speculation was supported by SEM observations and SEM/EDX analysis, which revealed that Al was present in the enamel and dentin of only those specimens that had undergone aluminablasting. These results were consistent with a previous study examining orthodontic bracket bonding that reported the presence of residual alumina on the aluminablasted enamel.<sup>25</sup> Furthermore, the elemental composition of oxygen was also seen to increase in the aluminablasted surfaces when compared with the surfaces of the specimens that did not undergo aluminablasting, irrespective of the tooth substrate considered. The additional Al and O are presumably derived from the blasting alumina particles.

The findings of the current study suggest that aluminablasting affected the bond strength and surface characteristics of enamel and dentin substrates differently. Therefore, the third null hypothesis,

that the effect of aluminablasting would not differ between enamel and dentin, was rejected. In general, larger Ra values and higher SFE are thought to be advantageous for bond performance because of enhanced wettability.<sup>19</sup> However, the presence of a rough surface and higher SFE by aluminablasting did not contribute to enamel and dentin bond performance; instead, the dentin specimens that had undergone aluminablasting exhibited a reduction in bond strength. Soares and others<sup>26</sup> investigated the effect of pretreatments on the dentin bond strength of two self-etching adhesives and reported that the aluminablasting group showed significantly lower micro tensile bond strength values than the no-treatment group (control) in both self-etch adhesives. However, they claimed that the reasons for the dentin bond strength reduction in self-etch adhesives were unclear. Integrating our laboratory results and those of the previous investigation, we may be able to explain this phenomenon. The dentin bond strength reduction after aluminablasting and the different trends observed with regard to the enamel and dentin bond strengths could be explained by the physical properties of the adherent substrates and the smear layer characteristics. Dentin substrate might suffer more damage than enamel substrate because of lower surface hardness and elastic modulus.<sup>27</sup> A previous study reported that air-powder polishing of dentin surfaces using sodium bicarbonate powder increased damage to the dentin substructure and reduced bond performance of two-step and single-step self-etching adhesive systems,<sup>28</sup> leading to the development of a prophylactic polishing powder.<sup>29,30</sup> In addition, since alumina particles are harder than sodium bicarbonate powder, aluminablasting may cause much more damage not only to dentin but also to an enamel substrate, such as micro cracks. The dentin smear layer is composed of disorganized organic debris with HAp minerals, while the enamel smear layer is

highly porous.<sup>31</sup> The SEM observations showed that the dentin smear layer was compressed after aluminablasting (Figure 4C,D). This layer contains collagen fragments that can block the penetration of adhesive functional monomers, and this may be related to the fact that the intermolecular spacing of collagen (1.3 nm) is smaller than the size of the functional monomers (approximately 2 nm).<sup>32</sup> In the case of self-etch universal adhesives, penetration of the resin monomers into the smear layer and demineralization of the tooth surface are essential for chemical bonding with HAp. Tamura and others<sup>33</sup> investigated how air-powder polishing influences bonding between dentin and universal adhesives. They suggested that the presence of residual sodium bicarbonate powder on dentin surfaces leads to chemical and/or mechanical changes to collagen fibrils and prevention of adhesive penetration into dentin. It can be speculated that although a different blasting material is used in this study, the same situation might occur. Moreover, a previous study comparing the enamel and dentin bond durability of self-etch adhesives with different smear layers showed that the dentin was more susceptible to the condition of the smear layer condition than the enamel.<sup>34</sup>

Therefore, it can be speculated from the integrated results that the compressed dentin smear layer and embedded alumina particles may interfere with the penetration of the resin monomer and interaction with the functional monomer of universal adhesives. These may lead to lower bond strength because the dentin HAp has a higher affinity for the functional monomer than enamel.<sup>35,36</sup>

Changes in the elemental composition after aluminablasting showed significantly lower wt% of P and Ca in dentin surfaces than enamel surfaces and dentin surfaces without aluminablasting. Although the mineral content of dentin was lower than that of enamel, the dentin HAp has a high affinity for the functional monomer, thus creating greater nano-layering between them.<sup>35,36</sup> Therefore, it might be inferred that the lower concentration of P and Ca and the consumption of functional monomers by residual alumina particles in the aluminablasted dentin surfaces may lead to a weaker chemical bond compared with those in the dentin surfaces of specimens that did not undergo aluminablasting. Notably, all debonded specimens of aluminablasted dentin exhibited adhesive failure patterns, and this was in contrast to the specimens that did not undergo aluminablasting.

Regarding clinical practice, the results of this study suggest that aluminablasting should normally not be used with universal adhesives, because dentin bond performance was noticeably affected. Further, the bond strength and surface characteristic tests were performed using flat specimens in this study. However, when considering the clinical situation, the cavity when repairing a restoration is likely to have a complex configuration. It is probable that many more alumina particles may remain in a cavity than on a flat specimen and would be difficult to remove. There is a possibility that remnant alumina particles might be an inhibiting factor for immediate and long-term bond durability of dentin. In addition, although the enamel bond effectiveness did not change in response to aluminablasting, there are still reasons to avoid it when repairing aged restorations surrounded by sound enamel structures, because enamel loss after aluminablasting is much higher than after phosphoric acid etching.<sup>25</sup> Wendler and others<sup>13</sup> reported that no significant difference in Ra value was observed between aluminablasting and bur roughening. Therefore, instead of aluminablasting the tooth structure, it may be better to roughen the surface of the aged restoration with burs, avoiding damage to enamel as far as possible, and use a suitable application primer.

The results of this study suggest that alumina-blasting should not be used when universal adhesives are used to make a repair restoration *in situ*. Thus, further work to determine the bonding characteristics of universal adhesives to bur-roughened aged restorations would be valuable. The flexibility of universal adhesives (used with or without phosphoric acid etching, bonding to multiple substrates), gives reason to hope that an effective and conservative protocol can be developed.

## CONCLUSION

In conclusion, most universal adhesives in self-etch mode with aluminablasting exhibited lower dentin SBS values compared with the specimens without aluminablasting. Moreover, the enamel and dentin substrates exhibited similar Ra and SFE results in both groups. These results suggest that although aluminablasting of the tooth surface is thought to be effective for modification of the adherent surface, it may not enhance enamel bond performance and may also adversely affect the dentin bond effectiveness of the universal adhesives tested in this study. Therefore, when considering repair techniques using aluminablasting, we should take into account both the interaction between functional monomers and

HAp and the infiltration capability of resin monomers beyond the smear layer.

### Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interests of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 7 October 2018)

### REFERENCES

1. Lynch CD, Opdam NJ, Hickel R, Brunton PA, Gurgan S, Kakaboura A, Shearer AC, Vanherle G, & Wilson NHF (2014) Guidance on posterior resin composites: Academy of Operative Dentistry—European section *Journal of Dentistry* **42**(4) 377-383, <https://doi.org/10.1016/j.jdent.2014.01.009>.
2. Loomans BAC, Cardoso MV, Roeters FJM, Opdam NJM, De Munck J, Huysmans MC, & Van Meerbeek B (2011) Is there one optimal repair technique for all composites? *Dental Materials* **27**(7) 701-709, <https://doi.org/10.1016/j.dental.2011.03.013>.
3. Hickel R, Brühshaver K, & Ilie N (2013) Repair of restorations—criteria for decision making and clinical recommendations *Dental Materials* **29**(1) 28-50, <https://doi.org/10.1016/j.dental.2012.07.006>.
4. Özcan M (2015) Intraoral repair protocol for chipping or fracture of veneering ceramic in zirconia fixed dental prostheses *Journal of Adhesive Dentistry* **17**(2) 189-190.
5. Yoshida T, Miyazaki M, Hirohata N, & Moore BK (2005) Influence of metal conditioner contamination of the dentin surface on bond strengths of dentin adhesive systems using self-etching primers *Operative Dentistry* **30**(3) 359-367.
6. Mamane T, Takahashi M, Nakajima M, Foxton RM, & Tagami J (2015) Initial and long-term bond strengths of one-step self-etch adhesives with silane coupling agent to enamel-dentin-composite in combined situation *Dental Materials Journal* **34**(5) 663-670, <https://doi.org/10.4012/dmj.2015-050>.
7. da Rosa WL, Piva E, & da Silva AF (2015) Bond strength of universal adhesives: A systematic review and meta-analysis *Journal of Dentistry* **43**(7) 765-776, <https://doi.org/10.1016/j.jdent.2015.04.003>.
8. Takamizawa T, Barkmeier WW, Tsujimoto A, Berry TP, Watanabe H, Erickson RL, Latta MA, & Miyazaki M (2016) Influence of different etching modes on bond strength and fatigue strength to dentin using universal adhesive systems *Dental Materials* **32**(2) e9-e21, <https://doi.org/10.1016/j.dental.2015.11.005>.
9. Suzuki T, Takamizawa T, Barkmeier WW, Tsujimoto A, Endo H, Erickson RL, Latta MA, & Miyazaki M (2016) Influence of etching mode on enamel bond durability of universal adhesive systems *Operative Dentistry* **41**(5) 520-530, <https://doi.org/10.2341/15-347-L>.
10. Tsujimoto A, Barkmeier WW, Takamizawa T, Wilwerding TM, Latta MA, & Miyazaki M (2017) Interfacial characteristics and bond durability of universal adhesives to various substrates *Operative Dentistry* **42**(2) e59-e70, <https://doi.org/10.2341/15-353-L>.
11. Al Jabbari YS, Zinelis S, & Eliades G (2012) Effect of sandblasting conditions on alumina retention in representative dental alloys *Dental Materials Journal* **31**(2) 249-255, <https://doi.org/10.4012/dmj.2011-210>.
12. Bühler J, Amato M, Weiger R, & Walter C (2016) A systematic review on the effects of air polishing devices on oral tissue *International Journal of Dental Hygiene* **14**(1) 15-28, <https://doi.org/10.1111/idh.12120>.
13. Wendler M, Belli R, Panzer R, Skibbe D, Petschelt A, & Lohbauer U (2016) Repair bond strength of aged resin composites after different surface and bonding treatments *Materials* **9**(7) 547-559, <https://doi.org/10.3390/ma9070547>.
14. Higashi M, Matsumoto M, Kawaguchi A, Miura J, Minamino T, Kabetani T, Takeshige F, Mine A, & Yatani H (2016) Bonding effectiveness of self-adhesive and conventional-type adhesive resin cements to CAD/CAM resin blocks. Part 1: Effects of sandblasting and silanization *Dental Materials Journal* **35**(1) 21-28, <https://doi.org/10.4012/dmj.2015-234>.
15. Yoshida K (2018) Influence of cleaning methods on resin bonding to saliva-contaminated zirconia *Journal of Esthetic and Restorative Dentistry* **30**(3) 259-264, <https://doi.org/10.1111/jerd.12369>.
16. Ersu B, Yuzugullu B, Yazici AR, & Canay S (2009) Surface roughness and bond strengths of glass-infiltrated alumina-ceramics prepared using various surface treatments *Journal of Dentistry* **37**(11) 848-856, <https://doi.org/10.1016/j.jdent.2009.06.017>.
17. ISO-Standards (2013) ISO 29022 2013 Dentistry-Adhesion-Notched-Edge Shear Bond Strength Test *Geneve: International Organization for Standardization 1st edition* 1-12.
18. Imai A, Takamizawa T, Sai K, Tsujimoto A, Nojiri K, Endo H, Barkmeier WW, Latta MA, & Miyazaki M (2017) Influence of application method on surface free-energy and bond strength of universal adhesive systems to enamel *European Journal of Oral Science* **125**(5) 385-395, <https://doi.org/10.1111/eos.12361>.
19. Sai K, Takamizawa T, Imai A, Tsujimoto A, Ishii R, Barkmeier WW, Latta MA, & Miyazaki M (2018) Influence of application time and etching mode of universal adhesives on enamel adhesion *Journal of Adhesive Dentistry* **20**(1) 65-77, <https://doi.org/10.3290/j.jad.a39913>.
20. Hata T, Kitazato Y, & Saito T (1987) Estimation of the surface energy of polymer solids *Journal of Adhesion* **21**(3-4) 177-194.
21. Hosoya Y, Tadokoro K, Inoue T, Miyazaki M, & Tay FR (2013) Effect of SI-R20401 to remineralize artificial incipient enamel lesions in primary teeth *Journal of Oral Science* **55**(4) 301-310, <https://doi.org/10.2334/josnurd.55.301>.
22. Sai K, Shimamura Y, Takamizawa T, Tsujimoto A, Imai A, Endo H, Barkmeier WW, Latta MA, & Miyazaki M (2016) Influence of degradation conditions on dentin bonding durability of three universal adhesives *Journal of Dentistry* **54** 56-61, <https://doi.org/10.1016/j.jdent.2016.09.004>.

23. Suzuki S, Takamizawa T, Imai A, Tsujimoto A, Sai K, Takimoto M, Barkmeier WW, Latta MA, & Miyazaki M (2018) Bond durability of universal adhesive to bovine enamel using self-etch mode *Clinical Oral Investigations* **22**(3) 1113-1122, <https://doi.org/10.1007/s00784-017-2196-x>.
24. Baumgartner S, Koletsi D, Verma C, & Eliades T (2017) The effect of enamel sandblasting on enhancing bond strength of orthodontic brackets: A systematic review and meta-analysis *Journal of Adhesive Dentistry* **19**(6) 463-473, <https://doi.org/10.3290/j.jad.a39279>.
25. Patcas R, Zinelis S, Eliades G, & Eliades T (2015) Surface and interfacial analysis of sandblasted and acid-etched enamel for bonding orthodontic adhesives *American Journal of Orthodontics Dentofacial Orthopedics* **147**(Supplement 4) S64-S75, <https://doi.org/10.1016/j.ajodo.2015.01.014>.
26. Soares CJ, Castro CG, Santos Filho PCF, & de Mota AS (2007) Effect of previous treatments on bond strength to two self-etching adhesive systems to dental substrate *Journal of Adhesive Dentistry* **9**(3) 291-296.
27. Powers JM & Wataha JC (2013) *Dental Materials: Properties and Manipulation 10th edition* Mosby, St Louis MO.
28. Frankenberger R, Lohbauer U, Tay FR, Teschner M, & Nikolaenko SA (2007) The effect of different air-polishing powders on dentin bonding *Journal of Adhesive Dentistry* **9**(4) 381-389.
29. Tada K, Kakuta K, Ogura H, & Sato S (2010) Effect of particle diameter on air polishing of dentin surfaces *Odontology* **98**(1) 31-36, <https://doi.org/10.1007/s10266-009-0113-8>.
30. Sahrman P, Ronay V, Schmidlin PR, Attin T, & Paqué F (2014) Three-dimensional defect evaluation of air polish-  
ing on extracted human roots *Journal of Periodontology* **85**(8) 1107-1114, <https://doi.org/10.1902/jop.2014.130629>.
31. Thanatvarakorn O, Nakajima M, Prasansuttiporn T, Ichinose S, Foxton RM, & Tagami J (2014) Effect of smear layer deproteinizing on resin-dentine interface with self-etch adhesive, *Journal of Dentistry* **42**(3) 298-304, <https://doi.org/10.1016/j.jdent.2013.11.026>.
32. Bertassoni LE, Orgel JP, Antipova O, & Swain MV (2012) The dentin organic matrix-limitations of restorative dentistry hidden on the nanometer scale *Acta Biomaterialia* **8**(7) 2419-2433, <https://doi.org/10.1016/j.actbio.2012.02.022>.
33. Tamura Y, Takamizawa T, Shimamura Y, Akiba S, Yabuki C, Imai A, Tsujimoto A, Kurokawa H, & Miyazaki M (2017) Influence of air-powder polishing on bond strength and surface-free energy of universal adhesive systems *Dental Materials Journal* **36**(6) 762-769, <https://doi.org/10.4012/dmj.2016-185>.
34. Takamizawa T, Barkmeier WW, Sai K, Tsujimoto A, Imai A, Erickson RL, Latta MA, & Miyazaki M (2018) Influence of different smear layers on bond durability of self-etch adhesives *Dental Materials* **34**(2) 246-259, <https://doi.org/10.1016/j.dental.2017.11.002>.
35. Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Irie M, Ogawa T, Van Landuyt KL, Osaka A, Suzuki K, Minagi S, & Van Meerbeek B (2011) Nanolayering of phosphoric acid ester monomer on enamel and dentin *Acta Biomaterialia* **7**(8) 3187-3195, <https://doi.org/10.1016/j.actbio.2011.04.026>.
36. Yaguchi T (2017) Layering mechanism of MDP-Ca salt produced in demineralization of enamel and dentin apatite *Dental Materials* **33**(1) 23-32, <https://doi.org/10.1016/j.dental.2016.09.037>.

# Effect of pH Cycling Followed by Simulated Toothbrushing on the Surface Roughness and Bacterial Adhesion of Bulk-fill Composite Resins

DC Somacal • FB Manfroí • MSG Monteiro • SD Oliveira  
HR Bittencourt • GA Borges • AM Spohr

## Clinical Relevance

Toothbrushing slightly increases the surface degradation of conventional and bulk-fill resins initially caused by a cariogenic challenge. However, these superficial changes do not suggest significant differences in bacterial adhesion on the composite resin surfaces.

## SUMMARY

The aim was to evaluate, *in vitro*, quantitatively and qualitatively, the effect of pH cycling and simulated toothbrushing on surface roughness (Ra) and bacterial adhesion (Cn) of bulk-fill composite resins. Thirty specimens of each composite resin, 5 mm wide and 4 mm high, were obtained: group 1 (control): Filtek Z250 (Z250); group 2: Filtek Bulk-Fill (FTK);

group 3: Tetric N-Ceram Bulk-Fill (TTC); and group 4: Aura Bulk-Fill (AUR). After 24 hours, the specimens were polished and then alternated with demineralization/remineralization solutions for 15 cycles of 24 hours each at 37°C. Then the specimens were submitted to simulated toothbrushing. The Ra and Cn measurements were quantitatively analyzed in three stages: after polishing (Ra0 and Cn0), after pH

Deise Caren Somacal, DDS, MS, Department of Restorative Dentistry, Pontifical Catholic University of Rio Grande do Sul, Porto Alegre, Brazil

Fernanda Borguetti Manfroí, DDS, MS, PhD, Department of Restorative Dentistry, Pontifical Catholic University of Rio Grande do Sul, Porto Alegre, Brazil

Marina Silveira Gregis Monteiro, graduate student, Department of Immunology and Microbiology, Pontifical Catholic University of Rio Grande do Sul, Porto Alegre, Brazil

Sílvia Dias de Oliveira, MS, PhD, Department of Immunology and Microbiology, Pontifical Catholic University of Rio Grande do Sul, Porto Alegre, Brazil

Hélio Radke Bittencourt, MS, PhD, Department of Statistics, Pontifical Catholic University of Rio Grande do Sul, Porto Alegre, Brazil

Gilberto Antonio Borges, DDS, MS, PhD, Department of Restorative Dentistry, Uberaba University, Uberaba, Brazil

\*Ana Maria Spohr, PhD, Department of Dental Materials, Pontifical Catholic University of Rio Grande do Sul, Porto Alegre, Brazil

\*Corresponding author: Avenida Ipiranga, 6681, Porto Alegre, Rio Grande do Sul 90619-900, Brazil; e-mail: anaspohr@terra.com.br

<https://doi.org/10.2341/19-012-L>

cycling (Ra1 and Cn1), and after simulated toothbrushing (Ra2 and Cn2). The Ra values were submitted to two-way analysis of variance, followed by the Tukey test ( $\alpha=0.05$ ). The Kruskal-Wallis test, followed by multiple comparisons, was applied for Cn analysis. Surface topography and bacterial adhesion were observed by scanning electron microscopy (SEM). Z250, FTK, and TTC showed no significant change in Ra regardless of the treatment performed; AUR obtained increased Ra at Ra2 ( $p<0.05$ ). FTK differed from the others at Cn0 and Cn1 ( $p<0.05$ ). At Cn2, there was no difference among the composite resins. SEM images showed the exposure of fillers and microcavities at Ra1 and Ra2. There was greater bacterial adhesion at Cn1 for Z250 and FTK. It was concluded that the pH cycling caused surface degradation of all composite resins, which was potentiated by simulated toothbrushing. However, only AUR presented an increased Ra. Bacterial adhesion occurred on all composite resins after pH cycling; however, after simulated toothbrushing, adhesion of dispersed bacteria was similar for all the composite resin groups.

## INTRODUCTION

Composite resins are widely used in restorative dentistry to restore tooth structure lost due to carious lesions, trauma, or abrasive processes and for aesthetic reasons.<sup>1</sup> The desire to facilitate clinical practice resulted in bulk-fill composite resins. These materials are suitable for insertion in a 4- or 5-mm bulk placement, depending on the brand, without prolonged polymerization time. Therefore, bulk-fill composite resins eliminate incremental techniques and potentially reduce the amount of work and clinical steps required.<sup>2</sup>

One of the factors related to the success of composite resin restoration is the capacity of the material to be resistant to degradation in the oral environment.<sup>3</sup> An imbalance between the demineralization and remineralization process, associated with the presence of cariogenic bacteria, salivary dysfunction, and ingestion of fermentable carbohydrates, favor the occurrence of dental caries.<sup>4</sup> These chemical characteristics of the oral cavity may influence the properties of restorative materials.<sup>5,6</sup>

The study of surface degradation of the materials is important since it can facilitate the accumulation of bacterial plaque.<sup>7</sup> An association between surface roughness and bacterial adhesion was found to exist

for resinous materials.<sup>8</sup> Greater bacterial adhesion was observed in direct composite resin restorations when compared with indirect composite resin restorations<sup>9</sup> and glass-ionomer cements.<sup>10</sup> This susceptibility increases the patient's risk of secondary caries adjacent to the restoration margins in addition to the formation of biofilm, which contributes to the chemical and mechanical degradation of composite resin restorations.<sup>11</sup>

Consequently, there is a necessity to evaluate restorative materials under the challenging chemical conditions that occur in the oral cavity, such as pH cycling. In addition, it is important to evaluate pH cycling in association with the mechanical process of toothbrushing since it is a common daily oral hygiene habit.<sup>12</sup> *In vitro* studies used the pH-cycling model to mimic the effect of oral conditions on the surface of restorative materials.<sup>13-17</sup> This model is characterized by the dynamics between periods of demineralization and remineralization, simulating a cariogenic challenge<sup>17,18</sup> proposed for laboratory studies by Featherstone and others<sup>19</sup> and modified by Serra and Cury.<sup>20</sup> On this research line, studies examined restorative materials such as glass ionomers and polyacid-modified resins throughout the pH cycling.<sup>13,15,16</sup> Other studies submitted composite resins to pH cycling and analyzed their effect on the materials' surface after simulated toothbrushing.<sup>14,17</sup> Modification of surface roughness occurred when composite resin was submerged in different acidic solutions, with pH values ranging between 6.6 and 2.5.<sup>21</sup> However, no studies were found in the consulted literature regarding the effect of pH cycling followed by simulated toothbrushing on bulk-fill composite resins.

Therefore, this study aimed to evaluate the effect of pH cycling on surface roughness and biofilm formation of three bulk-fill composite resins in comparison with a conventional composite resin, with and without simulated toothbrushing. Complementary analysis using scanning electron microscopy (SEM) to evaluate surface topography and biofilm formation was also performed. The study was conducted under the following null hypotheses: pH cycling followed by simulated toothbrushing does not significantly influence 1) surface roughness or 2) biofilm formation on the composite resins.

## METHODS AND MATERIALS

### Obtaining Composite Resin Specimens

Three bulk-fill composite resins and a conventional composite resin were used (Table 1). The composite

Table 1: Composite Resins Used in the Study

| Material/Shade                                 | Filler Content (% Wt/Vol)                                                     | Organic Matrix                 | Manufacturer                             | Lot Number |
|------------------------------------------------|-------------------------------------------------------------------------------|--------------------------------|------------------------------------------|------------|
| Filtek Z250, Microhybrid/A2 (Z250)             | Zirconia/silica, 0.01 $\mu\text{m}$ to 3.50 $\mu\text{m}$ (84.5/60)           | TEGDMA, Bis-GMA, UDMA, Bis-EMA | 3M ESPE (St Paul, MN, USA)               | 1628100449 |
| Filtek Bulk Fill, Nanofiller/A2 (FTK)          | Zirconia, 4 to 11 nm; silica, 20 nm; ytterbium fluoride, 100 nm (76.5/58.4)   | AUDMA, AFM, DDDMA, UDMA        | 3M ESPE                                  | 1632700708 |
| Tetric N-Ceram Bulk Fill, Nanohybrid/IVB (TTC) | Barium/silica/aluminum; ytterbium fluoride, 0.04 to 3 $\mu\text{m}$ , (77/55) | Bis-GMA, UDMA, Bis-EMA         | Ivoclar Vivadent (Schaan, Liechtenstein) | U27917     |
| Aura Bulk Fill, Nanohybrid/Universal (AUR)     | Barium alumino-borosilicate and silica (65/81)                                | UDMA, Bis-EMA, Bis-GMA, TEGDMA | SDI (Bayswater, Australia)               | 150931     |

Abbreviations: TEGDMA, triethyleneglycol dimethacrylate; Bis-GMA, bisphenol-A-glycidyl methacrylate; UDMA, urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol-A-dimethacrylate; AUDMA, high-molecular-weight aromatic dimethacrylate; AFM, addition-fragmentation monomers; DDDMA, 1,12-dodecanediol dimethacrylate.

resin specimens were made using a silicone matrix with orifices of 5 mm in diameter and 4 mm in height. Thirty-three specimens were obtained for each composite resin: group 1 (control) Z250 (Z250); group 2: Filtek Bulk Fill (FTK); group 3: Tetric N-Ceram Bulk Fill (TTC); and group 4: Aura Bulk Fill (AUR). The matrix was positioned on a glass plate and filled with composite resin. The Z250 composite resin was inserted in two increments of approximately 2-mm thickness. The FTK, TTC, and AUR composite resins were each inserted in one increment of 4-mm thickness. A polyester strip was placed on each composite resin, followed by a glass plate, in order to obtain a flat surface. The composite resin increments were light cured using the LED light unit Radii-cal (SDI, Bayswater, Australia) for 20 seconds at a distance of 1 mm from the specimen surface. The light intensity was 1000 mW/cm<sup>2</sup> and monitored by a radiometer (Model 100 Demetron, St Louis, MO, USA).

The composite resin surface in contact with the polyester strip was finished with polishing discs (Sof-Lex Pop On, 3M ESPE, St Paul, MN, USA) of medium, fine, and superfine grain. Each disc was applied for 15 seconds and by only one operator. After polishing, the specimens were ultrasonically cleaned in distilled water for 10 minutes and then stored in distilled water at 37°C for 24 hours.

### Surface Roughness Test

The initial surface roughness (Ra0) of the specimens in each group (n=15) was measured with a roughness tester SL-201 (Mitutoyo SurfTest Analyzer, Tokyo, Japan). Three consecutive measurements of the specimen were taken in different regions (one

central, one right, and one left), with a cutoff of 0.25. The mean values of roughness (Ra, in  $\mu\text{m}$ ) were obtained for each specimen.

### pH Cycling

Each specimen alternated with the cycles of demineralization and remineralization solutions simulating cariogenic alteration. The cariogenic alteration was composed of 15 cycles, and each cycle consisted of immersing the specimens in the demineralization solution for six hours and subsequently immersing them in the remineralization solution for 18 hours. Between one solution and the other, the specimens were washed with deionized water for one minute. Each specimen was immersed in 10 mL of the above-mentioned solutions, based on the methodology applied by Valinoti and others.<sup>22</sup> At the end of the pH cycles, the specimens were subjected to a new surface roughness reading (Ra1).

### Simulated Toothbrushing

A simulated toothbrushing machine, developed by the Idea Institute of the University, was used for this study. Each specimen was fixed in the center (orifice) of an acrylic plate (55×25×4 mm), enabling the specimen to remain 1 mm beyond the edge of the orifice that housed the specimen. Utility wax was applied to fix the specimens. Each plate was placed in an acrylic tank that was attached to the brushing machine. The acrylic tank was filled with a mixture composed of 1 g of toothpaste (Colgate Total 12, Colgate-Palmolive, São Bernardo do Campo, Brazil) per 1 mL of distilled water. Soft bristle Classic Colgate toothbrushes (Colgate-Palmolive) were used, and a load of 200g was applied. The speed of

brushing was 250 cycles per minute, carried out in 10,000 cycles of simulated toothbrushing. The toothbrushes were changed after every four brushed specimens from each group. After the brushing cycle, the specimens were washed in running water and ultrasonically cleaned in distilled water for 10 minutes, followed by drying with compressed air. The roughness of the surface was measured again (Ra2). The surface roughness reading was perpendicular to the brushing direction of the toothbrush bristles. For the correct positioning of the specimen in the brushing machine and to ensure repeatability in the same direction (perpendicular to the brushing), a mark with a diamond bur and high-speed hand piece was made on the border of each specimen.

### Surface Topography Analysis by SEM

Three specimens ( $n=3$ ) from each composite resin were used. Of the three specimens, one was analyzed by SEM after polishing, the second after pH cycling, and the third after pH cycling and simulated toothbrushing. The specimens were dried in a dehumidifier with silica gel for 72 hours, fixed with double-sided carbon adhesive tape (SPI, West Chester, PA, USA), and the top surface sputter coated with gold (Balzers, Balzers, Liechtenstein) before SEM observation (JSM 6060, JEOL, Tokyo, Japan) under 20,000 $\times$  magnification.

### Biofilm Analysis

The specimens of composite resin were sterilized with ethylene oxide gas (ETR Sterilizer, Porto Alegre, Brazil). The colony-forming units (CFU/mL) were evaluated in triplicate for each group ( $n=9$ ) after the following treatments: polishing (Cn0), pH cycling (Cn1), and pH cycling and simulated toothbrushing (Cn2). Biofilm adhesion was qualitatively evaluated by SEM in duplicates for each group ( $n=6$ ) after the following treatments: polishing, pH cycling, and pH cycling and simulated toothbrushing. Polishing, pH cycling, and toothbrushing of the specimens were performed according to the procedures described above.

*Streptococcus mutans* ATCC 25175, stored at  $-20^{\circ}\text{C}$ , was obtained and cultivated in BHI (brain-heart infusion) broth for 24 hours at  $37^{\circ}\text{C}$  in a bacteriological incubator. An aliquot of this primary culture was inoculated into a fresh BHI broth and incubated for an additional 24 hours at  $37^{\circ}\text{C}$ . Subsequently, 100  $\mu\text{L}$  of this culture, containing approximately  $10^6$  CFU/mL, were added to 1 mL of BHI broth supplemented with 1% sucrose. The composite resin specimen was then placed in the

culture and incubated for 24 hours under microaerophilic conditions at  $37^{\circ}\text{C}$ . The specimens were carefully removed from the culture and washed in 1 mL of 0.85% saline solution, removing the planktonic cells and keeping only the cells adhered to the surface of the specimens; this process was performed twice.

**Bacterial Counts**—After the washes, the specimens were placed in the ultrasonic bath for 10 minutes in order to disintegrate the biofilm. Subsequently, the saline solution containing the disaggregated cells was diluted to  $10^{-6}$ . The bacterial counts were determined spotting 10  $\mu\text{L}$  of the first three dilutions and spreading 100  $\mu\text{L}$  of the last three on BHI agar in triplicate. These two techniques were used according to the expected cellular concentrations at each dilution. The BHI agar plates were incubated at  $37^{\circ}\text{C}$  for 24 hours, followed by CFU/mL count. The assays were adapted from Nakamura and others<sup>23</sup> and Yoshihara and others.<sup>24</sup>

**Biofilm Analysis by SEM**—The specimens were immersed in the fixative solution (2.5% glutaraldehyde), where they were kept for seven days. After fixation, the specimens were washed three times for 30 minutes each in 0.2 mol/L phosphate buffer and distilled water in a ratio of 1:1. The specimens were then dehydrated by immersion in 30%, 50%, 70%, 90%, and 100% ethanol and dried in a silica gel dehumidifier for 72 hours. Surfaces were sputter coated with gold (Balzers) and observed by SEM (JSM 6060 LV, JEOL) under 500 $\times$  to 2000 $\times$  magnification. Qualitative analysis was started with the smaller magnification (500 $\times$ ), selecting areas with a higher concentration of bacterial biofilm.

For image assessment, a single observer, blinded to the identities of the experimental groups, classified them according to the presence of bacteria. Using PowerPoint (Microsoft Corp, Redmond, WA, USA), each image was placed on a slide and presented on a computer in the form of a presentation. The images were arranged in order according to the level of contamination found so that the first was the least contaminated and the last was the most contaminated. The image occupying the middle position in each group was then selected.<sup>25</sup>

### Statistical Analysis

Surface roughness data were analyzed by two-way repeated measures analysis of variance (ANOVA) (material $\times$ treatment), followed by the Tukey test. The CFU/mL count was analyzed by the Kruskal-Wallis nonparametric test, followed by multiple

Table 2: Surface Roughness (Mean ± Standard Deviation) of the Composite Resins After the Different Treatments<sup>a</sup>

| Material | Surface Roughness, μm |                |                |
|----------|-----------------------|----------------|----------------|
|          | Ra0                   | Ra1            | Ra2            |
| Z250     | 0.15 ± 0.07 aA        | 0.15 ± 0.06 Aa | 0.17 ± 0.05 aA |
| FTK      | 0.17 ± 0.08 aA        | 0.15 ± 0.09 aA | 0.18 ± 0.05 aA |
| TTC      | 0.15 ± 0.06 aA        | 0.19 ± 0.06 aA | 0.20 ± 0.06 aA |
| AUR      | 0.19 ± 0.08 aA        | 0.19 ± 0.08 aA | 0.27 ± 0.11 bB |

Abbreviations: Ra0, after polishing; Ra1, after pH cycling; Ra2, after pH cycling followed by toothbrushing.  
<sup>a</sup> Means followed by different lowercase and uppercase letters represent significant differences according to the Tukey test (α=0.05).

comparisons. The significance level was 5%. The software used was SPSS 10.0 (SPSS Inc, Chicago, IL, USA).

RESULTS

Surface Roughness

According to the two-way ANOVA, the material factor ( $p=0.016$ ) and treatment factor ( $p=0.001$ ) were significant. The interaction between the factors was not significant ( $p=0.265$ ) (Table 2).

The four composite resins did not differ significantly from each other at Ra0 and Ra1. At Ra2, AUR obtained significantly higher surface roughness than the other composite resins that was also higher than at Ra0 and Ra1 ( $p<0.05$ ).

Surface Topography Analysis by SEM

The polished surfaces of the composite resins Z250, FTK, TTC, and AUR are shown in Figure 1 (A1 and B1) and Figure 2 (C1 and D1), respectively. After pH cycles, there was removal of the organic matrix and exposure of the fillers (black arrows), especially on the surface of Z250 (Figure 1A2), TTC (Figure 2C2), and AUR (Figure 2D2), in which the fillers became more evident compared to the polished surfaces. It is also possible to observe cracks (circles) and microcavities (white arrows) on the surface of all bulk-fill composites (Figures 1B2, 2C2, 2D2). After pH cycles followed by simulated toothbrushing, greater degradation of the organic matrix occurred in all composite resins, and the filler exposure and microcavities were more evident (Figures 1A3, 1B3, 2C3, 2D3).

Bacterial Counts

According to the Kruskal-Wallis analysis, followed by multiple comparisons, there were higher bacterial counts for FTK in relation to those for Z250, TTC,

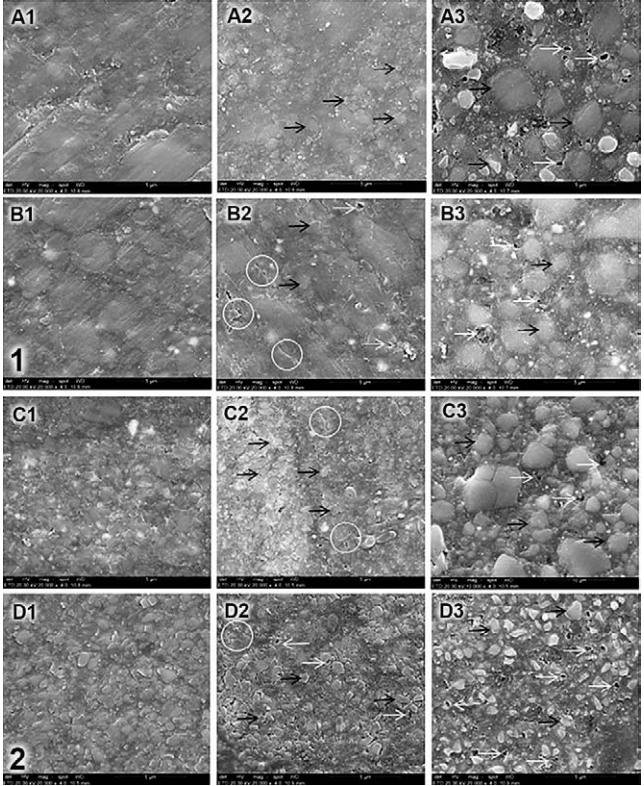


Figure 1. Scanning electron microscopic images (20,000×) of Filtek Z250 (A) and Filtek Bulk Fill (B) after polishing (1), after pH cycling (2), and after pH cycling and simulated toothbrushing (3). Circles, cracks; black arrows, fillers exposed; white arrows, microcavities.

Figure 2. Scanning electron microscopic images (20,000×) of Tetric N-Ceram Bulk Fill (C) and Aura Bulk Fill (D) after polishing (1), after pH cycling (2), and after pH cycling and simulated toothbrushing (3). Circles, cracks; black arrows, fillers exposed; white arrows, microcavities.

and AUR at Cn0 and Cn1 (Table 3). At Cn2, there were no significant differences in bacterial counts among the composite resins.

At Cn1, there was a significantly greater bacterial count for Z250 than at Cn0, followed by a significant decrease at Cn2. For FTK, there was a significantly decreased bacterial count at Cn2 than at Cn1. Bacterial counts for TTC and AUR did not differ significantly at Cn0, Cn1, or Cn2.

Biofilm Analysis by SEM

After polishing, dispersed bacteria were present (white arrows) on all composite resins and agglomerate formation (black arrows) on Z250 (Figure 3A1) and AUR (Figure 4D1).

After pH cycles, there was greater bacterial adhesion, with the formation of multiple agglomerates on Z250 (Figure 3A2), FTK (Figure 3B2), and

| Table 3: Median Values of Bacteria Count of the Composite Resins After the Different Treatments <sup>a</sup> |                                        |          |           |
|--------------------------------------------------------------------------------------------------------------|----------------------------------------|----------|-----------|
| Material                                                                                                     | Bacterial Count, CFU/mL10 <sup>6</sup> |          |           |
|                                                                                                              | Cn0                                    | Cn1      | Cn2       |
| Z250                                                                                                         | 7.00 aA                                | 14.00 aB | 1.80 aA   |
| FTK                                                                                                          | 31.60 bBC                              | 39.00 bC | 12.70 aAB |
| TTC                                                                                                          | 0.60 aA                                | 3.50 aA  | 2.30 aA   |
| AUR                                                                                                          | 2.00aA                                 | 2.60aA   | 2.00aA    |

Abbreviations: Ra0, after polishing; Ra1, after pH cycling; Ra2, after pH cycling followed by toothbrushing.  
<sup>a</sup> Medians followed by the same lowercase and uppercase letters do not differ significantly by the multiple comparison test.

AUR (Figure 4D2). There were dispersed bacteria on TTC (Figure 4C2).

Bacterial adhesion was less after simulated toothbrushing on Z250 (Figure 3A3), FTK (Figure 3B3), and AUR (Figure 4D3) than in the images obtained after pH cycles. There were dispersed bacteria on Z250, FTK, and TTC, while some bacterial adhesion remained on the surfaces of AUR (Figure 4D3).

DISCUSSION

The present study evaluated, qualitatively and quantitatively, the effect of pH cycling, followed by simulated toothbrushing, on surface changes and the bacterial adhesion of bulk-fill composite resins.

After pH cycling, which simulates cariogenic challenge, the SEM images showed modifications on the surface topography of the composite resins. Filler exposure, cracks, and microcavities occurred on the composite resins, with different intensities among the materials. These findings are related mainly to degradation of the organic matrix by the action of the cariogenic challenge solutions.<sup>26</sup>

The water in the solutions penetrates the polymer structure and chemically degrades the polymer, forming oligomers and monomers. The progressive degradation of this matrix leads to the formation of pores through which oligomers and monomers are released. In addition, there is degradation of the organic matrix by the acidic pH of the demineralization solution. Catalization of the ester groups of the dimethacrylates occurs, favoring the hydrolysis of these groups and formation of molecules of carboxylic acid and alcohol, which accelerate the degradation of the composite resin.<sup>26</sup> As a consequence, there is exposure of the fillers.

Absorption of the water in the solutions causes an increase in osmotic pressure at the organic matrix/filler interface, favoring formation of cracks on the

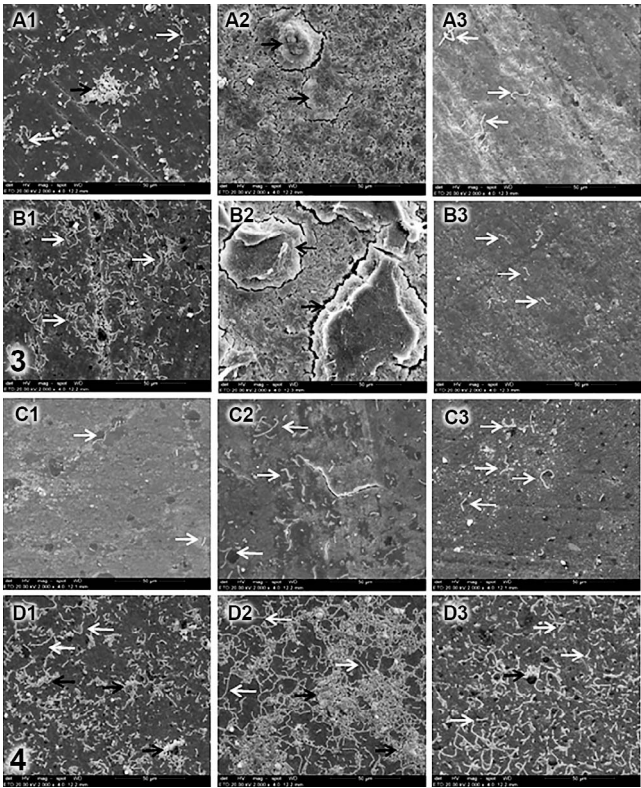


Figure 3. Scanning electron microscopic images (2,000X) of biofilm on Filtek Z250 (A) and Filtek Bulk Fill (B) after polishing (1), after pH cycling (2), and after pH cycling and simulated toothbrushing (3). White arrows, dispersed bacteria; black arrows, agglomerates of bacteria.

Figure 4. Scanning electron microscopic images (2,000X) of biofilm on Tetric N-Ceram Bulk Fill (C) and Aura Bulk Fill (D) after polishing (1), after pH cycling (2), and after pH cycling and simulated toothbrushing (3). White arrows, dispersed bacteria; black arrows, agglomerates of bacteria.

surface of the composite resins.<sup>27,28</sup> This absorption depends on the composition of the organic matrix and the quality of the bond between the organic matrix and fillers.<sup>29</sup> These cracks were also detected in another study.<sup>27</sup> Moreover, water absorption can generate hydrolytic degradation of the silane, favoring detachment of the fillers from the organic matrix and, consequently, the formation of microcavities.<sup>28</sup> Microcavities were also observed in another study that evaluated nanofiller and hybrid composite resins in contact with low-pH solutions.<sup>30</sup> According to Göpferich,<sup>26</sup> polymer degradation initially occurs superficially, with changes in surface morphology, followed by cracking and increased surface roughness. Although pH cycling caused changes in the composite resin surface in the present study, these changes did not result in a significant increase in surface roughness measurements, showing that the

composite resins evaluated were effective in maintaining the surface roughness against the pH variations.

The association between pH cycling and simulated toothbrushing was also evaluated since toothbrushing is part of common daily oral hygiene. Simulated toothbrushing abrasion is a methodology established in the literature; it is an important *in vitro* wear factor that simulates clinical conditions. According to Sexson and Phillips,<sup>31</sup> a patient performs approximately 15 cycles for each session of brushing. Thus, if maintaining an oral hygiene routine consisting of two daily brushing sessions, then 10,000 cycles are performed by the end of one year, which is the number of cycles applied to the specimens. The toothpaste used was Colgate Total 12, which has low abrasiveness on composite resins.<sup>32,33</sup>

SEM images demonstrated that the association of pH cycling with simulated toothbrushing caused a greater change on the surface topography of the composite resins. An increase in the exposure of fillers as well as the formation of microcavities was observed. Simulated toothbrushing with toothpaste causes wear on the composite resin surface through the abrasion process.<sup>33</sup> Several mechanisms are related to this wear: 1) organic matrix wear, 2) loss of fillers because of bond failure with the organic matrix, 3) loss of fillers due to shear of exposed fillers, 4) loss of fillers as a result of cracks in the organic matrix, and 5) exposure of voids intrinsic to the restorative process.<sup>34</sup> Therefore, toothbrushing potentiates the superficial degradation initially caused by pH cycling.

The pH cycling followed by simulated toothbrushing caused a significant increase in surface roughness only for the AUR composite resin. Therefore, the first hypothesis was partially rejected. Arithmetic roughness is a measure quantified by parameters of length (ampleness) and width (spacing) of irregularities or a combination of both.<sup>35</sup> It is suggested that the greater degradation of the organic matrix of the AUR favored the increase of its surface roughness when it was submitted to the toothbrushing abrasion mechanism. Some of the fillers exposed on the surface were removed by the shear process, leaving the surface with microcavities (Figure 2D3), which increased the parameters of height and width of the surface, consequently increasing surface roughness.<sup>34</sup> AUR contains the TEGDMA monomer, which reduces the material's viscosity while increasing its water absorption.<sup>36</sup> Degradation of the composite resins by hydrolysis is related to polymerization of the material and the monomeric composi-

tion. TEGDMA was shown to be more susceptible to hydrolysis than were Bis-GMA and Bis-EMA, leading to increased material wear and surface roughness.<sup>29</sup> While Z250 also contains TEGDMA, it did not show significant surface roughness alteration after pH cycling followed by simulated toothbrushing, which suggests that there may be differences in the percentages of this monomer in the compositions of the composite resins.<sup>29</sup>

One of the important aspects of the surface roughness study is related to bacterial adhesion and retention. After polishing (Ra0), all composite resins obtained surface roughness less than 0.2  $\mu\text{m}$ . After pH cycling (Ra1) and simulated toothbrushing (Ra2), surface roughness of the composite resins was approximately at the stated threshold surface roughness of 0.2  $\mu\text{m}$ ;<sup>7</sup> this can be considered a positive result since surface roughness above 0.2  $\mu\text{m}$  favors an increase in plaque formation on the material surfaces.<sup>37</sup>

The present study also evaluated bacterial adhesion on the composite resin surfaces. The accumulation of biofilm favors the formation of secondary caries at the restorative interfaces, which is the main factor responsible for failures of composite resin restorations.<sup>38</sup>

SEM images showed agglomerates of bacteria after pH cycling on Z250 and FTK composite resin surfaces (Figures 3A2 and 3B2, respectively). The agglomerates of bacteria were more intense for FTK. Conversely, there were only dispersed bacteria for TTC (Figure 4C2) and AUR (Figure 4D2). These findings corroborate the results of bacterial counts after pH cycling. According to the surface roughness means, similarities in relation to the bacterial adhesion would be expected, but this was not confirmed. Yuan and others<sup>39</sup> observed that bacterial adhesion is not always related only to the surface roughness but also to surface energy. Besides that, *S. mutans* have greater propensity for adhesion to substrates with high surface energy. One factor that influences the surface energy is the composition of the fillers of the composite resins.<sup>40</sup> The filler in Z250 and FTK is silica and zirconia particles, TTC uses barium-silica-aluminum particles, and AUR uses silica and barium alumino-borosilicate particles. However, other factors contribute to bacterial adhesion, such as size and shape of fillers,<sup>41,42</sup> monomer composition,<sup>42,43</sup> and amount of residual monomer.<sup>41</sup> A possible explanation for higher bacteria adhesion obtained for FTK could be related to the particular monomer mixture and fillers of this material. Further investigation is necessary to establish which

component is more relevant in the process of bacterial adhesion for each composite resin.

There were only dispersed bacteria on the surface of all composite resins after pH cycling followed by simulated toothbrushing. These findings are more evident for Z250 (Figure 3A3) and FTK (Figure 3B3), which showed a significant decrease in bacterial counts after toothbrushing in relation to pH cycling. There was no significant difference in bacterial counts between the treatments for TTC and AUR. The SEM images corroborate these findings since dispersed bacteria were predominant for the three treatments. Therefore, the second hypothesis was partially rejected.

Possible explanations can be drawn for the decrease of bacterial counts and the presence of dispersed bacteria instead of agglomerates of bacteria on the surface of the composite resins after toothbrushing. First, the presence of 0.3% Triclosan in the fluoridated toothpaste, which has a broad spectrum of antimicrobial action, may influence agglomeration. In bacteriostatic concentrations, this substance prevents protein synthesis and, in bactericidal concentrations, disorganizes the cytoplasmic membrane of the bacterium, leading to a loss of structural integrity and a leakage of intracellular content.<sup>44</sup> At lower concentrations (0.2% to 0.5%), Triclosan affects the metabolism of some species, such as *S mutans*, and may have influenced the lower bacterial adhesion on the surface of the composite resins.<sup>45</sup> In a second possible explanation, the presence of sodium fluoride in the toothpaste, in low concentrations, exerts subtle antimicrobial action,<sup>46</sup> presenting direct and indirect effects on *S mutans*. In the direct effect, sodium fluoride prevents an increase in the number of *S mutans* through the inhibition of critical metabolic processes.<sup>47</sup> In the indirect effect, sodium fluoride reduces environmental acidification in the biofilm. Another study found a reduction in *S mutans* when combining an acidic pH with the presence of sodium fluoride.<sup>46</sup>

Despite the easier and faster restorative procedures obtained with the bulk-fill composite resins, these materials are affected by pH cycling. Ideally, patients should maintain adequate oral hygiene and follow a low-sugar diet. Noncompliance with these factors increases the cariogenic challenges imposed on the restorative interfaces.

## CONCLUSIONS

Within the limitations of the present study, it is possible to conclude that pH cycling caused degradation of the surface of composite resins. Simulated

toothbrushing potentiated this degradation. However, only Aura Bulk Fill presented an increase in surface roughness after the simulated brushing regimen. Bacterial adhesion occurred on all composite resins after pH cycling; however, after simulated brushing, adhesion of dispersed bacteria was similar for all the composite resin groups.

## Acknowledgement

The authors are grateful to Coordination for the Improvement of Higher Education Personnel (CAPES, Brazil) for the scholarship for DCS.

## Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 25 May 2019)

## REFERENCES

1. Demarco FF, Collares K, Correa MB, Cenci MS, Moraes RR, & Opdam NJ (2017) Should my composite restorations last forever? Why are they failing? *Brazilian Oral Research* **31**(1) 92-99.
2. Walter R (2013) Critical appraisal: Bulk-fill flowable composite resins *Journal of Esthetic and Restorative Dentistry* **25**(1) 72-76.
3. Krüger J, Maletz R, Ottl P, & Warkentin. (2018) In vitro aging behavior of dental composites considering the influence of filler content, storage media and incubation time *PLoS One* **13**(4) e0195160.
4. Featherstone JD (2004) The continuum of dental caries—Evidence for a dynamic disease process *Journal of Dental Research* **83** (Special Issue C) 39-42.
5. Rahim TN, Mohamad D, Md Akil H, & Ab Rahman I (2012) Water sorption characteristics of restorative dental composites immersed in acidic drinks *Dental Materials* **28**(1) 63-70.
6. Neres É, Moda MD, Chiba EK, Briso A, Pessan JP, & Fagundes TC (2017) Microhardness and roughness of infiltrated white spot lesions submitted to different challenges *Operative Dentistry* **42**(4) 428-435.
7. Bollen CM, Lambrechts P, & Quirynen M (1997) Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: A review of the literature *Dental Materials* **13**(4) 258-269.
8. Glauser S, Astasov-Frauenhoffer M, Müller JA, Fischer J, Waltimo T, & Rohr N (2017) Bacterial colonization of resin composite cements: Influence of material composition and surface roughness *European Journal of Oral Science* **125**(4) 294-302.
9. Derchi G, Vano M, Barone A, Covani U, Diaspro A, & Salerno M (2017) Bacterial adhesion on direct and indirect dental restorative composite resins: An in vitro

- study on a natural biofilm *Journal of Prosthetic Dentistry* **117**(5) 669-676.
10. de Fúcio SB, Puppini-Rontani RM, de Carvalho FG, Mattos-Graner R de O, Correr-Sobrinho L, & Garcia-Godoy F (2009) Analyses of biofilms accumulated on dental restorative materials *American Journal of Dentistry* **22**(3) 131-136.
  11. Beyth N, Yudovin-Fearber I, Domb AJ, & Weiss EL (2010) Long-term antibacterial surface properties of composite resin incorporating polyethyleneimine nanoparticles *Quintessence International* **41**(10) 827-835.
  12. Sawlani K, Lawson NC, Burgess JO, Lemons JE, Kinderknecht KE, Givan DA, & Ramp L (2016) Factors influencing the progression of noncarious cervical lesions: A 5-year prospective clinical evaluation *Journal of Prosthetic Dentistry* **115**(5) 571-577.
  13. Komatsu H, Yamamoto H, Matsuda Y, Kijimura T, Kinugawa M, Okuyama K, Nomachi M, Yasuda K, Satoh T, & Oikawa S (2011) Fluorine analysis of human enamel around fluoride-containing materials under different pH-cycling by  $\mu$ -PIGE/PIXE system *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms* **269**(20) 2274-2277.
  14. Carvalho FG, Sampaio CS, Fúcio SB, Carlo HL, Correr-Sobrinho L, & Puppini-Rontani RM (2012) Effect of chemical and mechanical degradation on surface roughness of three glass ionomers and a nanofilled resin composite *Operative Dentistry* **37**(5) 509-517.
  15. Bahadure RN, Pandey RK, Kumar R, Gopal K, & Singh RK (2012) An estimation of fluoride release from various dental restorative materials at different pH: In vitro study *Journal of Indian Society of Pedodontics and Preventive Dentistry* **30**(2) 122-126.
  16. Paiva LF, Fidalgo TK, & Maia LC (2014) Mineral content of ionomer cements and preventive effect of these cements against white spot lesions around restorations *Brazilian Oral Research* **28**(1) 1-9.
  17. Ishikiriama SK, De Oliveira GU, Maenosono RM, Wang L, Duarte MA, & Mondelli RF (2014) Wear and surface roughness of silorane composites after pH cycling and toothbrushing abrasion *American Journal of Dentistry* **27**(4) 195-198.
  18. Pinto CF, Vermelho PM, Aguiar TR, Paes Leme AF, Oliveira MT, Souza EM, Cavalli V, & Giannini M (2015) Enamel and dentin bond strength, interfacial ultra-morphology and fluoride ion release of self-etching adhesives during a pH-cycling regime *Journal of Adhesive Dentistry* **17**(1) 27-34.
  19. Featherstone JDB, O'Really MM, Shariati M, & Brugler S (1989) Enhancement of remineralization *in vitro* and *in vivo*. In: Leach SA (ed), *Factors Relating to Demineralization and Remineralization of the Teeth* IRL Press, Oxford 23-24.
  20. Serra MC & Cury JA (1992) The in vitro effect of glass-ionomer cement restoration on enamel subjected to a demineralization and remineralization model *Quintessence International* **23**(2) 143-147.
  21. Münchow EA, Ferreira AC, Machado RM, Ramos TS, Rodrigues-Junior SA, & Zanchi CH (2014) Effect of acid solution on the surface degradation of a micro-hybrid composite resin *Brazilian Dental Journal* **25**(4) 321-326.
  22. Valinoti AC, Neves BG, Silva EM, & Maia LC (2008) Surface degradation of composite resins by acidic medicines and pH-cycling *Journal of Applied Oral Science* **16**(4) 257-265.
  23. Nakamura K, Shirato M, Kanno T, Lingström P, Örtengren U, & Niwano Y (2017) Photo-irradiated caffeic acid exhibits antimicrobial activity against *Streptococcus mutans* biofilms via hydroxyl radical formation *Scientific Reports* **7**(1) 6353-6366.
  24. Yoshihara K, Nagaoka N, Maruo Y, Sano H, Yoshida Y, & Van Meerbeek B (2017) Bacterial adhesion not inhibited by ion-releasing bioactive glass filler *Dental Materials* **33**(6) 723-734.
  25. Gründling GL, Zechin JG, Jardim WM, de Oliveira SD, & De Figueiredo JA (2011) Effect of ultrasonics on *Enterococcus faecalis* biofilm in a bovine tooth model *Journal of Endodontics* **37**(8) 1128-1133.
  26. Gopferich A (1996) Mechanisms of polymer degradation and erosion *Biomaterials* **17**(2) 103-114.
  27. Söderholm KJ, Zigan M, Ragan M, Fischlschweiger W, & Bergman M (1984) Hydrolytic degradation of dental composites *Journal of Dental Research* **63**(10) 1248-1254.
  28. Lepri CP, Ribeiro MVM, Dibb A, & Palma-Dibb RG (2014) Influence of mouth rinse solutions on the color stability and microhardness of a composite resin *International Journal of Esthetic Dentistry* **9**(2) 238-246.
  29. Ferracane JK (2006) Hygroscopic and hydrolytic effects in dental polymer networks *Dental Materials* **22**(3) 211-222.
  30. Almeida GS, Poskus LT, Guimarães JG, & Silva EM (2010) The effect of mouthrinses on salivary sorption, solubility and surface degradation of a nanofilled and a hybrid resin composite *Operative Dentistry* **35**(1) 105-111.
  31. Sexton JC & Phillips RW (1951) Studies on the effects of abrasives on acrylic resins *Journal of Prosthetic Dentistry* **1**(4) 454-471.
  32. Barbieri GM, Mota EG, Rodrigues-Junior SA, & Burnett LH Jr (2011) Effect of whitening dentifrices on the surface roughness of commercial composites *Journal of Esthetic and Restorative Dentistry* **23**(5) 338-345.
  33. Monteiro B & Spohr AM (2015) Surface roughness of composite resins after simulated toothbrushing with different dentifrices *Journal of International Oral Health* **7**(7) 1-5.
  34. O'Brien WJ & Yee J Jr (1980) Microstructure of posterior restorations of composite resin after clinical wear *Operative Dentistry* **5**(3) 90-94.
  35. Cilli R, de Mattos MC, Honorio HM, Rios D, de Araujo PA, & Prakki A (2009) The role of surface sealants in the roughness of composites after a simulated toothbrushing test *Journal of Dentistry* **37**(12) 970-977.
  36. Fonseca AS, Labruna Moreira AD, de Albuquerque PP, de Menezes LR, Pfeifer CS, & Schneider LF (2017) Effect of monomer type on the CC degree of conversion, water sorption and solubility, and color stability of model dental composites *Dental Materials* **33**(4) 394-401.

37. Quirynen M, Marechal M, Busscher HJ, Weerkamp AH, Darius PL, & van Steenberghe D (1990) The influence of surface free energy and surface roughness on early plaque formation. An in vivo study in man *Journal of Clinical Periodontology* **17**(3) 138-144.
38. Nedeljkovic I, Teughels W, De Munck J, Van Meerbeek B, & Van Landuyt KL (2015) Is secondary caries with composites a material-based problem? *Dental Materials* **31**(11) 247-277.
39. Yuan C, Wang X, Gao X, Chen F, Liang X, & Li D (2016) Effects of surface properties of polymer-based restorative materials on early adhesion of *Streptococcus mutans* in vitro *Journal of Dentistry* **54** 33-40.
40. Ionescu A, Wutscher E, Brambilla E, Schneider-Feyrer S, Giessibl FJ, & Hahnel S (2012) Influence of surface properties of resin-based composites on in vitro *Streptococcus mutans* biofilm development *European Journal of Oral Sciences* **120**(5) 458-465.
41. Ikeda M, Matin K, Nikaido T, Foxton RM, & Tagami J (2007) Effect of surface characteristics on adherence of *S. mutans* biofilms to indirect resin composites *Dental Materials Journal* **26**(6) 915-923.
42. Hao Y, Huang X, Zhou X, Li M, Ren B, Peng X, & Cheng L (2018) Influence of dental prosthesis and restorative materials interface on oral biofilms *International Journal of Molecular Sciences* **19**(10) 3157.
43. Hahnel S, Rosentritt M, Bürgers R, & Handel G (2008) Surface properties and in vitro *Streptococcus mutans* adhesion to dental resin polymers *Journal of Materials Science: Materials in Medicine* **19**(7) 2619-2627.
44. Anushree, B, Fawaz MA, Narahari R, Shahela T, & Syed A (2015) Comparison of antimicrobial efficacy of triclosan-containing, herbal and homeopathy toothpastes—An in vitro study *Journal of Clinical and Diagnostic Research* **9**(10) DC05-DC08.
45. Davies RM, Ellwood RP, & Davies GM (2004) The effectiveness of a toothpaste containing triclosan and polyvinyl-methyl ether maleic acid copolymer in improving plaque control and gingival health: A systematic review *Journal of Clinical Periodontology* **31**(12) 1029-1033.
46. Bradshaw DJ, Marsh PD, Hodgson RJ, & Visser JM (2002) Effects of glucose and fluoride on competition and metabolism within in vitro dental bacterial communities and biofilms *Caries Research* **36**(2) 81-86.
47. Marquis RE (1995) Antimicrobial actions of fluoride for oral bacteria. *Canadian Journal of Microbiology* **41**(11) 955-964.

# Influence of CAD/CAM Fabrication and Sintering Procedures on the Fracture Load of Full-Contour Monolithic Zirconia Crowns as a Function of Material Thickness

M Zimmermann • A Ender • A Mehl

## Clinical Relevance

Chairside sintering might be a suitable procedure to produce zirconia restorations with clinically adequate fracture loading values for specific restoration thicknesses.

## Summary

**Objective:** The purpose of this *in vitro* study was to analyze the effect of computer-aided design/computer-aided manufacturing (CAD/CAM) fabrication and sintering procedures on the fracture load of monolithic zirconia crowns with different material thicknesses

\*Moritz Zimmermann, Dr Med Dent, Division of Computerized Restorative Dentistry, Clinic for Preventive Dentistry, Periodontology and Cariology, Center of Dental Medicine, University of Zurich, Zurich, Switzerland

Andreas Ender, PD, Dr Med Dent, Division of Computerized Restorative Dentistry, Clinic for Preventive Dentistry, Periodontology and Cariology, Center of Dental Medicine, University of Zurich, Zurich, Switzerland

Albert Mehl, Dr Med Dent, Dr Rer Nat, Professor, Division of Computerized Restorative Dentistry, Clinic for Preventive Dentistry, Periodontology and Cariology, Center of Dental Medicine, University of Zurich, Zurich, Switzerland

\*Corresponding author: Plattenstrasse 11, CH-8032 Zurich, Switzerland; e-mail: moritz.zimmermann@zzm.uzh.ch

<https://doi.org/10.2341/19-086-L>

adhesively seated to methacrylate dies fabricated with stereolithography technology.

**Method:** Monolithic zirconia crowns were fabricated from inCoris TZI C material with a chairside CAD/CAM system (CEREC MCXL) comprising three material thicknesses (0.5/1.0/1.5 mm, n=8 each). Two CAD/CAM fabrication procedures (milling, MI; grinding, GR), two chairside sintering procedures (superspeed, SS; speedfire sintering, SF), and one labside sintering procedure (classic, CL) were evaluated. In total, 144 crowns were fabricated. Restorations were adhesively seated to methacrylate dies fabricated with SLA technology. Thermomechanical cycling (TCML) was performed before fracture testing. Loading forces until fracture were registered and statistically analyzed with one-way analysis of variance (ANOVA), *post hoc* Scheffé test, and three-way ANOVA ( $\alpha=0.05$ ).

**Results:** Test groups showed statistically significant differences ( $p<0.05$ ). The highest mean value was found for 1.5-mm crowns of

group GR\_SF with  $3678.6 \pm 363.9$  N. The lowest mean value was found for group 0.5-mm crowns of group MI\_SF with  $382.4 \pm 30.7$  N. There was a significant three-way interaction effect between thickness, sintering, and processing [ $F(4,126)=9.542$ ;  $p<0.001$ ; three-way ANOVA, significance level  $\alpha=0.05$ ].

**Conclusions:** CAD/CAM fabrication and sintering procedures influence the maximum loading force of monolithic zirconia crowns with different material thicknesses. A material thickness of 0.5 mm should be considered as a critical thickness for monolithic zirconia crown restorations.

INTRODUCTION

Zirconia ceramic materials have been described in literature both for single restoration and fixed dental prostheses (FDPs) indications.<sup>1</sup> Y-TZP (yttria-stabilized tetragonal zirconia polycrystal) is the most widely used type of zirconia material.<sup>2</sup> Marginal adaptation, internal fitting, and clinical long-term survival of zirconia restorations have been demonstrated to be clinically acceptable compared with conventional ceramic materials.<sup>3,4</sup> Zirconia materials have advantageous material characteristics such as a superior fracture resistance and a higher fracture toughness.<sup>5</sup> The extent of tooth preparation might be thus reduced for zirconia restorations.<sup>6,7</sup> Among the reported shortcomings for zirconia are a high opacity, a reduction of fracture strength as a result of low-temperature aging degradation, and the possibility of chipping of veneering ceramic.<sup>8–12</sup> Zirconia materials show superior wear behavior and lower antagonist wear compared with glass ceramics after grinding treatments.<sup>13</sup> Until now, few clinical studies are available investigating the clinical long-term success of monolithic zirconia restorations, and a recommended minimum thickness for monolithic zirconia restorations has not been reported.<sup>14–16</sup>

Zirconia restorations are normally fabricated in a presintered green body state with a geometric magnification of up to 20% to 30% using subtractive computer-aided design/computer-aided manufacturing (CAD/CAM) technology.<sup>1</sup> Subtractive fabrication can be performed either with milling technology using carbide burs or with grinding technology using diamond-coated instruments. Both fabrication methods can be executed in dry and wet surroundings. Most zirconia restorations today are produced using dry-milling CAM fabrication procedures. Few studies are available investigating a possible effect of the respective CAM fabrication method on the material

Table 1: Overview Test Groups

| Group | Fabrication Procedure | Sintering Procedure | Sintering Device |
|-------|-----------------------|---------------------|------------------|
| MI_CL | Milling               | Classic             | inFire HTC speed |
| GR_CL | Grinding              |                     |                  |
| MI_SS | Milling               | Superspeed          |                  |
| GR_SS | Grinding              |                     |                  |
| MI_SF | Milling               | Speedfire           | CEREC SpeedFire  |
| GR_SF | Grinding              |                     |                  |

Monolithic zirconia material inCoris TZI C was used for all groups; for each group three different subgroups with material thicknesses 0.5/1.0/1.5 mm were established with each (n=8). A total of n = 144 crowns were fabricated. There were two different CAM fabrication processes (MI = milling, GR = grinding) using a CEREC MCXL CAM device and three different sintering processes (CL = classic, SS = superspeed, SF = speedfire).

characteristics of zirconia.<sup>17,18</sup> Zirconia restorations obtain their final geometric form during a subsequent high temperature sintering process using special ceramic furnacing devices. Various sintering procedures are available for zirconia restorations both varying the sintering time and the sintering temperature. Few studies have investigated a possible effect of sintering procedures on the material characteristics of zirconia.<sup>19–22</sup>

The aim of this study was to analyze the effect of different fabrication and sintering procedures on the fracture load of full-contour monolithic zirconia crowns with different material thicknesses *in vitro*. The null hypothesis was that different sintering and fabrication procedures do not influence the maximum fracture load of monolithic zirconia crowns with different material thicknesses.

METHODS AND MATERIALS

In this study, fatigue loading and subsequent fracture loading of monolithic zirconia crowns adhesively seated to abutment dies fabricated with stereolithography (SLA) technology was performed. Three different crown material thicknesses (0.5/1.0/1.5 mm) were investigated for each specific test group. Monolithic partially stabilized zirconia material (Y-TZP) inCoris TZI C (Dentsply Sirona, York, USA) was used for all test groups. Test groups comprised two different CAM fabrication procedures and three different sintering procedures. For each test group, eight crowns were fabricated. In total, 144 crowns were fabricated. Specification of test groups investigated in this study is shown in Table 1.

All crowns were adhesively seated to abutment dies fabricated with SLA technology. Design of abutment dies was performed in accordance with preparation guidelines for full-ceramic restorations

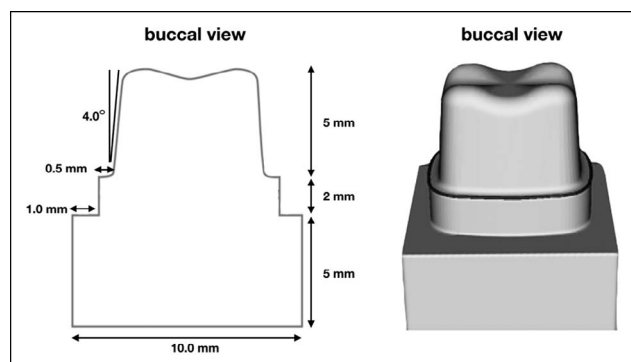


Figure 1. Design of abutment die for 0.5 mm crown; left: cross section with indication of metric values; right: digitized abutment die with indication of preparation margin highlighted in blue.

with CAD Software Pro Engineer Wildfire 4.0 (PTC, Boston, USA). Three different die geometries were designed in respect of full-contour crown material thicknesses 0.5/1.0/1.5 mm. The specification of the abutment design was that the restoration's outer contour could be designed identically for each material thickness. SLA printer Viper Si2 (3D Systems, Rock Hill, USA) was used for abutment die fabrication. z-axis resolution was 50  $\mu\text{m}$  for the abutment die. A methacrylate material was used as abutment material (E-Modulus, 2.5 GPa; fractural strength, 110-130 MPa; Shore hardness, 80-84 Shore D). The specifications of the designed abutment die for a 0.5 mm crown is shown in Figure 1.

The design of full-contour crowns was performed using CAD software CEREC v.4.0 (Dentsply Sirona) after initially scanning the abutment die for 0.5-mm material thickness with the intraoral scanning device CEREC Bluecam (Dentsply Sirona). Using the software tool "biocopy," the design of the 0.5-mm restoration could be transferred with an identical outer contour but an individual adaptation of the inner contour to the abutment dies for 1.0- and 1.5-mm restorations. Crown material thicknesses were controlled in all cases by applying CAD software tools "cursor details" and "show minimum thickness" so that an identical design could be ensured for the respective test groups. Restoration parameters were identical for all crowns with a spacer parameter setting of 80  $\mu\text{m}$ . Different material thicknesses of 0.5/1.0/1.5 mm always comprised the entire crown restoration.

All crowns were fabricated with a chairside CAD/CAM system using a 3+1 axis milling unit CEREC MCXL (Dentsply Sirona). There were two different CAM fabrication procedures: group MI (milling) and group GR (grinding). The CEREC MCXL milling machine was equipped with different carbide burs

for milling (Shaper 25/RZ, Finisher 10) and diamond-coated burs for grinding (StepBur 20, Cylinder Pointed Bur 20). Milling and grinding mode was set to "standard." Milling and grinding instruments were renewed after the fabrication of one test group (ie, after eight restorations to exclude a possible damaging effect of used instruments to the restoration while fabrication).

Monolithic zirconia crowns were forwarded to different sintering procedures after fabrication. There were three different sintering procedures: group CL (classic sintering with inFire HTC speed; Dentsply Sirona), group SS (superspeed sintering with inFire HTC speed; Dentsply Sirona), and group SF (speedfire sintering with CEREC SpeedFire; Dentsply Sirona). All sintering procedure parameters were strictly according to manufacturer's recommendations: CL, sintering temperature 1510°C, dwell time 120 minutes, total time eight hours, start from room temperature; SS, sintering temperature 1580°C, dwell time 10 minutes, total time 10 minutes, start at preheated furnace; SF, sintering temperature 1580°C, dwell time two minutes, total time 13.34 minutes, start from room temperature (example for inCoris TZI C, shade A3). No post-processing was performed to the crowns after the sintering process.

Crowns were cleaned ultrasonically for three minutes and degreased with ethanol after sintering. No phosphoric acid etching cleaning of restorations was performed. SLA-fabricated abutment dies and crowns were first sandblasted (Cojet, 3M, Saint Paul, USA) (diameter  $\leq 50 \mu\text{m}$ , 200 kPa) and then cleaned with ultrasonic using ethanol for three minutes. Abutment dies were silanized using Mono-bond Plus (Ivoclar Vivadent AG, Schaan, Lichtenstein) for 60 seconds and then dried with oil-free air. Crowns were then adhesively seated to SLA fabricated abutment dies following a standardized adhesive protocol using a dual cure luting resin cement (PANAVIA F 2.0, Kuraray Noritake, Tokyo, Japan). An oxygen layer inhibitor material (Oxyguard, Kuraray Noritake) was applied around the cervical margin of the crowns during polymerization. A Satelec MiniLED polymerization lamp (KaVo Dental, Biberach an der Riss, Germany) was used for polymerization of the luting resin cement with 1600 mW/cm<sup>2</sup> from the occlusal, mesial, distal, buccal, and lingual aspects for 60 seconds each. All crowns were then forwarded to fatigue loading followed by fracture loading in a universal testing machine.

Restorations were embedded using a methacrylate material (Paladur; Kulzer, Hanau, Germany) in

| Table 2: Results for Maximum Loading Force of Monolithic Zirconia Crowns With Material Thicknesses 0.5/1.0/1.5 mm (n=8)                                                  |               |        |       |         |                         |        |        |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------|--------|-------|---------|-------------------------|--------|--------|
| Group                                                                                                                                                                    | Loading Force |        |       |         | 95% Confidence Interval |        |        |
|                                                                                                                                                                          | Thickness     | Mean   | SD    | Minimum | Maximum                 | Lower  | Upper  |
| MI_CL                                                                                                                                                                    | 0.5           | 601.4  | 100.1 | 402.0   | 729.6                   | 517.7  | 685.1  |
|                                                                                                                                                                          | 1.0           | 1957.3 | 190.4 | 1673.8  | 2243.8                  | 1798.1 | 2116.5 |
|                                                                                                                                                                          | 1.5           | 3149.1 | 423.9 | 2738.2  | 4077.0                  | 2794.7 | 3503.5 |
| GR_CL                                                                                                                                                                    | 0.5           | 593.1  | 79.6  | 514.4   | 703.1                   | 526.5  | 659.6  |
|                                                                                                                                                                          | 1.0           | 2366.2 | 229.8 | 2121.0  | 2830.1                  | 2174.1 | 2558.3 |
|                                                                                                                                                                          | 1.5           | 3119.6 | 456.0 | 2643.4  | 4065.2                  | 2738.4 | 3500.9 |
| MI_SS                                                                                                                                                                    | 0.5           | 649.9  | 112.4 | 532.0   | 790.8                   | 555.9  | 743.9  |
|                                                                                                                                                                          | 1.0           | 1800.0 | 127.9 | 1669.0  | 2032.9                  | 1693.1 | 1907.0 |
|                                                                                                                                                                          | 1.5           | 2793.3 | 567.8 | 2075.1  | 3373.6                  | 2318.6 | 3268.0 |
| GR_SS                                                                                                                                                                    | 0.5           | 462.3  | 69.1  | 366.0   | 594.1                   | 404.5  | 520.0  |
|                                                                                                                                                                          | 1.0           | 1752.5 | 401.9 | 1426.1  | 2476.7                  | 1416.4 | 2088.5 |
|                                                                                                                                                                          | 1.5           | 2455.7 | 85.5  | 2250.8  | 2499.8                  | 2384.2 | 2527.2 |
| MI_SF                                                                                                                                                                    | 0.5           | 382.4  | 30.7  | 350.1   | 423.6                   | 356.8  | 408.1  |
|                                                                                                                                                                          | 1.0           | 1943.4 | 268.0 | 1656.5  | 2282.1                  | 1719.3 | 2167.5 |
|                                                                                                                                                                          | 1.5           | 2841.3 | 188.8 | 2603.0  | 3114.6                  | 2683.4 | 2999.1 |
| GR_SF                                                                                                                                                                    | 0.5           | 395.8  | 75.2  | 314.1   | 514.2                   | 332.9  | 458.7  |
|                                                                                                                                                                          | 1.0           | 1755.2 | 156.8 | 1555.7  | 1979.3                  | 1624.1 | 1886.4 |
|                                                                                                                                                                          | 1.5           | 3678.6 | 363.9 | 3206.7  | 4392.6                  | 3374.4 | 3982.8 |
| Two different CAM fabrication processes (MI = milling, GR = grinding) and three different sintering processes (CL = classic, SS = superspeed, SF = speedfire) were used. |               |        |       |         |                         |        |        |

pecially designed test blocks ensuring an identical central fixation of the crowns. Thermomechanical loading (TCML) of full-contour zirconia crowns (1.2 million cycles, 1.7 Hz, invariable occlusal load  $49 \pm 0.7$  N, thermal cycling 5-55°, dwell time 120 seconds, 12,000 cycles, water change time 10 seconds, human natural molar cusp antagonist, load to central fissure) was performed.<sup>23</sup> Crowns were investigated for cracks using a stereomicroscope with 14× magnification and transmitted light (Wild Leitz/M1B, Walter Products, Windsor, Canada) after fatigue loading. Fracture loading was performed using Allround Line z010 testing machine (Zwick Roell AG, Ulm, Germany) (crosshead speed, 1 mm/min; ball diameter, 5 mm). All crowns were loaded until fracture. Maximum loading forces were automatically recorded by the software. Fracture data values obtained were statistically evaluated using one-way analysis of variance (ANOVA), and *post hoc* Scheffé test, and three-way ANOVA ( $\alpha=0.05$ ) (IBM SPSS Statistics v25.0; IBM, Armonk, USA).

For each sintering group (CL, SS, and SF) a 2-mm-thickness disc (dimensions  $2 \times 2$  mm) was prepared with a low-speed diamond saw (Isomet; Buehler, Lake Bluff, USA) and sintered using the respective sintering parameters. Sintered discs were wolfram sputtered (CCU-010, Safematic GmbH, Bad Ragaz, Switzerland) and forwarded to Scanning Electron

Microscopy (SEM; Zeiss Supra V50, Carl Zeiss, Oberkochen, Germany) for qualitative microstructural imaging of the respective material structure.

RESULTS

Results for fracture loading forces of full-contour monolithic zirconia crowns are shown in Table 2. Data for fracture loading were normally distributed with homogeneity of variances (Shapiro-Wilk test and Levene test). Maximum loading forces statistically significantly varied among the different test groups (one-way ANOVA and *post hoc* Scheffé test,  $p<0.05$ ). For 0.5- and 1.0-mm-thickness restorations, both the fabrication procedure (MI and GR) and the sintering procedure (CL, SS, SF) showed no statistically significant effect ( $p>0.05$ ). However, for 1.5-mm-thickness restorations, statistically significant differences were found between test groups with different fabrication and sintering procedures ( $p<0.05$ ). There was a significant three-way interaction effect between thickness, sintering, and processing [ $F(4,126)=9.542$ ;  $p<0.001$ ; three-way ANOVA, significance level  $\alpha=0.05$ ]. The highest mean value was found for 1.5-mm crowns of group GR\_SF with  $3678.6 \pm 363.9$  N. The lowest mean value was found for group 0.5-mm crowns of group MI\_SF with  $382.4 \pm 30.7$  N. Based on the results found in this study, the null hypothesis that sintering and

Table 3: Results for Fracture Loading Force of Monolithic Zirconia Crowns With Material Thicknesses 0.5/1.0/1.5 mm<sup>a</sup>

| Crown Thickness | Sintering and Fabrication Procedures |                   |                   |                   |                  |                   |
|-----------------|--------------------------------------|-------------------|-------------------|-------------------|------------------|-------------------|
|                 | CL                                   |                   | SS                |                   | SF               |                   |
|                 | GR                                   | MI                | GR                | MI                | GR               | MI                |
| 0.5             | 593.1 ± 79.6 A                       | 601.4 ± 100.1 A   | 462.3 ± 69.1 A    | 649.9 ± 112.4 A   | 395.8 ± 75.2 A   | 382.4 ± 30.7 A    |
| 1.0             | 2366.2 ± 229.8 BC                    | 1957.3 ± 190.4 B  | 1752.5 ± 401.9 B  | 1800.0 ± 127.9 B  | 1755.2 ± 156.8 B | 1943.4 ± 268.0 B  |
| 1.5             | 3119.6 ± 456.0 DE                    | 3149.1 ± 423.9 DE | 2455.7 ± 85.5 BCD | 2793.3 ± 567.8 CD | 3678.6 ± 363.9 E | 2841.3 ± 188.8 CD |

<sup>a</sup> All values are indicated as mean ± SD in [N]. Statistical analysis with one-way ANOVA and post hoc Scheffé test (significance level  $\alpha=0.05$ ) is provided. Values with the same letters are not statistically significant different ( $p>0.05$ ). There is a significant three-way interaction effect between thickness, sintering, and processing [ $F(4,126)=9.542$ ;  $p<0.001$ ; three-way ANOVA, significance level  $\alpha=0.05$ ]. Two different CAM fabrication processes (MI = milling, GR = grinding) and three different sintering processes (CL = classic, SS = superspeed, SF = speedfire) were used.

fabrication procedures do not influence the maximum fracture load of monolithic zirconia crowns with different material thicknesses was rejected. An overview of the statistical results for fracture load values is shown in Table 3.

## DISCUSSION

In this study, the fracture load of full-contour monolithic zirconia crowns fabricated with two different fabrication methods, MI and GR, and three different sintering methods, CL, SS, and SF, was investigated as a function of three different material thicknesses (0.5/1.0/1.5 mm). InCoris TZI C zirconia material (Dentsply Sirona) was used for all test groups. Full-contour monolithic zirconia crowns were adhesively luted to methacrylate dies fabricated with SLA technology. Fracture loading was performed subsequent to fatigue loading. Maximum loading forces were statistically analyzed. Statistically significant differences were found for maximum fracture loading values between the different test groups (one-way ANOVA, *post hoc* Scheffé test,  $p<0.05$ ). Several aspects of this study need to be discussed.

In this study, abutment dies with a relatively low E-Modulus of 2.5 GPa were used. In the literature, there is unanimity that the stiffer the abutment die material, the higher the fracture load values for restorations might be. The E-Modulus of human dentin is reported to be between 7 and 13 GPa.<sup>24</sup> It is important to emphasize that the purpose of this study setup was to illustrate a worst-case scenario for the respective restoration material. Therefore, it would seem reasonable that if dentin abutment dies with a higher E-Modulus would have been used in this study, fracture load values for monolithic zirconia crowns might have been higher.

In this study, TCML was performed before load-to-fracture testing. TCML is an important test setup parameter when evaluating zirconia materials. In

the literature, the fracture strength of zirconia restorations has been demonstrated to be reduced by cyclic loading procedures.<sup>25</sup> Additionally, low-temperature aging degradation has been reported to reduce the fracture strength of zirconia.<sup>10</sup> It might thus be concluded that initial fracture load values for monolithic zirconia restorations would have been higher than the fracture load values found in this study.

In the literature, a recommended minimum thickness for monolithic zirconia restorations has not been reported.<sup>16</sup> Several studies are available, indicating that thicknesses of zirconia restorations can be reduced compared with conventional ceramics.<sup>6,7,26</sup> For the zirconia material InCoris TZI C used in this study, the manufacturer's recommendation for minimum material thickness is available describing wall thicknesses of at least 1.0 mm at the lowest point of the main fissure and the cusp and a circular ceramic thickness of 0.8 to 1.00 mm, with a tapering crown edge of 0.5 mm. Sorrentino and others<sup>6</sup> demonstrated that zirconia crown restorations with 0.5-mm thickness configuration showed sufficient fracture resistance. In this study, highest mean fractural loading force values for the 0.5-mm-thickness group was found for group MI\_SS with  $649.9 \pm 112.4$  N. Lowest mean fractural loading force values for the 0.5-mm-thickness group was found for group MI\_SF  $382.4 \pm 30.7$  N. Assuming a maximum chewing force of 600 N for a patient, results of this study suggest that a minimum thickness of 1.0 mm might be suitable for clinical application of full-contour monolithic zirconia crowns. However, it is important to state that *in vitro* findings cannot generally be transferred to *in vivo* conditions because of a more complex parameter setting. Results of this study demonstrate that fracture loading values are positively associated with restoration thicknesses. The highest mean fractural loading force values for the 1.5-mm test

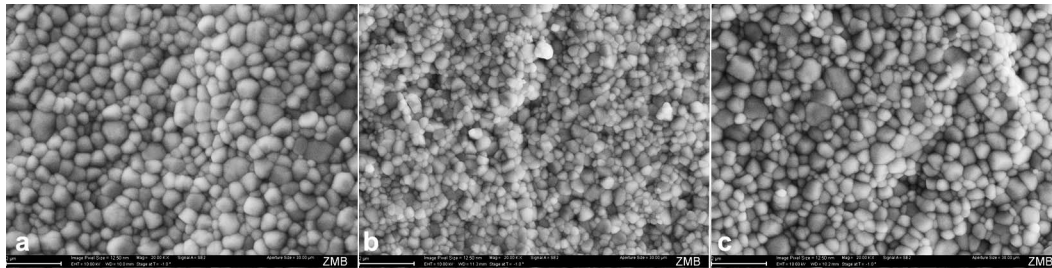


Figure 2. Qualitative SEM images for specimens sintered with different sintering modes: (A) classic sintering, group CL; (B) speedfire sintering, group SF; (C) superspeed sintering, group SS. SEM images with Zeiss Supra V50 SEM, acceleration speed 10 kV, magnification 20,000 $\times$ , specimens with wolfram sputtering; notice different grain size and contrast ratio for different sintering protocols.

group was found for group GR\_SF with  $3678.6 \pm 363.9$  N.

In the literature, contradictory results are available for the evaluation of the influence of the fabrication method on material characteristics of zirconia. Ozer and others<sup>16</sup> found that grinding fabrication did not affect the flexural strength of monolithic zirconia. Canneto and others<sup>18</sup> found that grinding fabrication can result in chip damage, with critical flaws subjected to tension and resulting in strength losses. Fraga and others<sup>27</sup> demonstrated that machining negatively affected the flexural strength of zirconia CAD/CAM ceramic up to 40% of a surface-optimized state. Al-Amleh and others<sup>28</sup> reported that zirconia material is sensitive to manufacturing processes that might lead to framework fractures, but the authors do not provide any recommendations about which processing method is least harmful. In the present study, no statistically different results were found between different fabrication procedures for 0.5- and 1.0-mm-thickness groups ( $p > 0.05$ ). For 1.5-mm thickness, statistically significant differences were only found within the sintering group SF when comparing milling and grinding procedures ( $p < 0.05$ ). Nevertheless, fracture loading values for the 1.5-mm-thickness restorations were within the range of clinical acceptability for both groups. Based on the limitations of this study, results for fracture loading forces thus suggest that both fabrication procedures produce clinically acceptable results for monolithic full-contour zirconia restorations.

In this study, three different sintering procedures were evaluated for the fracture load of monolithic zirconia restorations. For each group sintering temperature, dwell time and total oven time were different. In the literature, several studies are available evaluating the possible effect of sintering procedures on zirconia's material characteristics.<sup>19,20,22,29–31</sup> Kim and others<sup>19</sup> investigated the

effects of sintering conditions on grain size and found that grain size increased with prolonged sintering times. Results are thus in good accordance with results found in this study. Qualitative SEM image analysis of specimens using sintering protocols CL, SS, and SF revealed that grain size was highest for group CL and smallest for group SF (Figure 2). SEM image analysis revealed that the distribution of different grain sizes is different for the three sintering protocols. The best homogeneity for the matrix composition was found for group CL.

In the literature, contradictory results are published for the evaluation of different sintering procedures (ie, different grain sizes and matrix compositions of zirconia on the fracture loading force of zirconia restorations). Ebeid and others<sup>20</sup> reported that biaxial flexural strength is not affected by changes of sintering parameters. Sen and others<sup>31</sup> found that increased sintering temperature resulted in increased translucency with minimal impact on biaxial flexural strength. Inokoshi and others<sup>29</sup> reported that higher sintering temperatures and elongated dwell times increased the grain size. The authors concluded that the increase in grain size might favor low temperature degradation of zirconia.<sup>29</sup> Nakamura and others<sup>30</sup> described that mechanical strength of zirconia decreased with increasing grain size. Ersoy and others<sup>22</sup> used the identical zirconia material in Coris TZI C that was used in this study and demonstrated that a combination of high sintering temperatures and short sintering times increased the flexural strength of zirconia. In this study, no statistically significant differences were found within groups 0.5 and 1.0 mm for different sintering protocols ( $p > 0.05$ ). Statistically significant differences between different sintering protocols were only found between test groups with 1.5 mm thickness ( $p < 0.05$ ). Fracture loading values for 1.5-mm-thickness restorations were within a clinically acceptable range for all three groups. Based on the limitations of this study, the results suggest that

sintering procedures CL, SS, and SF produce clinically acceptable results for monolithic full-contour zirconia restorations.

## CONCLUSIONS

CAD/CAM fabrication and sintering procedures influence the maximum loading force of monolithic zirconia crowns with different material thicknesses. Chairside sintering might be a suitable procedure to produce zirconia restorations with clinically adequate fracture loading values for specific restoration thicknesses. A material thickness of 0.5 mm should be considered as a critical thickness for monolithic zirconia crown restorations.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 13 June 2019)

## References

- Miyazaki T, Nakamura T, Matsumura H, Ban S, & Kobayashi T (2013) Current status of zirconia restoration *Journal of Prosthodontic Research* **57**(4) 236-261.
- Zhang Y & Lawn BR (2018) Novel zirconia materials in dentistry *Journal of Dental Research* **97**(2) 140-147.
- Vigolo P & Mutinelli S (2012) Evaluation of zirconium-oxide-based ceramic single-unit posterior fixed dental prostheses (FDPs) generated with two CAD/CAM systems compared to porcelain-fused-to-metal single-unit posterior FDPs: a 5-year clinical prospective study *Journal of Prosthodontics* **21**(4) 265-269.
- Schley JS, Heussen N, Reich S, Fischer J, Haselhuhn K, & Wolfart S (2010) Survival probability of zirconia-based fixed dental prostheses up to 5 yr: a systematic review of the literature *European Journal of Oral Sciences* **118**(5) 443-450.
- Bachhav VC & Aras MA (2011) Zirconia-based fixed partial dentures: a clinical review *Quintessence International* **42**(2) 173-182.
- Sorrentino R, Triulzio C, Tricarico MG, Bonadeo G, Gherlone EF, & Ferrari M (2016) In vitro analysis of the fracture resistance of CAD-CAM monolithic zirconia molar crowns with different occlusal thickness *Journal of the Mechanical Behavior of Biomedical Materials* **61** 328-333.
- Sun T, Zhou S, Lai R, Liu R, Ma S, Zhou Z, & Longquan S (2014) Load-bearing capacity and the recommended thickness of dental monolithic zirconia single crowns *Journal of the Mechanical Behavior of Biomedical Materials* **35** 93-101.
- Beuer F, Stimmelmayer M, Gueth JF, Edelhoff D, & Naumann M (2012) In vitro performance of full-contour zirconia single crowns *Dental Materials* **28**(4) 449-456.
- Kohorst P, Butzheim LO, Dittmer MP, Heuer W, Borchers L, & Stiesch M (2010) Influence of preliminary damage on the load-bearing capacity of zirconia fixed dental prostheses *Journal of Prosthodontics* **19**(8) 606-613.
- Nakamura K, Harada A, Kanno T, Inagaki R, Niwano Y, Milleding P, & Örtengren U (2015) The influence of low-temperature degradation and cyclic loading on the fracture resistance of monolithic zirconia molar crowns *Journal of the Mechanical Behavior of Biomedical Materials* **47** 49-56.
- Sax C, Hammerle CH, & Sailer I (2011) 10-year clinical outcomes of fixed dental prostheses with zirconia frameworks *International Journal of Computerized Dentistry* **14**(3) 183-202.
- Sulaiman TA, Abdulmajeed AA, Donovan TE, Ritter AV, Lassila LV, Vallittu PK, & Närhi TO (2015) Degree of conversion of dual-polymerizing cements light polymerized through monolithic zirconia of different thicknesses and types *Journal of Prosthetic Dentistry* **114**(1) 103-108.
- Preis V, Behr M, Handel G, Schneider-Feyrer S, Hahnel S, & Rosentritt M (2012) Wear performance of dental ceramics after grinding and polishing treatments *Journal of the Mechanical Behavior of Biomedical Materials* **10** 13-22.
- Stober T, Bermejo JL, Rammelsberg P, & Schmitter M (2014) Enamel wear caused by monolithic zirconia crowns after 6 months of clinical use *Journal of Oral Rehabilitation* **41**(4) 314-322.
- Blatz MB, Vonderheide M, & Conejo J (2018) The effect of resin bonding on long-term success of high-strength ceramics *Journal of Dental Research* **97**(2) 132-139.
- Ozer F, Naden A, Turp V, Mante F, Sen D, & Blatz MB (2018) Effect of thickness and surface modifications on flexural strength of monolithic zirconia *Journal of Prosthetic Dentistry* **119**(6) 987-993.
- Baig MR, Tan KB, & Nicholls JI (2010) Evaluation of the marginal fit of a zirconia ceramic computer-aided machined (CAM) crown system *Journal of Prosthetic Dentistry* **104**(4) 216-227.
- Canneto JJ, Cattani-Lorente M, Durual S, Wiskott AH, & Scherrer SS (2016) Grinding damage assessment on four high-strength ceramics *Dental Materials* **32**(2) 171-182.
- Kim MJ, Ahn JS, Kim JH, Kim HY, & Kim WC (2013) Effects of the sintering conditions of dental zirconia ceramics on the grain size and translucency *Journal of Advanced Prosthodontics* **5**(2) 161-166.
- Ebeid K, Wille S, Hamdy A, Salah T, El-Etreby A, & Kern M (2014) Effect of changes in sintering parameters on monolithic translucent zirconia *Dental Materials* **30**(12) e419-e424.
- Khaledi AAR, Vojdani M, Farzin M, Pirouzi S, & Orandi S (2019) The effect of sintering time on the marginal fit of zirconia copings *Journal of Prosthodontics* **28**(1) e285-e289.
- Ersoy NM, Aydogdu HM, Degirmenci BU, Cokuk N, & Sevımay M (2015) The effects of sintering temperature and duration on the flexural strength and grain size of

- zirconia *Acta Biomaterialia Odontologica Scandinavica* **1(2-4)** 43-50.
23. Krejci I, Reich T, Lutz F, & Albertoni M (1990) [An in vitro test procedure for evaluating dental restoration systems. 1. A computer-controlled mastication simulator] *Schweizer Monatsschrift für Zahnmedizin* **100(8)** 953-960.
  24. Kinney JH, Marshall SJ, & Marshall GW (2003) The mechanical properties of human dentin: a critical review and re-evaluation of the dental literature *Critical Reviews in Oral Biology & Medicine* **14(1)** 13-29.
  25. Ghazy MH, Madina MM, & Aboushelib MN (2012) Influence of fabrication techniques and artificial aging on the fracture resistance of different cantilever zirconia fixed dental prostheses *Journal of Adhesive Dentistry* **14(2)** 161-166.
  26. Choi S, Yoon HI, & Park EJ (2017) Load-bearing capacity of various CAD/CAM monolithic molar crowns under recommended occlusal thickness and reduced occlusal thickness conditions *Journal of Advanced Prosthodontics* **9(6)** 423-431.
  27. Fraga S, Amaral M, Bottino MA, Valandro LF, Kleverlaan CJ, & May LG (2017) Impact of machining on the flexural fatigue strength of glass and polycrystalline CAD/CAM ceramics *Dental Materials* **33(11)** 1286-1297.
  28. Al-Amleh B, Lyons K, & Swain M (2010) Clinical trials in zirconia: a systematic review *Journal of Oral Rehabilitation* **37(8)** 641-652.
  29. Inokoshi M, Zhang F, De Munck J, Minakuchi S, Naert I, Vleugels J, Van Meerbeek B, & Vanmeensel K (2014) Influence of sintering conditions on low-temperature degradation of dental zirconia *Dental Materials* **30(6)** 669-678.
  30. Nakamura K, Adolfsson E, Milleding P, Kanno T, & Ortengren U (2012) Influence of grain size and veneer firing process on the flexural strength of zirconia ceramics *European Journal of Oral Sciences* **120(3)** 249-254.
  31. Sen N, Sermet IB, & Cinar S (2018) Effect of coloring and sintering on the translucency and biaxial strength of monolithic zirconia *Journal of Prosthetic Dentistry* **119(2)** 308.

## Online Only Articles

On occasion we receive manuscripts that we would like to publish, but do not have the page room to include in the print journal. For the full article, please go to [www.jopdentonline.org](http://www.jopdentonline.org) or enter the provided address into your address bar.

### Fracture Resistance and Marginal Adaptation of Capped and Uncapped Bulk-fill Resin-based Materials

HN Al-Nahedh • Z Alawami

Clinical Relevance: Resistance to marginal ridge fracture of class II restorations restored with some bulk-filled resin based composites can be significantly improved by capping with conventional composites and by higher radiant exposure.

<https://doi.org/10.2341/17-367-L>

### Efficacy of a Light-cured Tetracaine-based Anesthetic Gel for Rubber Dam Clamp Placement: A Triple-blind Randomized Clinical Trial

LM Wambier • CC Gonzaga • ACR Chibinski • DS Wambier • PV Farago • AD Loguercio • A Reis

Clinical Relevance: A light-cured anesthetic gel is an excellent alternative to decrease pain when used with a rubber dam clamp to restore noncarious cervical lesions.

<https://doi.org/10.2341/18-130-C>

### Effect of Analgesic Drugs on Tooth Sensitivity Induced by In-office Dental Bleaching: A Systematic Review and Meta-analysis

RTF Costa • SLD Moraes • CAA Lemos • JR SoutoMaior • BC do E Vasconcelos • EP Pellizzer

Clinical Relevance: Clinically, the most commonly observed side effect of bleaching is postoperative sensitivity. Thus, preemptive administration of analgesic drugs can reduce sensitivity and provide more comfort to the patient, without interfering with the results of bleaching.

<https://doi.org/10.2341/18-250-L>

### Efficacy and Adverse Effects of Whitening Dentifrices Compared With Other Products: A Systematic Review and Meta-analysis

A Devila • R Lasta • L Zanella • MA Dall Agnol • SA Rodrigues-Junior

Clinical Relevance: When advising patients, dentists should consider the extrinsic stain removal effect of whitening dentifrices that may ultimately result in color change and yet produce adverse effects more often than regular dentifrices.

<https://doi.org/10.2341/18-298-L>

### Effects of Finishing and Polishing Methods on the Surface Roughness and Surface Free Energy of Bulk-fill Resin Composites

R Ishii • T Takamizawa • A Tsujimoto • S Suzuki • A Imai • WW Barkmeier • MA Latta • M Miyazaki

Clinical Relevance: The finishing and polishing methods, along with the type of resin composite, significantly affected the surface roughness and surface free energy.

<https://doi.org/10.2341/18-246-L>

**This page was intentionally left blank.**

# Fracture Resistance and Marginal Adaptation of Capped and Uncapped Bulk-fill Resin-based Materials

HN Al-Nahedh • Z Alawami

## Clinical Relevance

Resistance to marginal ridge fracture of class II restorations restored with some bulk-filled resin based composites can be significantly improved by capping with conventional composites and by higher radiant exposure.

## SUMMARY

**Objectives:** This study tested the fracture resistance of capped and uncapped bulk-fill composite restorations and compared them to a conventional composite. Also, the effect of different radiant exposure was investigated.

**Methods and Materials:** Flowable and high-viscosity bulk-fill composites (SureFil SDR, Filtek Bulk-Fill Posterior, and Tetric N-Ceram Bulk-Fill) and a nanohybrid resin composite (Filtek Z350 XT) were used. Standardized class II cavities were prepared on extracted premolars, and different restoration protocols were used. In protocol 1 (control), restoration was applied using a layering technique; in protocol

2, restoration was applied in bulk with a capping layer; in protocol 3, restoration was applied in bulk without a capping layer; and in protocol 4, restoration was applied in bulk without a capping layer, and the light curing time was extended. After thermocycling, the restorations were examined for marginal gaps and then subjected to the fracture resistance test using a universal testing machine. Statistical analysis was carried out using two-way analysis of variance (ANOVA) followed by one-way ANOVA at a significance level of  $\alpha = 0.05$ .

**Results:** A statistically significant difference in the fracture resistance of the tested materials and protocols was detected. Filtek Bulk-Fill Posterior achieved the highest fracture resistance values regardless of the protocol used, and its results were comparable to those of Filtek Z350. SDR and Tetric N-Ceram Bulk-Fill achieved their highest strengths when a capping layer was added. Tetric N-Ceram Bulk-Fill showed improvement in fracture resistance with extended light curing, while SDR and Tetric N-Ceram Bulk-Fill achieved similar results with the addition of a capping layer.

\*Hend N Al-Nahedh, BDS, MSD, associate professor, Department of Restorative Dental Sciences, College of Dentistry, King Saud University, Riyadh, Saudi Arabia

Zainab Alawami, BDS, MSD, restorative specialist, Dentistry Division, Eastern Province, Ministry of Health, Eastern, Saudi Arabia

\*Corresponding author: PO Box 60169, Riyadh 11545, Saudi Arabia; e-mail: h\_nahed@yahoo.com

DOI: <http://doi.org/10.2341/17-367-L>

**The uncapped bulk-fill group showed more gap-free margins than the capped group.**

**Conclusion: The new high-viscosity bulk-fill composite restorations seem to have adequate fracture resistance. However, the results are material dependent, and some materials perform better with a capping layer and extended light curing.**

## INTRODUCTION

Resin-based composite (RBC) technology has undergone tremendous developments, producing restorative materials with excellent durability, wear resistance, and esthetics. Controlling the filler architecture through the incorporation of nanotechnology has resulted in dramatic improvements in these materials.<sup>1</sup> The first formulation of RBC was chemically activated, but the generations that followed are photoactivated composites.<sup>1</sup> One of the main problems encountered with resin composites is polymerization shrinkage. This is a reduction of the resin volume that occurs during the polymerization process. This shrinkage, coupled with other factors, such as the RBC modulus of elasticity and restoration confinement within the tooth structure, leads to the development of internal stress within the cavity, which is referred to as polymerization shrinkage stress.<sup>1,2</sup> Polymerization shrinkage stress has been associated with the development of marginal discrepancies, microleakage, postoperative sensitivity, and cusp deflection/enamel microcracks.<sup>2,3</sup> The incremental placement technique was introduced in an attempt to overcome some of the consequences of polymerization shrinkage stress.<sup>4</sup> Other strategies have been proposed in an effort to reduce polymerization shrinkage stress, such as use of different light-curing modes, the use of flowable RBC as an intermediate liner, and the development of new low-shrinkage monomers.<sup>5-7</sup>

Currently, the incremental placement technique is considered the standard of care in cavity preparations exceeding 2 mm. This technique was adopted to ensure sufficient light-curing exposure of the entire increment and to relieve, to some extent, part of the polymerization shrinkage stresses generated during light curing.<sup>8</sup> There are, however, some disadvantages associated with the incremental filling technique. In addition to the technique's requiring a longer time for placement and being labor intensive, there is the possibility of entrapping voids and/or contamination between the layers.<sup>9</sup> Bulk-fill RBCs reduce some of the problems associated with con-

ventional RBC materials occurring with the incremental filling technique.

The bulk-fill RBCs are a newly introduced category of direct resin-based restorative material advocated for use in posterior restorations. They can be applied and cured in a single layer of up to 4 or 5 mm, offering a faster restoration procedure.<sup>10-12</sup> With regard to their mechanical properties, the bulk-fill materials fall between conventional RBCs and flowable RBCs.<sup>13</sup> In general, when compared to conventional RBCs, the bulk-fill RBCs show inferior mechanical properties values.<sup>14</sup> There are two types of bulk-fill materials: flowable and high viscosity. Some manufacturers recommend adding a capping layer of 2 mm to the bulk-fill materials; others report that their materials do not require the addition of a capping layer. The indication for the addition of a capping layer can be attributed to the modulus of elasticity, indentation modulus, and measured hardness values of these materials.<sup>13</sup>

The composite-to-composite bonding during composite layering occurs by the formation of primary covalent bonds between the residual C=C bonds of the previously placed layer and the C=C bonds of the newly applied layer, development of interpenetrating networks, secondary bonding, and mechanical interlocking.<sup>15</sup> It is also believed that the oxygen-inhibited layer increases the bond strength between RBC increments.<sup>16</sup> Having a good bond between the RBC layers is crucial since adequate mechanical properties depend largely on the integrity between layers.<sup>17</sup> For the capping layer of bulk-fill RBCs, manufacturers usually advocate the use of any type of RBC; however, there are some concerns with regard to the bond strength between different types of composites. The microfilled RBC material associations show lower shear bond strength compared to hybrid/hybrid resin and hybrid/nanofiller resin associations. In addition, some materials show higher shear bond strength than others.<sup>18</sup>

The mechanical properties of RBC materials are impacted by the resin material composition, filler content, and coupling process. In addition, the inherent flaws present in RBC also influence mechanical properties. The use of the newly available bulk-fill RBC material is advocated to save time due to their ability to cure in thicker layers. However, several studies have reported low mechanical properties of some bulk-fill material, and caution is advocated when using these materials in areas of high occlusal load.<sup>13,14</sup> Furthermore, there is a lack of information in the literature about the fracture resistance of bulk-filled RBC restorations and the

Table 1: Resin Composite Materials Used in the Study

| Materials/Shade                                                                                                                                                                                                                                                                                                   | Lot Number | Material Type                           | Resin Matrix                                                                      | Filler                                                                                                                                                                                                                                                                   |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------|-----------------------------------------|-----------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| SDR<br>Universal shade                                                                                                                                                                                                                                                                                            | 1501000007 | Flowable bulk-fill composite            | Modified UDMA, di-methacrylate resin, di-functional diluents, EBPADMA, and TEGDMA | Filler content consists of barium and strontium alumino-fluoro-silicate glasses. Filler loading 68% by weight, 47.3% by volume. Inorganic filler particles range from 20 nm to 10 $\mu$ m.                                                                               |
| Tetric N-Ceram Bulk Fill<br>Shade IVB<br>(European trade name is Tetric EvoCeram Bulk-Fill)                                                                                                                                                                                                                       | T47219     | Packable hybrid bulk-fill composite     | Bis-GMA, Bis-EMA, and UDMA                                                        | Filler content consists of barium glass, prepolymer, ytterbium trifluoride, and mixed oxide. Filler loading 75% to 77% by weight, 53% to 55% by volume. Inorganic filler particle size is between 0.04 and 3 $\mu$ m, mean particle size is 0.6 $\mu$ m.                 |
| Filtek Bulk Fill Posterior Restorative<br>Shade A2                                                                                                                                                                                                                                                                | N682081    | Packable nanofilled bulk-fill composite | ERGP-DMA, diurethane-DMA, and 1,12-dodecane-DMA                                   | Filler content consists of nonagglomerated/nonaggregated 20-nm silica filler and 4- to 11-nm zirconia filler, aggregated zirconia/silica cluster filler, and ytterbium trifluoride filler agglomerate 100-nm particles. Filler loading 76.5% by weight, 58.4% by volume. |
| Filtek Z350 XT (3M ESPE), A2 Body shade<br>(North American trade name is Filtek Supreme Ultra)                                                                                                                                                                                                                    | N677462    | Nanohybrid paste composite              | Bis-GMA, UDMA, TEGDMA, PEGDMA, and Bis-EMA                                        | Filler content consists of nonagglomerated/nonaggregated 20-nm silica filler and 4- to 11-nm zirconia filler and aggregated zirconia/silica cluster filler. Filler loading 78.5% by weight, 63.3% by volume.                                                             |
| Abbreviations: UDMA, urethane dimethacrylate; EBPADMA, ethoxylated bisphenol A dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate; Bis-EMA, ethoxylated bisphenol-A dimethacrylate; ERGP-DMA, ERGP-dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate. |            |                                         |                                                                                   |                                                                                                                                                                                                                                                                          |

efficacy of capping bulk-filled materials with conventional RBCs.

The aims of the study were to compare the fracture resistance (or loads at fracture) of different capped and uncapped resin-based bulk-fill restorative materials in deep class II cavity preparations, to compare the fracture resistance of different bulk-fill composite restorations, to compare the fracture resistance of bulk-fill restorations to conventional nanohybrid RBC restorations, and to investigate the effect of using different radiant exposures on fracture resistance of bulk-fill composite materials. The null hypotheses were that 1) there is no significant difference in fracture resistance between capped and uncapped bulk-fill materials, 2) there are no significant differences in the fracture resistance between the bulk-fill materials tested, 3) there is no significant difference in fracture resistance between bulk-fill materials and nanohybrid composites, and 4) the radiant exposure does not influence the fracture resistance of bulk-fill materials.

## METHODS AND MATERIALS

The protocol for this study was registered and approved by the College of Dentistry Research

Center in King Saud University (CDRC registration #PR 0037). Three commercially available bulk-fill materials were used in the study: one flowable and two high-viscosity RBCs. A conventional nanohybrid RBC was used as a control (Table 1). Each RBC material was used with its corresponding bonding agent (Table 2). Eighty intact maxillary premolars were collected after extraction and stored in 0.05% thymol solution at a temperature of 4°C. The teeth were examined under a light microscope (Microscope series 80, SWIFT Instruments International, SA, Tokyo, Japan) at 10 $\times$  magnification to ensure that they were free of defects and fracture lines. The bucco-lingual dimension and crown length of the teeth at the interproximal areas were measured with a digital micrometer gauge (Mitutoyo, Kawasaki, Japan), and only teeth within a size range of less than 1 mm were selected. Later, they were stratified for tooth size and randomly distributed over the study groups (n=8) such that the variance of the mean measurements between groups was less than 5%. The sample size was estimated at  $\alpha = 0.05$  with an expected standard deviation of 0.12<sup>19</sup> and a power of 0.95. The number of specimens for each material was estimated to be eight in each protocol.

| Table 2: Adhesive Systems Used in the Study |            |                                                                                                                                                                                                                |
|---------------------------------------------|------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Adhesive System                             | Lot Number | Application                                                                                                                                                                                                    |
| Single Bond Universal                       | 590863     | Acid etching for 15 s; rinse and dry using cotton pellet and gentle blotting. Apply the adhesive and rub it in for 20 s. Gentle air drying for 5 s. Light cure for 10 s.                                       |
| Prime&Bond NT                               | 1307001099 | Acid etching for 15 s; rinse and dry using cotton pellet and gentle blotting. Apply a generous amount of the adhesive and keep surface wet for 20 s. Gentle air drying for 5 s. Light cure for 10 s.           |
| Tetric N-Bond                               | U34550     | Acid etching for 15 s; rinse and dry using cotton pellet and gentle blotting. Apply a thick layer of adhesive and gently brush for 10 s. Gentle air drying until surface is evenly shiny. Light cure for 10 s. |

Cavity Preparation

Under copious water irrigation, standardized class II occluso-distal cavities were prepared by one operator. To help standardize the preparations, the dimensions were confirmed using a periodontal probe and a digital micrometer with an accuracy level of up to 0.05 mm (Flexbar Tools, New York, NY, USA). The bucco-lingual width of the preparation was 2 mm with an occlusal depth of 2 mm; the distal box extended 6 mm gingivally, ending in dentin right below the cemento-enamel junction with a width of 3.5 mm at the marginal ridge; and the width of the gingival seat was 1 mm (Figure 1). The preparation was done using 1156 round-ended straight fissure carbide burs (Komet Dental, Lemgo, Germany). Each bur was replaced after four preparations.

Restoration Protocol

The teeth were randomly divided into 10 groups and restored by one operator. The restorative materials were placed with their respective bonding agents according to the manufacturers’ instructions (Table 2).

Protocol 1 (control): Eight cavities were incrementally filled with Single Bond Universal/Filtek Z350 XT nanohybrid composite using a horizontal incre-

mental placement technique with 2-mm layer thickness. Each layer was cured for 20 seconds.

Protocol 2 (capped): Twenty-four cavities were bulk-filled in 4-mm increments using different bulk-fill materials (Prime&Bond NT/SureFil SDR, Dentsply, Germany, Single Bond Universal/Filtek Bulk-Fill Posterior, 3M ESPE, USA, and Tetric N-Bond/Tetric N-Ceram Bulk-Fill, Ivoclar-Vivadent, Liechtenstein; n= 8) with the addition of a 2-mm capping layer using the Filtek Z350 XT composite without bonding agent. The first layer was cured for 20 seconds, and the second layer was cured for 10 seconds.

Protocol 3 (uncapped): Twenty-four cavities were restored with the three bulk-filled composite materials (n=8) in two increments: a 4-mm increment cured for 20 seconds and a 2-mm increment cured for 10 seconds.

Protocol 4 (extended light curing): Twenty-four cavities were restored with the three bulk-filled composite materials (n=8) in two increments: a 4-mm increment cured for 40 seconds and a 2-mm increment cured for 20 seconds.

A light-emitting diode light-curing unit was used (Bluephase G2, Ivoclar Vivadent AG, Schaan, Liechtenstein). The light-guide tip was placed in contact with the tooth cusps, and the distance was kept at 2 mm from the marginal ridge. Analysis and measurement of the irradiance values (1200 mW/cm<sup>2</sup>), spectrum emission, and total energy delivered for each specimen were performed with the use of a MARC-RC device (BlueLight Analytics Inc, Halifax, NS, Canada). After restoring the teeth, excess material was removed, and finishing and polishing were done using diamond composite finishing burs (flame-shaped DOS1F, Brasseler, Savannah, GA, USA), Soflex polishing discs (3M ESPE, St Paul, MN, USA), a scalpel, and a number 12 blade.

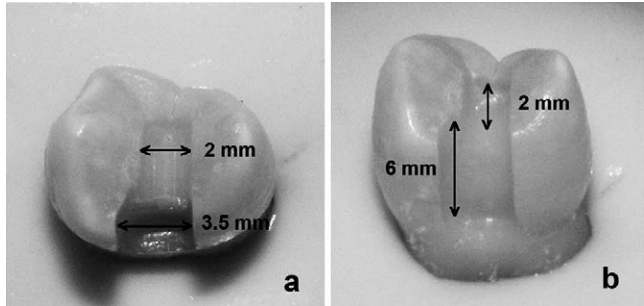


Figure 1. Standard cavity preparation.

Table 3: Mean and Standard Deviation Values of the Mean Fracture Resistance (N) of the Different Groups

| Material                   | Type        | N | Mean  | Standard Deviation | Standard Error | 95% Confidence Interval for Mean |             | Minimum | Maximum |
|----------------------------|-------------|---|-------|--------------------|----------------|----------------------------------|-------------|---------|---------|
|                            |             |   |       |                    |                | Lower Bound                      | Upper Bound |         |         |
| Filtek Z350 XT             | Control     | 8 | 67.55 | 15.66              | 3.25           | 60.9                             | 74.2        | 42.59   | 83.91   |
| SDR                        | Capped      | 8 | 68.95 | 2.59               | 0.91           | 58.04                            | 79.86       | 65.30   | 72.61   |
|                            | Uncapped    | 8 | 48.65 | 6.48               | 2.29           | 44.58                            | 71.70       | 42.41   | 59.58   |
|                            | Extended LC | 8 | 51.81 | 10.52              | 3.72           | 48.24                            | 75.35       | 36.84   | 60.48   |
| Filtek Bulk Fill Posterior | Capped      | 8 | 74.52 | 4.13               | 1.46           | 70.00                            | 79.04       | 65.78   | 78.71   |
|                            | Uncapped    | 8 | 70.61 | 4.19               | 1.48           | 59.70                            | 81.52       | 61.35   | 74.05   |
|                            | Extended LC | 8 | 70.10 | 7.21               | 2.55           | 59.19                            | 81.02       | 57.72   | 80.60   |
| Tetric N-Ceram Bulk Fill   | Capped      | 8 | 71.50 | 2.11               | 0.75           | 60.59                            | 82.42       | 66.98   | 73.93   |
|                            | Uncapped    | 8 | 53.08 | 8.23               | 2.91           | 46.44                            | 73.56       | 40.70   | 65.23   |
|                            | Extended LC | 8 | 63.10 | 7.10               | 2.51           | 56.97                            | 84.09       | 57.48   | 78.38   |

### Examination of Restoration Margins

After restoration, the teeth were stored in a distilled water solution at 37°C for two weeks. During this time, the teeth underwent thermocycling (Thermocycler 1106/1206, SD Mechatronik, Feldkirchen-Westerham, Germany) for 5000 cycles, with water baths between 5°C and 55°C, a dwell time of 30 seconds, and a transfer time of five seconds. Later, the marginal gaps along the restoration margins were measured using a digital microscope (HIROX Digital Microscope, Hirox Co Ltd, Tokyo, Japan) at a magnification of 150× to 200×. The restoration margins were divided into sections: the buccal and lingual margins divided into two sections, the gingival margin divided into three sections, and the interface between the composite layers was divided into two sections. An average reading for each section was taken in micrometers (μm), and the mean marginal gap was calculated.

### Fracture Resistance Test

After the thermocycling and marginal examination were done, the teeth were embedded into a plastic ring 2.5 cm in diameter and 3 cm in length. An autopolymerizing resin (Orthoresin, DeguDent GmbH, Hanau, Germany) was used up to 3 mm below the cemento-enamel junction in order to be able to fit the specimens into the jig of the Instron machine.

A flame-shaped diamond finishing bur was used to create a small flat area in the middle of the restoration's marginal ridge that was used as the point for force loading. Care was taken to ensure that the occluso-gingival length of the restoration was standardized at 6 mm. A universal testing machine (ElectroPuls E3000, Instron, Norwood, MA,

USA) with a smooth, 0.5-mm tipped, round-ended stainless-steel rod attached to its upper member was used to fracture the specimens. They were subjected to a compressive axial loading with a crosshead speed of 1 mm/min. The failure loads of the restorations were determined and recorded in newtons (N). The fracture mode of each specimen was evaluated under a stereomicroscope (SMZ 1000, Nikon, Tokyo, Japan) at 3× magnification and photographed. It was classified into three groups: cohesive fracture of tooth structure, cohesive fracture of the filling material, and mixed fracture of both tooth structure and the filling material. In addition, three representative samples from each group were coated with gold-palladium and examined under a scanning electron microscope (JSM-6360LV, JEOL, Tokyo, Japan).

### Statistical Analysis

The Kolmogorov-Smirnov normality test was carried out and satisfied. The statistical analysis was done using SPSS version 22.0 for Windows (IBM, Chicago, IL, USA). Two-way analysis of variance (ANOVA) and the Tukey honestly significant difference multiple comparisons test were carried out to compare the fracture resistance of different bulk-fill RBCs and determine the influence of the different protocols used. One-way ANOVA was carried out to compare the control with the bulk-fill groups prepared according to the manufacturers' instructions. The level of significance was set at  $\alpha = 0.05$  for all tests.

## RESULTS

The mean fracture loads of the different groups are presented in Table 3 and depicted in Figure 2. The two-way ANOVA revealed a statistically significant

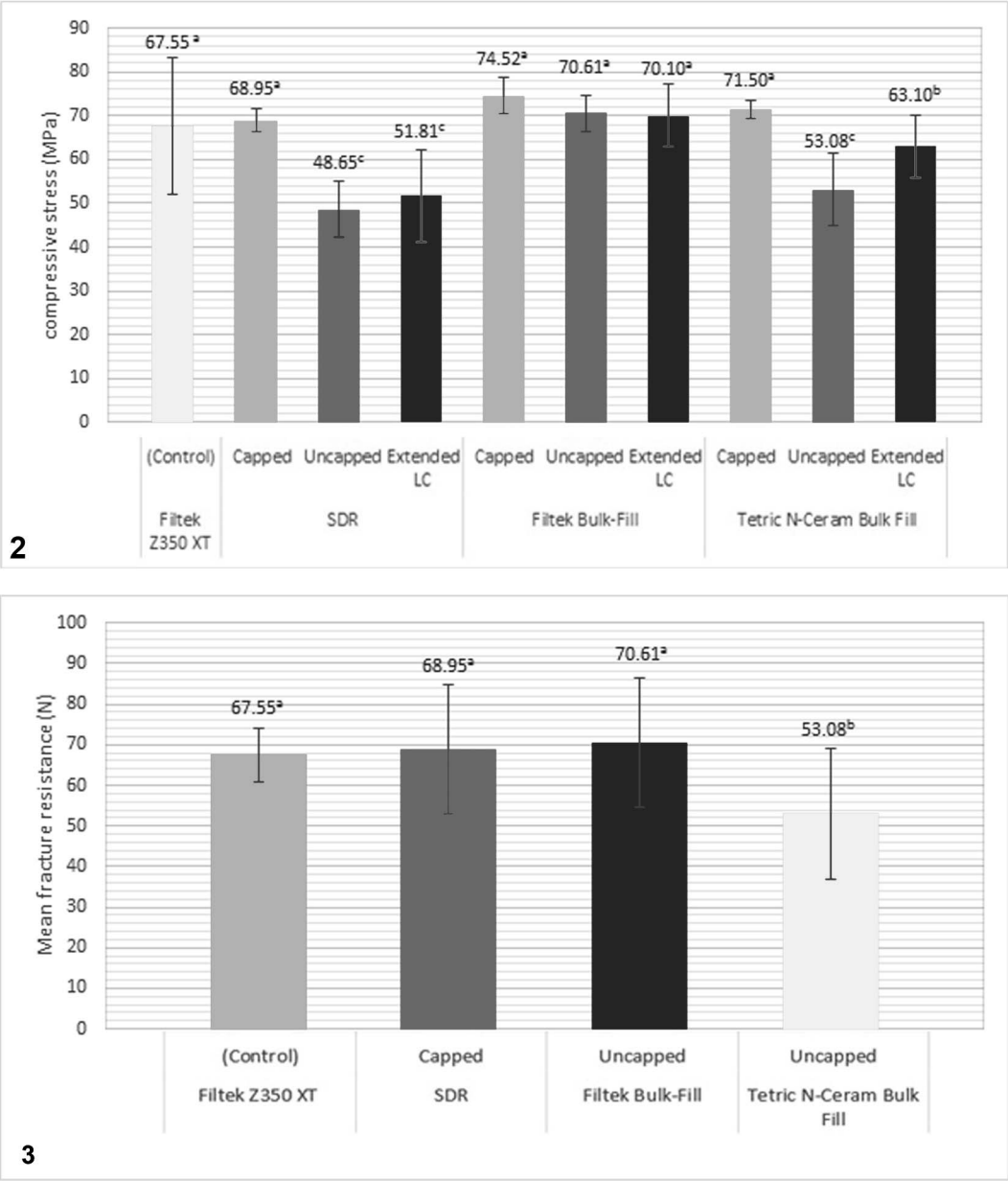


Figure 2. Chart presenting the mean values of fracture resistance (N) of each material within the different protocols with standard deviation bars. Different superscript letters indicate statistical differences between the bulk-fill restorations and protocols.

Figure 3. Chart presenting the mean values of fracture resistance (N) and results of one-way analysis of variance comparing groups prepared following the manufacturers' instructions. Different superscript letters indicate statistical differences.

difference among the experimental groups and among the different materials within the different protocols ( $p<0.001$ ). Also, the interaction between material and protocol was highly significant ( $p=0.0016$ ) (Table 4; Figure 2). The Filtek Bulk-Fill Posterior composite showed the highest fracture resistance values, while SDR showed the lowest values. Filtek Bulk-Fill Posterior was not affected by the different restoration protocols used. Capped SDR

and Tetric N-Ceram bulk-fill groups showed significantly higher fracture resistance values than uncapped groups ( $p<0.000$ ), and the capped SDR group was higher than the extended light cure group ( $p<0.000$ ).

When taking into consideration the protocol effect for all of the bulk-fill materials' fracture resistance values, the statistically significant difference between Filtek Bulk-Fill Posterior and the other bulk-

Table 4: Results of Two-Way Analysis of Variance Showing the Interaction Between the Different Materials and Protocols

| Source              | Type III Sum of Squares | Degrees of Freedom | F-Value   | Significance (p) |
|---------------------|-------------------------|--------------------|-----------|------------------|
| Intercept           | 44,423                  | 1                  | 1085.2062 | 0.000 (2.2e-16)  |
| Material            | 124                     | 2                  | 1.5192    | 0.227            |
| Protocol            | 93                      | 2                  | 1.1405    | 0.326            |
| Material × protocol | 807                     | 4                  | 4.9295    | 0.0016           |
| Residuals           | 2579                    | 63                 |           |                  |

fill materials disappeared when a capping layer was used. Also, a significant difference between SDR and Tetric N-Ceram Bulk-Fill was seen only when a higher radiant exposure was used ( $p=0.02$ ). The radiant exposure increase had no statistically significant effect on the fracture resistance values of bulk-fill restorations; nonetheless, there was a noticeable increase in the fracture resistance of Tetric N-Ceram Bulk-Fill composite, although the increase was not significant statistically ( $p=0.061$ ).

The one-way ANOVA comparing the four groups that were prepared following the manufacturers' instructions revealed a significant difference ( $p=0.002$ ) between them. Tetric N-Ceram Bulk-Fill mean fracture resistance was lower than the other bulk-fill materials. No statistically significant difference was found between the incrementally placed conventional nanohybrid RBC (control) and the Filtek Bulk-Fill Posterior or capped SDR bulk-fill composite groups (Table 5; Figure 3).

Examination of the fractured parts under a light microscope revealed that the fracture mode was mostly a mixed fracture of both tooth structure and the filling material (Figure 4). Severe fractures involving both composite and tooth structure were observed.

The mean marginal gap values for the tested groups are shown in Figure 5. Gaps were highest in the capped groups followed by the extended light cure groups, with uncapped groups demonstrating the fewest number of gaps. The enamel margins (buccal and lingual) showed lower marginal gap values compared to the dentin/cementum margin (gingival) in all tested groups. The gap values of gingival margin of the tested groups were similar to each other. Gaps between increments were found only in the control group and capped bulk-fill group. Figure 6 shows examples of some of the marginal gaps.

Macroscopic examination of the fractured specimens did not reveal any significant plastic deformation. The higher energy involved in fast fracture led to rough fracture surfaces and evidence of multiple crack planes (Figure 7a,b). Fractographic features, such as hackle lines, radial marks, and rip marks, were easily found on the fractured surfaces. Localized crushing at the point of contact was clearly evident at the surface and subsurface (Figure 7c). Also, in many specimens, a semiellipse was observed around the fracture origin. Crack lines started at the surface of the composite and propagated toward the gingival and then deflected, developing a compress-

Table 5: Results of One-Way Analysis of Variance and Tukey Honest Significant Difference Multiple Comparisons of the Mean Fracture Load of Groups Prepared Following the Manufacturers' Instructions

|                                  | Sum of Squares | Degrees of Freedom | F-Value     | Significance (p)  |
|----------------------------------|----------------|--------------------|-------------|-------------------|
| (Intercept)                      | 36,505         | 1                  | 432.9259    | 0.000 (< 2.2e-16) |
| Materials                        | 1566           | 3                  | 6.1895      | 0.0023            |
| Residuals                        | 2361           | 28                 |             |                   |
| Materials                        | Difference     | Lower Bound        | Upper Bound | Significance (p)  |
| Filtek Bulk Fill, Z350 XT        | 3.057625       | -9.47811           | 15.593360   | 0.909             |
| SDR capped, Z350 XT              | 1.397375       | -11.13836          | 13.933110   | 0.990             |
| Tetric N-Ceram, Z350 XT          | -14.474500     | -27.01024          | -1.938765   | 0.019             |
| SDR capped, Filtek Bulk Fill     | -1.660250      | -14.19599          | 10.875485   | 0.983             |
| Tetric N-Ceram, Filtek Bulk Fill | -17.532125     | -30.06786          | -4.996390   | 0.004             |
| TetricN-SDR capped               | -15.871875     | -28.40761          | -3.336140   | 0.009             |

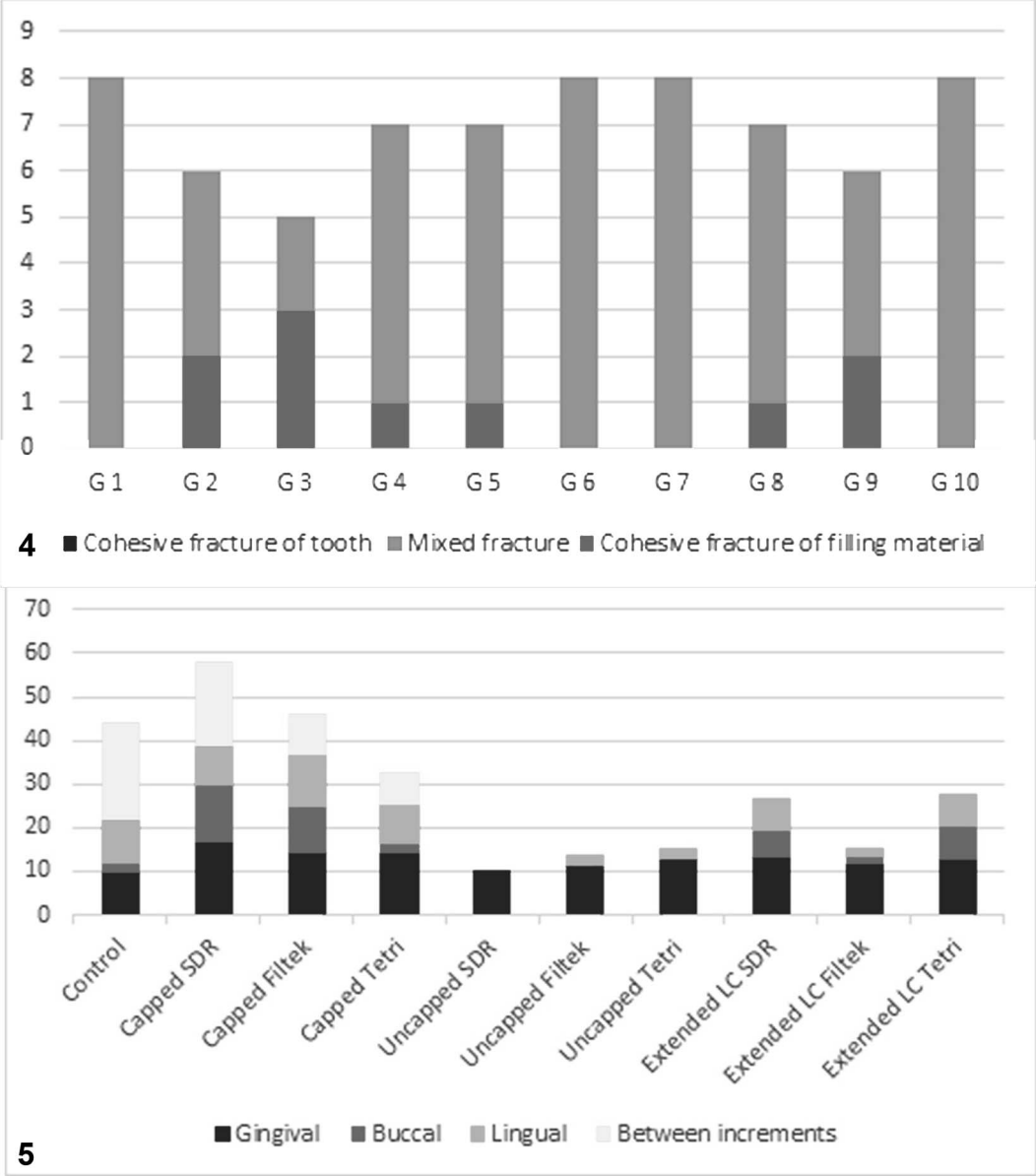


Figure 4. Chart presenting the fracture mode of tested groups.

Figure 5. Mean marginal gap values of tested materials.

sion curl that was observed in many specimens close to the gingival floor (Figure 8). In some specimens, chipping and steplike cracking patterns were seen (Figure 7d). A mirror at a crack origin was not evident on any of the specimens except one. This sample showed two sites of failures: one in the marginal ridge where the force was applied and another in the buccal wall, showing a clear mirror region where some marginal enamel had fractured (Figure 8).

DISCUSSION

Sarrett<sup>20</sup> has reported that the two main concerns with dental RBCs are bulk fracture and secondary caries. The propensity of a particular restoration to fracture in the oral cavity is very difficult to measure since it is affected by many factors. These factors include the material composition and properties, cavity size, integrity of the tooth structure, and the effectiveness of bonding. The restorative procedures allow transfer of the compressive masticatory stress-

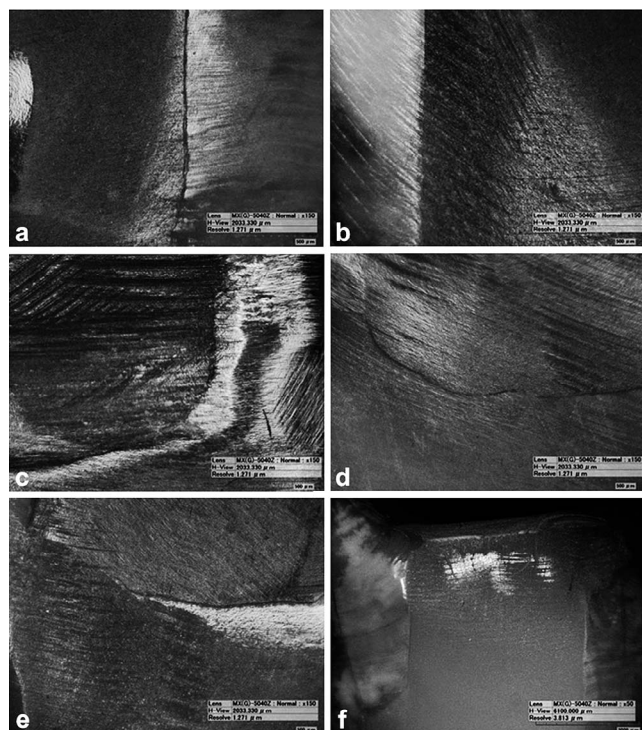


Figure 6. Digital microscope images of marginal gaps. (a): Marginal gaps in lingual wall, capped SDR. (b): Continuous margin in buccal wall, SDR extended LC. (c): Gingival margin gaps, incremental Filtek Z350 XT. (d): Gingival margin gaps, capped Filtek Bulk-Fill. (e): Marginal gaps between increments, capped SDR. (f): Continuous margin between increments, SDR extended LC.

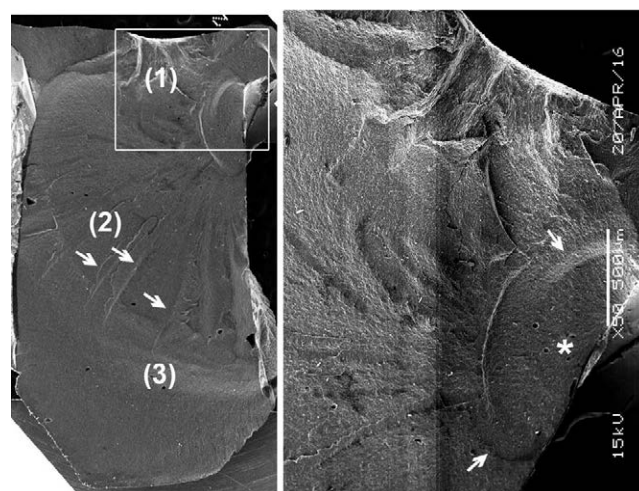


Figure 8. Photomicrograph of a fractured sample showing two areas of fracture: one in the marginal ridge, where the force was applied, and another in the buccal wall, where some marginal enamel fractured. Left: (1): Fracture origin. (2): Mist and hackle region; radial marks can be seen (white arrows). (3): Compression curl. Right: A mirror region can be seen, indicated by the asterisk (\*), and rip marks (semielliptical curved lines) are indicated by double arrows.

es to the tooth structure and alter the original stress distributions. In addition, residual post-gel polymerization shrinkage stresses are affected by the modulus of elasticity of the composite and tooth structures, polymerization conversion rate, bonding conditions, and geometrical features.<sup>21,22</sup> Therefore,

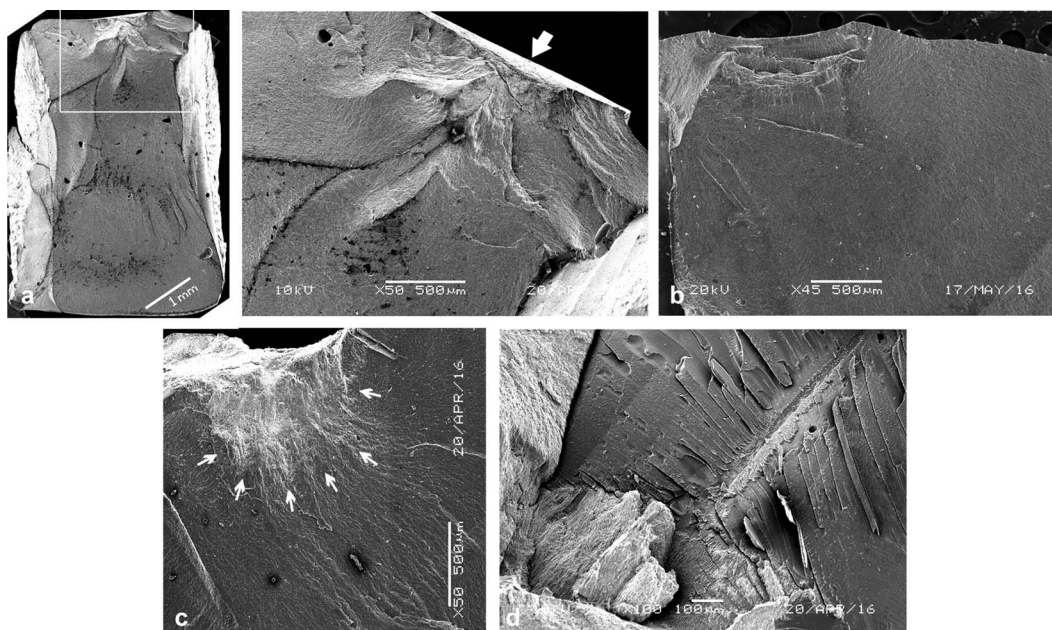


Figure 7. (a) Fractured restoration (left) and crack initiation site represented by the arrow (right); chevron points/pattern (V-shaped radial marks) pointing toward the crack origin site, demonstrating crack branching or crack bifurcation. (b): A sample showing multiple crack planes and hackles radiating from the site of fracture origin. (c): Localized crushing and radiating hackles at the site of fracture origin; a semiellipse is also seen represented by arrows. (d): Steplike cracking patterns.

in studying the fracture resistance (or loads at fracture) of restorations in clinical simulation conditions, the confounding factors are unavoidable, and the results should be carefully interpreted, as it is the outcome of the effects of composite resin mechanical properties, tooth factors, bond strength, and filling techniques.

Bulk-fill RBCs were developed to provide a faster restoration procedure and to minimize the possibility of void entrapment or contamination between the layers. Some studies have also reported reduced cusp deformation and higher fracture resistance with bulk-fill composites.<sup>23,24</sup> The addition of a 2-mm capping layer was recommended by some manufacturers.<sup>13,14</sup> However, the newer generation of bulk-fill RBCs are promoted to be used without a capping layer. In this study, the effect of adding a capping layer and extended light curing on fracture resistance of bulk-fill composite restorations were tested.

The capped bulk-fill composites showed fracture resistance values comparable to that of conventional nanohybrid RBC Filtek Z350 XT. Capping increased the fracture resistance values for SDR and Tetric N-Ceram Bulk-Fill composites. On the other hand, the performance of the Filtek Bulk-Fill composite was not significantly improved by the addition of a capping layer; it achieved consistently high fracture resistance values regardless of capping or light curing. Therefore, the first and third null hypotheses are partially rejected.

The Filtek Bulk-Fill composite's fracture resistance was higher than that of both SDR and Tetric N-Ceram Bulk-Fill; thus, the second null hypothesis is rejected. The performance of Tetric N-Ceram Bulk-Fill improved with the addition of a capping layer, and it was able to achieve fracture resistance values comparable to that of the Filtek Bulk-Fill composite. Higher filler loading has been associated with higher mechanical properties.<sup>25-27</sup> Although the filler content of Filtek Bulk-Fill (58.4% by volume) and Tetric N-Ceram Bulk-Fill (53% to 55% by volume) are comparable and both are promoted to be used without the need of a capping layer, Filtek Bulk-Fill showed a significantly higher fracture resistance. Tetric N-Ceram Bulk-Fill, with its higher filler content, was also expected to achieve higher results than SDR. However, the two materials showed similar performances. This could be related to the filler content of Tetric N-Ceram Bulk-Fill, which included prepolymerized filler particles.

Prepolymerized fillers consist of organic resin and inorganic fillers that are cured and then milled or

ground down, producing resin filler particles that are added to the nonpolymerized resin and inorganic fillers. They are usually added to increase filler loading.<sup>8,28,29</sup> However, the inclusion of prepolymerized fillers in the filler count is believed to overestimate the actual inorganic filler content.<sup>13,28</sup> It has even been questioned whether prepolymerized filler particles should be considered fillers.<sup>26</sup> Lower mechanical properties have been reported with composites containing prepolymerized fillers.<sup>26,30</sup> One of the major limitations of these fillers is the weak bond between the prepolymerized particles and the clinically cured resin matrix that decreases the tensile strength of the composite.<sup>28</sup> The poor integration between prepolymerized fillers and the resin matrix is related to the difficulty of silanization. This is due to a lack of active binding sites since they have already been cured.<sup>26</sup> Another probable cause for the lower fracture resistance values of Tetric N-Ceram Bulk-Fill is the monomer component bisphenol A-glycidyl methacrylate (Bis-EMA). Tetric N-Ceram Bulk-Fill contains Bis-EMA as a comonomer, and lower flexural strength was documented for composites containing this comonomer.<sup>31</sup>

The higher fracture resistance of Filtek Bulk-Fill composites compared to other bulk-fill composites could be related to the incorporation of the aggregated zirconia/silica cluster filler within the filler system. In a study by Curtis and others<sup>32</sup> that investigated the mechanical properties of discrete filler particles by subjecting them to compression forces, the nanocluster showed the capability to withstand higher fracture forces than conventional fillers. They also showed an ability to modify the crack propagation and failure mechanisms, thus contributing to the resin matrix reinforcement. In another study, the nanocluster showed a unique response to cyclic fatigue preloading regimes, increasing the composite's resistance to fracture and strength degradation.<sup>33</sup>

SDR has the lowest filler content (47% by volume) of the bulk-fill materials tested in this study, which may explain the lower fracture resistance values it attained. In this study, the SDR performance was improved when it was capped, reaching values comparable to that of Filtek Bulk-Fill and capped and extended curing Tetric N-Ceram Bulk-Fill. The performances of SDR and Tetric N-Ceram Bulk-Fill were similar. Extending the light curing time did not increase the fracture strength of SDR and Tetric N-Ceram Bulk-Fill composites significantly. However, the additional time allowed uncapped Tetric N-Ceram Bulk-Fill to achieve significantly higher

fracture resistance values than uncapped SDR. According to the manufacturers, Filtek Bulk-Fill and SDR are available in semitranslucent shades that help provide the desired depth of cure,<sup>34,35</sup> while Tetric N-Ceram Bulk-Fill has a more enamel-like translucency. An excellent depth of cure has been previously reported for SDR.<sup>10,36</sup> Son and others<sup>37</sup> reported a lower translucency parameter for Tetric N-Ceram Bulk-Fill compared to SDR and flowable Filtek Bulk-Fill. In addition, lower light attenuation—and thus a higher light transmission—was documented for SDR compared to Tetric N-Ceram Bulk-Fill.<sup>11,37,38</sup>

Li and others<sup>11</sup> suggested that translucency could be the main variable that controls curing efficiency at depth. Translucent materials reportedly allow lower light scattering<sup>39</sup> and have been associated with a higher depth of cure.<sup>40,41</sup> Tetric N-Ceram Bulk-Fill might allow less light penetration during the early polymerization period due to its higher opacity and higher viscosity; however, an increase in light transmittance was observed with the polymerized compared to the unpolymerized composite resin. Additionally, the more organized polymer network permits easier passage of light through the composite bulk.<sup>41,42</sup> The limited depth of cure of Tetric N-Ceram Bulk-Fill and the increase in depth of cure with extended light curing have been observed in several studies.<sup>10,36,41,43,44</sup>

SDR and flowable Filtek Bulk-Fill show consistent polymerization properties at different increment thicknesses, demonstrating their ability to cure sufficiently without the need to extend the light curing process. Garoushi and others<sup>38</sup> measured the degree of conversion of bulk-fill RBC through different increment thicknesses. Both SDR and flowable Filtek Bulk-Fill showed no correlation between thickness and degree of conversion, unlike Tetric N-Ceram Bulk-Fill, which showed a significant correlation as the degree of conversion decreased with the increase in increment thickness. Likewise, Flury and others<sup>45</sup> reported a decrease in Tetric N-Ceram Bulk-Fill Vickers microhardness with the increase in increment thickness, while SDR and flowable Filtek Bulk-Fill showed no significant difference in Vickers microhardness.

When reviewing the literature that compared the mechanical properties of Tetric N-Ceram Bulk-Fill and SDR, Tetric N-Ceram Bulk-Fill was reported to have a higher flexural modulus,<sup>44</sup> Vickers microhardness,<sup>13,14,23</sup> indentation modulus,<sup>13</sup> and modulus of elasticity.<sup>14</sup> SDR seems to have higher flexural strength and fracture toughness.<sup>44</sup> On the other

hand, some studies documented comparable flexural modulus,<sup>13</sup> flexural strength,<sup>14</sup> compressive strength values, and fracture resistance for SDR and Tetric N-Ceram Bulk-Fill.<sup>23</sup> Tetric N-Ceram Bulk-Fill and SDR seem to have comparable mechanical properties. However, when both were compared to conventional RBCs, higher flexural strength, flexural modulus,<sup>46</sup> modulus of elasticity, and Vickers microhardness values<sup>14</sup> were reported for conventional RBCs. Nonetheless, Rosatto and others<sup>23</sup> recorded comparable compressive strength of Tetric N-Ceram Bulk-Fill and conventional RBCs. Another study reported no differences in fracture strength of SDR and a conventional submicron hybrid composite RBC.<sup>47</sup> In this study, only capped Tetric N-Ceram Bulk-Fill and SDR were able to achieve fracture resistance values comparable to that of Filtek Z350 XT.

No differences in marginal integrity between the different bulk-fill materials or between conventional nanohybrid RBC and bulk-fill materials (SonicFill, Tetric EvoCeram Bulk-Fill, and SDR) were detected in previous studies,<sup>48,49</sup> which is in agreement with the findings of this study. Additionally, higher marginal integrities in enamel than in dentin were reported in several studies,<sup>48-50</sup> consistent with the current study.

In bulk-fill materials, better adaptation without gaps between the first and second increments was observed when the same material was used, whereas when a capping layer of a different material was used, gaps were seen between the increments. Furthermore, the gap values of buccal and lingual walls increased when a capping layer was used and when the light cure exposure was extended. Gap development has been associated with polymerization contraction,<sup>49</sup> and extended light curing was correlated with an increase in contraction stress,<sup>10,50</sup> which explains the increase in gap size with extended exposure time. A higher polymerization contraction was reported for SDR than Tetric N-Ceram Bulk-Fill and conventional RBC;<sup>49</sup> nonetheless, the gap values of SDR in this study were comparable to that of other materials. This could be attributed to the advanced stress-decreasing resin technology incorporated within it in the form of a polymerization modulator embedded in the center of the resin monomer backbone.<sup>51</sup> In addition, the flowable nature of the SDR material might have allowed some stress relief during the polymerization contraction. Calheiros and others<sup>52</sup> and Kleverlaan and Feilzer<sup>53</sup> have identified the material's viscoelastic properties as the most influential factor in

polymerization contraction stress development. RBC materials with higher viscosity showed higher contraction stress. The gaps at the gingival margins were wider than at the proximal margins, which is in accordance with the results reported by a previous study.<sup>49</sup>

Polymers can show brittle behavior, which is not significantly influenced by the strain rate. The polymer's susceptibility to cracking and brittle fracture was also found during compression testing. This susceptibility was due to any local inhomogeneity in the specimen that could lead to local tensile stress, which initialized the brittle failure. As the crack propagates, it reaches the maximum speed, which has enough energy to initiate secondary cracks, known as hackle lines. This feature was frequently seen in the scanning electron microscopic images. During the brittle fracture, the cleavage stops, and the fracture planes recombine in the course of further propagation. Chips are formed in front of the crack propagation line, and they explain the occurrence of steplike cracking patterns in some specimens.<sup>54,55</sup>

Fractographic features common to plastics are radial marks and rip marks. Radial marks are lines on a fracture surface that radiate outward from the origin and are formed by the intersection of brittle fractures propagating at different levels. Rip marks are shown around the mirror area as semiellipses curving around the site of impact, suggestive of subcritical crack growth.<sup>54,55</sup>

## CONCLUSIONS

Within the limitations of this study, the following conclusions can be made:

- Filtek Bulk-Fill Posterior restorations showed consistently high fracture resistance under all conditions.
- Filtek Bulk-Fill Posterior and Filtek Z350 XT conventional composite restorations have comparable fracture resistance values.
- With restorations of SDR bulk-fill and Tetric N-Ceram Bulk-Fill, the addition of a capping layer is necessary for adequate fracture resistance.
- Tetric N-Ceram Bulk-Fill restorations can benefit from extending the curing time from 20 to 40 seconds.
- Gap formation does not differ from the bulk-fill composite and the conventional composite.
- Better adaptation between increments/layers is achieved when using the same material.

## Acknowledgements

The authors would like to thank the College of Dentistry Research Center and Deanship of Scientific Research at King Saud University, Saudi Arabia, for approving and funding this research; the College of Dentistry physical laboratory staff for their cooperation and support; Professors Widad Awliya, Mohammad AlQahtain, and Maan AlShaafi for their valuable contribution to this work; Mr Ahmed Alawami and Mr Nasser AlMeflehi for his help with the statistical analysis.

## Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the College of Dentistry Research Center at King Saud University. The approval code for this study is: registration #PR 0037.

## Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 27 March 2019)

## REFERENCES

1. Sakaguchi RL & Powers JM (2012) *Craig's Restorative Dental Materials 13th Edition* Mosby, St Louis MO.
2. Ferracane JL (2005) Developing a more complete understanding of stresses produced in dental composites during polymerization *Dental Materials* **21**(1) 36-42. 10.1016/j.dental.2004.10.004
3. Hilton TJ (2002) Can modern restorative procedures and materials reliably seal cavities? In vitro investigations. Part 1 *American Journal of Dentistry* **15**(3) 198-210
4. Lutz E, Krejci I, & Oldenburg TR (1986) Elimination of polymerization stresses at the margins of posterior composite resin restorations: A new restorative technique *Quintessence International* **17**(12) 777-784.
5. Taubock TT, Feilzer AJ, Buchalla W, Kleverlaan CJ, Krejci I, & Attin T (2014) Effect of modulated photoactivation on polymerization shrinkage behavior of dental restorative resin composites *European Journal of Oral Sciences* **122**(4) 293-302. 10.1111/eos.12139
6. Cara RR, Fleming GJ, Palin WM, Walmsley AD, & Burke FJ (2007) Cuspal deflection and microleakage in premolar teeth restored with resin-based composites with and without an intermediary flowable layer *Journal of Dentistry* **35**(6) 482-489. 10.1016/j.jdent.2007.01.005
7. Cramer NB, Stansbury JW, & Bowman CN (2011) Recent advances and developments in composite dental restorative materials *Journal of Dental Research* **90**(4) 402-416. 10.1177/0022034510381263
8. Ferracane JL (2011) Resin composite—State of the art *Dental Materials* **27**(1) 29-38. 10.1016/j.dental.2010.10.020
9. Campodonico CE, Tantbirojn D, Olin PS, & Versluis A (2011) Cuspal deflection and depth of cure in resin-based composite restorations filled by using bulk, incremental

- and transtooth-illumination techniques *Journal of the American Dental Association* **142**(10) 1176-1182.
10. Zorzin J, Maier E, Harre S, Fey T, Belli R, Lohbauer U, Petschelt A, & Taschner M (2015) Bulk-fill resin composites: Polymerization properties and extended light curing *Dental Materials* **31**(3) 293-301. 10.1016/j.dental.2014.12.010
  11. Li X, Pongprueksa P, Van Meerbeek B, & De Munck J (2015) Curing profile of bulk-fill resin-based composites *Journal of Dentistry* **43**(6) 664-672. 10.1016/j.jdent.2015.01.002
  12. Czasch P & Ilie N (2013) In vitro comparison of mechanical properties and degree of cure of bulk fill composites *Clinical Oral Investigations* **17**(1) 227-235. 10.1007/s00784-012-0702-8
  13. Ilie N, Bucuta S, & Draenert M (2013) Bulk-fill resin-based composites: An in vitro assessment of their mechanical performance *Operative Dentistry* **38**(6) 618-625. 10.2341/12-395-1
  14. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, & Leloup G (2014) Physico-mechanical characteristics of commercially available bulk-fill composites *Journal of Dentistry* **42**(8) 993-1000. 10.1016/j.jdent.2014.05.009
  15. Eliades GC & Caputo AA (1989) The strength of layering technique in visible light-cured composites *Journal of Prosthetic Dentistry* **61**(1) 31-38.
  16. Truffier-Boutry D, Place E, Devaux J, & Leloup G (2003) Interfacial layer characterization in dental composite *Journal of Oral Rehabilitation* **30**(1) 74-77.
  17. Shawkat ES, Shortall AC, Addison O, & Palin WM (2009) Oxygen inhibition and incremental layer bond strengths of resin composites *Dental Materials* **25**(11) 1338-1346. 10.1016/j.dental.2009.06.003
  18. Ribeiro JC, Gomes PN, Moyses MR, Dias SC, Pereira LJ, & Ribeiro JG (2008) Shear strength evaluation of composite-composite resin associations *Journal of Dentistry* **36**(5) 326-330. 10.1016/j.jdent.2008.01.015.
  19. Ornaghi BP, Meier MM, Lohbauer U, & Braga RR (2014) Fracture toughness and cyclic fatigue resistance of resin composites with different filler size distributions *Dental Materials* **30**(7) 742-751. 10.1016/j.dental.2014.04.004
  20. Sarrett DC (2005) Clinical challenges and the relevance of materials testing for posterior composite restorations *Dental Materials* **21**(1) 9-20. 10.1016/j.dental.2004.10.001
  21. Versluis A & Versluis-Tantbirojn D (2011) Filling cavities or restoring teeth? *Journal of the Tennessee Dental Association* **91**(2) 36-42.
  22. Soares CJ, Bicalho AA, Tantbirojn D, & Versluis A (2013) Polymerization shrinkage stresses in a premolar restored with different composite resins and different incremental techniques *Journal of Adhesive Dentistry* **15**(4) 341-350 10.3290/j.jad.a29012
  23. Rosatto CM, Bicalho AA, Verissimo C, Braganca GF, Rodrigues MP, Tantbirojn D, Versluis A, & Soares CJ (2015) Mechanical properties, shrinkage stress, cuspal strain and fracture resistance of molars restored with bulk-fill composites and incremental filling technique *Journal of Dentistry* **43**(12) 1519-1528. 10.1016/j.jdent.2015.09.007
  24. Moorthy A, Hogg CH, Dowling AH, Grufferty BF, Benetti AR, & Fleming GJ (2012) Cuspal deflection and microleakage in premolar teeth restored with bulk-fill flowable resin-based composite base materials *Journal of Dentistry* **40**(6) 500-505. 10.1016/j.jdent.2012.02.015
  25. Beun S, Glorieux T, Devaux J, Vreven J, & Leloup G (2007) Characterization of nanofilled compared to universal and microfilled composites *Dental Materials* **23**(1) 51-59. 10.1016/j.dental.2005.12.003
  26. Randolph LD, Palin WM, Leloup G, & Leprince JG (2016) Filler characteristics of modern dental resin composites and their influence on physico-mechanical properties *Dental Materials* **32**(12) 1586-1599. 10.1016/j.dental.2016.09.034
  27. Ilie N & Hickel R (2009) Investigations on mechanical behaviour of dental composites *Clinical Oral Investigations* **13**(4) 427-438. 10.1007/s00784-009-0258-4
  28. Anusavice KJ, Phillips RW, Shen C, & Rawls HR (2013) *Phillips' Science of Dental Materials 12th edition* Elsevier/Saunders, St Louis MO
  29. Salazar DC, Dennison J, & Yaman P (2013) Inorganic and prepolymerized filler analysis of four resin composites *Operative Dentistry* **38**(6) E201-E209. 10.2341/12-474-1
  30. Blackham JT, Vandewalle KS, & Lien W (2009) Properties of hybrid resin composite systems containing prepolymerized filler particles *Operative Dentistry* **34**(6) 697-702. 10.2341/08-118-L
  31. Goncalves F, Kawano Y, Pfeifer C, Stansbury JW, & Braga RR (2009) Influence of BisGMA, TEGDMA, and BisEMA contents on viscosity, conversion, and flexural strength of experimental resins and composites *European Journal of Oral Sciences* **117**(4) 442-446. 10.1111/j.1600-0722.2009.00636.x
  32. Curtis AR, Palin WM, Fleming GJ, Shortall AC, & Marquis PM (2009) The mechanical properties of nano-filled resin-based composites: Characterizing discrete filler particles and agglomerates using a micromanipulation technique *Dental Materials* **25**(2) 180-187. 10.1016/j.dental.2008.05.013
  33. Curtis AR, Palin WM, Fleming GJ, Shortall AC, & Marquis PM (2009) The mechanical properties of nano-filled resin-based composites: The impact of dry and wet cyclic pre-loading on bi-axial flexure strength *Dental Materials* **25**(2) 188-197. 10.1016/j.dental.2008.06.003
  34. 3M ESPE (2015) Filtek Bulk Fill Posterior Restorative Technical Product Profile. *St Paul, MN: 3M ESPE.*
  35. Dentsply (2015) SureFil SDR Flow DFU. *York, PA: Dentsply.*
  36. Jang JH, Park, SH & Hwang IN (2015) Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin *Operative Dentistry* **40**(2) 172-180. 10.2341/13-307-L
  37. Son SA, Park JK, Seo DG, Ko CC, & Kwon YH (2017) How light attenuation and filler content affect the microhardness and polymerization shrinkage and translucency of

- bulk-fill composites? *Clinical Oral Investigations*. **21(2)** 559-565. 10.1007/s00784-016-1920-2
38. Garoushi S, Vallittu P, Shinya A, & Lassila L (2016) Influence of increment thickness on light transmission, degree of conversion and micro hardness of bulk fill composites *Odontology* **104(3)** 291-297. 10.1007/s10266-015-0227-0
  39. Leloup G, Holvoet PE, Bebelman S, & Devaux J (2002) Raman scattering determination of the depth of cure of light-activated composites: Influence of different clinically relevant parameters *Journal of Oral Rehabilitation* **29(6)** 510-515.
  40. Ferracane JL, Aday P, Matsumoto H, & Marker VA (1986) Relationship between shade and depth of cure for light-activated dental composite resins *Dental Materials* **2(2)** 80-84.
  41. Ilie N & Stark K (2014) Curing behaviour of high-viscosity bulk-fill composites *Journal of Dentistry* **42(8)** 977-985. 10.1016/j.jdent.2014.05.012
  42. dos Santos GB, Alto RV, Filho HR, da Silva EM, & Fellows CE (2008) Light transmission on dental resin composites *Dental Materials* **24(5)** 571-576. 10.1016/j.dental.2007.06.015
  43. Flury S, Hayoz S, Peutzfeldt A, Husler, J & Lussi A (2012) Depth of cure of resin composites: Is the ISO 4049 method suitable for bulk fill materials? *Dental Materials* **28(5)** 521-528, 10.1016/j.dental.2012.02.002
  44. Garoushi S, Sailynoja E, Vallittu PK, & Lassila L (2013) Physical properties and depth of cure of a new short fiber reinforced composite *Dental Materials* **29(8)** 835-841. 10.1016/j.dental.2013.04.016
  45. Flury S, Peutzfeldt A, & Lussi A (2014) Influence of increment thickness on microhardness and dentin bond strength of bulk fill resin composites *Dental Materials* **30(10)** 1104-1112. 10.1016/j.dental.2014.07.001
  46. El-Damanhoury H & Platt J (2014) Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites *Operative Dentistry* **39(4)** 374-382. 10.2341/13-017-L
  47. de Assis FS, Lima SN, Tonetto MR, Bhandi SH, Pinto SC, Malaquias P, Loguercio AD, & Bandeca MC (2016) Evaluation of bond strength, marginal integrity, and fracture strength of bulk- vs incrementally-filled restorations *Journal of Adhesive Dentistry* **18(4)** 317-323. 10.3290/j.jad.a36516
  48. Al-Harbi F, Kaisarly D, Bader D, & El Gezawi M (2016) Marginal integrity of bulk versus incremental fill class II composite restorations *Operative Dentistry* **41(2)** 146-156. 10.2341/14-306-L
  49. Benetti AR, Havndrup-Pedersen C, Honore D, Pedersen MK, & Pallesen U (2015) Bulk-fill resin composites: Polymerization contraction, depth of cure, and gap formation *Operative Dentistry* **40(2)** 190-200. 10.2341/13-324-L
  50. Calheiros FC, Kawano Y, Stansbury JW, & Braga RR (2006) Influence of radiant exposure on contraction stress, degree of conversion and mechanical properties of resin composites *Dental Materials* **22(9)** 799-803. 10.1016/j.dental.2005.11.008
  51. Dentsply (2011) SureFil SDR Flow Scientific Compendium. York, PA: Dentsply.
  52. Calheiros FC, Sadek FT, Braga RR, & Cardoso PE (2004) Polymerization contraction stress of low-shrinkage composites and its correlation with microleakage in class V restorations *Journal of Dentistry* **32(5)** 407-412. 10.1016/j.jdent.2004.01.014
  53. Kleverlaan CJ & Feilzer AJ (2005) Polymerization shrinkage and contraction stress of dental resin composites *Dental Materials* **21(12)** 1150-1157. 10.1016/j.dental.2005.02.004
  54. Becker W & Lampman S (2002) Fracture Appearance and Mechanisms of Deformation and Fracture *Materials Park, OH: ASM International* 559-586
  55. Callister WD & Rethwisch DG (2011) Materials Science and Engineering *New York, NY: John Wiley & Sons*.

# Efficacy of a Light-cured Tetracaine-based Anesthetic Gel for Rubber Dam Clamp Placement: A Triple-blind Randomized Clinical Trial

LM Wambier • CC Gonzaga • ACR Chibinski  
DS Wambier • PV Farago • AD Loguercio • A Reis

## Clinical Relevance

A light-cured anesthetic gel is an excellent alternative to decrease pain when used with a rubber dam clamp to restore noncarious cervical lesions.

## SUMMARY

**Objectives:** To evaluate the efficacy of a new light-cured anesthetic gel for pain control in adults undergoing rubber dam isolation for the restorative treatment of noncarious cervical lesions (NCCLs).

**Methods and Materials:** This study was a randomized, split-mouth, triple-blind, con-

trolled trial. The sample comprised 50 adults with at least one pair of NCCLs located in the same arch but on opposite sides. Simple randomization defined the tooth to receive the light-cured tetracaine-based anesthetic gel or the placebo gel. After cotton roll isolation, the gels were applied in the gingival tissue around the tooth with the aid of the applicator tip of a syringe, left in place for 15 seconds, and light-cured for 15 seconds. Then, a #212 clamp was positioned on the tooth. If the patient reported pain, the clamp was removed, the patient filled

Letícia Maíra Wambier, DDS, MS, PhD, professor, Graduate Program in Dentistry, Positivo University, Curitiba, Brazil

Carla Castiglia Gonzaga, DDS, MS, PhD, professor, Graduate Program in Dentistry, Positivo University, Curitiba, Brazil

Ana Cláudia Rodrigues Chibinski, DDS, MS, PhD, professor, Department of Pediatric Dentistry, State University of Ponta Grossa, Ponta Grossa, Brazil

Denise Stadler Wambier, DDS, MS, PhD, professor, Department of Pediatric Dentistry, State University of Ponta Grossa, Ponta Grossa, Brazil

Paulo Vitor Farago, MSc, PhD, professor, Postgraduate Program in Pharmaceutical Sciences, Department of Pharmaceutical Sciences, State University of Ponta Grossa, Ponta Grossa, Brazil

Alessandro Dourado Loguercio, DDS, MS, PhD, professor, Department of Restorative Dentistry, State University of Ponta Grossa, Ponta Grossa, Brazil

\*Alessandra Reis, DDS, PhD, professor, Department of Restorative Dentistry, State University of Ponta Grossa, Ponta Grossa, Brazil

\*Corresponding author: Av General Carlos Cavalcanti, 4748 Ponta Grossa, Paraná, Brazil; e-mail: reis\_ale@hotmail.com

DOI: <http://doi.org/10.2341/18-130-C>

out a pain intensity form (a 0-10 visual analog scale [VAS] and a 0-4 verbal rating scale [VRS]) and an injectable anesthetic was applied before rubber dam isolation for the restorative procedure. The absolute risk, intensity of pain, and need for rescue anesthesia were analyzed by the McNemar test and the Wilcoxon signed rank test ( $\alpha=5\%$ ).

**Results:** The odds ratio [OR] for pain (OR=3.5; 95% confidence interval [CI]=1.1 to 14.6;  $p=0.03$ ) showed lower reports of pain for the light-cured anesthetic gel. One in five patients will benefit from placement of the light-cured anesthetic gel. On average, pain intensity was one VAS unit lower in those using the light-cured anesthetic gel than in those using the placebo gel. For the VRS, the pain intensity for the light-cured anesthetic gel was 0.4 units lower than the pain intensity for the placebo gel (95% CI=-0.9 to 0.07). The OR for rescue anesthesia was 2.5 (95% CI=0.7 to 10.9;  $p=0.18$ ).

**Conclusions:** The light-cured, tetracaine-based anesthetic gel reduced the absolute risk of pain by 20% in NCCLs.

## INTRODUCTION

The use of restorative treatment for noncarious cervical lesions (NCCLs) has increased, especially in the elderly population, as the occurrence of NCCLs has increased.<sup>1,2</sup> For instance, in the Chinese population, the occurrence of NCCLs was reported to be 63%.<sup>3</sup> This high proportion of patients with NCCLs increased the use of restorative treatment for these specific lesions.<sup>4</sup>

Restorative treatment can be performed with different restorative materials,<sup>5-7</sup> but adhesive systems and composite resins are the most commonly used because of their superior esthetic properties.<sup>8</sup> However, adhesive systems are sensitive to moisture and contaminants, such as saliva,<sup>9</sup> gingival fluid, and blood.<sup>9-11</sup> Adequate moisture control can be achieved with rubber dam isolation, which avoids external contamination and may improve the performance of restorations.<sup>12-14</sup>

Unfortunately, a rubber dam is kept in place by a metal clamp that also aims to retract the gingival tissue to expose the gingival margin of the NCCL. This procedure may be painful and may require the use of local anesthesia for the patient's comfort during the procedure.<sup>15</sup> A recent study demonstrated that approximately 70% of the patients undergoing

restorative treatment of NCCLs required anesthesia for clamp placement.<sup>11</sup>

The problem is that approximately 18% of patients are "very much" afraid of needles,<sup>16</sup> which can be an obstacle for restorations of NCCLs under rubber dam isolation. Apart from that, the numbing produced by the injectable anesthesia, the pain during puncture, and its longer duration are additional disadvantages that lead patients to dislike injectable anesthesia.<sup>17,18</sup> The restorative procedure in NCCLs is usually performed quickly and is not painful as the procedure usually does not require cavity preparation. Therefore, topical anesthesia could be an alternative to produce analgesia for clamp placement.

Topical anesthetics have been used in dentistry to alleviate patient fears and provide pain control.<sup>15</sup> There are some commercially available topical anesthetics. They are suitable to replace injectable anesthesia in some dental procedures, such as probing,<sup>19</sup> scaling and root planning,<sup>20-22</sup> clamp placement in children,<sup>23,24</sup> and placement of mini-implants in orthodontics.<sup>25</sup> However, these products have a very short duration of action (10-15 minutes),<sup>26-28</sup> they flow to the neighboring areas causing an unpleasant anesthesia sensation, and they can be easily washed out by saliva.<sup>23,29,30</sup> Thus, it would be interesting to develop an anesthetic gel capable of overcoming these disadvantages and replacing injectable anesthesia.

This article presents an alternative for topical anesthesia based on a newly developed light-cured anesthetic gel. We developed a topical light-cured anesthetic gel using a more potent anesthetic salt than those used in the commercially available products. This triple-blind, randomized clinical trial evaluated the efficacy of this new light-cured tetracaine-based anesthetic gel compared with a placebo on the risk and intensity of pain in adults undergoing clamp placement for rubber dam isolation before treatment of NCCLs.

## METHODS AND MATERIALS

### Protocol Registration

We registered this research protocol in the Brazilian clinical trials registry (REBEC) under identification number RBR-6HXHX7. The article was written based on the CONSORT (Consolidated Standards of Reporting Trials) statement for randomised trials.<sup>31</sup>

### Trial Design, Settings, and Data Collection Locations

We conducted a triple-blind, randomized, split-mouth, placebo-controlled clinical trial with an equal

allocation ratio. The study was performed from September 18, 2015, to November 11, 2016, in the dental clinics of the School of Dentistry of the local university.

### Recruitment

The participants in the clinical trial (convenience sample) presented themselves for treatment at the School of Dentistry at the local university. No type of advertisement was done in any type of media.

### Eligibility Criteria

For inclusion in the trial, participants had to be at least 18 years old and have good general and oral health. The participants had at least one pair of NCCLs, without undercuts, that needed restorative treatment to avoid excessive dental wear and to eliminate hypersensitivity. These lesions were located in the same arch, but on opposite sides, and the gingival margin had a similar size, depth, and location.

Participants were excluded if the teeth with the NCCLs were endodontically treated. Participants with gingivitis, periodontitis, dental mobility, and bruxism habits were also excluded, as were adults with a history of sensitivity or allergic reaction to ester-based anesthetics.

### Sample-size Calculation

The primary outcome of this study was the absolute risk of pain caused by clamp placement. The risk of pain caused by clamp placement was reported to be 70% in an earlier study.<sup>11</sup> Therefore, at least 40 patients were required to detect a difference in pain risk of 30% with a power of 80% and a significance level of 5%. All calculations for determining the sample size were carried out with a freely available online program for this purpose ([www.sealedenvelope.com](http://www.sealedenvelope.com)).

The sample-size calculation did not account for potential correlation between the paired treatment outcomes. This approach resulted in a larger sample size than if the correlation coefficient between treatment outcomes was not zero. We took this approach because published within-person trials do not report this correlation coefficient; thus, we opted to be conservative.

### Random Sequence Generation and Allocation Concealment

The gel to be used in each tooth was determined by simple randomization. The random sequence was

generated from the same website used for sample-size calculation ([www.sealedenvelope.com](http://www.sealedenvelope.com)) by an investigator who was not involved in implementing the study.

The random sequence generated was individually placed in opaque, consecutively numbered, and sealed envelopes, which were only opened by the operator immediately before the intervention. The envelope contained the group to be used in teeth located in the quadrant with the lowest two-digit World Dental Federation (FDI) numbering system, while the opposite-side teeth received the alternative treatment.

### Blinding

This was a triple-masked clinical trial, in which the patient, operator, and statistician were blinded to the group assignment. Delivery and guidance on the administration of the light-cured gels was performed by a researcher not involved in the implementation.

A single investigator prepared the topical light-cured tetracaine-based anesthetic gel following the description of the patent (BR 10 2016 007724 9).<sup>32</sup> The placebo gel was formulated similarly, following all the steps of the light-cured tetracaine-based anesthetic gel except for inclusion of the anesthetic salt. The light-cured gels were formulated in the Pharmacy School of the local university with the following reagents: tetracaine hydrochloride (5%), inhibitors, monomers, photoinitiators, coinitiators, dyes, and inert filler. The gels had a viscosity similar to that used in light-cured gingival barriers used for in-office bleaching. The anesthesia produced by the gel took approximately 40 minutes, reaching peak 10 minutes after application.

Both gels were transferred to dark syringes to avoid contact with light and to prevent foreknowledge of the group assignment during application. The syringes were marked only with numbered codes so that neither the operators nor the patients could identify them.

### Study Intervention

One researcher was responsible for applying the gels and placing and adapting the rubber dam clamp. Another researcher was responsible for the restorative treatment. First, patients were instructed about all the steps of the treatment and the possible sensations they could experience during clamp adaptation. We made it clear that if there was any objectionable discomfort, an injectable anesthesia would be applied.

The side of the dental arch to be treated was isolated with cotton rolls and saliva ejectors, and the anesthetic gel or placebo gel was placed, with the aid of a syringe tip, in the gingival tissue around the tooth to be restored. The gel was placed 2 mm away from the gingival margin and extended approximately 2 mm beyond the gingival margin on both sides (facial and lingual). The gel was left in place for 15 seconds and then light-cured for 15 seconds with a light-emitting diode light-curing unit (Radii-cal, SDI, Bayswater, Australia) set at 1200 mW/cm<sup>2</sup>. A #212 clamp (Duflex-SS White, Rio de Janeiro, Brazil) was positioned with a door-clamp gripper for adaptation check. At this moment, we asked the patient if he or she felt pain or discomfort. If the answer was positive, the clamp was removed, the intensity of pain/discomfort was recorded (as described in the next section), and an injectable anesthesia (2% lidocaine with epinephrine 1:100,000; Alphacaine; DFL, Rio de Janeiro, Brazil) was applied. The rubber dam was then installed and the clamp placed. Placement of the anesthetic gel did not interfere with clamp placement.

For restoration placement, the enamel surface was etched with 37% phosphoric acid for 15 seconds (Condac 37; FGM, Joinville, Brazil), followed by water washing (15 seconds) and drying with an air stream (10 seconds) keeping the dentin moist. Two coats of the two-step etch-and-rinse adhesive Ambar (FGM) were applied and the solvent evaporated with an air stream for five seconds. The adhesive was light cured for 20 seconds with the same light curing device. The composite resin Opallis (FGM) was incrementally placed and each increment light-cured for 20 seconds. The restorative steps were performed by a calibrated investigator.

### Assessment of the Outcome

All outcomes were measured just after placement of the #212 clamp, as it is the most critical phase in terms of pain sensation during the procedure. The odds of having pain (yes or no) was obtained for each group and organized in a 2 × 2 table for paired data to allow the calculation of the odds ratio. Pain intensity was further evaluated using two different pain scales:

1. Visual analog scale (VAS)<sup>33</sup>: This scale is a 10-cm horizontal line labeled from 0 to 10, where 0 = no pain and 10 = unbearable pain. The patient marked the intensity of the pain with a vertical line across the horizontal line of the scale. Then, the distance in millimeters from 0 to the vertical marked line was measured with the aid of a millimeter ruler.
2. Verbal rating scale (VRS)<sup>20,34,35</sup>: The patient was asked to indicate the numeric value of the degree of pain using a five-point numeric rating scale in which 0 = none, 1 = mild, 2 = moderate, 3 = considerable, and 4 = severe.

### Statistical Analysis

We performed the analysis following the intention-to-treat protocol, and we involved all participants who were randomly assigned.<sup>31</sup> We compared the odds of pain and need for rescue anesthesia for both groups using the McNemar test ( $\alpha=0.05$ ). We calculated the odds ratio (OR) of pain and the need for rescue anesthesia along with the confidence interval (CI) for the effect size. Correlation coefficients were calculated using the Spearman correlation for data on risk of pain and for rescue anesthesia.

We performed the comparison of pain intensity (VAS and VRS) using the Wilcoxon signed-rank test, as data did not follow normal distribution. Correlation coefficients for the paired data for each outcome were calculated. In all statistical tests, the significance level was set at 0.05. We performed all analyses by using the software Sigma Plot version 11.0 (Systat Software Inc, San Jose, CA, USA).

## RESULTS

### Characteristics of Included Participants

Clinical dental examinations were performed for a total of 120 adults; 50 met the eligibility criteria (Figure 1). Patient age ranged from 25 to 66 years (mean age, 40.4 ± 1.0 years); the percentage of men was 40% , and the most treated teeth were the two lower first premolars (FDI two-digit notation: teeth number 34 and 44), which represented 36% of the sample.

### Adherence to the Protocol and Dropouts

Treatment was performed on all participants who qualified, and no missing data were observed. Figure 1 depicts the flow diagram for the different phases of the study design.

No patient felt pain during the restorative procedure and the light-cured gel was not reapplied during any restorative procedure. The restorative procedure took approximately 20 ± 5 minutes.

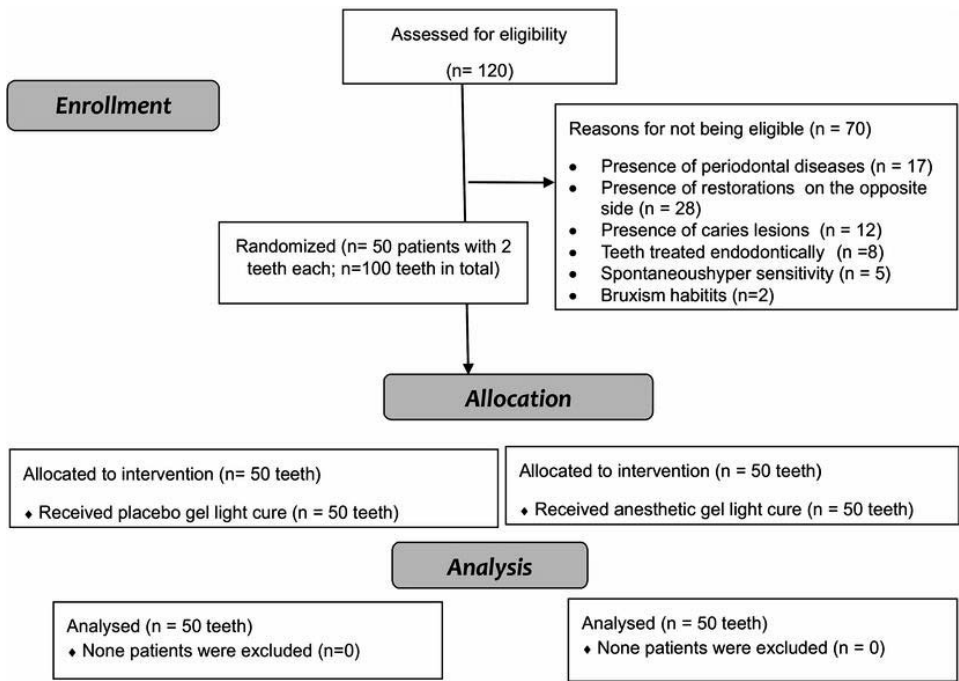


Figure 1. Flow diagram of the split-mouth clinical trial, including detailed information regarding the excluded participants.

**Odds and Intensity of Pain**

The absolute risk of pain in the side where placebo was applied was 56% (95% CI=42% to 68%); while for the anesthetic gel it was 36% (95% CI=24% to 50%) with an absolute risk difference of 20% (95% CI=0.5% to 37%). The number needed to treat was five, meaning that one patient will not report pain for every five patients being treated.

A total of 28 patients presented pain in the placebo group, and from this total, 14 patients reported pain exclusively in the side where the placebo gel was applied, immediately after the placement of the #212 clamp.

When the light-cured tetracaine-based anesthetic gel was used, the number of patients who reported pain was only 18. In comparative terms between groups, the OR for pain (Table 1; OR=3.5; 95% CI=1.1 to 14.6) was on average 3.5 times lower for the light-cured tetracaine-based anesthetic gel, and

a statistically significant difference between groups was detected (Table 1;  $p=0.03$ ).

The phi correlation coefficient using the Spearman test for pairs of binary data was low but significant ( $-0.2$ ;  $p=0.04$ ).

**Pain Intensity**

Pain intensity was positively correlated in both groups. The correlation was moderate for the VAS scale (Table 2,  $r=0.50$ ;  $p<0.0001$ ) and weak for the VRS scale (Table 2;  $r=0.43$ ;  $p=0.001$ ). The mean differences in pain intensity between light-cured tetracaine-based anesthetic gel and placebo gel groups were significant for both scales (VAS: Table 3;  $p=0.005$  and VRS: Table 3;  $p=0.015$ ). On average, pain intensity reduction was small, being one VAS unit lower in the light-cured tetracaine-based anesthetic group compared with the placebo group (95% CI=-2.3 to -0.3; Table 2). For the VRS scale, the

| Table 1: Matched Tabulation of Outcomes With the Two Treatments Along with the Odds Ratio* |                            |          |       |                                      |
|--------------------------------------------------------------------------------------------|----------------------------|----------|-------|--------------------------------------|
| Placebo Gel                                                                                | Light-cured Anesthetic Gel |          |       | Odds Ratio (95% Confidence Interval) |
|                                                                                            | Positive                   | Negative | Total |                                      |
| Positive                                                                                   | 14                         | 14       | 28    | 3.5 (1.1 to 14.6)                    |
| Negative                                                                                   | 4                          | 18       | 22    |                                      |
| Total                                                                                      | 18                         | 32       | 50    |                                      |
| * McNemar test (p=0.03). Correlation coefficient using the Spearman test = -0.2 (p=0.04).  |                            |          |       |                                      |

| Table 2: Means ± Standard Deviations of Pain Intensity for Both Groups Using the Two Pain Scales, as well as, mean difference and the Correlation Coefficient of the Paired Data |                            |             |                                              |                                       |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------|-------------|----------------------------------------------|---------------------------------------|
| Pain Scale                                                                                                                                                                       | Means ± Standard Deviation |             | Mean Difference<br>(95% Confidence Interval) | Correlation Coefficient<br>(p-Value)* |
|                                                                                                                                                                                  | Light-cured Anesthetic Gel | Placebo Gel |                                              |                                       |
| Visual analog scale (0-10 cm)                                                                                                                                                    | 2.4±2.9                    | 3.4±2.8     | −1 (−2.3 to −0.3)                            | 0.50 (p<0.0001)                       |
| Verbal rating scale (0-4)                                                                                                                                                        | 0.8±1.0                    | 1.2±1.1     | −0.4 (−0.9 to −0.07)                         | 0.43 (p=0.001)                        |
| * Wilcoxon signed-rank test.                                                                                                                                                     |                            |             |                                              |                                       |

light-cured tetracaine-based gel was 0.4 units lower than placebo (95% CI=−0.9 to −0.07; Table 2).

**Odds of Rescue Anesthesia**

In all patients who required rescue anesthesia, the injection was performed immediately after placement of the #212 clamp. In comparative terms, the OR for rescue anesthesia (Table 4; OR=2.5; 95% CI=0.7 to 10.9) was on average 2.5 times lower for the light-cured tetracaine-based anesthetic gel, but it did not reach statistical significance (Table 4; p=0.18). The phi correlation coefficient using Spearman test for pairs of binary data was low but significant (−0.12; p=0.20).

**Adverse Effects**

No adverse effects were observed or related by patients during this study.

**DISCUSSION**

This study demonstrated that the newly developed light-cured tetracaine based anesthetic gel can provide a slight reduction in the risk for and intensity of pain related to clamp placement. This finding is a result of an appropriately designed trial that followed the principles of proper randomization, allocation, and blinding of operator, patient, and statistician, thereby reducing selection and performance bias.<sup>36</sup>

This new light-cured tetracaine-based anesthetic gel has two main differences from topical anesthetic gels available in the market: the salt used for the anesthetic purpose and the vehicle of application. In this new light-cured anesthetic gel, we used tetra-

caine instead of prilocaine and lidocaine as tetracaine is more potent.<sup>29,37</sup> Tetracaine is an ester with higher analgesic efficacy<sup>38</sup> and long duration, which is related to its higher hydrophobicity that promotes a prolonged interaction with the sodium channel binding site, resulting in a higher efficacy than other anesthetics.<sup>17,18,39</sup>

Although we have not compared this new light-cured tetracaine-based anesthetic gel with others in the market, from a clinical standpoint, it is clear that the light-cured gel does not flow away from the applied site. This keeps the product from being diluted by saliva and keeps it in the site to be anesthetized, which is an advantage over products marketed as spray, ointment, gel, or adhesive.<sup>30,40,41</sup>

The purpose of this study was to assess the efficacy of the gel against a placebo. We are currently conducting a randomized clinical trial to compare this light-cured tetracaine-based anesthetic gel with other commercial products on the dental market.

Even though the use of a clamp to restore NCCLs is very common, studies that compare the pain associated with this procedure are rare. A recent article showed that 70% of patients reported pain when adapting a #212 clamp or retraction cord without any topical anesthetic. In these cases, injectable anesthesia was required most of the time.<sup>11</sup> The risk of pain during clamp placement in this earlier study<sup>11</sup> was very similar to the results of the present study; 56% of patients reported pain during clamp placement. Therefore, the performance of our new light-cured topical anesthetic gel is an interesting finding because a 20% reduction in the risk of pain was observed. It may be an alternative to replace injectable anesthesia in some cases, thereby

| Table 3: Medians and Interquartile Range of Pain Intensity for Both Groups Using the Two Pain Scales |                               |             |          |
|------------------------------------------------------------------------------------------------------|-------------------------------|-------------|----------|
| Pain Scale                                                                                           | Medians (Interquartile Range) |             | p-Value* |
|                                                                                                      | Light-cured Anesthetic Gel    | Placebo Gel |          |
| Visual analog scale (0-10 cm)                                                                        | 1.2 (0-3.8)                   | 3 (1.0-5.6) | 0.005    |
| Verbal rating scale (0-4)                                                                            | 0 (0-1)                       | 1 (0-2)     | 0.015    |
| * Wilcoxon signed-rank test.                                                                         |                               |             |          |

Table 4: Comparison of the Number of Patients Who Required Rescue Anesthesia in Both Groups Along With the Absolute and the Odds Ratio\*

| Placebo Gel | Light-cured Anesthetic Gel |          |       | Odds Ratio<br>(95% Confidence Interval) |
|-------------|----------------------------|----------|-------|-----------------------------------------|
|             | Positive                   | Negative | Total |                                         |
| Positive    | 10                         | 10       | 20    | 2.5 (0.7 to 10.9)                       |
| Negative    | 4                          | 26       | 30    |                                         |
| Total       | 14                         | 36       | 50    |                                         |

\* McNemar test ( $p=0.18$ ). Correlation coefficient using Spearman test  $-0.12$  ( $p=0.2$ ).

overcoming the patient's fear of needles and the numbness related to local anesthesia.

Some studies have tested the adaptation of a clamp in children undergoing rubber dam isolation for the placement of sealants<sup>23,24,30</sup> and have had positive results for light-cured tetracaine-based anesthetic gel, corroborating the findings of our study. It is known that because of the anatomy of NCCLs, which often present subgingival margins, there is need of gingival retraction, and retraction requires an effective gel containing a potent anesthetic salt.

From a clinical perspective, we may say that a reduction of 20% was observed in the risk of pain. This means that for every five patients needing treatment, one did not experience pain with the anesthetic gel. The number of patients that required for rescue anesthesia was not different between groups as it depends on whether the patient considers the discomfort bearable or unbearable.<sup>22</sup>

Finally, we should discuss the limitation of the present study. The intensity and risk of pain are subjective measures, and therefore depend on individual interpretation.<sup>42,43</sup> When the patient reported pain on the first side treated, there was a tendency to feel pain on the opposite side. As the gels were randomized, this bias was minimized, but probably not completely eliminated. Further well-delineated clinical studies should be conducted to compare the use of different gels for clamp adaptation in NCCLs to minimize pain.

## CONCLUSION

The new light-cured tetracaine-based anesthetic reduced the risk of pain in 20% of patients. Those who experienced pain had a pain intensity that was one unit lower (VAS 0-10) than the placebo gel.

## Acknowledgements

This study was conducted during the doctoral studies of Leticia Maira Wambier under the supervision of Professor Alessandra Reis. This study was partially supported by the National Council for Scientific and Technological Develop-

ment, from the Brazilian government, under grants 304105/2013-9 and 305588/2014-1, Coordination of Improvement of Higher Level Personnel (CAPES), Brazilian Ministry of Education.

## Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the State University of Ponta Grossa. The approval code for this study is 974.479.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 11 April 2019)

## REFERENCES

- Bartlett DW & Shah P (2006) A critical review of non-carious cervical (wear) lesions and the role of abfraction, erosion, and abrasion *Journal of Dental Research* **85**(4) 306-312.
- Wood I, Jawad Z, Paisley C, & Brunton P (2008) Non-carious cervical tooth surface loss: a literature review *Journal of Dentistry* **36**(10) 759-766.
- Yang J, Cai D, Wang F, He D, Ma L, Jin Y, & Que K (2016) Non-carious cervical lesions (NCCLs) in a random sampling community population and the association of NCCLs with occlusive wear *Journal of Oral Rehabilitation* **43**(12) 960-966.
- Reis A, de Geus JL, Wambier L, Schroeder M, & Loguercio AD (2018) Compliance of randomized clinical trials in noncarious cervical lesions with the CONSORT statement: A systematic review of methodology *Operative Dentistry* **43**(3) E129-E151.
- Loguercio AD, Reis A, Barbosa AN, & Roulet JF (2003) Five-year double-blind randomized clinical evaluation of a resin-modified glass ionomer and a polyacid-modified resin in noncarious cervical lesions *Journal of Adhesive Dentistry* **5**(4) 323-332.
- Pena CE, Rodrigues JA, Ely C, Giannini M, & Reis AF (2016) Two-year randomized clinical trial of self-etching adhesives and selective enamel etching *Operative Dentistry* **41**(3) 249-257.
- Zander-Grande C, Amaral RC, Loguercio AD, Barroso LP, & Reis A (2014) Clinical performance of one-step self-etch

- adhesives applied actively in cervical lesions: 24-month clinical trial *Operative Dentistry* **39**(3) 228-238.
8. Schroeder M, Correa IC, Bauer J, Loguercio AD, & Reis A (2017) Influence of adhesive strategy on clinical parameters in cervical restorations: A systematic review and meta-analysis *Journal of Dentistry* **62** 36-53.
  9. Cobanoglu N, Unlu N, Ozer FF, & Blatz MB (2013) Bond strength of self-etch adhesives after saliva contamination at different application steps *Operative Dentistry* **38**(5) 505-511.
  10. Aboushelib MN (2011) Clinical performance of self-etching adhesives with saliva contamination *Journal of Adhesive Dentistry* **13**(5) 489-493.
  11. Loguercio AD, Luque-Martinez I, Lisboa AH, Higashi C, Queiroz VA, Rego RO, & Reis A (2015) Influence of isolation method of the operative field on gingival damage, patients' preference, and restoration retention in noncarious cervical lesions *Operative Dentistry* **40**(6) 581-593.
  12. Brunthaler A, Konig F, Lucas T, Sperr W, & Schedle A (2003) Longevity of direct resin composite restorations in posterior teeth *Clinical Oral Investigations* **7**(2) 63-70.
  13. Heintze SD, Ruffieux C, & Rousson V (2010) Clinical performance of cervical restorations—A meta-analysis *Dental Materials* **26**(10) 993-1000.
  14. Raskin A, Setcos JC, Vreven J, & Wilson NH (2000) Influence of the isolation method on the 10-year clinical behaviour of posterior resin composite restorations *Clinical Oral Investigations* **4**(3) 148-152.
  15. Boyce RA, Kirpalani T, & Mohan N (2016) Updates of topical and local anesthesia agents *Dental Clinics of North America* **60**(2) 445-471.
  16. Armfield JM & Heaton LJ (2013) Management of fear and anxiety in the dental clinic: A review *Australian Dental Journal* **58**(4) 390-407; quiz 531.
  17. Kudo M (2005) Initial injection pressure for dental local anesthesia: effects on pain and anxiety *Anesthesia Progress* **52**(3) 95-101.
  18. Kumar M, Chawla R, & Goyal M (2015) Topical anesthesia *Journal of Anaesthesiology, Clinical Pharmacology* **31**(4) 450-456.
  19. Winning L, Polyzois I, Nylund K, Kelly A, & Claffey N (2012) A placebo-controlled trial to evaluate an anesthetic gel when probing in patients with advanced periodontitis *Journal of Periodontology* **83**(12) 1492-1498.
  20. Mayor-Subirana G, Yague-Garcia J, Valmaseda-Castellon E, Arnabat-Dominguez J, Berini-Ayres L, & Gay-Escoda C (2013) Anesthetic efficacy of oraqix® versus hurricaine® and placebo for pain control during non-surgical periodontal treatment *Medicina Oral, Patología Oral y Cirugía Bucal* **19**(2) 192-201.
  21. Wambier LM, de Geus JL, Boing TF, Chibinski ACR, Wambier DS, Rego RO, Loguercio AD, & Reis A (2017) Intrapocket topical anesthetic versus injected anesthetic for pain control during scaling and root planing in adult patients: Systematic review and meta-analysis *Journal of the American Dental Association* **148**(11) 814-824.e812.
  22. Wambier LM, de Geus JL, Chibinski AC, Wambier DS, Rego RO, Loguercio AD & Reis A (2016) Intra-pocket anaesthesia and pain during probing, scaling and root planing: a systematic review and meta-analysis *Journal of Clinical Periodontology* **43**(9) 754-766.
  23. Lim S & Julliard K (2004) Evaluating the efficacy of EMLA topical anesthetic in sealant placement with rubber dam *Pediatric Dentistry* **26**(6) 497-500.
  24. Yoon RK & Chussid S (2009) Topical anesthesia for rubber dam clamp placement in sealant placement: Comparison of lidocaine/prilocaine gel and benzocaine *Pediatric Dentistry* **31**(5) 377-381.
  25. Valieri MM, de Freitas KM, Valarelli FP, & Cancado RH (2014) Comparison of topical and infiltration anesthesia for orthodontic mini-implant placement *Dental Press Journal of Orthodontics* **19**(2) 76-83.
  26. Daneshkazemi A, Abrisham SM, Daneshkazemi P & Davoudi A (2016) The efficacy of eutectic mixture of local anesthetics as a topical anesthetic agent used for dental procedures: A brief review *Anesthesia, Essays and Researches* **10**(3) 383-387.
  27. Friskopp J, Nilsson M, & Isacson G (2001) The anesthetic onset and duration of a new lidocaine/prilocaine gel intra-pocket anesthetic (Oraqix®) for periodontal scaling/root planing *Journal of Clinical Periodontology* **28**(5) 453-458.
  28. Nayak R & Sudha P (2006) Evaluation of three topical anaesthetic agents against pain: A clinical study *Indian Journal of Dental Research* **17**(4) 155-160.
  29. Meechan JG (2000) Intra-oral topical anaesthetics: A review *Journal of Dentistry* **28**(1) 3-14.
  30. Stecker SS, Swift JQ, Hodges JS, & Erickson PR (2002) Should a mucoadhesive patch (DentiPatch) be used for gingival anesthesia in children? *Anesthesia Progress* **49**(1) 3-8.
  31. Pandis N, Chung B, Scherer RW, Elbourne D, & Altman DG (2017) CONSORT 2010 statement: Extension checklist for reporting within person randomised trials *BMJ* **357** j2835.
  32. Wambier LM, Farago PV, Loguercio AD, & Reis A (2016) Processo de obtenção de barreira gengival anestésica fotoativada **BR 10 2016 007724 9** 1-22 [in Portuguese].
  33. Herdevall BM, Klinge B, Persson L, Huledal G, & Abdel-Rehim M (2003) Plasma levels of lidocaine, o-toluidine, and prilocaine after application of 8.5 g Oraqix in patients with generalized periodontitis: Effect on blood methemoglobin and tolerability *Acta Odontologica Scandinavica* **61**(4) 230-234.
  34. Breivik H, Borchgrevink PC, Allen SM, Rosseland LA, Romundstad L, Hals EK, Kvarstein G, & Stubhaug A (2008) Assessment of pain *British Journal of Anaesthesia* **101**(1) 17-24.
  35. Derman SHM, Lowden CE, Kaus P, & Noack MJ (2013) Pocket-depths-related effectiveness of an intrapocket anaesthesia gel in periodontal maintenance patients *International Journal of Dental Hygiene* **1**(1) 1-4.
  36. Higgins JP, Altman DG, Gotzsche PC, Juni P, Moher D, Oxman AD, Savovic J, Schulz KF, Weeks L, & Sterne JA

- for the Cochrane Bias Methods Group & Cochrane Statistical Methods Group (2011) The Cochrane Collaboration's tool for assessing risk of bias in randomised trials *British Medical Journal* **343** d5928.
37. Giordano D, Raso MG, Pernice C, Agnoletti V, & Barbieri V (2015) Topical local anesthesia: focus on lidocaine-tetracaine combination *Local and Regional Anesthesia* **8** 95-100.
38. Milam SB & Giovannitti JA, Jr. (1984) Local anesthetics in dental practice *Dental Clinics of North America* **28(3)** 493-508.
39. Becker DE & Reed KL (2012) Local anesthetics: Review of pharmacological considerations *Anesthesia Progress* **59(2)** 90-101; quiz 102-103.
40. Koppolu P, Mishra A, Swapna LA, Butchibabu K, Bagalkokar A, & Baroudi K (2016) Comparison of efficacy among various topical anesthetics: An approach towards painless injections in periodontal surgery *Saudi Journal of Anaesthesia* **10(1)** 55-57.
41. Roh J, Han M, Kim KN, & Kim KM (2016) The in vitro and in vivo effects of a fast-dissolving mucoadhesive bilayered strip as topical anesthetics *Dental Materials Journal* **35(4)** 601-605.
42. Chesterton LS, Barlas P, Foster NE, Baxter GD, & Wright CC (2003) Gender differences in pressure pain threshold in healthy humans *Pain* **101(3)** 259-266.
43. Renton T (2015) Pain. Part 1: Introduction to pain *Dental Update* **42(2)** 109-112, 114-106, 118-120 passim.

# Effect of Analgesic Drugs on Tooth Sensitivity Induced by In-office Dental Bleaching: A Systematic Review and Meta-analysis

RTF Costa • SLD Moraes • CAA Lemos  
JR SoutoMaior • BC do E Vasconcelos • EP Pellizzer

## Clinical Relevance

Clinically, the most commonly observed side effect of bleaching is postoperative sensitivity. Thus, preemptive administration of analgesic drugs can reduce sensitivity and provide more comfort to the patient, without interfering with the results of bleaching.

## SUMMARY

**Objective:** This systematic review evaluates the effect of preemptive analgesia on tooth sensitivity induced by in-office tooth bleaching.

**Methods:** The review was structured based on the Preferred Reporting Items for Systematic Reviews and Meta-analyses (PRISMA) checklist. The methods were recorded at PROSPERO

Rayanna Thayse Florêncio Costa, DDS, MS student, School of Dentistry, Pernambuco University (UPE), Camaragibe, Pernambuco, Brazil

\*Sandra Lúcia Dantas de Moraes, DDS, MS, PhD, adjunct professor, School of Dentistry, Pernambuco University (UPE), Camaragibe, Pernambuco, Brazil

Cleidiel Aparecido Araujo Lemos, DDS, MS, PhD, Dental Materials and Prosthodontics, São Paulo State University (UNESP), School of Dentistry, Araçatuba, São Paulo, Brazil

Juliana Raposo Souto-Maior, DDS, MS, PhD, School of Dentistry, Pernambuco University, Camaragibe, Pernambuco, Brazil

(CRD42018095440). Randomized clinical trials, studies published in English, and studies in which the efficacy of preemptive analgesia with analgesic and anti-inflammatory medications prior to in-office tooth bleaching was compared with that of placebo were included. PubMed/MEDLINE, Scopus, Web of Science, and Cochrane Library were used for searching. The electronic search provided 373 articles, and seven of them were selected based on the inclusion criteria.

Belmiro Cavalcanti do Egito Vasconcelos, DDS, MS, PhD, associate professor, School of Dentistry, Pernambuco University (UPE), Camaragibe, Pernambuco, Brazil

Eduardo Piza Pellizzer, DDS, MS, PhD, full professor, Dental Materials and Prosthodontics, São Paulo State University (UNESP), School of Dentistry, Araçatuba, São Paulo, Brazil

\*Corresponding author: PE 54.756-220, Brazil; e-mail: sandra.moraes@upe.br

DOI: 10.2341/18-250-L

**Results:** Immediately after time point, a significant reduction of dental sensitivity was observed in the drug group compared to the control group ( $p=0.02$ ; mean difference [MD]:  $-0.90$ ; confidence interval [CI]:  $-1.63$  to  $-0.16$ ), while there was no significant difference at up to one-hour ( $p=0.22$ ; MD:  $-0.42$ ; CI:  $-1.09$  to  $-0.25$ ), at 1-24-hour ( $p=0.88$ ; MD:  $-0.05$ ; CI:  $-0.61$  to  $0.72$ ), or 24-48-hour ( $p=0.69$ ; MD:  $0.05$ ; CI:  $-0.21$  to  $0.32$ ) time points. The incidence of sensitivity during the procedure was not statistically different between the groups ( $p=0.64$ ; MD:  $0.91$ ; CI:  $0.92$  to  $1.15$ ). The nonsteroidal anti-inflammatory drug group showed a statistically significant reduction ( $p=0.04$ ; MD:  $-0.69$ ; CI:  $-1.36$  to  $-0.03$ ) in tooth sensitivity compared with the other groups.

**Conclusions:** This systematic review and meta-analysis demonstrated that the medications analyzed did not interfere with the incidence of sensitivity symptoms. Regarding the intensity, no difference was observed between the drug and placebo groups at the up to one-hour, 1-24-hour, or 24-48-hour time points, and there was a statistically significant difference at the zero-hour time point in favor of the drug group. However, based on the variables that influenced this result, it should be considered with prudence because a small difference was observed.

## INTRODUCTION

Tooth bleaching is a simple, successful, and widely used technique that aims to improve the esthetics of the smile by lightening the coloration of teeth.<sup>1-3</sup> The most effective techniques are performed in office and at home under the supervision of a dentist.<sup>4</sup> The main agents that promote bleaching are hydrogen peroxide<sup>5</sup> and carbamide peroxide.<sup>6</sup>

At hydrogen peroxide concentrations higher than 35%, tooth sensitivity is a common side effect during and after the procedure.<sup>1,3,7</sup> This effect is attributed to the free radicals released by  $H_2O_2$ . As a result of its low molecular weight, the hydrogen peroxide molecule can traverse the dentin and reach the pulp cavity, resulting in inflammatory reactions of the pulp cells. Patients perceive the manifestation of an acute, temporary, and short-term painful reaction to an external stimulus as tooth sensitivity.<sup>2,3,5,8</sup>

To reduce the sensitivity induced by dental bleaching, clinical studies have tested the use of bleaching gels at lower concentrations with/without a shorter

application time,<sup>9-11</sup> desensitizing dentifrices,<sup>12</sup> and topical desensitizing agents.<sup>13-15</sup> In addition, the use of preprocedure medication to control tooth sensitivity has also been studied<sup>1-5,7,8,16</sup> as a different approach for the control of tooth sensitivity induced by in-office dental whitening.

Therefore, the aim of this systematic review was to evaluate the effectiveness of drugs with analgesic effect on tooth sensitivity induced by in-office dental bleaching when hydrogen peroxide is used. The null hypothesis states that the medications with analgesic effect do not interfere with tooth sensitivity when in-office bleaching is performed using hydrogen peroxide.

## METHOD

### Registry Protocol

This systematic review was structured based on the Preferred Reporting Items for Systematic Reviews and Meta-analyses (PRISMA) checklist.<sup>17</sup> The methods for this systematic review were recorded on the international prospective register of systematic reviews (PROSPERO-CRD42018095440).

### Eligibility Criteria

The selected studies are in accordance with the PICO strategy. The following elements were included: (P) Population: patients subjected to in-office tooth bleaching; (I) Intervention: patients subjected to in-office tooth bleaching with preemptive administration of analgesic drugs; (C) Comparison: Patients subjected to in-office tooth bleaching without the use of preemptive analgesia (negative control); and (O) Outcome: reduction of in-office tooth bleaching-induced sensitivity. Thus, the question asked was "Does the preoperative use of analgesic or anti-inflammatory drugs with analgesic effects minimize the sensitivity induced by in-office tooth bleaching?"

The following inclusion criteria were applied: 1) randomized clinical trials; 2) studies published in English; and 3) studies comparing the efficacy of preemptive analgesic drugs—that is, analgesic and anti-inflammatory medications—administered prior to in-office tooth bleaching with a placebo.

The following exclusion criteria were applied: 1) *in vitro* studies; 2) animal studies; 3) case series or case reports; 4) retrospective studies; 5) revisions and commentaries of revisions; 6) studies evaluating other nonanalgesic and anti-inflammatory drugs; and 7) studies that did not have a control group.

## Information Sources

Two authors (RTFC and CAAL) independently performed an electronic search in the following databases: PubMed/MEDLINE, Scopus, Web of Science, and Cochrane Library; searches were performed using the following terms: bleaching, anti-inflammatory, teeth bleaching, tooth bleaching, whitening, in-office bleaching, sensitivity, analgesic, and nonsteroidal.

In each database, studies were selected based on the title and abstract. To determine inclusion, each article was read in its entirety. The choices made by the two evaluators were analyzed by the third author (SLDM), and consensus was reached through discussion.

The same author performed a manual search for the articles published in the following journals: *Operative Dentistry*, *Journal of Dentistry*, *Journal of Dental Research*, *Clinical Oral Investigations*, and *Quintessence International*, in addition to the grey literature.

## Data Collection Process

One author (RTFC) collected the information from the articles, and another author (CAAL) reviewed the results. The choices made by the two evaluators were analyzed by the third author (SLDM), and consensus was reached through discussion.

The variables collected from the articles were as follows: author, type of study, bleaching agent, number of bleaching sessions, bleaching agent protocol, mean age, drug used to perform preemptive analgesia, dosage, mode of administration, time points of sensitivity assessment, type of scale used for the measurement of sensitivity, mean degree of dental sensitivity, absolute risk, and relative risk.

## Risk of Bias

Two investigators (RTFC and CAAL) rated the quality and risk of bias of the randomized controlled trials using the Cochrane risk-of-bias tool.<sup>18</sup> This tool evaluates selection bias (random sequence generation and allocation), performance bias (blinding of participants and personnel), bias detection, attrition bias (incomplete outcome data), reporting bias (selective reporting), and biases from other sources.

## Summary Measures

The meta-analysis (Reviewer Manager 5 software, Cochrane Group) was based on the Mantel-Haenzel

and inverse variance methods. Data from eligible studies were either dichotomous (tooth sensitivity incidence), evaluated using the risk ratio (RR), or continuous (tooth sensitivity intensity), evaluated using mean differences (MDs), with corresponding 95% confidence intervals (CIs). RR and MD values were considered statistically significant at the  $p < 0.05$  level.

Heterogeneity analyses evaluated the  $I^2$  value (25%=low, 50%=moderate, and 75%=high). The meta-analyses effects were based on the heterogeneity study. When the heterogeneity was statistically significant ( $p < 0.10$ ), random effects meta-analyses were conducted; otherwise, meta-analyses were performed using fixed effects.<sup>19</sup>

## Additional Analysis

The Kappa score was used to evaluate concordance between the examiners in determining the studies that must be included among those identified through the initial database search. It was performed as an additional analysis with the objective of verifying agreement between the two examiners in the search for articles in the databases. Any disagreement between authors was discussed and consensus reached.

## RESULTS

### Literature Search

The electronic search provided 373 articles: 236 from PubMed/Medline, 119 from Web of Science, 10 from Cochrane Library, and 16 from Scopus. After duplicate articles were removed, the remaining 280 articles were searched manually for those published in the journals; one study was selected. The titles and abstracts of the articles were then read, and the eligibility criteria were applied; 12 articles were subjected to analysis. After reading the full articles, five were excluded for the following reasons: the medication used was an antimicrobial<sup>20</sup> or an antioxidant drug;<sup>6</sup> the article was a commentary on a study;<sup>21</sup> the drug used was banned in the United States and most of Europe;<sup>3</sup> and the clinical trial did not include a control group.<sup>22</sup> Thus, seven studies were selected for this systematic review. The flow chart in Figure 1 details the search strategy.

The Kappa scores for the articles selected from PubMed/MEDLINE (0.8), Scopus (1.0), Web of Science (1.0), and the Cochrane Library (1.0) suggested a high level of agreement among the examiners. Based on the results of the Kappa test,

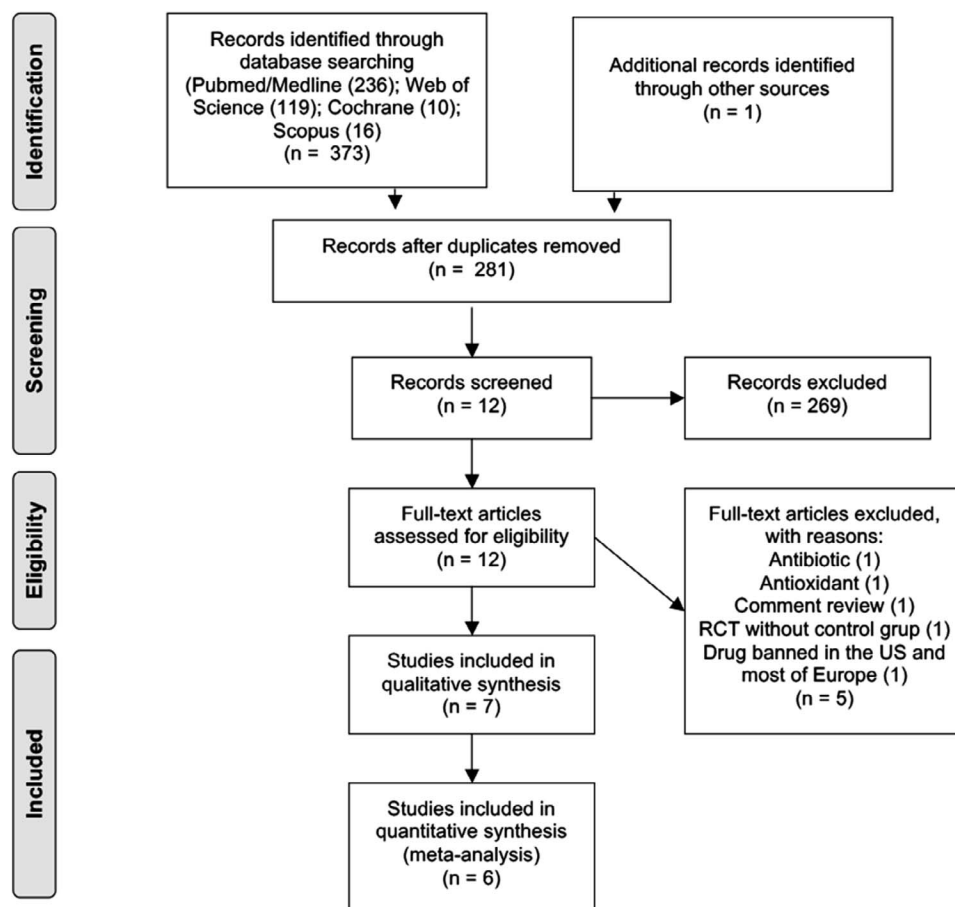


Figure 1. Flowchart describing the search and selection strategies.

there was an “almost perfect agreement” between the reviewers.<sup>23</sup>

### Description of the Studies

Tables 1 and 2 summarize the information from the seven studies included. Six studies were randomized, triple-blinded clinical trials and one was a double-blinded clinical trial. All studies included a placebo control group. The bleaching agent used in all of the studies was hydrogen peroxide, and it was applied in office at a concentration ranging from 35% to 38%. A total of 359 patients were evaluated.

The most commonly used bleaching agent was Whitening HP Maxx (FGM, Joinville, Brazil).<sup>4,7,8,16</sup> In-office bleaching was the procedure of choice in all of the studies, and it was performed in two sessions in all of the studies,<sup>2,4,5,7,8,16</sup> with the exception of one study, in which the procedure was performed in one session.<sup>1</sup> The majority of the studies used the drug only preoperatively,<sup>1,2,5,7</sup> whereas the remaining studies<sup>4,8,16</sup> administered the drug both preoperatively and postoperatively. The drugs selected for

the studies varied; however, ibuprofen<sup>1,8</sup> was used in two studies.

The pharmacokinetic information on each medicine was obtained from the package leaflet, except when a medicine was no longer manufactured by the company. In such an instance, the package leaflet of a drug with the same constitution, but different pharmaceutical manufacturer, was used.

### Quality Assessment of the Studies

A low risk of bias in random sequence generation and allocation concealment (selection bias) was verified for all the studies, with the exception of one;<sup>1</sup> this unverified study was classified as “uncertain” risk of bias. Under the category of blinding the participants and personnel (performance bias), all the studies were verified. Blinding of the outcome assessment (bias detection) was verified in most studies; under this category, only one study<sup>1</sup> was judged as “unclear” risk of bias. Incomplete outcome data was verified in most studies, with the exception of one; this unverified study was classified as “high” risk of bias. All of the selected studies were

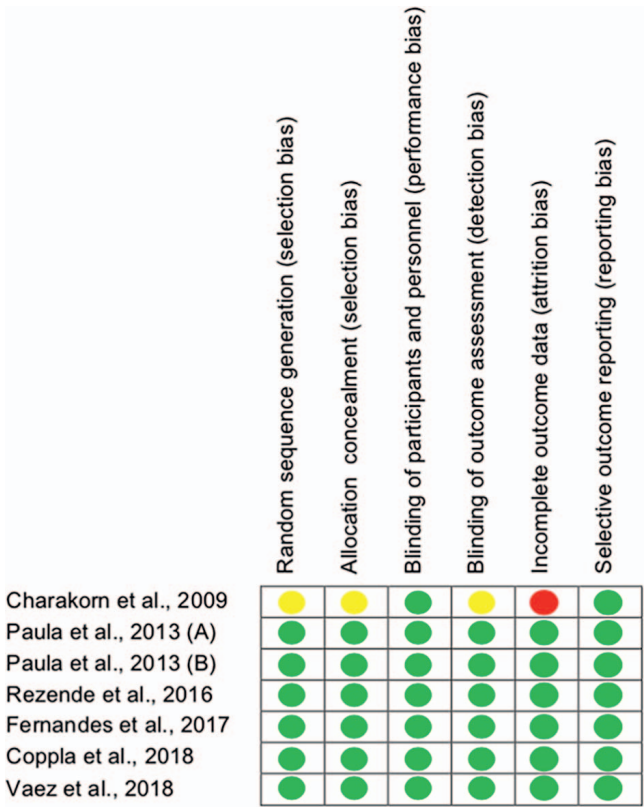


Figure 2. Assessment of the risk of bias in the included studies based on the Cochrane Risk of Bias Tools. (red), High risk of bias; (yellow), Uncertain risk of bias; (green), Low risk of bias.

considered to have low risk of selective reporting (Figure 2).

Meta-analysis

**Sensitivity Level**—Tooth sensitivity was measured using the visual analogue scale (VAS) method. Sensitivity was measured immediately after the end of bleaching procedure (0h) and up to one hour (up to 1h), from 1 to 24 hours (1-24h), and from 24 to 48 hours (24-48h) after the procedure.

Immediately After

Two studies evaluated the sensitivity immediately after tooth bleaching (0h).<sup>1,5</sup> In both of the studies random effects showed statistically significant differences between the drug and placebo groups ( $p=0.02$ ; MD:  $-0.90$ ; CI:  $-1.63$  to  $-0.16$ ). The data were heterogeneous ( $X^2$ :  $3.03$ ;  $I^2=67\%$ ;  $p=0.08$ ), and both the studies showed a common effect size; this suggests that there are differences in the incidence of tooth sensitivity between the analgesic drug and placebo groups (Figure 3).

Up to 1h

Five studies assessed tooth bleaching-induced sensitivity up to 1h after the procedure based on continuous outcome data via visual analogue scales using visual measurements.<sup>1,4,7,8,16</sup> Random effects did not show significant differences between the drug and placebo groups ( $p=0.22$ ; MD:  $-0.42$ ; CI:  $-1.09$  to  $-0.25$ ). Heterogeneity was not significant ( $X^2$ :  $3.09$ ;  $I^2=0\%$ ;  $p=0.54$ ), and all studies included in the analysis shared a common effect size (Figure 3).

1-24h

As shown in Figure 3, five studies<sup>1,4,7,8,16</sup> reported bleaching sensitivity from 1-24 hours after the procedure; sensitivity was assessed by visual measurements. In all of these studies, random effects showed no significant difference between the drug and placebo groups ( $p=0.88$ ; MD:  $-0.05$ ; CI:  $-0.61$  to  $0.72$ ). The data were not heterogeneous ( $X^2$ :  $1.12$ ;  $I^2=0\%$ ;  $p=0.89$ ), and all studies included in the analysis shared a common effect size.

24-48h

Four studies reported tooth bleaching sensitivity from 24 to 48 hours after the procedure; in these studies sensitivity was assessed by visual measurements.<sup>4,7,8,16</sup> The results showed no difference between the drug and placebo groups ( $p=0.69$ ; MD:  $0.05$ ; CI:  $-0.21$  to  $0.32$ ). The data were not heterogeneous ( $X^2$ :  $0.66$ ;  $I^2=0\%$ ;  $p=0.42$ ), and all the studies included in the analysis shared a common effect size (Figure 3).

**Sensitivity Incidence**—Six studies assessed the incidence of sensitivity (Figure 4).<sup>2,4,5,7,8,16</sup> The results showed no significant difference between the drug and placebo groups ( $p=0.64$ ; MD:  $0.91$ ; CI:  $0.92$  to  $1.15$ ). The data were heterogeneous ( $X^2$ :  $6.29$ ;  $I^2=21\%$ ;  $p=0.28$ ), and all the studies shared a common effect size.

**Drug Groups**—Subgroup analyses were conducted to examine the effect of medication on the intensity of sensitivity. The nonsteroidal anti-inflammatory drug (NSAID) group showed a significant decrease in tooth sensitivity immediately after the bleaching procedure ( $p=0.04$ ; MD:  $-0.69$ ; CI:  $-1.36$  to  $-0.03$ ). The data did not show heterogeneity ( $X^2$ :  $2.03$ ;  $I^2=0\%$ ;  $p=0.57$ ) (Figure 5).

DISCUSSION

The current results show that analgesia with preemptive medication was effective in decreasing tooth sensitivity immediately after the in-office

Table 1: Characteristics of Included Studies

| Study                      | Type of Study                 | Bleaching Agent                            | Session | Bleaching Protocol |
|----------------------------|-------------------------------|--------------------------------------------|---------|--------------------|
| Charakorn and others, 2009 | RCT (double-blind)            | 38% HP (Opalescence Xtra Boost, Ultradent) | 1       | 2 × 20'            |
| Paula and others, 2013 (A) | RCT (triple-blind)            | 35% HP (Whiteness HP Maxx, FGM)            | 2       | 3 × 15'            |
| Paula and others, 2013 (B) | RCT (triple-blind)            | 35% HP (Whiteness HP Maxx, FGM)            | 2       | 3 × 15'            |
| Rezende and others, 2016   | RCT (triple-blind)            | 35% HP (Whiteness HP Maxx, FGM)            | 2       | 3 × 15'            |
| Fernandes and others, 2017 | RCT (triple-blind; crossover) | 35% HP (Whiteness HP Blue, FGM)            | 2       | 1 × 40'            |
| Coppla and others, 2018    | RCT (triple-blind)            | 35% HP (Whiteness HP Maxx, FGM)            | 2       | 3 × 15'            |
| Vaez and others, 2018      | RCT (triple-blind)            | 35% HP (Whiteness HP Blue, FGM)            | 2       | 1 × 40'            |

Abbreviations: HP, hydrogen peroxide; RCT, randomized clinical trial; SD, single dose; BTP, before the procedure; ', minutes.

bleaching procedure. However, there was no difference in the incidence of sensitivity-associated symptoms between the drug and placebo groups at the 1-24h or 24-48h time points. Thus, the null hypothesis is partially rejected in this study.

The decreased sensitivity immediately after the bleaching procedure can be attributed not only to the peak plasma concentration of the drug during this period<sup>5</sup> but also to the fact that the production of prostaglandins, which signal inflammation, occurs

Table 2: Characteristics of Included Studies

| Study                      | Moment of Evaluations | Scale         | Tooth Sensitivity Intensity Mean (Standard Deviation) |              |
|----------------------------|-----------------------|---------------|-------------------------------------------------------|--------------|
|                            |                       |               | Placebo                                               | Drug         |
| Charakorn and others, 2009 | 0 h                   | VAS (1-10 cm) | 2.66 (±3.10)                                          | 0.50 (±0.99) |
|                            | Up to 1 h             |               | 3.09 (±3.05)                                          | 3.15 (±3.21) |
|                            | 24 h                  |               | 3.11 (±3.26)                                          | 2.58 (±3.08) |
| Paula and others, 2013 (A) | Up to 1 h             | VAS (1-10 cm) | 3.3 (±2.9)                                            | 2.6 (±2.4)   |
|                            | 1-24 h                |               | 2.4 (±2.7)                                            | 2.2 (±2.8)   |
|                            | 24-48 h               |               | 0 (±0)                                                | 0.1 (±0.4)   |
| Paula and others, 2013 (B) | Up to 1 h             | VAS (1-10 cm) | 3.3 (±2.9)                                            | 1.5 (±2.0)   |
|                            | 1-24 h                |               | 2.4 (±2.7)                                            | 3.0 (±2.8)   |
|                            | 24-48 h               |               | 0.0 (±0.0)                                            | 0.0 (±0.0)   |
| Rezende and others, 2016   | During bleaching      | VAS (1-10 cm) | 2.7 (±2.3)                                            | 3.2 (±2.7)   |
|                            | Up to 1 h             |               | 3.5 (±2.9)                                            | 3.5 (±2.7)   |
|                            | 24 h                  |               | 2.2 (±2.8)                                            | 1.9 (2.6)    |
|                            | 48 h                  |               | 0.4 (±1.7)                                            | 0.2 (±0.8)   |
| Fernandes and others, 2017 | During bleaching      | VAS (1-10 cm) | 1.77 (±1.94)                                          | 1.75 (±2.11) |
|                            | 0 h                   |               | 2.12 (±2.26)                                          | 1.56 (±1.95) |
|                            | 24 h                  |               | NR                                                    | NR           |
| Coppla and others, 2018    | Up to 1 h             | VAS (1-10 cm) | 3.8 (±2.8)                                            | 3.6 (±2.7)   |
|                            | 1-24 h                |               | 2.3 (±2.4)                                            | 2.6 (±2.9)   |
|                            | 24-48 h               |               | 0.2 (±0.7)                                            | 0.3 (±0.8)   |
| Vaez and others, 2018      | During bleaching      | VAS (1-10 cm) | NR                                                    | NR           |
|                            | 0 h                   |               |                                                       |              |
|                            | 24 h                  |               |                                                       |              |

Abbreviations: NR, Not related; CI, confidence interval; VAS, Visual analog scale.

Table 1: Characteristics of Included Studies (ext.)

| Study                      | Mean Age ( $\pm$ SD) | n  | Drug                                 | Dosage                                                         | Administration |
|----------------------------|----------------------|----|--------------------------------------|----------------------------------------------------------------|----------------|
| Charakorn and others, 2009 | 25.0 ( $\pm$ 4.2)    | 15 | Ibuprofen 600 mg                     | SD: 30' BTP                                                    | Oral           |
|                            |                      | 16 | Placebo capsules                     |                                                                |                |
| Paula and others, 2013 (A) | 26.7 ( $\pm$ 6.1)    | 15 | Etoricoxib 60 mg                     | First dose: 1 h BTP<br>+ extra dose: after 24 h                | Oral           |
|                            | 26.4 ( $\pm$ 6.8)    | 15 | Placebo capsules                     |                                                                |                |
| Paula and others, 2013 (B) | 32.9 ( $\pm$ 9.9)    | 15 | Ibuprofen 400 mg                     | First dose: 1 h BTP<br>+ extra doses: 8/8 h for 2 d            | Oral           |
|                            | 26.4 ( $\pm$ 6.8)    | 15 | Placebo capsules                     |                                                                |                |
| Rezende and others, 2016   | 22.7 ( $\pm$ 4.5)    | 33 | Dexamethasone 4 mg                   | First dose: 2 capsules 1 h BTP<br>+ extra doses: 6/6 h for 2 d | Oral           |
|                            | 22.4 ( $\pm$ 3.6)    | 30 | Placebo capsules                     |                                                                |                |
| Fernandes and others, 2017 | NR                   | 50 | Naproxen 500 mg                      | SD: 1 h BTP                                                    | Oral           |
|                            |                      |    | Placebo capsules                     |                                                                |                |
| Coppla and others, 2018    | 22.3 ( $\pm$ 6.3)    | 53 | Acetaminophen 500 mg + Codeine 30 mg | SD: 1 h BTP                                                    | Oral           |
|                            | 22.5 ( $\pm$ 5.0)    | 52 | Placebo capsules                     |                                                                |                |
| Vaez and others, 2018      | NR                   | 25 | Etodolac 400 mg                      | SD: 1 h BTP                                                    | Oral           |
|                            |                      | 25 | Placebo capsules                     |                                                                |                |

primarily within the first hour after the stimulus of damage is initiated. According to de Paula and others,<sup>8</sup> another explanation for the long-term ineffectiveness of preemptive analgesia in tooth whitening is the possibility that other inflammatory mediators (other than COX-1 and COX-2) are

activated over time and are involved in the mechanism of pain, with the possibility of a neurogenic reaction.<sup>2,8,24,25</sup>

The administration of preemptive medication has been suggested<sup>1-8,16,20</sup> to minimize discomfort imme-

Table 2: Characteristics of Included Studies (ext.)

| Study                      | No. of Participants Who Experienced Tooth Sensitivity During the Tooth Bleaching |         |      | Absolute Risk (95% CI) |                | Relative Risk (95% CI) |
|----------------------------|----------------------------------------------------------------------------------|---------|------|------------------------|----------------|------------------------|
|                            |                                                                                  | Placebo | Drug | Placebo                | Drug           |                        |
| Charakorn and others, 2009 |                                                                                  | NR      | NR   | NR                     | NR             | NR                     |
| Paula and others, 2013 (A) | Yes                                                                              | 9       | 11   | 60 (35-80)             | 73 (48-89)     | 1.2 (0.73-2.0)         |
|                            | No                                                                               | 6       | 4    |                        |                |                        |
| Paula and others, 2013 (B) | Yes                                                                              | 12      | 13   | 80 (55-93)             | 87 (62-96)     | 0.92 (0.67-1.3)        |
|                            | No                                                                               | 3       | 2    |                        |                |                        |
| Rezende and others, 2016   | Yes                                                                              | 27      | 30   | 90 (76-97)             | 91 (74-97)     | 1.01 (0.86-1.18)       |
|                            | No                                                                               | 3       | 3    |                        |                |                        |
| Fernandes and others, 2017 | NR                                                                               | NR      | NR   | NR                     | NR             | NR                     |
|                            | Yes                                                                              | 52      | 50   |                        |                |                        |
| Coppla and others, 2018    | No                                                                               | 1       | 2    | 96.2 (87.2-98.9)       | 96.2 (87-98.4) | 1.0 (0.92-1.07)        |
| Vaez and others, 2018      |                                                                                  | NR      | NR   | NR                     | NR             | NR                     |

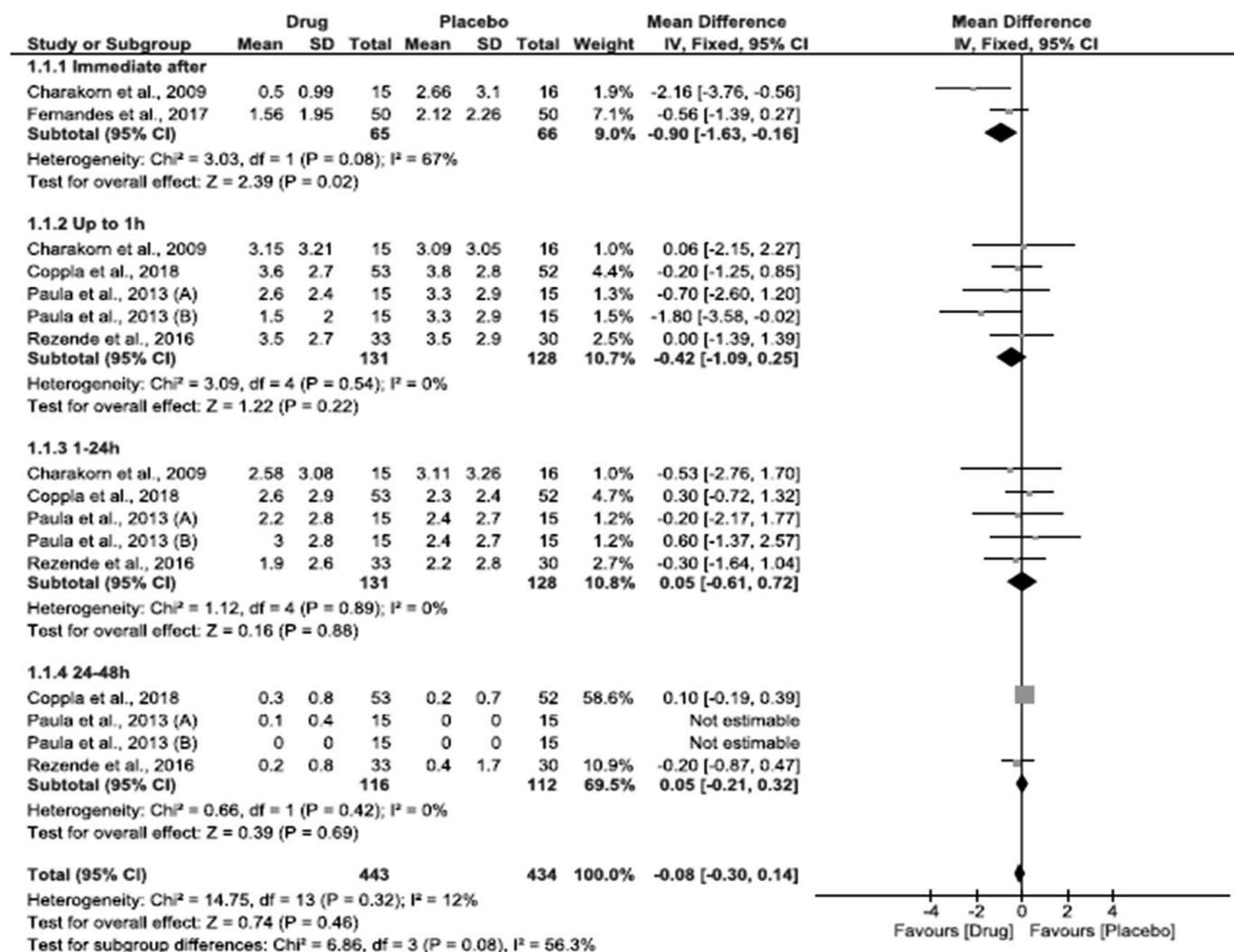


Figure 3. Forest plot for the event "sensitivity measurements."

diately after in-office bleaching. The oral route of administration is associated with good bioavailability, and it is the most common route of administration used in clinical trials.<sup>1-5,7,8,16</sup> NSAIDs, corticosteroids, and analgesics are all oral medications. The meta-analysis found that the one drug category that had a positive effect on sensitivity was NSAIDs when a single preprocedure dose is administered alone or with accompanying additional doses at the 24h time point after the procedure. This finding is not fully understood. However, Fernandes and others<sup>5</sup> state that it may be related to the effect of NSAIDs on bradykinin, which inhibits the synthesis of prostaglandin, a mediator of the inflammatory process. Preemptive administration of other doses or combinations of medications, in addition to a longer lead time, are possibly more effective. However,

further studies are needed to identify new drug regimens that could eliminate odontogenic pain.

Despite the effectiveness of bleaching techniques, tooth sensitivity is the main adverse effect during in-office procedures.<sup>9,13,20,26</sup> The gels used in office are predominantly hydrogen peroxide-based,<sup>2,4,5,27</sup> with  $H_2O_2$  concentrations ranging from 15% to 38%;<sup>2</sup> the most commonly used concentration is 35%. According to Bortolatto and others,<sup>9</sup> the gel concentration may have a significant influence on sensitivity. This product acts by penetrating the dental structure and promoting the lysis of macromolecules in the pigment through an oxy-reduction reaction.<sup>9</sup> However, the gel is also capable of inducing changes in the odontoblasts layer and triggering an inflammatory reaction in the dental pulp.<sup>22,28</sup> This can lead to the release of inflammatory mediators that are

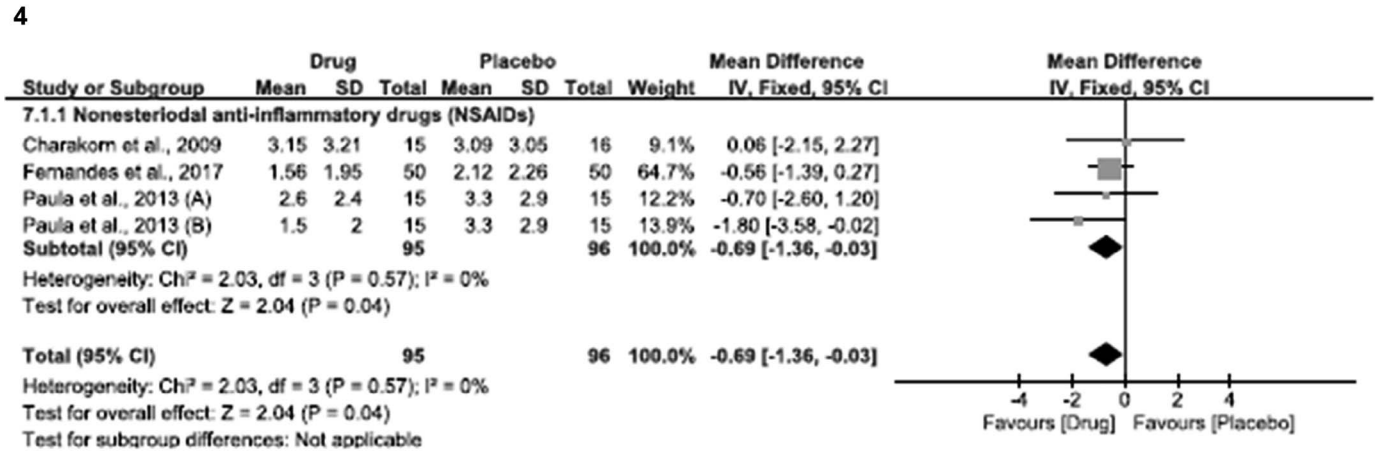
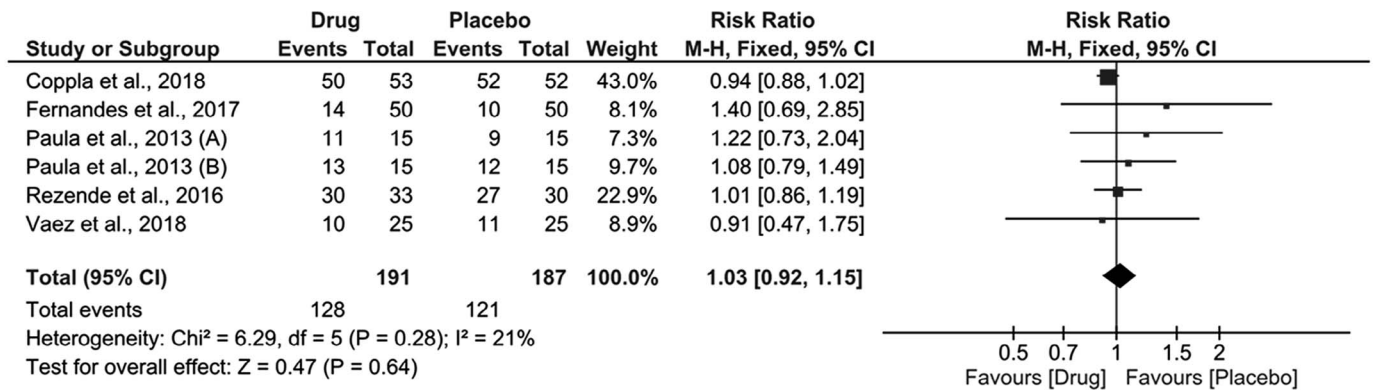


Figure 4. Forest plot for the event “sensitivity incidence.”

Figure 5. Forest plot for the event “subgroup medication group (NSAIDs) analyses to evaluate the effect on the intensity of sensitivity.”

responsible for painful sensations, local vasodilatation, and increased vascular permeability.<sup>16</sup>

Although the mechanisms involved in hydrogen peroxide-induced pain are not fully understood yet,<sup>2,24,29</sup> the sensitivity caused by tooth bleaching tends to increase during the first hours after the procedure, and this sensitivity is described as “zingers” by Haywood.<sup>30</sup>

The studies included in this systematic review and meta-analysis reported the presence of sensitivity during and up to 48 hours after the bleaching procedure and showed that the peak of pain is experienced within this time interval. As reported in the literature, tooth sensitivity occurs predominantly during the first 24 hours postprocedure,<sup>4,6,25</sup> and the symptoms may persist for several days.<sup>16,31</sup> However, Charakorn and others<sup>1</sup> have suggested that the peak of dental sensitivity is experienced between one and six hours after bleaching.

Regardless of the type of drug administered, no difference was observed in the incidence of dental

sensitivity between the drug and placebo groups, confirming the results reported in previous studies.<sup>6,10,32,33</sup> Regarding the intensity of the tooth sensitivity, the only observed statistically significant result was that the analgesic drugs can reduce the intensity of tooth sensitivity at the immediately after time point ( $p=0.02$ ). However, as only two studies were used in this comparison and one of them presented a high risk of bias, this led us to consider that not only the  $p$ -value must be taken into consideration, according to Glick and Greenberg<sup>34</sup> and Best and others.<sup>35</sup> It is believed that the quality of the studies and the CI must also be taken under consideration to interpret the  $p$ -value.

Importantly, the included studies present strong evidence; all of these studies were double-blinded or triple-blinded randomized clinical trials (RCTs). According to Higgins and others,<sup>18</sup> RCTs provide optimal scientific evidence, and when carried out following preestablished standards, they can be decisive in the construction of a systematic review. In addition, the absence of heterogeneity and the

high quality of RCTs confirmed by the bias risk analysis were significant; these results allow us to state that the studies shared the same effect size. Moher and others<sup>17</sup> stated that the absence of heterogeneity is highly relevant in the verification of scientific evidence.

One limiting factor of this study was that the age range of the study participants was narrow. This review predominantly included young adults, which hinders the universalization of these results to the general population. However, the response to pulp sensitivity is more evident in young individuals than in older individuals because they present wider dentinal tubules and thinner dentin layers.<sup>36,37</sup> Therefore, to determine the efficacy of preemptive analgesia in reducing bleaching-induced dental sensitivity, further randomized clinical trials evaluating different routes of administration and drugs classes should be carried out in varied age groups.

## CONCLUSIONS

This systematic review and meta-analysis demonstrated that the medications analyzed did not interfere with the incidence of sensitivity symptoms. Regarding the intensity, no difference was observed between the drug and placebo groups at the up to 1h, 1-24h, or 24-48h time points, and there was a statistically significant difference at the 0h time point in favor of the drug group. However, based on the variables that influenced this result, it should be considered with prudence because a small difference was observed. This study also confirmed the need for more trials of the effect of the drugs with analgesic effect on tooth sensitivity induced by in-office dental bleaching, especially with NSAIDs.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 17 April 2019)

## REFERENCES

1. Charakorn P, Cabanilla LL, Wagner WC, Foong W-C, Shaheen J, Pregitzer R, & Schneider D (2009) The effect of preoperative ibuprofen on tooth sensitivity caused by in-office bleaching *Operative Dentistry* **34**(2) 131-135, <https://doi.org/10.2341/12-107-C>.
2. Vaez SC, Faria-e-Silva AL, Loguercio AD, Fernandes MTG, & Nahsan FPS (2018) Preemptive use of etodolac on tooth sensitivity after in-office bleaching: A randomized clinical trial abstract *Journal of Applied Oral Science* **26** 1-9, <https://doi.org/10.1590/1678-7757-2016-0473>.
3. Rezende M, Chemin K, Vaez SC, Peixoto AC, Rabelo J de F, Braga SSL, Faria-e-silva AL, Da Silva GR, Soares CJ, Loguercio AD, & Reis A (2018) Effect of topical application of dipyrone on dental sensitivity reduction after in-office dental bleaching *Journal of the American Dental Association* **149**(5) 363-371, <https://doi.org/10.1016/j.adaj.2017.11.003>.
4. de Paula EA, Loguercio AD, Fernandes D, Kossatz S, & Reis A (2013) Perioperative use of an anti-inflammatory drug on tooth sensitivity caused by in-office bleaching: A randomized, triple-blind clinical trial *Clinical Oral Investigations* **17**(9) 2091-2097, <https://doi.org/10.1007/s00784-013-0918-2>.
5. Fernandes MTG, Vaez SC, Lima CM, Nahsan FPS, Loguercio AD, & Faria-e-Silva AL (2017) Preemptive use of naproxen on tooth sensitivity caused by in-office bleaching: A triple-blind, crossover, randomized clinical trial *Operative Dentistry* **42**(5) 486-496, <https://doi.org/10.2341/16-100-C>.
6. de Paula EA, Kossatz S, Fernandes D, Loguercio AD, & Reis A (2014) Administration of ascorbic acid to prevent bleaching-induced tooth sensitivity: A randomized triple-blind clinical trial *Operative Dentistry* **39**(2) 128-135, <https://doi.org/10.2341/12-483-C>.
7. Coppla FM, Rezende M, de Paula EA, Farago PV, Loguercio AD, Kossatz S, & Reis A (2018) Combination of acetaminophen/codeine analgesics does not avoid bleaching-induced tooth sensitivity: A randomized, triple-blind two-center clinical trial *Operative Dentistry* **43**(2) 53-63, <https://doi.org/10.2341/17-092-C>.
8. de Paula EA, Kossatz S, Fernandes D, Loguercio AD, & Reis A (2013) The effect of perioperative ibuprofen use on tooth sensitivity caused by in-office bleaching *Operative Dentistry* **38**(6) 601-608, <https://doi.org/10.2341/12-107-C>.
9. Bortolatto JF, Pretel H, Floros MC, Luiz ACC, Dantas AAR, Fernandez E, & Moncada G (2014) Low concentration H2O2/TiO<sub>2</sub>N in office bleaching: A randomized clinical trial *Journal of Dental Research* **93**(7) 66S-71S, <https://doi.org/10.1177/0022034514537466>.
10. Rezende M, Ferri L, Kossatz S, Loguercio AD, & Reis A (2016) Combined bleaching technique using low and high hydrogen peroxide in-office bleaching gel *Operative Dentistry* **41**(4) 1-9, <https://doi.org/10.2341/15-266-C>.
11. Chemin K, Rezende M, Loguercio AD, Reis A, & Kossatz S (2018) Effectiveness of and dental sensitivity to at-home bleaching with 4% and 10% hydrogen peroxide: A randomized, triple-blind clinical trial *Operative Dentistry* **43**(3) 232-240, <https://doi.org/10.2341/16-260-C>.
12. Thiesen CH, Rodrigues Filho R, Prates LHM, & Sartori N (2013) The influence of desensitizing dentifrices on pain induced by in-office bleaching *Brazilian Oral Research* **27**(6) 517-523, <https://doi.org/http://dx.doi.org/10.1590/S1806-83242013000600012>.
13. Públío J do C, D'Arce MBF, Ambrosano GMB, Aguiar FHB, Lovadino JR, Paulillo LAMS, & Lima DANL (2015) Efficacy of tooth bleaching with the prior application of a desensitizing agent *Journal of Investigative and Clinical*

- Dentistry* **6**(2) 133-140, <https://doi.org/10.1111/jicd.12074>.
14. Palé M, Mayoral JR, Llopis J, Vallès M, Basilio J, & Roig M (2014) Evaluation of the effectiveness of an in-office bleaching system and the effect of potassium nitrate as a desensitizing agent *Odontology* **102**(2) 203-210, <https://doi.org/10.1007/s10266-013-0132-3>.
  15. Bonafé E, Loguercio AD, Reis A, & Kossatz S (2014) Effectiveness of a desensitizing agent before in-office tooth bleaching in restored teeth *Clinical Oral Investigations* **18**(3) 839-845, <https://doi.org/10.1007/s00784-013-1055-7>.
  16. Rezende M, Bonafé E, Vochikovski L, Farago PV, Loguercio AD, Reis A, & Kossatz S (2016) Pre- and postoperative dexamethasone does not reduce bleaching-induced tooth sensitivity: A randomized, triple-masked clinical trial *Journal of the American Dental Association* **147**(1) 41-49, <https://doi.org/10.1016/j.adaj.2015.07.003>.
  17. Moher D, Liberati A, Tetzlaff J, Altman DG, & Prisma T (2010) Preferred reporting items for systematic reviews and meta-analyses: The PRISMA statement *International Journal of Surgery* **8**(5) 336-341, <https://doi.org/10.1016/j.ijso.2010.02.007>.
  18. Higgins JPT, Altman DG, Gøtzsche PC, Jüni P, Moher D, & Oxman AD (2011) The Cochrane Collaboration's tool for assessing risk of bias in randomised trials *BMJ* **343** 1-9, <https://doi.org/10.1136/bmj.d5928>.
  19. Egger MSG & Altman DG (2013) Principles of and procedures for systematic reviews. In: Egger MSG (ed) *Systematic Reviews in Health Care: Meta-Analysis in Context* BMJ Books, London, UK.
  20. Alexandrino LD, Alencar C de M, Silveira ADS da, Alves EB, & Silva CM (2017) Randomized clinical trial of the effect of NovaMin and CPP-ACPF in combination with dental bleaching *Journal of Applied Oral Science* **25**(3) 335-340, <https://doi.org/http://dx.doi.org/10.1590/1678-7757-2016-0408>.
  21. Devji T (2018) Use of naproxen before in-office tooth bleaching may result in a small decrease in tooth sensitivity during and immediately after the procedure *Journal of the American Dental Association* **149**(4) 77, <https://doi.org/10.1016/j.adaj.2017.12.017>.
  22. Poubel LA da C, Gouvea CVD, Calazans FS, Dip EC, Alves WV, Marins SS, Barcelos R, & Barceiro MO (2019) Pre-operative use of dexamethasone does not reduce incidence or intensity of bleaching-induced tooth sensitivity. A triple-blind, parallel-design, randomized clinical trial *Clinical Oral Investigations* **43**(1) 435-444, <https://doi.org/https://doi.org/10.1007/s00784-018-2452-8>.
  23. Landis JR & Koch GG (1977) The measurement of observer agreement for categorical data *Biometrics* **33** 159-174.
  24. Caviedes-Bucheli J, Ariza-García G, Restrepo-Méndez S, Ríos-Ororio N, Lombana N, & Muñoz HR (2008) The effect of tooth bleaching on substance P expression in human dental pulp *Journal of Endodontics* **34**(12) 1462-1465, <https://doi.org/10.1016/j.joen.2008.09.013>.
  25. Markowitz K (2010) Pretty painful: Why does tooth bleaching hurt? *Medical Hypotheses* **74**(5) 835-840, <https://doi.org/10.1016/j.mehy.2009.11.044>.
  26. Reis A, Kossatz S, Martins GC, & Loguercio AD (2013) Efficacy of and effect on tooth sensitivity of in-office bleaching gel concentrations: A randomized clinical trial *Operative Dentistry* **38**(4) 386-393, <https://doi.org/10.2341/12-140-C>.
  27. SoutoMaior J, de Moraes S, Lemos C, Vasconcelos B, Monte M, & Pellizzer E (2018) Effectiveness of light sources on in-office dental bleaching: A systematic review and meta-analyses *Operative Dentistry* **44**(3) E105-E117, <https://doi.org/10.2341/17-280-L>.
  28. Kose C, Calixto AL, Bauer JRO, Reis A, & Loguercio AD (2016) Comparison of the effects of in-office bleaching times on whitening and tooth sensitivity: A single blind, randomized clinical trial *Operative Dentistry* **41**(2) 138-145, <https://doi.org/10.2341/15-085-C>.
  29. Costa CA de S, Riehl H, Kina JF, Sacono NT, & Hebling J (2010) Human pulp responses to in-office tooth bleaching *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology* **109**(4) e59-e64, <https://doi.org/10.1016/j.tripleo.2009.12.002>.
  30. Haywood VB (2005) Treating sensitivity during tooth whitening *Compendium of Continuing Education in Dentistry* **26**(9 Supplement 3) 11-20.
  31. Carey CM (2014) Tooth whitening: What we now know *Journal of Evidence Based Dental Practice* **14** (Supplement) 70-76, <https://doi.org/10.1016/j.jebdp.2014.02.006>.
  32. Reis A, Tay LY, Herrera DR, Kossatz S, & Loguercio AD (2011) Clinical effects of prolonged application time of an in-office bleaching gel *Operative Dentistry* **36**(6) 590-596, <https://doi.org/10.2341/10-173-C>.
  33. Correa AC, Santana TR, Nahsan FP, Loguercio AD, & Faria-e-Silva AL (2015) The impact of a customized tray on in-office bleaching tooth sensitivity: a randomized clinical trial *Operative Dentistry* **40**(5) 1-8, <https://doi.org/10.2341/15-029-C>.
  34. Glick M & Greenberg BL (2010) The hermeneutic pitfalls of P *Journal of the American Dental Association* **141**(12) 1404, 1406-1407, <https://doi.org/10.14219/jada.archive.2010.0087>.
  35. Best AI M, Greenberg BL, & Glick M (2016) From tea tasting to t test: A P value ain't what you think it is *Journal of the American Dental Association* **147**(7) 527-529, <https://doi.org/10.1016/j.adaj.2016.05.004>.
  36. Maran BM, Vochikovski L, Hortkoff DR de A, Stanislawczuk R, Loguercio AD, & Reis A (2018) Tooth sensitivity with a desensitizing-containing at-home bleaching gel—A randomized triple-blind clinical trial *Journal of Dentistry* **72**(2018) 64-70, <https://doi.org/10.1016/j.jdent.2018.03.006>.
  37. Roderjan DA, Stanislawczuk R, Hebling J, Costa CA de S, Reis A, & Loguercio AD (2015) Response of human pulps to different in-office bleaching techniques: Preliminary findings *Brazilian Dental Journal* **26**(3) 242-248, <https://doi.org/http://dx.doi.org/10.1590/0103-6440201302282>.

# Efficacy and Adverse Effects of Whitening Dentifrices Compared With Other Products: A Systematic Review and Meta-analysis

A Devila • R Lasta • L Zanella • MA Dall Agnol • SA Rodrigues-Junior

## Clinical Relevance

When advising patients, dentists should consider the extrinsic stain removal effect of whitening dentifrices that may ultimately result in color change and yet produce adverse effects more often than regular dentifrices.

## SUMMARY

Whitening dentifrices (WDs) are widespread and accessible worldwide, claiming to whiten teeth. Therefore, this systematic review aimed to assess the extrinsic stain removal (ESR), the whitening potential, and the adverse effects of WDs. Randomized controlled trials comparing WDs with regular dentifrices (RDs) and other

home-based whitening products were searched at NCBI-PubMed, Cochrane-CENTRAL, EBS-CO-Host, and clinicaltrials.gov. The studies were screened and had data extracted by two independent researchers. Eligible studies presented outcomes of ESR, color change, and adverse effects, with no restriction of publication date. Data were meta-analyzed using RevMan 5.3, and the level of evidence was rated according to GRADE criteria. Eleven studies ( $n=1962$ ) assessed reduction of stain area and intensity through Lobene Stain index, with a mean difference (MD) of  $-0.33$  ( $[-0.41; -0.25]$ ;  $p=0.00001$ ) and  $-0.34$  ( $[-0.44; -0.25]$ ;  $p=0.00001$ ), respectively. When the modified Lobene Stain index was used (six studies;  $n=2576$ ), MD was  $-0.42$  ( $[-0.58; -0.25]$ ;  $p=0.00001$ ) and  $-0.30$  ( $[-0.39; -0.21]$ ;  $p=0.00001$ ), respectively. Mean color change through shade guide tabs (three studies;  $n=1322$ ) was  $-1.80$  ( $[-2.33; -1.26]$ ;  $p=0.00001$ ). All differences were in favor of the WDs, which also produced a risk of adverse effects ( $RR=1.74$ ;  $[1.20, 2.52]$ ;  $p=0.003$ ; four studies;  $n=1322$ ). The comparison of WDs with paint-on gel (two studies;  $n=58$ ) yielded similar efficacy and adverse effects ( $p>0.05$ ), whereas the

Angélica Devila, DDS, private dentist, Chapecó, Santa Catarina, Brazil

Rafaela Lasta, DDS, private dentist, Chapecó, Santa Catarina, Brazil

Luana Zanella, DDS, MsC, private dentist, Chapecó, Santa Catarina, Brazil

Mauro A. Dall Agnol, DDS, MsC, PhD, School of Dentistry, Universidade Comunitária da Região de Chapecó (Unochapecó), Chapecó, Santa Catarina, Brazil

\*Sinval A. Rodrigues-Junior, DDS, MS, PhD, Health Sciences Postgraduate Program, Universidade Comunitária da Região de Chapecó (Unochapecó), Chapecó, Santa Catarina, Brazil

\*Corresponding author: Servidão Anjo da Guarda, n° 295-D, Bairro Efapi, PO Box 1141, Chapecó, Santa Catarina 89809-000, Brazil; e-mail: rodriguesjunior.sa@unochapeco.edu.br

<https://doi.org/10.2341/18-298-L>

**comparison of WDs with white strips (two studies; n=87) yielded higher efficacy of the latter ( $p=0.00001$ ) and similar adverse effects ( $p=0.52$ ). The quality of evidence varied from low to moderate. WDs are more effective in reducing extrinsic stain and producing a whitening-like effect in teeth than RDs, although they also produce more adverse effects. Whitening efficacy of WDs is similar to paint-on gel and lower than white strips. Higher-quality evidence demands larger, well-conducted, independent studies.**

## INTRODUCTION

Dentifrices have been a powerful adjunct in dental hygiene, acting as a vehicle for fluoride, among other active ingredients.<sup>1</sup> Their cosmetic effect is inherent to their cleansing effect through biofilm and extrinsic stain removal and relies on the presence of abrasives.<sup>2</sup> Throughout the last two decades, dentifrices have also become a means of responding to the strong esthetic appeal as over-the-counter (OTC) whitening products for at-home tooth whitening, without professional supervision.<sup>3,4</sup>

Dentifrices that claim to whiten teeth have represented more than 50% of the OTC tooth whitening products<sup>5</sup> and their supposedly whitening properties depend on mechanical biofilm removal with a high quantity of abrasives.<sup>3,5,6</sup> Additionally, they may contain chemicals that break down organic molecules of the biofilm, removing chromogens or preventing their accumulation. Surfactants, enzymes, citrates, pyrophosphates, and hexametaphosphates are components of whitening dentifrices (WDs) and help degrade stained biofilms, aiding its mechanical removal.<sup>7</sup> However, the presence of peroxides in dentifrices is less common and is challenging, mainly due to formulation aspects.<sup>7</sup> Therefore, the limited amount of whitening constituents and the short contact time with the tooth surface have raised questions about the real whitening potential of WDs.<sup>8</sup>

Even so, WDs have been recently shown as more effective than regular dentifrices (RDs) in removing extrinsic staining at six weeks, regardless of whether adjunctive chemical antidiscoloration agents were present.<sup>9</sup> This suggests a potential esthetic effect resulting from removal of tooth surface discoloration by using WDs, although it may not translate into significant color improvement.<sup>5</sup> It also has been shown that, even in the absence of a real objective whitening effect, WDs may generate a subjective patient perception of an improved, whiter smile.<sup>10</sup>

However, time required for WDs to reach a whitening-like effect demands further clarification. For instance, although the ADA Acceptance Program Guidelines for Home-Use Tooth Stain Removal Products<sup>11</sup> recommends a minimum six-week assessment time for studies involving WDs, studies have shown significant effect at earlier periods<sup>12</sup> and improved effect with increasing treatment time.<sup>13</sup>

The basis for the widespread recommendation of any therapeutic agent involves the production of clinically proven effects,<sup>2</sup> regardless of the mechanism of action involved. In this sense, how WDs compare with other home-whitening products, including those that contain whitening substances and those that require professional supervision,<sup>5</sup> is paramount to recommend its clinical use. Additionally, safety issues such as allergic responses, abrasion of teeth and restorations, and other undesirable responses should be crystal clear, because these whitening products require no prescription or professional supervision.<sup>4</sup>

Therefore, this systematic review aimed to verify whether WDs are as effective and safe as RDs and as other home-based tooth whitening products and to verify the effect of application time on whitening efficacy. The hypotheses were that 1) WDs are more effective than RDs and produce more adverse effects; 2) the whitening effect improves as application time increases; and 3) WDs are as effective and safe as other home-based tooth whitening products.

## METHODS

This systematic review was built based on the recommendations of the *Cochrane Handbook for Systematic Reviews of Interventions* and the *Transparent Reporting of Systematic Reviews and Meta-analyses*<sup>14,15</sup> and focused on answering the questions that follow: are WDs as effective and as safe as 1) RDs and 2) other home-based whitening products; and what is the effect of application time on tooth whitening efficacy?

### Search Strategy and PICOS Criteria

PICOS criteria involved the following:

Problem: tooth whitening

Intervention: whitening dentifrices

Comparison: regular dentifrices or other home-based whitening methods

Outcomes: extrinsic stain removal (ESR), color change, and adverse effects

Studies were randomized controlled clinical trials written in English, with no restriction of application

time and outcome measure. Exclusion criteria involved the following: studies that compared WD with associated methods or protocols of tooth whitening and that compared WD with WD, studies with induced staining, studies without baseline data, studies that did not assess the desired outcomes, studies focusing on stain prevention, and studies with immediate color assessment.

Searches for articles were conducted in March 2017 and June 2018 at NCBI-PubMed, Cochrane-CENTRAL, EBSCO-Host, and clinicaltrials.gov. The following search strategy was customized to each database searched: Dentifrices[MeSH Terms] OR Toothpastes[MeSH Terms] OR whitening dentifrice OR whitening toothpaste OR bleaching toothpaste AND Tooth bleaching[MeSH Terms] OR tooth whitening OR bleaching, teeth OR tooth stain removal OR extrinsic stain removal OR pigment removal. The studies were screened by two independent reviewers (AD and RL) in the databases and in the references of the included studies. Disagreements were solved by two other reviewers (LZ and SARJ). Additionally, other potentially relevant studies were searched in the references of the included studies.

### Data Extraction

Data were collected independently by AD and RL into previously prepared extraction worksheets with information relative to the published article and characteristics of the study, participants, interventions, and outcomes in each experimental group. Authors of the studies were contacted by email to solve methodologic concerns or to obtain outcome data. Studies in which the data variability was expressed using SE had it converted to SD using the following equation:  $SD = SE\sqrt{n}$ , where  $n$  is the number of participants in each group.

### Outcome Measures

Efficacy of the interventions was expressed as the capacity of ESR or color change. The former was assessed using the Lobene stain index,<sup>16</sup> the Lobene stain index modified by Macpherson,<sup>17</sup> and the Shaw and Murray index.<sup>18</sup> The first two indexes generate ordinal measures of stain intensity and area and may also merge both into a composite index. The Shaw and Murray index sums the number of stained squares from a standard grid superposed to the tooth surface. Color variation was assessed through the yellowing change summary measure  $\Delta b^*$ , the lightness change summary measure  $\Delta L^*$ , and the nondirectional color change summary measure  $\Delta E^*$

resulting from the tridimensional color space CIE- $L^*a^*b^*$ <sup>19</sup> and by the score difference of shade tabs in Vita Classical and Vita 3D Master shade guides. All efficacy measures generated continuous variables.

Safety was assessed as the occurrence of adverse effects resulting from either using the WD or the RD/ other whitening intervention and was analyzed as a dichotomous variable.

### Methodologic Quality of the Studies

The Risk of Bias tool 2.0 from the Cochrane Collaboration<sup>20</sup> was applied to each individual study to determine the presence of bias resulting from 1) the randomization process, 2) deviations from intended interventions, 3) missing outcome data, 4) measurement of the outcome, and 5) selection of the reported result. Each study was classified as low risk, high risk, or some concerns for these domains, and an overall risk of bias was designated.

### Data Analysis

ESR was expressed as change in staining area, intensity, or in the composite mean score after the interventions. The mean difference was calculated as follows:

$$D = \mu_1 - \mu_2$$

where  $\mu_1$  and  $\mu_2$  are the mean area, intensity, or composite score before and after the interventions, respectively.<sup>21</sup> Because the composite score appeared to have been calculated differently in different studies, and considering that it derives from original data of stain area and intensity, it is described in the tables; however, it was not meta-analyzed.

The SD of the difference between before and after the interventions was determined through the following equation:

$$S_{\text{diff}} = \sqrt{S_1^2 + S_2^2 - 2 \times r \times S_1 \times S_2},$$

where  $S_1$  and  $S_2$  are the SDs of before and after the interventions, respectively, and  $r$  is the coefficient of correlation between before and after scores.<sup>14,21</sup> Because no study provided data of the difference to calculate  $r$ , sensitivity analysis was undertaken involving the comparisons at six weeks and simulating  $r=0$ ,  $r=0.5$ , and  $r=0.9$ . Because no difference in the pooled effect size measured or in the 95% confidence interval (95% CI) was detected, one adopted the  $r$  value that generated the lowest heterogeneity between data ( $r=0$ ). Heterogeneity

| Table 1: Results of the Comparison Between WD and RD                                                                                    |                           |                                   |                                   |         |                                 |
|-----------------------------------------------------------------------------------------------------------------------------------------|---------------------------|-----------------------------------|-----------------------------------|---------|---------------------------------|
| Outcome                                                                                                                                 | Comparison (n)            | Number of Studies and Comparisons | Effect Size Measure [95% CI]      | p       | Heterogeneity (I <sup>2</sup> ) |
| Reduction of stained area (LSI)                                                                                                         | WD (n=992) × RD (n=970)   | 11 studies; 17 comparisons        | −0.33 [−0.41; −0.25] <sup>a</sup> | 0.00001 | 72%                             |
| Reduction of stained area (MLSI)                                                                                                        | WD (n=1300) × RD (n=1276) | 6 studies; 8 comparisons          | −0.42 [−0.58; −0.25] <sup>a</sup> | 0.00001 | 91%                             |
| Reduction of stain intensity (LSI)                                                                                                      | WD (n=992) × RD (n=970)   | 11 studies; 17 comparisons        | −0.34 [−0.44; −0.25] <sup>a</sup> | 0.00001 | 78%                             |
| Reduction of stain intensity (MLSI)                                                                                                     | WD (n=1300) × RD (n=1276) | 6 studies; 8 comparisons          | −0.30 [−0.39; −0.21] <sup>a</sup> | 0.00001 | 78%                             |
| Vita Classic shade guide                                                                                                                | WD (n=674) × RD (n=648)   | 3 studies; 4 comparisons          | −1.80 [−2.33; −1.26] <sup>a</sup> | 0.00001 | 96%                             |
| Adverse effects                                                                                                                         | WD (n=664) × RD (n=658)   | 4 studies; 5 comparisons          | 1.74 [1.20;2.52] <sup>b</sup>     | 0.003   | 0%                              |
| Abbreviations: LSI, Lobene stain index; MLSI, Macpherson modified Lobene stain index; RD, regular dentifrice; WD, whitening dentifrice. |                           |                                   |                                   |         |                                 |
| <sup>a</sup> Mean difference [95% CI]: negative results favor the WD.                                                                   |                           |                                   |                                   |         |                                 |
| <sup>b</sup> Risk ratio [95% CI]: results above 1 favor the RD.                                                                         |                           |                                   |                                   |         |                                 |

was verified using the *I*<sup>2</sup> test with a significance level <0.10. Data were meta-analyzed with the inverse variance method and random effects model. To meta-analyze data generated using the Vita Classical shade guide, the SD data of Ghassemi and others<sup>22</sup> was used to calculate *r* to obtain the SD of the difference for Ghassemi and others.<sup>13</sup>

In the presence of high heterogeneity, sensitivity analysis was undertaken using the “leave-one-out” method to identify the role of each individual study on the final effect size measure. Subgroup meta-analyses were undertaken to analyze the hypothesis that whitening efficacy of the WD would rely on application time.

Adverse effects data were meta-analyzed using Mantel-Haenszel with 95% CI and a random effects model. Studies with no adverse effects reported in either group were not included in the meta-analysis. All meta-analyses were performed in RevMan 5.3.<sup>23</sup> The quality of evidence generated for each outcome was analyzed using *The Grading of Recommendations Assessment, Development and Evaluation* (GRADE), considering as parameters the study design, risk of bias, inconsistency, indirect evidence, imprecision, and publication bias.<sup>14,24</sup> Publication bias was assessed by analyzing the symmetry of distribution of the studies around the effect size measure in funnel plots when at least 10 studies were present.

RESULTS

One hundred forty-four potential studies were identified out of 8329 references. Another 15 studies were found by hand search in the references of the

included studies (Figure 1). Full-text reading of 76 studies led to exclusion of 36 studies. The remaining 40 studies were included in the qualitative analysis. The Online Appendix file contains complementary information on sensitivity analysis adopted to calculate the SD of studies that did not provide this data; the reasons for exclusion of studies; qualitative description of study design, participants, interventions and results; details on decisions related to risk of bias; publication bias; and grading of evidence.

Comparison WD × RD

Twelve studies qualitatively expressed ESR assessed through Lobene, Lobene modified by Macpherson (composite index), and the Shaw and Murray index.<sup>13,25–35</sup> All 12 studies revealed higher stain removal with the WD than with the RD, behavior that was confirmed by the meta-analysis of stain area and intensity using either Lobene (11 studies<sup>12,36–45</sup> with 1962 participants) or Lobene modified by Macpherson (6 studies<sup>13,22,46–49</sup> with 2576 participants) indexes (Table 1). For either outcome, the heterogeneity level was considered high (72%–91%). The risk of bias was rated serious, because most studies had raised concerns about the randomization process, with possible deviations from intended interventions that could include allocation concealment and about blinding of outcome assessors, leading to concerns in the overall bias. Indirectness and imprecision were not relevant issues; publication bias was not considered an issue, because the distribution of the studies in the funnel plots of the Lobene stain index was fairly symmetrical. The level of evidence was rated low.

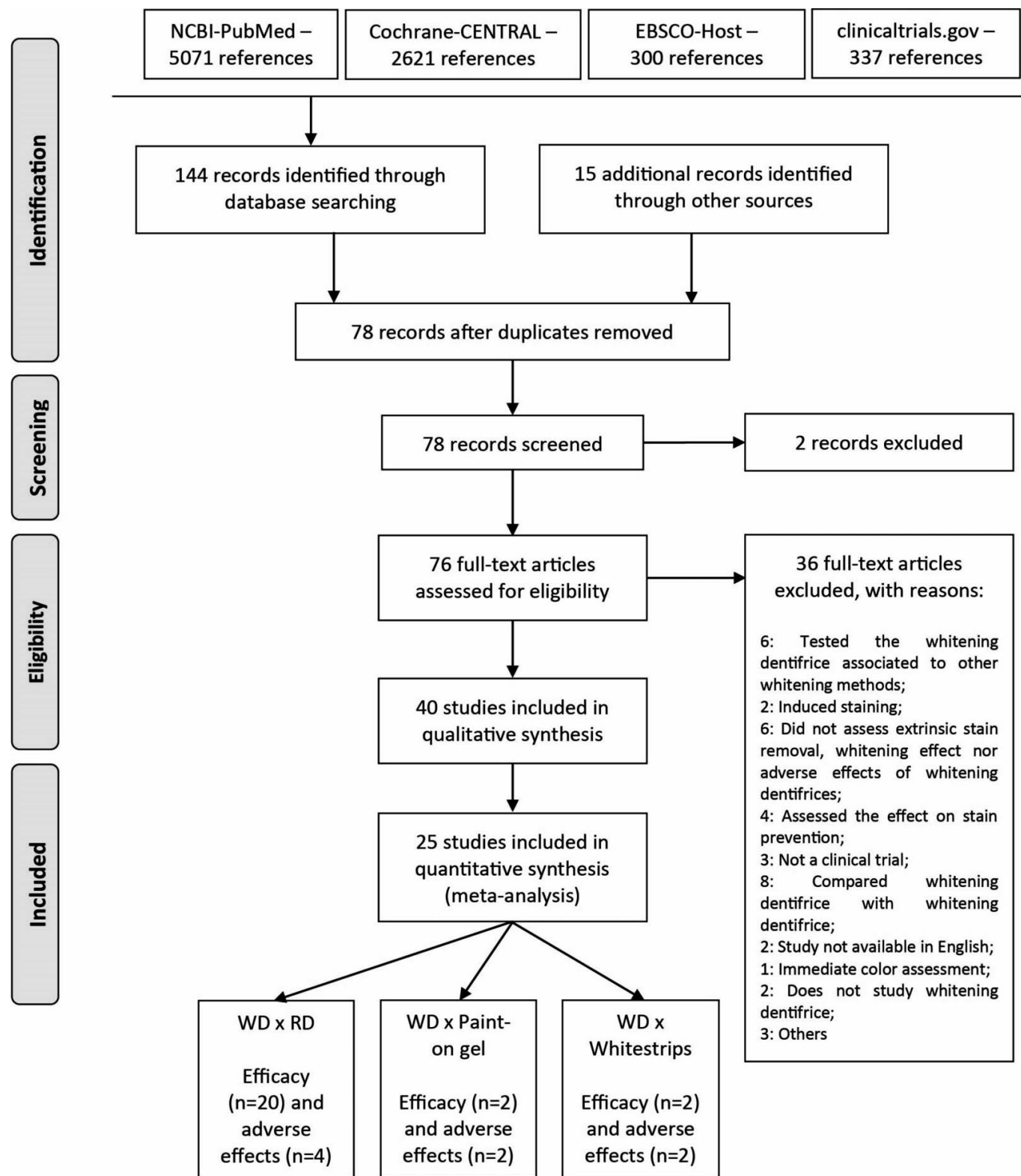


Figure 1. Flow diagram of the study.

| Table 2: Difference in Area of ESR (MD [95% CI]) With Either WD or RD in LSI or MLSI Considering Application Time                                                                          |                         |                                   |                              |         |                                 |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------|-----------------------------------|------------------------------|---------|---------------------------------|
| Time                                                                                                                                                                                       | LSI                     |                                   |                              |         |                                 |
|                                                                                                                                                                                            | Comparison (n)          | Number of Studies and Comparisons | Effect Size Measure [95% CI] | p       | Heterogeneity (I <sup>2</sup> ) |
| 5 days                                                                                                                                                                                     | —                       | —                                 | —                            | —       | —                               |
| 1 week                                                                                                                                                                                     | WD (n=74) × RD (n=70)   | 1 study; 1 comparison             | −0.12 [−0.29;0.05]           | 0.17    | —                               |
| 2 weeks                                                                                                                                                                                    | WD (n=74) × RD (n=70)   | 1 study; 1 comparison             | −0.24 [−0.41; −0.07]         | 0.006   | —                               |
| 3 weeks                                                                                                                                                                                    | —                       | —                                 | —                            | —       | —                               |
| 4 weeks                                                                                                                                                                                    | WD (n=46) × RD (n=46)   | 1 study; 1 comparison             | −0.28 [−0.58;0.02]           | 0.07    | 0%                              |
| 6 weeks                                                                                                                                                                                    | WD (n=666) × RD (n=652) | 9 studies; 15 comparisons         | −0.34 [−0.45; −0.24]         | 0.00001 | 78%                             |
| 8 weeks                                                                                                                                                                                    | WD (n=46) × RD (n=46)   | 1 study; 1 comparison             | −0.42 [−0.74; −0.10]         | 0.010   | —                               |
| 12 weeks                                                                                                                                                                                   | WD (n=43) × RD (n=43)   | 1 study; 1 comparison             | −0.35 [−0.51; −0.19]         | 0.00001 | —                               |
| 24 weeks                                                                                                                                                                                   | WD (n=43) × RD (n=43)   | 1 study; 1 comparison             | −0.38 [−0.53; −0.23]         | 0.00001 | —                               |
| Abbreviations: ESR, extrinsic stain removal; LSI, Lobene stain index; MD, mean difference; MLSI, Macpherson modified Lobene stain index; RD, regular dentifrice; WD, whitening dentifrice. |                         |                                   |                              |         |                                 |

One study assessed color change using the Vita 3D Master shade guide and found no significant influence of the type of dentifrice.<sup>10</sup> On the other hand, color change assessed using the Vita Classical shade guide, when meta-analyzed (three studies<sup>13,22,50</sup> involving 1322 participants), generated a mean 1.80 whiter shade tab, favoring the WD (Table 1). Heterogeneity was rated high, and risk of bias was rated serious due to concerns regarding the randomization process and potential deviations from intended interventions. Again, indirectness and imprecision were not serious; publication bias was not assessed due to the limited number of studies. The level of evidence was rated low.

Three studies assessed color change through Δs (which represented the before-after color change in each CIEL\*a\*b\* axis and the overall color change).<sup>8,25,51</sup> Koertge and others<sup>25</sup> observed a higher Δb\* (reduction of yellowness) with the whitening dentifrices. Horn and others,<sup>8</sup> comparing three WD with a RD, noticed that only one WD produced significantly higher ΔE\* than the RD. Pintado-Palomino and others<sup>51</sup> observed no significant difference in ΔE\* comparing two WD and a RD.

Eleven studies assessed the occurrence of adverse effects associated with the treatment.<sup>13,25–31,35,51,52</sup> In nine studies, no adverse effect was identified throughout the study.<sup>13,25–28,30,31,35,51</sup> The other two studies reported similar adverse events associated to either treatment group.<sup>29,52</sup> On the other hand, when meta-analyzed, the adverse effects of four other studies<sup>53–56</sup> involving five comparisons were lower with the RD (Table 1). No heterogeneity between studies was identified. Risk of bias was rated serious due to concerns regarding the random-

ization process and potential deviations from intended interventions that led to concerns in overall risk of bias. Indirectness and imprecision were not relevant issues; however, publication bias was not assessed due to the limited number of studies. The level of evidence was rated moderate. Gingivitis, gingival hyperplasia, gingival ulcer, lip ulcer, desquamation, tooth sensitivity, pain, and stomatitis were adverse effects reported as a consequence of dentifrice use.

Sensitivity analysis revealed no significant shift of effect size using the leave-one-out method with each individual study. Subgroup meta-analyses of ESR considering application time varied from five days to 24 months, and revealed that, for extrinsic stain area, with the exception of the one-week assessment, the other time periods favored the WD in at least one stain index (*p*<0.05; Table 2). The same behavior was observed regarding the extrinsic stain intensity (*p*<0.05; Table 3). Color change measured by means of shade tab position was assessed from five days to six weeks and also favored the WD (*p*<0.05; Table 4). Heterogeneity, when measurable, varied from 0% to 81% for ESR and from 0% to 98% for color change based on shade guide measures.

Comparison WD × Paint-on Gel

Two studies<sup>57,58</sup> involving 58 participants assessed color change through ΔE\* and adverse effects comparing the WD with the paint-on gel. The meta-analysis revealed no significant difference in color change or adverse effects measures (*p*>0.05). There was no serious concern about heterogeneity (Table 5). On the other hand, the risk of bias was

Table 2: Difference in Area of ESR (MD [95% CI]) With Either WD or RD in LSI or MLSI Considering Application Time (ext.)

| Time     | MLSI                    |                                   |                              |         |                         |
|----------|-------------------------|-----------------------------------|------------------------------|---------|-------------------------|
|          | Comparison (n)          | Number of Studies and Comparisons | Effect Size Measure [95% CI] | p       | Heterogeneity ( $I^2$ ) |
| 5 days   | WD (n=143) × RD (n=152) | 2 studies; 3 comparisons          | -0.18 [-0.32; -0.03]         | 0.02    | 0%                      |
| 1 week   | —                       | —                                 | —                            | —       | —                       |
| 2 weeks  | WD (n=115) × RD (n=126) | 1 study; 2 comparisons            | -0.33 [-0.48; -0.17]         | 0.0001  | 0%                      |
| 3 weeks  | WD (n=59) × RD (n=60)   | 1 study; 1 comparison             | -1.36 [-1.59; -1.13]         | 0.00001 | —                       |
| 4 weeks  | WD (n=662) × RD (n=639) | 4 studies; 6 comparisons          | -0.26 [-0.42; -0.10]         | 0.001   | 79%                     |
| 6 weeks  | WD (n=260) × RD (n=235) | 3 studies; 4 comparisons          | -0.69 [-0.88; -0.51]         | 0.00001 | 70%                     |
| 8 weeks  | WD (n=61) × RD (n=64)   | 1 study; 1 comparison             | -0.10 [-0.25; 0.05]          | 0.19    | —                       |
| 12 weeks | —                       | —                                 | —                            | —       | —                       |
| 24 weeks | —                       | —                                 | —                            | —       | —                       |

rated serious, because both studies raised concerns about the randomization process, with possible deviations from the intended interventions that could include allocation concealment and missing outcome data, leading to concerns in the overall bias. Indirectness and imprecision were not relevant issues; publication bias was not assessed due to the limited number of studies. The level of evidence was rated low.

### Comparison WD × Whitening Strips

Two studies<sup>57,59</sup> involving 87 participants assessed color change through  $\Delta b^*$  and  $\Delta L^*$  and also assessed adverse effects comparing the WD with whitening strips. Reduction of yellowness ( $\Delta b^*$ ) and increase of lightness ( $\Delta L^*$ ) were significantly higher using the whitening strips ( $p < 0.00001$ ; Table 5). The occurrence of adverse effects was not significantly different in either group; heterogeneity was a concern only for adverse effects ( $I^2 = 59\%$ ). On the other hand, the risk of bias was rated serious, because both studies raised concerns about the randomization process and possible deviations from intended interventions that could include allocation concealment; one of them also generated concerns about missing outcome data, leading to concerns in the overall bias. Indirectness and imprecision were not relevant issues; however, publication bias was not assessed due to the limited number of studies. The level of evidence was rated moderate for color change and low for adverse effects.

### Comparison WD × Custom Tray

One study<sup>58</sup> involving 29 participants analyzed the influence of 1% hydrogen peroxide containing activated WD × the tray system containing 5% carbam-

ide peroxide gel. The  $\Delta E^*$  was 1.49 higher with the tray system than with the WD ( $p < 0.0014$ ). Ten participants of the tray system group manifested oral irritation or tooth sensitivity (66.7%) vs three in the WD group (21.4%).

## DISCUSSION

Whitening dentifrices have been largely commercialized, claiming to contribute to the whitening effect of teeth. This study proved this claim to be true, either by ESR or color change. Therefore, the hypothesis that a whitening-like effect would be better provided by WDs compared with RDs was accepted. Also, compared with RDs, WDs induced more adverse effects. The study also showed that the whitening effect is not time dependent, with results showing up as early as five days of dentifrice use, leading to the rejection of the second hypothesis. Finally, the whitening efficacy and the potential of producing adverse effects of the WDs were similar to most of the other home-based whitening products, leading to the acceptance of the third hypothesis.

Efficacy was expressed in this study as the capacity of ESR and color change. Extrinsic dental staining differs from the intrinsic stain by being a discolored pellicle adhered to the tooth surface, as opposed to the inner discoloring caused by medicines, such as tetracycline, dental traumatism, or aging.<sup>60</sup> ESR requires cleansing and polishing actions produced by abrasive-containing dentifrices or professional prophylaxis products. Also, the presence of agents responsible for releasing the adherent stain or for bleaching chromogens has been suggested.<sup>7</sup>

ESR was analyzed using predefined standard stain indexes. The Lobene stain index<sup>16</sup> provides

| Table 3: Difference in Intensity Reduction of Extrinsic Stain (MD [95% CI]) With Either WD or RD in LSI or MLSI Considering Application Time                 |                         |                                   |                              |         |                                 |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------|-----------------------------------|------------------------------|---------|---------------------------------|
| Time                                                                                                                                                         | LSI                     |                                   |                              |         |                                 |
|                                                                                                                                                              | Comparison (n)          | Number of Studies and Comparisons | Effect Size Measure [95% CI] | p       | Heterogeneity (I <sup>2</sup> ) |
| 5 days                                                                                                                                                       | —                       | —                                 | —                            | —       | —                               |
| 1 week                                                                                                                                                       | WD (n=74) × RD (n=70)   | 1 study; 1 comparison             | −0.26 [−0.42; −0.10]         | 0.001   | —                               |
| 2 weeks                                                                                                                                                      | WD (n=74) × RD (n=70)   | 1 study; 1 comparison             | −0.63 [−0.79; −0.47]         | 0.00001 | —                               |
| 3 weeks                                                                                                                                                      | —                       | —                                 | —                            | —       | —                               |
| 4 weeks                                                                                                                                                      | WD (n=46) × RD (n=46)   | 1 study; 1 comparisons            | −0.16 [−0.45; 0.13]          | 0.29    | 0%                              |
| 6 weeks                                                                                                                                                      | WD (n=666) × RD (n=652) | 9 studies; 15 comparisons         | −0.34 [−0.46; −0.22]         | 0.00001 | 81%                             |
| 8 weeks                                                                                                                                                      | WD (n=46) × RD (n=46)   | 1 study; 1 comparison             | −0.26 [−0.54; 0.02]          | 0.07    | —                               |
| 12 weeks                                                                                                                                                     | WD (n=43) × RD (n=43)   | 1 study; 1 comparison             | −0.35 [−0.53; −0.17]         | 0.0001  | —                               |
| 24 weeks                                                                                                                                                     | WD (n=43) × RD (n=43)   | 1 study; 1 comparison             | −0.36 [−0.54; −0.18]         | 0.0001  | —                               |
| Abbreviations: LSI, Lobene stain index; MD, mean difference; MLSI, Macpherson modified Lobene stain index; RD, regular dentifrice; WD, whitening dentifrice. |                         |                                   |                              |         |                                 |

an ordinal scale that varies from 0 (no stain) to 3 (heavy stain) for stain intensity and 0 (no stain) to stain over one third, two thirds, or more than two thirds of the region (scores 1, 2, and 3, respectively) for stain area. This is assessed in the gingival region and the body region of the tooth. Its modification in 2000 by Macpherson and others<sup>17</sup> differs by including the proximal surfaces of the tooth in the index and by considering different distributions of stain in buccal/labial surfaces and lingual/palatal surfaces. In this sense, the buccal/labial surfaces are categorized as follows: 1 (stain limited to pits/grooves), 2 (stain outside pits/grooves, up to 10% of the area), and 3 (more than 10% of the area affected), and the lingual/palatal surfaces are categorized similarly to the original index. A total of 97% of the studies that assessed ESR used the Lobene stain index or its modification. The meta-analysis of stain area and intensity results favored the WD, regardless of the stain index used. Area and intensity results varied 0.09 and 0.04 between indexes, respectively, and this variation was not significant, as revealed by the overlapping CIs (Table 1). The only study that assessed ESR with the Shaw and Murray index<sup>33</sup> confirmed the higher ESR capacity with the WDs, proving this significant effect regardless of the measurement instrument used. The composite index assessed by Lobene and Macpherson-modified Lobene is a product of the stain area and intensity scores and was reported by a sufficient number of studies to provide a meta-analysis. However, the lack of information about a standardized procedure for calculation and the evidence of a different scaling for this index between studies led to the decision for qualitative presentation of these results only.

The Δs from the tridimensional color space CIEL\*a\*b\*, widely used in tooth color change assessments,<sup>51,61</sup> revealed significant color change toward a lighter tooth using some WDs in comparison to the baseline color.<sup>8,25,51</sup> In one study, color change was significant from baseline and similar with the WDs and the RD,<sup>51</sup> whereas in another study, no color change was observed with either dentifrice through a four-week assessment period.<sup>8</sup> It has been suggested that any overall color change resulting from WD use implies ESR and should not be confounded with a real bleaching effect.<sup>51</sup> This effect relies more on the amount and type of abrasive in the dentifrice<sup>51</sup> than on the presence of additional antidiscoloration chemical agents.<sup>9</sup>

When color change was assessed by the difference of shade tab score of the Vita Classical shade guide, meta-analysis revealed that WDs produced almost two shade tabs lighter than the RDs (Table 1). Although the difference of shade tab score was lower than the difference produced by peroxide-containing whitening products,<sup>61</sup> it was significant toward a higher color change effect by the WD. In summary, most studies that assessed color change as a relevant outcome presented some whitening-like effect as consequence of WD use.

Considerable heterogeneity, as measured by the I<sup>2</sup>, was identified between studies regarding the efficacy outcomes of the comparison between WD and RD (Table 1). The presence of significant heterogeneity indicated that variability higher than the expected in the effect size measure existed between studies. This variability should be incorporated to the model adopting the random effects

Table 3: Difference in Intensity Reduction of Extrinsic Stain (MD [95% CI]) With Either WD or RD in LSI or MLSI Considering Application Time (ext.)

| Time     | MLSI                    |                                   |                              |         |                         |
|----------|-------------------------|-----------------------------------|------------------------------|---------|-------------------------|
|          | Comparison (n)          | Number of Studies and Comparisons | Effect Size Measure [95% CI] | p       | Heterogeneity ( $I^2$ ) |
| 5 days   | WD (n=143) × RD (n=152) | 2 studies; 3 comparisons          | −0.15 [−0.27; −0.03]         | 0.01    | 0%                      |
| 1 week   | —                       | —                                 | —                            | —       | —                       |
| 2 weeks  | WD (n=115) × RD (n=126) | 1 study; 2 comparisons            | −0.29 [−0.41; −0.16]         | 0.00001 | 0%                      |
| 3 weeks  | WD (n=59) × RD (n=60)   | 1 study; 1 comparison             | −0.38 [−0.51; −0.25]         | 0.00001 | —                       |
| 4 weeks  | WD (n=662) × RD (n=639) | 4 studies; 6 comparisons          | −0.21 [−0.35; −0.08]         | 0.001   | 75%                     |
| 6 weeks  | WD (n=260) × RD (n=235) | 3 studies; 4 comparisons          | −0.49 [−0.58; −0.40]         | 0.00001 | 0%                      |
| 8 weeks  | WD (n=61) × RD (n=64)   | 1 study; 1 comparison             | −0.30 [−0.41; −0.28]         | 0.00001 | 41%                     |
| 12 weeks | —                       | —                                 | —                            | —       | —                       |
| 24 weeks | —                       | —                                 | —                            | —       | —                       |

model, which assumes that the true effect size varies from one study to another.<sup>14</sup> Also, in the presence of considerable heterogeneity, sensitivity analysis and subgroup meta-analysis are recommended to aid in the identification of possible sources of heterogeneity, either clinical or methodologic. Demographic and behavioral characteristics of the participants represent important sources of heterogeneity,<sup>61</sup> as well as the constitution of the WDs and RDs studied. As to the latter, it has been previously shown that the presence of antidiscoloring chemical adjuncts in the WD does not interfere in the effect size measure.<sup>9</sup> The other information, as presented by the primary studies, does not allow subgroup meta-analysis that could explain clinical heterogeneity.

A subgroup meta-analysis was set *a priori* based on the hypothesis that the total study time and, as a consequence, the cumulative treatment time, could improve color change produced by the WDs, as previously observed.<sup>13</sup> Treatment time did not influence significantly the ESR analyzed using either Lobene or Macpherson modified Lobene stain indexes, because in all times tested, starting at five days and throughout 24 weeks, at least one index revealed significant results in favor of the WD

(Tables 2 and 3). Similar behavior was observed when color change was analyzed using the shade tabs of Vita Classical shade guide (Table 4). From five days to four weeks, an increasing effect size was observed toward the WD, stabilizing from four to six weeks. This suggests that a whitening-like effect based on superficial abrasion and removal of surface pigmentation may be observed short term, earlier than the six-week period recommended by the ADA guidelines.<sup>11</sup>

Adverse effects were almost twice more likely to occur when using the WD (Table 1), with emphasis on tooth sensitivity and soft tissues repercussions. Compositional differences of WDs compared with RDs mainly involve the amount, shape, size, acuteness, and hardness of abrasives<sup>3</sup> and are probably related to the occurrence of adverse effects that may affect quality of life of users, such as pain or discomfort, and eventually lead to discontinuation of the treatment. Other adverse effects were not identified in the primary studies, because they require other detection approaches. Wear of the tooth and of restorations, for instance, are seldom identified by patient report or visual examination, but they may affect long-term biofilm retention due

Table 4: Difference in Color Change (MD [95% CI]) With Either WD or RD Considering Application Time

| Outcome        | Time    | Comparison (n)          | Number of Studies and Comparisons | Effect Size Measure [95% CI] | p       | Heterogeneity ( $I^2$ ) |
|----------------|---------|-------------------------|-----------------------------------|------------------------------|---------|-------------------------|
| Shade position | 5 days  | WD (n=115) × RD (n=126) | 1 study; 2 comparisons            | −0.47 [−0.77; −0.17]         | 0.002   | 0%                      |
| Shade position | 2 weeks | WD (n=136) × RD (n=149) | 2 studies; 3 comparisons          | −1.83 [−3.30; −0.35]         | 0.02    | 96%                     |
| Shade position | 4 weeks | WD (n=222) × RD (n=198) | 3 studies; 4 comparisons          | −2.28 [−3.30; −1.25]         | 0.0001  | 98%                     |
| Shade position | 6 weeks | WD (n=201) × RD (n=175) | 2 studies; 3 comparisons          | −2.03 [−2.73; −1.34]         | 0.00001 | 92%                     |

Abbreviations: MD, mean difference; RD, regular dentifrice; WD, whitening dentifrice.

| Table 5: Comparison Between the WD and Other Home-Based Whitening Products |                 |                                   |                                 |         |                                 |
|----------------------------------------------------------------------------|-----------------|-----------------------------------|---------------------------------|---------|---------------------------------|
| Comparison (n)                                                             | Outcome         | Number of Studies and Comparisons | Effect Size Measure [95% CI]    | p       | Heterogeneity (I <sup>2</sup> ) |
| WD (n=28) × paint-on gel (n=29)                                            | ΔE*             | 2 studies; 2 comparisons          | 0.15 [−1.87;2.18] <sup>a</sup>  | 0.88    | 0%                              |
| WD (n=28) × paint-on gel (n=30)                                            | Adverse effects | 2 studies; 2 comparisons          | 3.74 [0.31;44.98] <sup>b</sup>  | 0.30    | 40%                             |
| WD (n=45) × white strips (n=42)                                            | Δb*             | 2 studies; 3 comparisons          | 2.37 [2.07;2.66] <sup>a,c</sup> | 0.00001 | 0%                              |
| WD (n=45) × white strips (n=42)                                            | ΔL*             | 2 studies; 3 comparisons          | 2.14 [1.78;2.49] <sup>a,c</sup> | 0.00001 | 0%                              |
| WD (n=45) × white strips (n=42)                                            | Adverse effects | 2 studies; 3 comparisons          | 0.60 [0.13;2.80] <sup>b</sup>   | 0.52    | 59%                             |
| Abbreviation: WD, whitening dentifrice.                                    |                 |                                   |                                 |         |                                 |
| <sup>a</sup> Mean difference [95% CI].                                     |                 |                                   |                                 |         |                                 |
| <sup>b</sup> Risk ratio [95% CI].                                          |                 |                                   |                                 |         |                                 |
| <sup>c</sup> Favored the white strips.                                     |                 |                                   |                                 |         |                                 |

to increased roughness, and even affect the cosmetic aspect of the smile.<sup>3,62</sup>

The comparisons of the WD with other home-based whitening products revealed higher efficacy of the hydrogen peroxide-containing white strips and a carbamide peroxide-based tray system. The 18% carbamide peroxide paint-on gel and the WD produced similar color change (Table 5). All tooth whitening systems, including the WD compared here, had their whitening mechanism based on liberation of oxygen free radicals through the tooth structure from peroxide-containing substances.<sup>63</sup> These systems have been shown to rely on the concentration of oxygen-releasing substances and on time of contact with the tooth structure for their whitening capacity.<sup>61</sup> In this sense, the higher whitening performance of the white strips and the tray system is explained by the prolonged contact time of the whitening peroxides with the tooth structure, given each whitening regimen (twice a day for 30 minutes and once a day for six to eight hours, respectively).<sup>57,58</sup> Interestingly, the similar performance of the paint-on gel in comparison with the WD may be due to the short application time regimen (twice a day for 15-30 minutes), regardless of the substantial difference of hydrogen peroxide content between them. No significant difference in occurrence of adverse effects was observed between the WD and the other whitening methods, excepting the tray system, which presented a 40% higher occurrence of oral irritation or tooth sensitivity,<sup>58</sup> also possibly associated with the concentration of peroxide.

Overall risk of bias generated concerns in 85% of the primary studies. Risk of bias is related to whether key aspects of the randomized controlled trial designs were taken into consideration and how, and these may influence methodologic heterogeneity.<sup>61</sup> Frequent concerns in primary studies were

related to random sequence generation and deviations from the intended interventions. The latter is related to allocation concealment to participants, care providers, and trial personnel and the likelihood of unbalanced interventions between groups due to lack of allocation concealment.<sup>20</sup> Risk of bias was considered serious in both comparisons because of concerns generated in key methodologic issues of the randomized controlled trials.

Imprecision relates to the sample size required to correctly reject the null hypothesis<sup>21</sup> and was rated serious for the comparison between the WD and the paint-on gel. Rating took into consideration the small sample size based on two studies and the 95% CI that failed to exclude the threshold of no effect for both efficacy and adverse effects measurement. For the other comparisons, imprecision was not an issue, considering that the pooled number of participants in the meta-analyses reached the minimum required by the Acceptance Program Guidelines for Home-Use Tooth Stain Removal Products,<sup>11</sup> and the 95% CI of the estimate of treatment effect excluded the no effect threshold. However, only 7.5% of the included studies presented a sample size calculation or a rationale to justify the sample size.

Publication bias is present when the dataset of the literature misrepresents the true body of evidence because of a biased sample of relevant studies.<sup>21,64</sup> It may be caused by various reasons, including the difficulty of publication of negative results, and may lead to conclusions of significant effects due to nonidentification of studies with nonsignificant results.<sup>64</sup> Funnel plots display the relationship between the study size and effect size and represent absence of publication bias through symmetric distribution of studies around the mean effect size.<sup>21</sup> Funnel plots were generated for ESR analyzed by the Lobene stain index, because it included more

than 10 studies and revealed a symmetric distribution of study sizes around the mean effect size. For the other outcomes, publication bias was not assessable due to the limited number of studies. Caution during interpretation of results is recommended, because approximately 80% of the studies were conducted or sponsored by the manufacturers of the WDs.

The quality of evidence generated was rated based on GRADE criteria<sup>65</sup> for the recommendation of WD compared with RD, paint-on gel, and white strips. The level of evidence for efficacy and adverse effects produced by the WD in comparison with the RD was low and moderate, respectively. Risk of bias and inconsistency was responsible for downgrading evidence of efficacy. The comparison with the paint-on gel generated a low level of evidence for recommendation of the WD, based on a serious risk of bias and imprecision. Compared with the white strips, a moderate level of evidence of efficacy and low level of adverse effects favored the white strips. The risk of bias and inconsistency downgraded the level of evidence for adverse effects. Noteworthy, previous studies called attention to the low-quality evidence base of home-based tooth whitening procedures, highlighting the preponderance of industry-conducted studies.<sup>61,66</sup>

This review presented evidence of a whitening-like effect produced by WDs, taking into consideration all possible outcome measures and follow-up times reported. It confirmed the higher ESR capacity of WDs reported elsewhere<sup>9</sup> and revealed greater adverse effects in comparison to RDs. Finally, it highlights the demand for high-quality, high-sample, independent studies to further improve the quality of the existing evidence.

## CONCLUSION

Whitening dentifrices are more effective in reducing extrinsic stain and producing a whitening-like effect in teeth than regular dentifrices. Also, they do produce more adverse effects. The efficacy of the whitening dentifrice does not seem to rely on application time. Both, whitening efficacy and production of adverse effects of whitening dentifrices are similar to most of the other whitening products.

Caution is recommended when interpreting these conclusions, because they are impacted by the low quality of the primary studies, most of which are sponsored by industry. The limited number of studies and limited sample size hinder statistically sound comparisons between whitening dentifrices

and other home-based whitening products. High-quality evidence requires larger, well-conducted, independent studies.

## Acknowledgements

The authors thank Dr. Stephen Siegel, editor in chief of the *Journal of Clinical Dentistry*, and Daniel Perkins on behalf of AEGIS Communications for retrieval of full-text papers published in the *Journal of Clinical Dentistry* and *Compendium of Continuing Education in Dentistry*. The project was funded by FAPESC Grant 06/2017.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 20 July 2019)

## References

- Clarkson J, Walsh T, & Worthington H (2011) Cochrane reviews on the benefits/risks of fluoride toothpastes *Journal of Dental Research* **90**(5) 573-579.
- Cury JA & Tenuta LMA (2014) Evidence-based recommendation on toothpaste use *Brazilian Oral Research* **28**(Special Issue 1) 1-7.
- Barbieri GM, Mota EG, Rodrigues-Junior SA, & Burnett Jr LH (2011) Effect of whitening dentifrices on the surface roughness of commercial composites *Journal of Esthetic and Restorative Dentistry* **23**(5) 338-345.
- ADA Council on Scientific Affairs (2009) Tooth whitening/bleaching: treatment considerations for dentists and their patients *American Dental Association (Chicago)* 2009(September 2009) 1-12.
- Demarco FF, Meireles SS, & Masotti AS (2009) Over-the-counter whitening agents: critical assessment of the OTC products for home-use bleaching *Brazilian Oral Research* **23**(Special Issue 1) 64-70.
- Moran J, Claydon NC, Addy M, & Newcombe R (2005) Clinical studies to determine the effectiveness of a whitening toothpaste at reducing stain (using a forced stain model). *International Journal of Dental Hygiene* **3**(1) 25-30.
- Joiner A (2010) Whitening toothpastes: a review of the literature *Journal of Dentistry*. **38**(Supplement 2) e17-e24.
- Horn BA, Bittencourt BF, Maria Mongrue Gomes O, & Farhat PA (2014) Clinical evaluation of the whitening effect of over-the-counter dentifrices on vital teeth *Brazilian Dental Journal* **25**(3) 203-206.
- Soeteman G, Valkenburg C, Van der Weijden G, Van Loveren C, Bakker E, & Slot DE (2018) Whitening dentifrice and tooth surface discoloration: a systematic review and meta-analysis *International Journal of Dental Hygiene* **16**(1) 24-35.
- Raoufi S & Birkhed D (2010) Effect of whitening toothpastes on tooth staining using two different colour-

- measuring devices: a 12-week clinical trial. *International Dental Journal* **60**(6) 419-423.
11. ADA Council on Scientific Affairs (2012) Acceptance program requirements: Home-use tooth stain removal products. ADA Guidelines *American Dental Association (Chicago)* 2012(April 2012) 1-10.
  12. Sreenivasan P, DeVizio W, Prasad KVV, Rajesh G, Kalyanpur R, & Rao CB (2009) Clinical stain removal efficacy of two dentifrices after one and two weeks of use *Journal of Clinical Dentistry* **20**(7) 1-4.
  13. Ghassemi A, Vorwerk L, Cirigliano A, Hooper W, Co D, & Nathoo S (2015) Clinical effectiveness evaluation of a new whitening dentifrice *Journal of Clinical Dentistry* **26**(3) 66-71.
  14. Higgins J & Green S (2011) *Cochrane Handbook for Systematic Reviews of Interventions*. Version 5.1.0 [updated March 2011]. The Cochrane Collaboration, 2011. Available from [www.handbook.cochrane.org](http://www.handbook.cochrane.org).
  15. Moher D, Liberati A, Tetzlaff J, & Altman DG (2009) Preferred reporting items for systematic reviews and meta-analyses: the PRISMA statement *Journal of Clinical Epidemiology* **62**(10) 1006-1012.
  16. Lobene R (1968) Effect of dentifrices on tooth stains with controlled brushing *Journal of the American Dental Association* **77**(4) 849-855.
  17. Macpherson LMD, Stephen KW, Joiner A, Schafer F, & Huntington E (2000) Comparison of a conventional and modified tooth stain index *Journal of Clinical Periodontology* **27**(11) 854-859.
  18. Shaw L & Murray JJ (1977) A new index for measuring extrinsic stain in clinical trials. *Community Dentistry and Oral Epidemiology* **5**(3) 116-120.
  19. Commission Internationale d'Eclairage (2004) CIE technical report: colorimetry. CIE Pub No. 15.3, Vienna, Austria: CIE Central Bureau, 2004.
  20. Higgins JPT, Sterne JAC, Savovic J, Page MJ, Hróbjartsson A, Boutron I, Reeves B & Eldridge S (2016) A revised tool for assessing risk of bias in randomized trials In: Chandler J, McKenzie J, Boutron I, Welch V (editors). *Cochrane Methods*. Cochrane Database of Systematic Reviews Suppl 1(10) 1-21.
  21. Borenstein M, Rothstein HR, Hedges LV, & Higgins JP (2009) *Introduction to Meta-Analysis*. John Wiley and Sons, Ltd: West Sussex.
  22. Ghassemi A, Hooper W, Vorwerk L, Domke T, DeSciscio P, & Nathoo S (2012) Effectiveness of a new dentifrice with baking soda and peroxide in removing extrinsic stain and whitening teeth *Journal of Clinical Dentistry* **23**(3) 86-91.
  23. Review Manager (RevMan) [Computer program]. Version 5.3. Copenhagen: The Nordic Cochrane Centre, The Cochrane Collaboration, 2014. 43.
  24. Guyatt G, Oxman AD, Akl EA, Kunz R, Vist G, Brozek J, Norris S, Falck-Ytter Y, Glasziou P, Debeer H, Jaeschke R, Rind D, Meerpohl J, Dahm P, & Schünemann HJ (2011) GRADE guidelines: 1. Introduction: GRADE evidence profiles and summary of findings tables *Journal of Clinical Epidemiology* **64**(4) 383-394.
  25. Koertge TE, Brooks CN, Sarbin AG, Powers D, & Gunsolley JC (1998) A longitudinal comparison of tooth whitening resulting from dentifrice use *Journal of Clinical Dentistry* **9**(3) 67-71.
  26. Nathoo S, Mateo LR, Delgado E, Zhang YP, & DeVizio W (2011) Extrinsic stain removal efficacy of a new dentifrice containing 0.3% triclosan, 2.0% PVM/MA copolymer, 0.243% NaF and specially-designed silica for sensitivity relief and whitening benefits as compared to a dentifrice containing 0.3% triclosan, 2% PVM/MA *American Journal of Dentistry* **24**(Special Issue A) 28A-31A.
  27. Buelo A, Ghassemi A, Vorwerk L, Hooper W, & Nathoo S (2016) Clinical study to determine the stain removal effectiveness of a new dentifrice formulation *Journal of Clinical Dentistry* **27**(3) 80-83.
  28. Shao Y, Xue J, Peng C, Li C, Zhang S, & Zhou H (2017) Clinical evaluation of a toothpaste containing lysozyme for removal of extrinsic stains on the tooth surface: an 8-week, double-blind, randomized study *American Journal of Dentistry* **30**(3) 147-150.
  29. Young S, Mason S, Milleman JL, & Milleman KR (2017) A randomized clinical study to evaluate the effect of an ultra-low abrasivity dentifrice on extrinsic dental stain *American Journal of Dentistry* **30**(5) 255-261.
  30. Ayad F, Arcuri H, Brevilieri E, Laffi S, Lemos AM, Yoshioka M, Baines E, Sheth J, & DeVizio W (1999) Efficacy of two dentifrices on removal of natural extrinsic stain *American Journal of Dentistry* **12**(4) 164-166.
  31. Gerlach RW, Ramsey LL, & White DJ (2002) Extrinsic stain removal with a sodium hexametaphosphate-containing dentifrice: comparisons to marketed controls *Journal of Clinical Dentistry* **13**(1) 10-14.
  32. Collins L, Naeeni M, Schäfer F, Brignoli C, Schiavi A, Roberts J, & Colgan P (2005) The effect of a calcium carbonate/perlite toothpaste on the removal of extrinsic tooth stain in two weeks *International Dental Journal* **55**(3) 179-182.
  33. Walsh TF, Rawlinson A, Wildgoose D, Marlow I, Haywood J, & Ward JM (2005) Clinical evaluation of the stain removing ability of a whitening dentifrice and stain controlling system *Journal of Dentistry* **33**(5) 413-418.
  34. Li YC, He T, Sun L, Zhang Y, Li X, Wang Y, Zhao S, & Tang RY (2007) Extrinsic stain removal efficacy of a dual-phase dentifrice *American Journal of Dentistry* **20**(4) 227-230.
  35. Xie P, Lu J, Wan H, & Hao Y (2010) Effect of toothpaste containing d-limonene on natural extrinsic smoking stain: a 4-week clinical trial *American Journal of Dentistry* **23**(4) 196-200.
  36. Sharma N, Galustians J, Qaqaish J, Rustogi KN, Petrone ME, Chaknis P, DeVizio W, Volpe AR, & Proskin HM (1999) A six-week clinical efficacy study of three commercially available dentifrices for the removal of extrinsic tooth stain *Journal of Clinical Dentistry* **10**(3) 111-114.
  37. Yin W, Li X, He S, Ma H, Hu D, Zhang YP, Delgado E, DeVizio W, & Mateo LR (2010) Extrinsic stain removal efficacy of a new desensitizing dentifrice containing 8.0% arginine, calcium carbonate and 1450 ppm fluoride

- American Journal of Dentistry* **23(Special Issue A)** 36A-40A.
38. Yankell SL, Emling RC, Petrone ME, Rustogi K, Volpe AR, & Proskin HM (1999) A six-week clinical efficacy study of four commercially available dentifrices for the removal of extrinsic tooth stain *Journal of Clinical Dentistry* **10(3)** 115-118.
  39. Ayad F, Demarchi B, Khalaf A, Petrone ME, Chaknis P, DeVizio W, Volpe AR, & Proskin HM (1999) A six-week clinical efficacy study of a new dentifrice for the removal of extrinsic tooth stain *Journal of Clinical Dentistry* **10(3)** 103-106.
  40. Mankodi S, Sowinski J, Davies R, Ellwood R, Bradshaw B, Petrone ME, DeVizio W, Chaknis P, Volpe AR, & Proskin HM (1999) A six-week clinical efficacy study of a tooth whitening tartar control dentifrice for the removal of extrinsic tooth stain *Journal of Clinical Dentistry* **10(3)** 99-102.
  41. Ayad F, Demarchi B, Khalaf A, Davies R, Ellwood R, Bradshaw B, Petrone ME, Chaknis P, DeVizio W, Volpe AR, & Proskin HM (2000) A six-week clinical tooth whitening study of a new calculus-inhibiting dentifrice formulation *Journal of Clinical Dentistry* **11(3)** 84-87.
  42. Ayad F, Khalaf A, Chaknis P, Petrone ME, DeVizio W, Volpe AR, & Proskin HM (2002) Clinical efficacy of a new tooth whitening dentifrice *Journal of Clinical Dentistry* **13(2)** 82-85.
  43. Nathoo S, Petrone ME, DeVizio W, Chaknis P, & Volpe A (2002) A six-week clinical study to compare the stain removal efficacy of three dentifrices *Journal of Clinical Dentistry* **13(2)** 91-94.
  44. Singh S, Mankodi S, Chaknis P, Petrone ME, DeVizio W, Volpe AR, & Proskin HM (2002) The clinical efficacy of a new tooth whitening dentifrice formulation: a six-month study in adults *Journal of Clinical Dentistry* **13(2)** 86-90.
  45. Sielski C, Berta RL, Petrone ME, Chaknis P, DeVizio W, Volpe AR, & Proskin HM (2002) A study to assess the tooth whitening efficacy of a new dentifrice formulation variant containing a special grade of silica: a six-week study on adults *Journal of Clinical Dentistry* **13(2)** 77-81.
  46. Matheson J, Cox T, Baylor N, Joiner A, Patil R, Karad V, Ketkar V, & Bijlani N (2004) Effect of toothpaste with natural calcium carbonate/perlite on extrinsic tooth stain *International Dental Journal* **54(5)** 321-325.
  47. Nathoo S, Singh S, Petrone DM, Wachs GN, Chaknis P, DeVizio W, & Proskin HM (2008) Clinical studies to assess the extrinsic stain prevention and stain removal efficacy of a variant of a commercially available dentifrice containing a new dual silica system *Journal of Clinical Dentistry* **19(3)** 95-101.
  48. Prasad KVV, Sreenivasan PK, Rajesh G, Ramya K, Bhaskar Rao C, & de Vizio W (2011) The efficacy of dentifrices on extrinsic tooth stains among community dwelling adults in India: a randomised controlled trial *Community Dental Health* **28** 201-205.
  49. Ghassemi A, Vonverk L, Hooper W, Cirigliano A, De Sciscio P, & Nathoo S (2015) Extrinsic stain removal effectiveness of a new whitening dentifrice *Journal of Clinical Dentistry* **26(3)** 72-75.
  50. Kakar A, Rustogi K, Petrone ME, Yun PZ, DeVizio W, & Proskin HM (2004) A clinical investigation of the tooth whitening efficacy of a new hydrogen peroxide-containing dentifrice *Journal of Clinical Dentistry* **15(2)** 41-45.
  51. Pintado-Palomino K, Vasconcelos CVM, da Silva RJ, de Fressatti AL, da Motta BJG, de Pires-de-Souza FCP, & Tirapelli C (2016) Effect of whitening dentifrices: a double-blind randomized controlled trial *Brazilian Oral Research* **30(1)** e82.
  52. Volpe AR, Mooney R, Zumbrennen C, Stahl D, & Goldman HM (1975) A long term clinical study evaluating the effect of two dentifrices on oral tissues *Journal of Periodontology* **46(2)** 113-118.
  53. Isaacs RL, Bartizek RD, Owens TS, Walters PA, & Gerlach RW (2001) Maintenance of tooth color after prophylaxis: comparison of three dentifrices *Journal of Clinical Dentistry* **12(2)** 51-55.
  54. Gerlach RW & White DJ (2001) Removal of extrinsic stain using a tartar control whitening dentifrice: a randomized clinical trial *Journal of Clinical Dentistry* **12(2)** 42-46.
  55. Gerlach RW, Liu H, Prater ME, Ramsey LL, & White DJ (2002) Removal of extrinsic stain using a 7.0% sodium hexametaphosphate dentifrice: a randomized clinical trial *Journal of Clinical Dentistry* **13(1)** 6-9.
  56. Young S, Parkinson C, Hall C, Wang N, Milleman JL, & Milleman KR (2015) A randomized clinical study investigating the stain-removal potential of two experimental dentifrices *Journal of Clinical Dentistry* **26(4)** 96-103.
  57. Gerlach RW, & Barker ML (2003) Clinical response of three direct-to-consumer whitening products: strips, paint-on gel, and dentifrice *Compendium of Continuing Education in Dentistry* **24(6)** 458-466.
  58. Gerlach RW, Barker ML, & Tucker HL (2004) Clinical response of three whitening products having different peroxide delivery: comparison of tray, paint-on gel, and dentifrice *Journal of Clinical Dentistry* **15(4)** 112-117.
  59. Yudhira R, Peumans M, Barker ML, & Gerlach RW (2007) Clinical trial of tooth whitening with 6% hydrogen peroxide whitening strips and two whitening dentifrices *American Journal of Dentistry* **20(Special Issue A)** 32A-36A.
  60. Volpe AR, Petrone ME, DeVizio W, Emling RC, & Yankell SL (1999) A new perspective on the methodology for clinical efficacy evaluations of tooth whitening dentifrices *Journal of Clinical Dentistry* **10(Special No. 3)** 95-98.
  61. Serraglio CR, Zanella L, Dalla-Vecchia KB, & Rodrigues-Junior SA (2016) Efficacy and safety of over-the-counter whitening strips as compared to home-whitening with 10 % carbamide peroxide gel: systematic review of RCTs and metaanalysis *Clinical Oral Investigations* **20(1)** 1-14.
  62. Da Costa J, Adams-Belusko A, Riley K, & Ferracane JL (2010) The effect of various dentifrices on surface roughness and gloss of resin composites *Journal of Dentistry* **38(Supplement 2)** e123-e128.
  63. Kwon SR & Wertz PW (2015) Review of the mechanism of tooth whitening *Journal of Esthetic and Restorative Dentistry* **27(5)** 240-257.

64. Coleman BG, Johnson TM, Erley KJ, Topolski R, Rethman M, & Lancaster DD (2016) Preparing dental students and residents to overcome internal and external barriers to evidence-based practice *Journal of Dental Education* **80(10)** 1161-1169.
65. Guyatt G, Oxman AD, Akl EA, Kunz R, Vist G, Brozek J, Norris S, Falck-ytter Y, Glasziou P, & Jaeschke R (2011) GRADE guidelines: 1. Introduction GRADE evidence profiles and summary of findings tables *Journal of Clinical Epidemiology* **64(4)** 383-394.
66. Hasson H, Ismail AI, & Neiva G (2007) Home-based chemically-induced whitening of teeth in adults *Cochrane Database of Systematic Reviews* **52(1)** 71-72.

# Effects of Finishing and Polishing Methods on the Surface Roughness and Surface Free Energy of Bulk-fill Resin Composites

R Ishii • T Takamizawa • A Tsujimoto • S Suzuki  
A Imai • WW Barkmeier • MA Latta • M Miyazaki

## Clinical Relevance

The finishing and polishing methods, along with the type of resin composite, significantly affected the surface roughness and surface free energy.

## SUMMARY

The purpose of this study was to determine the influence of finishing and polishing methods on surface properties of bulk-fill resin composites through surface roughness (*Ra*) and surface free energy (SFE) measurements, and scanning electron microscopy (SEM) observations. Three bulk-fill resin composites, Tetric EvoCeram Bulk Fill (TB), Filtek Bulk Fill (FB), and Filtek Bulk Fill Flowable Restorative (FF), and two conventional resin composites, Clearfil AP-X

(AP) and Estelite  $\Sigma$  Quick (EQ) were used. Seventy cured specimens of each resin composite were prepared and divided into seven groups of 10 specimens. *Ra*, SFE measurements, and SEM observations were conducted after finishing and polishing procedures. Three groups of specimens were finished with a fine grit diamond bur (FDB), and three with a tungsten carbide bur (CBB). After finishing, one group from each type of finishing was polished with aluminum oxide flexible disks (SSD) and one group from each type of finishing was polished with diamond particles embedded

Ryo Ishii, DDS, PhD, Department of Operative Dentistry, Nihon University Graduate School of Dentistry, Tokyo, Japan

\*Toshiki Takamizawa, DDS, PhD, Department of Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Akimasa Tsujimoto, DDS, PhD, Department of Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Soshi Suzuki, DDS, Department of Operative Dentistry, Nihon University Graduate School of Dentistry, Tokyo, Japan

Arisa Imai, DDS, Department of Operative Dentistry, Nihon University Graduate School of Dentistry, Tokyo, Japan

Wayne W Barkmeier, DDS, MS, Department of General Dentistry, Creighton University School of Dentistry, Omaha, NE, USA

Mark A Latta, DMD, MS, Department of General Dentistry, Creighton University School of Dentistry, Omaha, NE, USA

Masashi Miyazaki, DDS, PhD, Department of Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

\*Corresponding author: 1-8-13, Kanda-Surugadai, Chiyoda-ku, Tokyo, Japan; e-mail: Takamizawa.toshiki@nihon-u.ac.jp

DOI: 10.2341/18-246-L

in a silicone point (CMP). A baseline group of samples that were neither finished nor polished after removing the translucent strips from the surface was examined. Although the baseline group showed significantly lower *Ra* values than the other groups, most resin composites showed lower *Ra* values with CBB+SSD than with the other finishing and polishing groups. Among the tested resin composites, EQ showed significantly lower *Ra* values than the other resin composites, regardless of the finishing and polishing methods. On the other hand, AP showed significantly higher *Ra* values than the other resin composites in all finishing and polishing groups, apart from FB with FDB. For the finished specimens, most resin composites showed higher SFE values with CBB than with FDB. For the polished specimens, all the tested resin composites with CMP showed lower  $\gamma_s$  values than those with SSD, regardless of the finishing method. The baseline groups of TB and FB showed significantly lower SFE values than the other finished and polished groups. In the SEM observations, all the examined resin composites showed rougher surfaces after finishing with FDB than with CBB. However, when comparing the different polishing methods (CMP and SSD), surface smoothness appeared to be material dependent.

## INTRODUCTION

Recent improvements in direct resin composite technology have led to their increased use in the treatment of posterior lesions.<sup>1</sup> Various types of resin composites with distinctive features, such as esthetics, low shrinkage, favorable mechanical properties, and good handling features, have been introduced in clinical settings.<sup>2,3</sup> In particular, bulk-fill resin composites have been extensively adopted because of their ability to reduce contraction stress along with the number of necessary clinical steps.<sup>4,5</sup> Single-layer bulk-fill resin composites can be used to create thicknesses of up to 4 mm with adequate light irradiation.<sup>6-8</sup> These composites show rapid activation of polymerization thanks to modified initiation systems, and they also have increased translucency due to decreased filler load and increased filler size.<sup>9,10</sup> In addition, high-viscosity bulk-fill resin composites that can be used efficiently as conventional resin composites are also available.<sup>3,11</sup> Although the surface and mechanical properties of these materials are sufficient for use in high-stress-bearing areas, the information available

on their surface characteristics is limited compared with low-viscosity bulk-fill resin composites.

Irregularities in restoration surfaces resulting from inappropriate finishing and polishing may create problems in clinical use, including staining, plaque accumulation, restoration degradation, and gingival inflammation.<sup>12-16</sup> Furthermore, rough surfaces on occlusal contact areas also induce friction, leading to wear on the surface of the opposing tooth.<sup>17</sup> Thus, appropriate finishing and polishing procedures are important in order to obtain the desired esthetics and to ensure the properties of the resin composite restorations.

The surface properties of polished resin composites are typically evaluated on the basis of surface roughness, gloss, and morphologic observation. Although these characteristics provide useful information about the surface properties after finishing and polishing, they are less helpful for understanding the chemical interactions between the polished surfaces and adhering substances in the oral environment (eg, salivary glycoproteins, plaque, and stains).<sup>18</sup> In solids, the surface free energy (SFE) is an important parameter that determines the chemical interactions that occur at the surface. SFE is related to the work of adhesion and interfacial tension with liquids<sup>19,20</sup> and can be calculated by measuring contact angles with different liquids of known SFE (eg, 1-bromonaphthalene, diiodomethane, and distilled water). The measurement of contact angles is an established method to understand changes in surface characteristics resulting from chemical interactions between mineralized tissue and functional monomers or the influence of the oxygen-inhibited layer of adhesives.<sup>19-21</sup> However, few studies have been carried out on polishing methods for resin composites from the perspective of interface science. Therefore, it would be valuable to determine the SFEs of the polished surfaces of resin composites.

The objectives of this study were to determine the effects of different finishing and polishing methods on the surface roughness and SFE of bulk-fill resin composites and to compare their surface properties with those of conventional resin composites. The null hypothesis was that the surface roughness and SFE of resin composites are not affected by the finishing and polishing methods or the type of resin composite.

## METHODS AND MATERIALS

### Study Materials

The components of resin composites used in this study are shown in Table 1. Three bulk-fill resin

Table 1: *Materials Used in This Study*

| Code                    | Resin Composite<br>(Shade: Lot No.)                  | Main Components                                                                                                                                                                   | Type of Resin Composite              | Manufacturer                          |
|-------------------------|------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------|---------------------------------------|
| TB                      | Tetric EvoCream Bulk Fill (A2: T21387)               | bis-GMA, UDMA, ytterbium trifluoride, Ba glass filler, mixed oxide, prepolymer filler, EBPADMA, additives, catalysts, stabilizers, pigments                                       | High-viscosity bulk-fill             | Ivoclar vivadent Schaan, Lichtenstein |
| FB                      | Filtek Bulk Fill Posterior restorative (A2: N701975) | Silane-treated ceramic, aromatic UDMA, ytterbium fluoride, UDMA, silane-treated silica, DDDMA, silane-treated zirconia, water, modified methacrylate monomer, EDMAB, benzotriazol | High-viscosity bulk-fill             | 3M ESPE, St Paul, MN, USA             |
| FF                      | Filtek Bulk Fill Flowable restorative (A2: N865208)  | Silane-treated ceramic, bis-GMA, UDMA, EDMAB, substituted dimethacrylate, bis-EMA, TEGDMA, benzotriazol, ytterbium fluoride,                                                      | Low-viscosity bulk-fill              | 3M ESPE                               |
| AP                      | Clearfil AP-X (A2: CC0043)                           | bis-GMA, TEGDMA, silane barium glass filler, silane silica filler, silanated colloidal silica, catalysts, accelerators, CQ, pigments, others                                      | Conventional (small particle hybrid) | Kuraray Noritake Dental Tokyo, Japan  |
| EQ                      | Estelite $\Sigma$ quick (A2:154095P)                 | bis-GMA, TEGDMA, silica-zirconia filler, dibutyl hydroxy toluene                                                                                                                  | Conventional (supra-nano filled)     | Tokuyama Dental Tokyo, Japan          |
| <b>Finishing Bur</b>    |                                                      | <b>Model</b>                                                                                                                                                                      |                                      |                                       |
| FDB                     | Super fine grit diamond bur                          | SF102R                                                                                                                                                                            |                                      | Shofu, Kyoto, Japan                   |
| CBB                     | Tungsten carbide bur                                 | FG7714 (12 blades)                                                                                                                                                                |                                      | Kerr, Orange, CA, USA                 |
| <b>Polishing System</b> |                                                      |                                                                                                                                                                                   |                                      |                                       |
| CMP                     | Compomaster (one-step system)                        | One step diamond polisher (6- $\mu$ m) silicone base (25%), diamond particles (75%)                                                                                               |                                      | Shofu                                 |
| SSD                     | Super-Snap (multi-step system)                       | Fine: green: $\phi$ 12-mm disk; 20- $\mu$ m aluminium oxide<br>Superfine: red: $\phi$ 12-mm disk; 7- $\mu$ m aluminium oxide                                                      |                                      | Shofu                                 |

Abbreviations: AP, Clearfil AP-X; bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy) phenyl] propane; CBB, tungsten carbide bur; CMB, Compomaster; CQ, dl-camphorquinone; DDDMA, 1,12-dodecane dimethacrylate; EBPADMA, ethoxylated bisphenol A dimethacrylate; EDMAB, ethyl 4-dimethyl aminobenzoate; EQ, Estelite  $\Sigma$  quick; FB, Filtek Bulk Fill Posterior restorative; FDB, super fine grit diamond bur; FF, Filtek Bulk Fill Flowable restorative; SSD, Super-Snap; TB, Tetric EvoCream Bulk; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

composites (Tetric EvoCeram Bulk Fill [TB; Ivoclar Vivadent, Schaan, Liechtenstein], Filtek Bulk Fill [FB; 3M ESPE, St Paul, MN, USA], and Filtek Bulk Fill Flowable Restorative [FF; 3M ESPE]) along with two conventional resin composites (Clearfil AP-X [AP; Kuraray Noritake Dental, Tokyo, Japan] as a hybrid resin composite and Estelite  $\Sigma$  Quick [EQ; Tokuyama Dental, Tokyo, Japan] as a supra-nano-filled resin composite) were tested.<sup>1</sup> A halogen quartz tungsten curing unit (Optilux 501, SDS Kerr, Danbury, CT, USA) was used to avoid any influence of the reported nonuniformity of light-emitting diode (LED) curing units.<sup>22,23</sup> The light irradiance (average=600 mW/cm<sup>2</sup>) of the curing unit was confirmed using a dental radiometer (Model 100, Kerr).

### Inorganic Filler Content

The inorganic filler content of the resin composites was evaluated using thermogravimetry and differential thermal analysis (TG/DTA6300, Seiko Instruments, Tokyo, Japan). A paste of each resin composite (50 mg) was placed in a pure platinum cylindroid crucible (diameter=7 mm, depth=10

mm) and heated in the thermogravimeter from 25°C to 800°C at a heating rate of 10°C/min in atmospheric air until the organic components were completely incinerated. The weight of the residual resin paste was automatically measured using the built-in differential balance, which has a high sensitivity and horizontal accuracy. The inorganic filler content (wt%) was then calculated on the basis of the compensated blank curve. Six measurements per test material were evaluated to obtain an average inorganic filler content.

### Specimen Preparation for Finishing and Polishing

The specimens for finishing and polishing were prepared in cylindrical Teflon molds (height=2.0 mm, diameter=10.0 mm; Sanplatec Corp, Osaka, Japan). One end was sealed with a translucent polyester matrix tape (Matrix Tape and Dispenser, 3M ESPE), and the resin paste was inserted from the open end. The other end was then sealed in the same way, and pressure was applied manually through a glass plate. Subsequently, both the top and bottom of

the mold were irradiated with light for 30 seconds. The mold was then cut open with a scalpel and removed from the cured resin composite. The specimens were stored in the dark at 25°C for 24 hours before finishing and polishing to allow postirradiation polymerization to reach a stable state.<sup>24</sup> Seventy specimens were prepared for each resin composite.

### Finishing and Polishing Procedures

The 70 specimens of each resin composite were treated in the following way. The specimens were divided into seven groups of 10, and one group was set aside without further treatment to serve as a baseline measurement. The specimens in the remaining six groups were ground flat with 320-grit silicon carbide paper (Fuji Star Type DDC, Sankyo Rikagaku Co, Saitama, Japan) under water as a coolant. Three of the groups were finished using a superfine-grit diamond bur (FDB; SF102R, Shofu, Kyoto, Japan), and the other three were finished using a tungsten carbide bur (CBB; FG7714, long tapered trimming, Kerr). The finishing procedures were performed using a high-speed handpiece under spraying water as a coolant. The finishing procedures were carried out with a light hand pressure in multiple directions, and the burs were changed after five uses. In addition, in order to guarantee the flatness of the finished and polished samples, the sample thickness was measured using a dial gauge micrometer (CPM15-25DM; Mitutoyo, Tokyo, Japan) at five locations. From the three groups finished by each method, one was set aside for measurement. One of the two remaining groups was polished using the one-step point-type polishing system CompoMaster (CMP; Shofu), and the other was polished using the multistep polishing system Super-Snap Rainbow Technique Kit (SSD; Shofu). All polishing procedures were carried out using a slow-speed handpiece (5000 rpm) with a contact pressure of 1.0 N monitored by a digital balance (AT200, Mettler, Greifensee, Switzerland) underneath the specimen. The specimens were finished and polished by a single operator to reduce variability between samples. The final groups of specimens for each composite were as follows: a) no treatment; b) ground, finished with FDB; c) ground, finished with CBB, d) ground, finished with FDB, polished with CMP; e) ground, finished with CBB, polished with CMP; f) ground, finished with FDB, polished with SSD; g) ground, finished with CBB, polished with SSD.

### Measurement of Surface Roughness (*Ra*)

Before the measurement of surface roughness (*Ra*), the specimens were cleaned with distilled water in an

ultrasonic cleaner for 1 min and dried with oil-free air. The surfaces of all specimens were observed using a three-dimensional laser scanning microscope (VK-8700, Keyence, Osaka, Japan). The spectral maximum of the excitation light was 658 nm, and the intensity of the excitation light, along with the amplification of the photomultiplier, was kept constant during the observation period. Using software (VK Analyzer, Keyence), the *Ra* values of 10 specimens in each group in 1.0 mm × 1.0 mm regions were measured. Profilometric measurements were conducted in five regions near the centers of the specimens. The means were then determined for each group.

### SFE Measurements

Specimens for SFE measurements were prepared as for the *Ra* measurements, as described earlier. Each resin composite surface was treated in accordance with the experimental protocol for the finishing and polishing procedures. The contact angles of the specimens, including those in the baseline group, were measured in order to evaluate the surface characteristics of each specimen surface. SFE was determined by measuring the contact angles on the specimen surfaces using three test liquids with known SFE parameters: 1-bromonaphthalene, diiodomethane, and distilled water.<sup>19-21</sup> A contact angle meter (Drop Master DM500, Kyowa Interface Science, Saitama, Japan) connected to a charge-coupled device camera was used for automatic measurements of the contact angles.

The equilibrium contact angle ( $\theta$ ) was measured for each test liquid on 10 specimens for each condition using the sessile-drop method at  $23 \pm 1^\circ\text{C}$ . Sessile drops (volume = 1.0  $\mu\text{L}$ ) of each liquid were dispensed using a micropipette. The SFE parameters of the solids were then determined on the basis of the following fundamental concepts of wetting. The Young–Dupré equation relates the work of adhesion for a solid (S) and liquid (L) that are in contact ( $W_{\text{SL}}$ ), the interfacial free energy between the solid and the liquid ( $\gamma_{\text{SL}}$ ), and the free energies of the liquid and solid ( $\gamma_{\text{L}}$  and  $\gamma_{\text{S}}$ , respectively) as follows:

$$W_{\text{SL}} = \gamma_{\text{L}} + \gamma_{\text{S}} - \gamma_{\text{SL}} = \gamma_{\text{L}}(1 + \cos\theta).$$

Extending the Fowkes equation following the Kitazaki–Hata approach<sup>25</sup> gives

$$\gamma_{\text{SL}} = \gamma_{\text{L}} + \gamma_{\text{S}} - 2(\gamma_{\text{L}}^{\text{d}}\gamma_{\text{S}}^{\text{d}})^{1/2} - 2(\gamma_{\text{L}}^{\text{p}}\gamma_{\text{S}}^{\text{p}})^{1/2} - 2(\gamma_{\text{L}}^{\text{h}}\gamma_{\text{S}}^{\text{h}})^{1/2},$$

$$\gamma_{\text{L}} = \gamma_{\text{L}}^{\text{d}} + \gamma_{\text{L}}^{\text{p}} + \gamma_{\text{L}}^{\text{h}}; \gamma_{\text{S}} = \gamma_{\text{S}}^{\text{d}} + \gamma_{\text{S}}^{\text{p}} + \gamma_{\text{S}}^{\text{h}},$$

where  $\gamma^d$ ,  $\gamma^p$ , and  $\gamma^h$  are the components of the SFE ( $\gamma$ ) arising from the dispersion force, the polar (permanent and induced) force, and the hydrogen-bonding force, respectively. The  $\theta$  values were determined for the three test liquids, and the surface-energy parameters of the treated enamel surfaces were calculated using the earlier equations using add-on software along with an interface measurement and analysis system (FAMAS, Kyowa Interface Science).

**Scanning Electron Microscopy (SEM) Observations**—After polishing the cured resin composite specimens to a high gloss using abrasive discs (Fuji Star Type DDC), they were further polished using a series of diamond pastes down to a particle size of 0.25  $\mu\text{m}$  (DP-Paste; Struers, Ballerup, Denmark). The polished surfaces were then subjected to argon-ion beam etching (IIS-200ER; Elionix Inc, Tokyo, Japan) for 40 seconds with the ion beam directed perpendicular to the polished specimen surface (accelerating voltage=1 kV, ion current density=0.4 mA/cm<sup>2</sup>). The surfaces were then coated with a thin film of gold in a vacuum evaporator (Quick Coater Type SC-701; Sanyu Denchi, Tokyo, Japan). Scanning electron microscopy (SEM; FE-8000, Elionix Inc) observations were conducted at an operating voltage of 10 kV and magnifications of 5000 $\times$  and 30,000 $\times$ .

Representative samples of the five resin composites polished using different methods were also examined by SEM. The surfaces of the samples were rinsed with tap water after the finishing or polishing procedures followed by evaporation coating. The coated surfaces were visualized by SEM at an operating voltage of 10 kV and a magnification of 2,500 $\times$ .

### Statistical Analysis

A power analysis indicated that at least four samples were necessary for effective measurement of inorganic filler content and eight samples were necessary for *Ra* and SFE measurements. Thus, this study was initially performed with sample sizes of five for inorganic filler content measurement and 10 for *Ra* and SFE measurements. After gathering the data, post hoc power tests were performed using two statistical software systems (G Power calculator version 3.1; <http://www.gpower.hhu.de/>, and Sigma Plot version 13.0; Systat Software, Inc, Chicago, IL, USA). These tests indicated that the sample size was adequate.

Based on the normal distribution (Kolmogorov–Smirnov test), the data for each test were subjected

Table 2: Inorganic filler contents (wt%)

|    | Inorganic Filler Content, mean (SD) | Tukey Group <sup>a</sup> |
|----|-------------------------------------|--------------------------|
| TB | 77.4 (0.4)                          | b                        |
| FB | 75.2 (0.3)                          | c                        |
| FF | 63.8 (0.4)                          | e                        |
| AP | 83.8 (0.5)                          | a                        |
| EQ | 67.3 (0.4)                          | d                        |

Abbreviations: AP, Clearfil AP-X; EQ, Estelite  $\Sigma$  quick; FB, Filtek Bulk Fill Posterior restorative; FDB, super fine grit diamond bur; FF, Filtek Bulk Fill Flowable restorative; TB, Tetric EvoCream Bulk.  
<sup>a</sup> Same lowercase letter in vertical columns indicates no difference at 5% significance level.  
 Values in parenthesis indicates standard deviation.

to analysis of variance (ANOVA) followed by the Tukey honestly significant difference test at a significance level of 0.05. One-way ANOVA was used for inorganic filler content, whereas two-way ANOVA, with the polishing method and type of resin composite as the factors, was used for the *Ra* and SFE data. Statistical analyses were carried out using commercially available statistical software (Sigma-Plot version 11.0; SPSS Inc, Chicago, IL, USA).

## RESULTS

### Inorganic Filler Content

The inorganic filler content of the resin composites are shown in Table 2. The average inorganic filler content ranged from 63.8 to 83.8 wt% and depended on the material. The inorganic filler content decreased in the following order: AP > TB > FB > EQ > FF. The hybrid conventional resin composite AP showed a significantly higher inorganic filler content than the other resin composites. In contrast, the low-viscosity bulk-fill resin composite FF showed a significantly lower inorganic filler content than the other resin composites.

### Ra Values

The *Ra* values of the resin composites are shown in Table 3. The low *Ra* values observed in the baseline groups are artifacts of the experimental protocol and are not representative of unfinished composites in clinical settings. The two-way ANOVA results indicate that the finishing and polishing methods, along with the type of resin composite, significantly affected *Ra* ( $p < 0.001$ ). The two-way interaction between these two factors was also significant ( $p < 0.001$ ). All the finished and polished groups showed significantly higher *Ra* values than the baseline groups, regardless of the type of resin composite. In particular, the resin composites fin-

| Table 3: Influence of Finishing and Polishing Procedures on Surface Roughness, Ra, mean $\mu\text{m}$ (SD)                                                                                                                                                                      |                |                |                |                |                |                |                |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
|                                                                                                                                                                                                                                                                                 | Baseline       | FDB            | CBB            | FDB+CMP        | CBB+CMP        | FDB+SSD        | CBB+SSD        |
| TB                                                                                                                                                                                                                                                                              | 0.08 (0.01) aE | 0.68 (0.05) cA | 0.39 (0.02) dC | 0.43 (0.01) dB | 0.32 (0.05) cD | 0.29 (0.01) bD | 0.31 (0.01) bD |
| FB                                                                                                                                                                                                                                                                              | 0.09 (0.01) aF | 0.99 (0.02) aA | 0.68 (0.01) bB | 0.49 (0.01) cC | 0.48 (0.01) bC | 0.31 (0.01) bD | 0.25 (0.01) cE |
| FF                                                                                                                                                                                                                                                                              | 0.08 (0.01) aG | 0.88 (0.02) bA | 0.59 (0.01) cB | 0.54 (0.01) bC | 0.45 (0.02) bD | 0.31 (0.01) bE | 0.26 (0.01) cF |
| AP                                                                                                                                                                                                                                                                              | 0.09 (0.01) aE | 0.87 (0.02) bA | 0.80 (0.01) aB | 0.86 (0.04) aA | 0.76 (0.02) aC | 0.75 (0.02) aC | 0.64 (0.01) aD |
| EQ                                                                                                                                                                                                                                                                              | 0.08 (0.02) aD | 0.62 (0.02) dA | 0.30 (0.02) eB | 0.28 (0.01) eB | 0.28 (0.01) dB | 0.22 (0.01) cC | 0.21 (0.01) dC |
| Abbreviations: AP, Clearfil AP-X; CBB, tungsten carbide bur; CMB, Compomaster; EQ, Estelite $\Sigma$ quick; FB, Filtek Bulk Fill Posterior restorative; FDB, super fine grit diamond bur; FF, Filtek Bulk Fill Flowable restorative; SSD, Super-Snap; TB, Tetric EvoCream Bulk. |                |                |                |                |                |                |                |
| <sup>a</sup> Same lowercase letter in vertical columns indicates no difference at 5% significance level. Same capital letter in horizontal rows indicates no difference at 5% significance level.                                                                               |                |                |                |                |                |                |                |

ished with FDB showed higher  $Ra$  values than the other finishing and polishing groups. In contrast, the CBB+SSD groups showed lower  $Ra$  values than the other groups, with the exception of the baseline groups. Upon comparing the different finishing procedures (FDB and CBB), CBB resulted in significantly lower  $Ra$  values than FDB, regardless of the type of resin composite. Among the polishing methods, SSD resulted in significantly lower  $Ra$  values than CMP, regardless of the finishing method. Among the tested resin composites, EQ showed significantly lower  $Ra$  values than the other resin composites, regardless of the finishing and polishing methods. In contrast, AP showed significantly higher  $Ra$  values than the other resin composites in all finishing and polishing groups, with the exception of FB with FDB. For the bulk-fill resin composites, FB groups without polishing showed significantly higher  $Ra$  values than the other bulk-fill resin composites.

**SFE Parameters**—The total SFE ( $\gamma_S$ ) values and the three corresponding parameters are shown in Table 4 and Figure 1. Two-way ANOVA revealed that the finishing and polishing methods, along with the type of resin composite, significantly affected the  $\gamma_S$  value ( $p<0.001$ ). The two-way interaction between the factors was also significant ( $p<0.001$ ).

For the finished specimens, most resin composites showed higher  $\gamma_S$  values with CBB than with FDB.

For the polished specimens, all the tested resin composites with CMP showed lower  $\gamma_S$  values than those with SSD, regardless of the finishing method. Considering each resin composite across different finishing and polishing groups, the baseline groups of TB and FB showed significantly lower  $\gamma_S$  values than the other finished and polished groups. Comparing the resin composites in the groups finished with FDB and CBB, AP with SF resulted in a significantly higher  $\gamma_S$  value than the other resin composites; however, the  $\gamma_S$  value of AP was not significantly higher than those of FB and EQ with CBB. On the other hand, TB and FF showed lower  $\gamma_S$  values than the other resin composites with CBB. For groups polished with CMP, EQ showed significantly lower  $\gamma_S$  than the other resin composites. In contrast, for SSD, AP had significantly lower  $\gamma_S$  values than the other resin composites. In both cases, these trends did not depend on the finishing method.

Next, we examined the different components of  $\gamma_S$ . In all groups, the dispersion force ( $\gamma_S^d$ ) showed a similar value of approximately  $41\text{ mN}\cdot\text{m}^{-1}$ , whereas the polar force ( $\gamma_S^p$ ) and hydrogen-bonding force ( $\gamma_S^h$ ) varied by group. In terms of  $\gamma_S^p$  (Table 5), the resin composites could be classified into three groups: 1) for TB and FB, although the baseline groups showed somewhat lower  $\gamma_S^p$  values, finishing and polishing tended to increase  $\gamma_S^p$ ; 2) for AP, although the groups

| Table 4: Influence of Finishing and Polishing Procedures on Total Surface Free Energy, mean (SD). <sup>a</sup>                                                                                                                                                                  |                |                |                |                 |                |                |                 |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------|----------------|----------------|-----------------|----------------|----------------|-----------------|
|                                                                                                                                                                                                                                                                                 | Baseline       | FDB            | CBB            | FDB+CMP         | CBB+CMP        | FDB+SSD        | CBB+SSD         |
| TB                                                                                                                                                                                                                                                                              | 44.1 (0.5) cD  | 48.2 (1.2) bAB | 46.8 (1.1) bBC | 47.9 (1.4) bABC | 46.5 (1.2) aC  | 48.4 (1.3) cA  | 47.3 (0.9) cABC |
| FB                                                                                                                                                                                                                                                                              | 42.6 (0.8) dE  | 49.1 (0.8) bC  | 54.2 (1.3) aA  | 49.2 (1.4) aC   | 47.3 (0.8) aD  | 51.7 (1.1) aB  | 52.4 (1.1) aB   |
| FF                                                                                                                                                                                                                                                                              | 48.3 (1.1) aA  | 45.2 (1.1) cBC | 47.7 (0.9) bAB | 43.9 (1.0) cC   | 46.4 (2.0) aB  | 49.0 (1.3) bcA | 49.3 (1.3) bA   |
| AP                                                                                                                                                                                                                                                                              | 47.3 (0.9) abC | 50.9 (1.2) aB  | 55.3 (1.1) aA  | 43.8 (1.1) cE   | 44.7 (0.6) bDE | 45.3 (0.9) dD  | 45.8 (0.8) dD   |
| EQ                                                                                                                                                                                                                                                                              | 47.1(1.1) bC   | 48.6 (1.2) bC  | 54.9 (1.2) aA  | 42.2 (1.0) dD   | 42.4 (1.3) cD  | 50.2 (1.1) bB  | 49.6 (1.1) bBC  |
| Abbreviations: AP, Clearfil AP-X; CBB, tungsten carbide bur; CMB, Compomaster; EQ, Estelite $\Sigma$ quick; FB, Filtek Bulk Fill Posterior restorative; FDB, super fine grit diamond bur; FF, Filtek Bulk Fill Flowable restorative; SSD, Super-Snap; TB, Tetric EvoCream Bulk. |                |                |                |                 |                |                |                 |
| <sup>a</sup> Same lowercase letter in vertical columns indicates no difference at 5% significance level. Same capital letter in horizontal rows indicates no difference at 5% significance level.                                                                               |                |                |                |                 |                |                |                 |

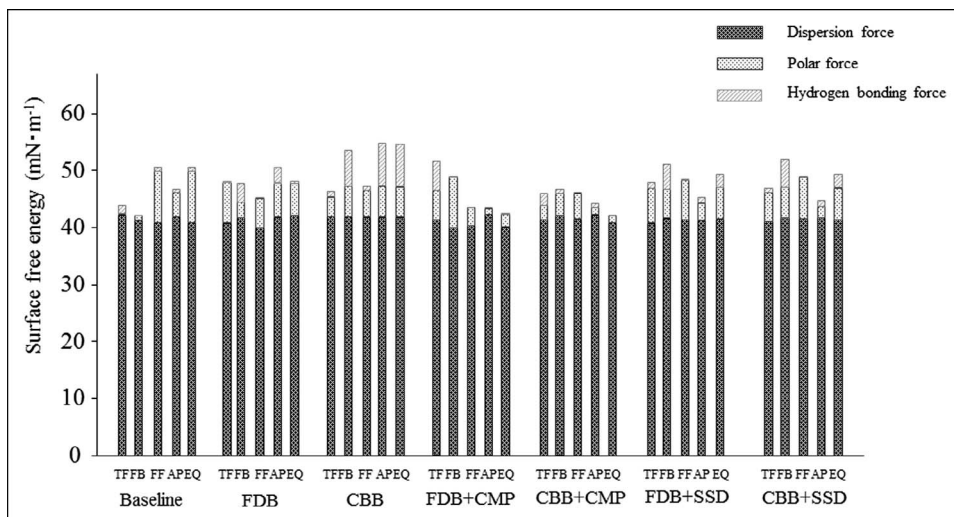


Figure 1. The total SFE ( $\gamma_s$ ) values and the three corresponding parameters.

finished with FDB and CBB showed significantly higher  $\gamma_s^p$  values than the baseline group, all polished groups showed significantly lower  $\gamma_s^p$  values than the baseline and finishing groups; 3) for FF and EQ, the SSD groups showed somewhat higher  $\gamma_s^p$  values than the other groups, whereas the CMP groups showed lower  $\gamma_s^p$  values.

The observed changes in  $\gamma_s^h$  (Table 6) depended strongly on the composite material, along with the finishing and polishing methods. Overall, FF showed low  $\gamma_s^h$  and significantly lower  $\gamma_s^h$  values in the finished and polished groups than the baseline group. In contrast, the other resin composites generally showed higher  $\gamma_s^h$  values after finishing or polishing. AP and EQ showed significantly higher  $\gamma_s^h$  values with CBB than the other groups. FB and EQ showed significantly higher  $\gamma_s^h$  values with SSD than with CMP, regardless of the finishing method. Compared with the other resin composites, the TB groups showed significantly higher  $\gamma_s^h$  values with CMP than with SSD, and the  $\gamma_s^h$  values were significantly higher than those for the other resin composites, regardless of the finishing method.

**SEM Observations**—The SEM images of polished surfaces after argon-ion etching are shown in Figure 2. The shapes, sizes, and distributions of inorganic fillers depended on the composite material. In the high-viscosity bulk-fill resin composite TB, 0.5–2.0  $\mu\text{m}$  irregular fillers and nanosized spherical fillers were packed at a high density (Figure 2A). In addition to the nanosized spherical fillers used in TB, FB included 0.5–5.0  $\mu\text{m}$  aggregates of filler particles (Figure 2B). FF was similar to FB but had larger interspaces between filler particles (Figure 2C). The hybrid composite AP had 0.5–10.0  $\mu\text{m}$  irregular fillers (Figure 2D). EQ had nanosize spherical fillers and pre-polymerized fillers that use the same nanosize spherical fillers (Figure 2E).

Representative SEM images of the resin composite surfaces after different finishing and polishing methods are shown in Figures 3 through 7. All the examined resin composites showed rougher surfaces after finishing with FDB than with CBB (Figures 3AB, 4AB, 5AB, 6AB, and 7AB). Evidence of scratches and plucked-out filler was obvious after FDB finishing. More specifically, the plucking out of aggregated fillers in FF and FB, pre-polymerized

Table 5: Influence of Finishing and Polishing Procedures on Polar Force, mean (SD) <sup>a</sup>

|    | Baseline      | FDB           | CBB            | FDB+CMF       | CBB+CMF       | FDB+SSD        | CBB+SSD      |
|----|---------------|---------------|----------------|---------------|---------------|----------------|--------------|
| TB | 0.2 (0.1) cD  | 7.2 (1.8) aA  | 3.5 (0.9) bC   | 3.2 (0.7) bC  | 2.7 (1.0) bcC | 6.2 (1.2) abAB | 5.2 (1.3) bB |
| FB | 1.0 (0.6) cD  | 2.8 (0.7) cC  | 5.4 (1.1) aB   | 9.0 (1.4) aA  | 3.9 (0.8) abC | 5.2 (0.9) bB   | 5.4 (0.9) bB |
| FF | 4.3 (1.0) bBC | 5.4 (0.9) bB  | 4.8 (0.9) abBC | 3.3 (1.1) cBC | 4.7 (1.9) aC  | 7.1 (0.8) aA   | 7.5 (1.3) aA |
| AP | 4.3 (0.8) bB  | 6.0 (1.0) abA | 5.5 (0.9) aA   | 1.3 (0.8) dD  | 1.4 (0.5) cD  | 3.1 (0.7) cC   | 2.0 (0.7) cD |
| EQ | 5.5 (1.0) aA  | 5.8 (1.0) abA | 5.4 (1.3) aA   | 2.4 (1.3) cdB | 1.3 (1.0) cB  | 5.7 (1.0) bA   | 5.7 (1.0) bA |

Abbreviations: AP, Clearfil AP-X; CBB, tungsten carbide bur; CMB, Compomaster; EQ, Estelite  $\Sigma$  quick; FB, Filtek Bulk Fill Posterior restorative; FDB, super fine grit diamond bur; FF, Filtek Bulk Fill Flowable restorative; SSD, Super-Snap; TB, Tetric EvoCream Bulk.

<sup>a</sup> Same lowercase letter in vertical columns indicates no difference at 5% significance level.

Same capital letter in horizontal rows indicates no difference at 5% significance level.

| Table 6: Influence of Finishing and Polishing Procedures on Hydrogen Bonding Force, mean (SD) <sup>a</sup>                                                                                                                                                                      |              |               |              |              |              |              |               |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------|---------------|--------------|--------------|--------------|--------------|---------------|
|                                                                                                                                                                                                                                                                                 | Baseline     | FDB           | CBB          | FDB+CMP      | CBB+CMP      | FDB+SSD      | CBB+SSD       |
| TB                                                                                                                                                                                                                                                                              | 1.7 (0.3) aA | 0.1 (0.1) cE  | 1.1 (0.3) cB | 2.3 (0.2) aA | 2.2 (0.5) aA | 1.1 (0.5) cC | 0.8 (0.3) cCD |
| FB                                                                                                                                                                                                                                                                              | 0.2 (0.1) cE | 3.4 (0.3) aC  | 6.5 (0.6) bA | 0.2 (0.1) bE | 1.0 (0.2) bC | 4.6 (0.6) aB | 5.0 (0.6) aB  |
| FF                                                                                                                                                                                                                                                                              | 2.0 (0.3) aA | 0.1 (0.1) cC  | 0.8 (0.2) cB | 0.2 (0.1) bC | 0.1 (0.1) cC | 0.3 (0.2) dC | 0.2 (0.1) dC  |
| AP                                                                                                                                                                                                                                                                              | 0.8 (0.1) bC | 2.8 (0.3) bB  | 7.6 (0.6) aA | 0.2 (0.1) bD | 0.9 (0.5) bC | 1.2 (0.6) cC | 1.2 (0.5) cC  |
| EQ                                                                                                                                                                                                                                                                              | 1.0 (0.3) bC | 0.5 (0.2) cCD | 7.6 (0.6) aA | 0.1 (0.1) bD | 0.2 (0.2) cD | 2.4 (0.4) bB | 2.6 (0.4) bB  |
| Abbreviations: AP, Clearfil AP-X; CBB, tungsten carbide bur; CMB, Compomaster; EQ, Estelite $\Sigma$ quick; FB, Filtek Bulk Fill Posterior restorative; FDB, super fine grit diamond bur; FF, Filtek Bulk Fill Flowable restorative; SSD, Super-Snap; TB, Tetric EvoCream Bulk. |              |               |              |              |              |              |               |
| <sup>a</sup> Same lowercase letter in vertical columns indicates no difference at 5% significance level. Same capital letter in horizontal rows indicates no difference at 5% significance level.                                                                               |              |               |              |              |              |              |               |

fillers in EQ, and large glass fillers in AP was observed. In contrast, with the exception of AP specimens, which exhibited some superficial scratches, the surfaces finished with CBB were relatively smooth.

FB specimens finished with FDB exhibited rougher surfaces than those finished with CBB, regardless of the polishing method. For the other resin composites, no clear differences in morphology were observed between the samples finished with FDB and CBB, regardless of the polishing method. When comparing the different polishing methods (CMP

and SSD), smoother surfaces were obtained with SSD for TB, FF, and EQ, regardless of the finishing method. For FB, polishing with CMP after finishing with CBB produced the smoothest surface among the different polishing methods. For AP, no clear differences in morphology were observed between the samples polished with CMP and SSD.

DISCUSSION

In recent years, LED-based curing sources have been used extensively in clinics instead of halogen quartz tungsten curing units due to their compactness and easy handling. However, the range of wavelengths of an LED curing unit is narrower than that of a conventional halogen quartz-tungsten based curing unit. In addition, it has been reported that some LED curing units have uneven light intensity and wavelength, depending on the location of the curing light guide.<sup>22,23</sup> In this study, to evaluate the basic surface characteristics of bulk-fill and conventional resin composite using a stable curing light source, a conventional halogen quartz-tungsten based curing unit was used.

In practice, the surfaces of resin composite restorations are directly exposed to degradation by biofilm attack, acid erosion, water sorption, occlusal and thermal stresses, enzymatic degradation, and other sources.<sup>1,26-28</sup> Thus, it is necessary to determine the surface properties of resin composite restorations in order to understand the relationship between degradation sources and the restoration surface. In this study, we evaluated the chemical and morphologic changes of bulk-fill resin composites and compared them with those of conventional resin composites after different finishing and polishing methods.

The results indicated that the finishing and polishing methods, along with the type of resin composite, significantly affected *Ra*, in agreement with previous reports.<sup>12-17,29-31</sup> Thus, the null hy-

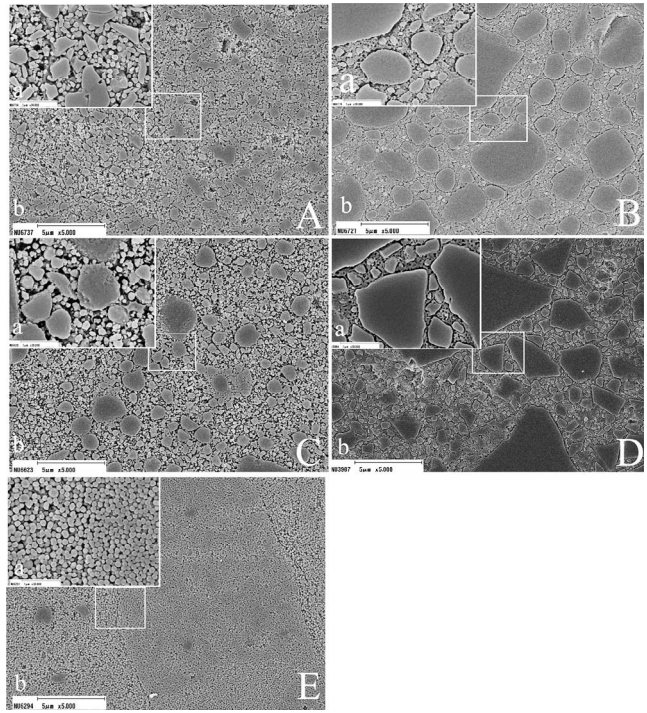


Figure 2. SEM images of resin composite surfaces after argon-ion etching. (A): Tetric EvoCeram Bulk Fill (TB)—(a) 5000 $\times$  and (b) 30,000 $\times$ . (B): Filtek Bulk Fill posterior restorative (FB)—(a) 5000 $\times$  and (b) 30,000 $\times$ . (C): Filtek Bulk Fill Flowable Restorative (FF)—(a) 5000 $\times$  and (b) 30,000 $\times$ . (D): Clearfil AP-X (AP)—(a) 5000 $\times$  and (b) 30,000 $\times$ . (E): Estelite  $\Sigma$  Quick (EQ)—(a) 5000 $\times$  and (b) 30,000 $\times$ .

pothesis that the surface roughness is not affected by the finishing or polishing method or the type of resin composite was rejected. Finishing with FDB resulted in significantly higher  $Ra$  values than finishing with CBB, regardless of the type of resin composite. Although FB, FF, and EQ use nano-sized spherical fillers, FB and FF exhibited rougher surfaces than the conventional hybrid resin composite AP with FDB, and the roughness of EQ with FDB was nearly twice that of EQ with CBB. Based on SEM observations, scratches and filler plucking were more obvious with FDB than with CBB for all resin composites. This may be attributed to the different finishing mechanisms of CBB and FDB<sup>32,33</sup>; tungsten carbide burs have several fine blades that cut away the surface, whereas diamond burs grind the surface with many abrasive diamond particles. When considering SEM images (FDB and CBB), the abrasive particles might have promoted the plucking of filler particles, generating voids in the resin surface and making it rougher (Figures 3A, 4A, 5A, 6A, and 7A). In contrast, the tungsten carbide burs cut the filler particles so that part of the filler particle was embedded in the surface, which resulted in a smoother surface (Figures 3B, 4B, 5B, 6B, and 7B). It has been reported that although tungsten carbide burs do not cut as efficiently as diamond burs, tungsten carbide burs are best suited for smoothing and finishing.<sup>32</sup> Therefore, the CBB-finished specimens exhibited less scratching and filler plucking than the FDB-finished specimens. The FB and FF composite materials use 0.5–5.0  $\mu\text{m}$  cluster fillers that aggregate nano-sized fillers, and EQ also uses pre-polymerized fillers. Because the adhesion strength between pre-polymerized or cluster fillers and the resin matrix might be low, the plucking of pre-polymerized or cluster fillers after finishing with FDB might result in a rougher surface.<sup>34</sup>

To date, various polishing systems using different materials and shapes have been introduced.<sup>33</sup> In this study, a one-step polishing system with diamond particles impregnated in a silicone point (CMP) and a multiple-step polishing system using aluminum oxide abrasive disks (SSD) were examined. In the results for  $Ra$  after polishing, SSD showed significantly lower  $Ra$  values than CMP for most resin composites used, regardless of the finishing method. In particular, polishing with SSD after finishing with CBB produced the smoothest surfaces, irrespective of the type of resin composite. Earlier studies also showed that aluminum oxide flexible disks created the smoothest surfaces on resin

composite restorations due to their tendency to abrade filler particles and resin matrix equally without plucking filler particles or gouging into the material.<sup>32,33</sup> In addition, it has also been reported that  $Ra$  and gloss strongly depend on polishing time and application force.<sup>35,36</sup> The polishing system SSD takes multiple steps, and total polishing time was twice as long as CMP in this experimental protocol. These differences in polishing mechanism and polishing time might explain the differences in results between SSD and CMP.

In this study, most tested composites polished with SSD showed  $Ra$  values of approximately 0.3  $\mu\text{m}$  or lower. The exception is AP, which showed a value over 0.6  $\mu\text{m}$  with every polishing combination. AP uses irregular fillers with a wide range of sizes, and the interspaces between filler particles are larger than in EQ. EQ showed significantly lower  $Ra$  values than the other resin composites, and its  $Ra$  values after polishing with SSD were close to 0.2  $\mu\text{m}$ . After argon-ion etching, the surface of EQ appeared homogeneous, despite the fact that EQ uses pre-polymerized fillers and has a relatively low inorganic filler content. The high polishability of EQ can be attributed to its fine and even spherical fillers and the small interspaces between filler particles (Figure 2E [see a and b]).<sup>37</sup>

Another aim of this study was to investigate the surface chemical properties through SFE measurements after different finishing and polishing methods. As for  $Ra$ , the finishing and polishing methods, along with the type of resin composite, significantly affected SFE. Thus, the null hypothesis that the SFEs of resin composites are not influenced by the finishing or polishing method or type of resin composite was rejected. For most resin composites, the baseline specimens showed lower  $\gamma_s$  values than the finishing samples. The baseline high-viscosity TB and FB composites showed significantly lower  $\gamma_s$  compared with the finished and polished TB and FB, along with the other resin composites. Baseline surfaces produced by translucent matrix tape in contact with resin composites make up a resin-rich layer. In general, the hydrophilic nature of inorganic fillers gives them high wettability, whereas the resin matrix is hydrophobic. FF, AP, and EQ contain the hydrophilic resin monomer triethylene glycol dimethacrylate (TEGDMA)<sup>23</sup>; however, TB and FB each use different resin monomers with a hydrophobic backbone and increased molecular mobility, ethoxy-lated bisphenol A dimethacrylate (EBDADMA) and 1,12-dodecane dimethacrylate (DDDMA), respectively.<sup>3,35</sup> This may explain the lower  $\gamma_s$  values observed

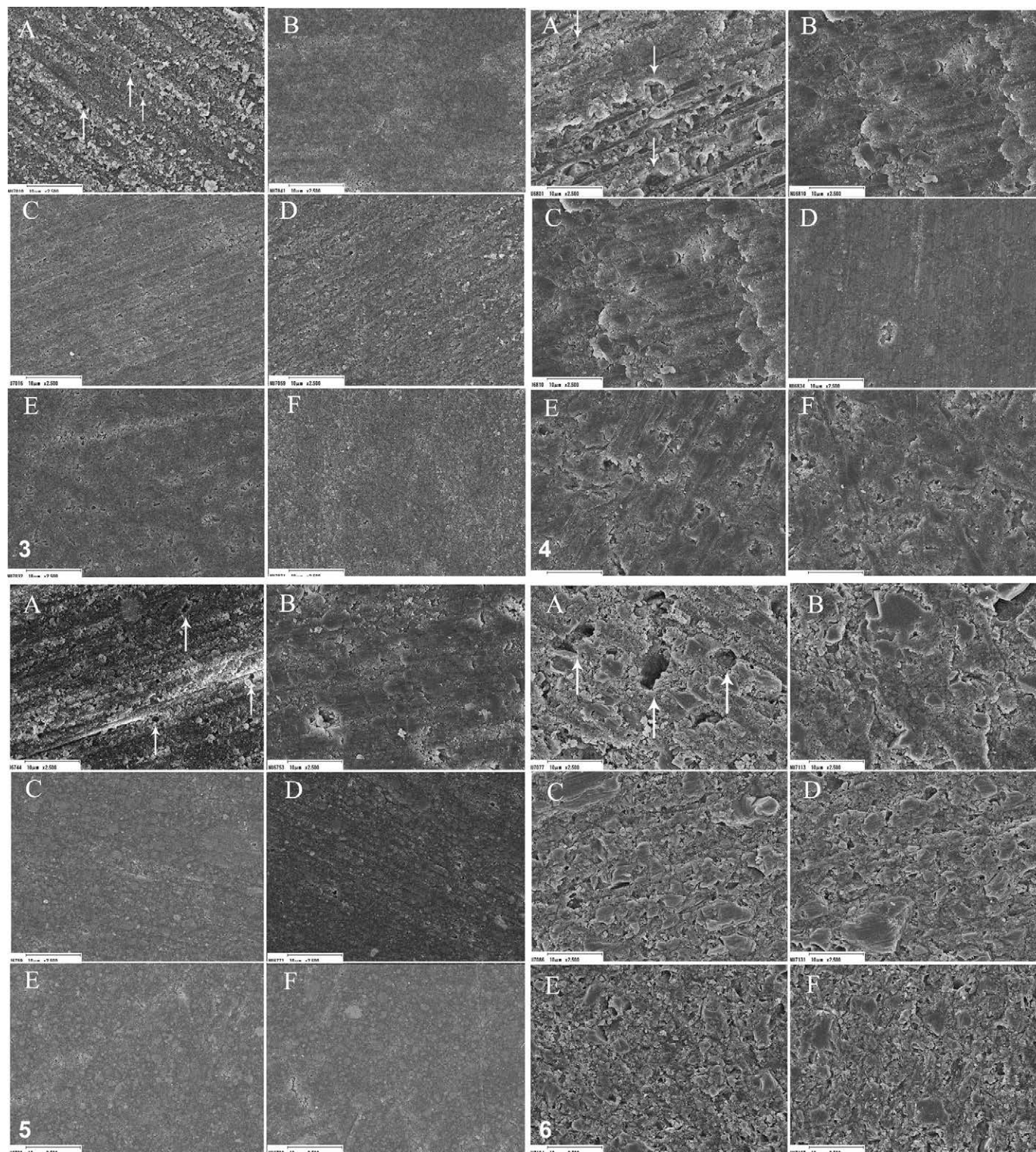


Figure 3. Representative SEM images of resin composite surfaces after different finishing and polishing procedures. (A): TB finished with FDB (2500 $\times$ ). (B): TB finished with CBB (2500 $\times$ ). (C): TB polished with CMP after finishing with FDB (2500 $\times$ ). (D): TB polished with CMP after finishing with CBB (2500 $\times$ ). (E): TB polished with SSD after finishing with FDB (2500 $\times$ ). (F): TB polished with SSD after finishing with CBB (2500 $\times$ ). The arrows indicate evidence of plucked-out filler.

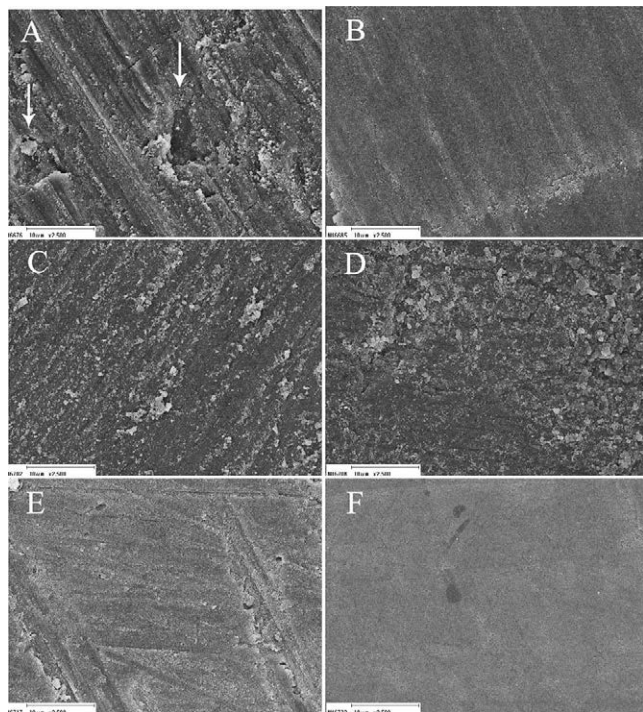


Figure 7. Representative SEM images of resin composite surfaces after different finishing and polishing procedures. (A): EQ finished with FDB (2500 $\times$ ). (B): EQ finished with SF CBB (2500 $\times$ ). (C): EQ polished with CMP after finishing with FDB (2500 $\times$ ). (D): EQ polished with CMP after finishing with CBB (2500 $\times$ ). (E): EQ polished with SSD after finishing with FDB (2500 $\times$ ). (F): EQ polished with SSD after finishing with CBB (2500 $\times$ ). The arrows indicate evidence of plucked-out filler.

for TB and FB. In particular, their baseline  $\gamma_S^P$  was significantly lower than that of the other resin composites. The polar force ( $\gamma_S^P$ ) is thought to be related to electric and metallic interactions, in addition to dipolar interactions.<sup>19</sup>

In the finished groups, AP, EQ, and FB showed significantly higher  $\gamma_S$  and  $\gamma_S^h$  values with CBB than with FDB. This phenomenon might be explained by the different finishing mechanisms of the burs used. Although AP, FB, and EQ exhibited many large

voids after finishing with SF, finishing with CBB resulted in relatively flat and even surfaces, as discussed earlier. Hence, the cutting of the filler particles, leaving parts of the filler particles exposed at the surface, might have contributed to the higher  $\gamma_S$  and  $\gamma_S^h$  values. The hydrogen-bonding force ( $\gamma_S^h$ ) represents water and hydroxyl components of the resin composite surface.<sup>19</sup> In the polished groups, most resin composites showed higher  $\gamma_S$  values with SSD compared with CMP, regardless of the finishing method. This suggests that different polishing methods result in different surface chemistries. Specifically, most resin composites polished with CMP had lower  $\gamma_S^P$  and  $\gamma_S^h$  values than those of composites polished with SSD. In addition to the different polishing mechanisms of CMP and SSD, the  $\gamma_S$  values may have been influenced by remnants of silicone from CMP, making the surface more hydrophobic.

The use of inappropriate finishing and polishing procedures on resin composite restorations compromises the long-term clinical performance of the restorations.<sup>17,32,36</sup> Finishing is necessary to produce a precise anatomical form, good contours, appropriate occlusion, and healthy embrasure form.<sup>12-14</sup> Finishing also eliminates the upper resin-rich and oxygen-inhibited layer of the resin composite.<sup>38</sup> Polishing after finishing is critical in order to obtain a smooth surface with gloss and to modify the surface to resist degradation.<sup>36,37,39</sup> The results of this study indicate that the size and shape of the filler of a resin composite can influence its surface roughness, and the type of resin matrix may be the dominant factor in determining SFE. Therefore, the combination of finishing method, polishing method, and type of resin composite should be carefully considered for clinical applications. In addition, the results of this study are consistent with findings of previous reports on conventional resin composites,

Figure 4. Representative SEM images of resin composite surfaces after different finishing and polishing procedures. (A): FB finished with FDB (2500 $\times$ ). (B): FB finished with CBB (2500 $\times$ ). (C): FB polished with CMP after finishing with FDB (2500 $\times$ ). (D): FB polished with CMP after finishing with CBB (2500 $\times$ ). (E): FB polished with SSD after finishing with FDB (2500 $\times$ ). (F): FB polished with SSD after finishing with CBB (2500 $\times$ ). The arrows indicate evidence of plucked-out filler.

Figure 5. Representative SEM images of resin composite surfaces after different finishing and polishing procedures. (A): FF finished with FDB (2500 $\times$ ). (B): FF finished with CBB (2500 $\times$ ). (C): FF polished with CMP after finishing with FDB (2500 $\times$ ). (D): FF polished with CMP after finishing with CBB (2500 $\times$ ). (E): FF polished with SSD after finishing with FDB (2500 $\times$ ). (F): FF polished with SSD after finishing with CBB (2500 $\times$ ). The arrows indicate evidence of plucked-out filler.

Figure 6. Representative SEM images of resin composite surfaces after different finishing and polishing procedures. (A): AP finished with FDB (2500 $\times$ ). (B): AP finished with CBB (2500 $\times$ ). (C): AP polished with CMP after finishing with FDB (2500 $\times$ ). (D): AP polished with CMP after finishing with CBB (2500 $\times$ ). (E): AP polished with SSD after finishing with FDB (2500 $\times$ ). (F): AP polished with SSD after finishing with CBB (2500 $\times$ ). The arrows indicate evidence of plucked-out filler.

suggesting that bulk-fill and conventional composites respond similarly to finishing and polishing.

On the other hand, recent studies have indicated that biofilm formation is more influenced by material characteristics and composition than surface roughness.<sup>40,41</sup> In particular, filler size, filler shape, distribution, and matrix composition are critical factors for biofilm formation.<sup>42</sup> Although matrix components are not influenced by finishing and polishing procedures, it is probable that different finishing and polishing procedures may have an effect on not only on surface topography but also on the surface chemistry of resin composite restorations.

While SFE reflects some of the surface characteristics of materials, further research is needed to investigate the influence of different finishing and polishing methods and types of resin composites on the surface characteristics of resin composite using different methodologies.

## CONCLUSION

This study demonstrated that the finishing and polishing methods, along with the type of resin composite, significantly affect the surface properties of the composite in terms of both *Ra* and SFE. Finishing and polishing significantly increased the SFEs of the high-viscosity bulk-fill resin composites. Finishing with FDB resulted in significantly higher *Ra* values than finishing with CBB, regardless of the type of resin composite. However, most resin composites finished with FDB showed lower SFEs than those finished with CBB. Polishing with SSD resulted in significantly lower *Ra* values than polishing with CMP, regardless of the finishing method. On the other hand, most resin composites polished with SSD showed higher  $\gamma_s$  than those polished with CMP. SEM observation revealed that the shapes, sizes, and distributions of inorganic fillers depended on the composite material. Regardless of the type of resin composite, polishing with SSD after finishing with CBB resulted in a smoother surface than other combinations of finishing and polishing.

## Acknowledgements

This work was supported in part by a Grant-in-Aid for Scientific Research (C) (16K11565 and 17K11716) and a Grant-in-Aid for Young Scientists (B) (17K17141, and 17K17142) from the Japan Society for the Promotion of Science. This project was also supported in part by the Sato Fund and by a grant from the Dental Research Center of the Nihon University School of Dentistry, Japan.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 30 November 2018)

## REFERENCES

1. Ferracane JL (2011) Resin composite-state of the art *Dental Materials* **27**(1) 29-38. <https://doi.org/10.1016/j.dental.2010.10.020>
2. Ilie N, Hilton TJ, Heintze SD, Hickel R, Watts DC, Silikas N, Stansbury JW, Cadenaro M, & Ferracane JL (2017) Academy of Dental Materials guidance—Resin composites: Part I—Mechanical properties *Dental Materials* **33**(8) 880-894. <https://doi.org/10.1016/j.dental.2017.04.013>
3. Shibasaki S, Takamizawa T, Nojiri K, Imai A, Tsujimoto A, Endo H, Suzuki S, Suda S, Barkmeier WW, Latta MA, & Miyazaki M (2017) Polymerization behavior and mechanical properties of high-viscosity bulk fill and low shrinkage resin composites *Operative Dentistry* **42**(6) E177-187. <https://doi.org/10.2341/16-385-L>
4. El-Damanhoury HM & Platt JA (2014) Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites *Operative Dentistry* **39**(4) 374-382. <https://doi.org/10.2341/13-017-L>
5. Al Sunbul H, Silikas N, & Watts DC (2016) Polymerization shrinkage kinetics and shrinkage-stress in dental resin-composites *Dental Materials* **32**(8) 998-1006. <https://doi.org/10.2341/13-017-L>
6. Ilie N, Keßler A, & Durner J (2013) Influence of various irradiation processes on the mechanical properties and polymerization kinetics of bulk-fill resin based composites *Journal of Dentistry* **41**(8) 695-702. <https://doi.org/10.1016/j.jdent.2013.05.008>
7. Jang JH, Park SH, & Hwang IN (2015) Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin *Operative Dentistry* **40**(2) 172-80. <https://doi.org/10.2341/13-307-L>
8. Li X, Pongprueksa P, Van Meerbeek B, & De Munck J (2015) Curing profile of bulk-fill resin-based composites *Journal of Dentistry* **43**(6) 664-672. <https://doi.org/10.1016/j.jdent.2015.01.002>
9. Moszner N, Fischer UK, Ganster B, Liska R, & Rheinberger V (2008) Benzoyl germanium derivatives as novel visible light photoinitiators for dental materials *Dental Materials* **24**(7) 901-907. <https://doi.org/10.1016/j.dental.2007.11.004>
10. Ilie N, Bucuta S, & Dreaenert M (2013) Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance *Operative Dentistry* **38**(6) 618-625. <https://doi.org/10.2341/12-395-L>
11. Kim RJY, Kim YJ, Choi NS, & Lee IB (2015) Polymerization shrinkage, modulus, and shrinkage stress related to tooth-restoration interfacial debonding in bulk-fill composite *Journal of Dentistry* **43**(4) 430-439. <https://doi.org/10.1016/j.jdent.2015.02.002>

12. Reis AF, Giannini M, Lovadino JR, & Ambrosano GM (2003) Effects of various finishing systems on the surface roughness and staining susceptibility of packable composite resins *Dental Materials* **19**(1) 12-18. [https://doi.org/10.1016/S0109-5641\(02\)00014-3](https://doi.org/10.1016/S0109-5641(02)00014-3)
13. Ikeda M, Matin K, Nikaido T, Foxton RM, & Tagami J (2007) Effect of surface characteristics on adherence of *S. mutans* biofilms to indirect resin composites *Dental Materials Journal* **26**(6) 915-923. <https://doi.org/10.4012/dmj.26.915>
14. Jefferies SR (2007) Abrasive finishing and polishing in restorative dentistry; a state-of-the-art review *Dental Clinics North America* **51**(2) 379-397. <https://doi.org/10.1016/j.cden.2006.12.002>
15. Gharechahi M, Moosavi H, Forghani M (2012) Effect of surface roughness and materials composition on biofilm formation *Journal of Biomaterials and Nanobiotechnology* **3**(4A) 541-546. <http://dx.doi.org/10.4236/jbnb.2012.324056>
16. Glauser S, Astasov-Frauenhoffer M, Müller JA, Fischer J, Walimo T, & Rohr N (2017) Bacterial colonization of resin composite cements: Influence of material composition and surface roughness *European Journal of Oral Science* **125**(4) 294-302. <https://doi.org/10.1111/eos.12355>
17. Rodrigues-Junior SA, Chemin P, Piaia PP, & Ferracane JL (2015) Surface roughness and gloss of actual composites as polished with different polishing systems *Operative Dentistry* **40**(4) 418-429. <https://doi.org/10.2341/14-014L>
18. Cazzaniga G, Ottobelli M, Ionescu A, & Garcia-Godoy F (2015) Surface properties of resin-based composite materials and biofilm formation: A review of the current literature *American Journal of Dentistry* **28**(6) 311-320.
19. Imai A, Takamizawa T, Sai K, Tsujimoto A, Nojiri K, Barkmeier WW, Latta MA, & Miyazaki M (2017) Influence of application method on surface free-energy and bond strength of universal adhesive systems to enamel *European Journal of Oral Science* **125**(5) 385-395. <https://doi.org/10.1111/eos.12361>
20. Tsujimoto A, Barkmeier WW, Takamizawa T, Latta MA, & Miyazaki M (2016) Influence of the oxygen-inhibited layer on bonding performance of dental adhesive systems: Surface free energy perspective *Journal of Adhesive Dentistry* **18**(1) 51-58. <https://doi.org/10.3290/jad.a35515>
21. Ouchi H, Tsujimoto A, Nojiri K, Hirai K, Takamizawa T, Barkmeier WW, Latta MA, & Miyazaki M (2017) Effect of oxygen inhibition layer of universal adhesives on enamel bond fatigue durability and interfacial characteristics with different etching modes *Operative Dentistry* **42**(6) 636-645. <https://doi.org/10.2341/16-255-L>
22. Price RBT, Rugeberg FA, Labrie D, & Felix CM (2010) Irradiance uniformity and distribution from dental light curing units *Journal of Esthetic and Restorative Dentistry* **22**(2) 86-103. <https://doi.org/10.1111/j.1708-8240.2010.00318.x>
23. Michaud PL, Price RBT, Labrie D, Rueggeberg FA, & Sullivan B (2014) Localised irradiance distribution found in dental light curing units. *Journal of Dentistry* **42**(2) 129-139. <https://doi.org/10.1016/j.jdent.2013.11.014>. Epub 2013 Nov 25.
24. Al-Ahdal K, Ilie N, Silikas N, & Watts DC (2015) Polymerization kinetics and impact of post polymerization on the degree of conversion of bulk-fill resin-composite at clinically relevant depth *Dental Materials* **31**(10) 1207-1213. <https://doi.org/10.1016/j.dental.2015.07.004>. Epub 2015 Aug 19.
25. Hata T, Kitazato Y, & Saito T (1987) Estimation of the surface energy of polymer solids *Journal of Adhesion* **21**(3-4) 177-194.
26. Ferracane JL (2006) Hygroscopic and hydrolytic effects in dental polymer networks *Dental Materials* **22**(3) 211-22. <https://doi.org/10.1016/j.dental.2005.05.005>
27. Delaviz Y, Finer Y, & Santerre JP (2014) Biodegradation of resin composites and adhesives by oral bacteria and saliva: A rationale for new material designs that consider the clinical environment and treatment challenges *Dental Materials* **30**(1) 16-32. <https://doi.org/10.1016/j.dental.2013.08.201>
28. Yokokawa M, Rikuta A, Tsujimoto A, Tsuchiya K, Shibasaki S, Matsuyoshi S, & Miyazaki M (2015) Influence methyl mercaptan on the repair bond strength of composites fabricated using self-etch adhesives *European Journal of Oral Science* **123**(1) 46-52. <https://doi.org/10.1111/eos.12164>
29. Carnerio PMA, Ramos TM, de Azevedo CS, de Lima E, de Souza SHJ, Turbino KL, Cesar PF, & Matos AB (2016) Influence of finishing and polishing techniques and abrasion on transmittance and roughness of composite resins *Operative Dentistry* **41**(6) 634-41. <https://doi.org/10.2341/15-281-L>
30. Dutra DAM, Pereira GKR, Kantorski KZ, Valandro LF, & Zanatta FB (2018) Does finishing and polishing of restorative materials affect bacterial adhesion and biofilm formation? A systematic review *Operative Dentistry* **43**(1) 37-52. <https://doi.org/10.2341/17-073-L>
31. Daud A, Gray G, Lynch CD, Wilson NHF, & Blum IR (2018) A randomised controlled study on the use of finishing and polishing systems on different resin composites using 3D contact optical profilometry and scanning electron microscopy *Journal of Dentistry* **71**(Apr) 25-30. <https://doi.org/10.1016/j.jdent.2018.01.008>
32. Jung M (1997) Surface roughness and cutting efficacy of composite finishing instruments *Operative Dentistry* **22**(3) 98-104.
33. Roeder LB, Tate WH, & Powers JM (2000) Effect of finishing and polishing procedures on the surface roughness of packable composites *Operative Dentistry* **25**(6) 534-543.
34. Sumino N, Tsubota K, Takamizawa T, Shiratsuchi K, Miyazaki M, & Latta MA (2013) Comparison of the wear and flexural characteristics of flowable resin composites for posterior lesions *Acta Odontologica Scandinavica* **71**(3-4) 820-827. <https://doi.org/10.3109/00016357.2012.734405>
35. Skrtic D, Antonucci JM, & Liu DW (2006) Ethoxylated bisphenol dimetacrylate-based amorphous calcium phosphate composites *Acta Biomaterialia* **2**(1) 85-94. <https://doi.org/10.1016/j.actbio.2005.10.004>

36. Waheeb N, Silikas N, & Watts D (2012) Initial polishing time affects gloss retention in resin composites *American Journal of Dentistry* **25**(5) 303-306.
37. Hosoya Y, Shiraishi T, Odatsu T, Nagafuji J, Kotaku M, Miyazaki M, & Powers JM (2011) Effects of polishing on surface roughness, gloss and color of resin composites *Journal of Oral Science* **53**(3) 283-291. <https://doi.org/10.2334/josnugd.53.283>
38. Patel SB, Gordan VV, Barrett AA, & Shen C (2004) The effect of surface finishing and storage solution on color stability of resin-based composites *Journal of the American Dental Association* **135**(5) 587-594. <https://doi.org/10.14219/jada.archive.2004.0246>
39. Moda MD, Godas AGL, Fernandes JC, Suzuki TYU, Guedes APA, Briso ALF, Bedran-Russo AK, & Dos Santos PH (2018) Comparison of different polishing methods on the surface roughness of microhybrid, microfill, and nanofill composite resin *Journal of Investigative and Clinical Dentistry* **9**(1) 1-9. <https://doi.org/10.1111/jicd.12287>. Epub 2017 Aug 1
40. Song F, Koo H, & Ren D (2015) Effects of material properties on bacterial adhesion and biofilm formation *Journal of Dental Research* **94**(8) 1027-1034. <https://doi.org/10.1177/0022034515587690jdr.sagepub.com>
41. Cazzaniga G, Ottobelli M, Ionescu AC, Paolone G, Gherlone E, Ferracane JL, & Brabilla E (2017) In vitro biofilm formation on resin-based composites after different finishing and polishing procedures *Journal of Dentistry* **67** 43-52. <https://doi.org/10.1016/j.jdent.2017.07.012>Get rights and content
42. Pereira CA, Eskelson E, Cavalli V, Liporoni PC, Jorge AO, & do Rego MA (2011) Streptococcus mutans biofilm adhesion on composite resin surfaces after different finishing and polishing techniques *Operative Dentistry* **36**(3) 311-317. <https://doi.org/10.2341/10-285-L>

# OPERATIVE DENTISTRY

---

## CORPORATE SPONSORS

These Dental Manufacturers have joined Operative Dentistry in our commitment to publish quality dental literature in a timely manner. We thank them for their support.



# OPERATIVE DENTISTRY

Volume 45 / Number 2  
March/April 2020

www.jopdent.org  
113–228

## EDITORIAL

- 113 Conservative Tooth Preparation, Minimal Intervention, and/or Narrow-parallel Preparations: A Narrative  
*JW Osborne*

## CLINICAL TECHNIQUE/CASE REPORT

- 117 Multidisciplinary Management and Pulp Vitality Preservation of a Tooth With Extensive Iatrogenic Furcal Root Perforation and Biologic Width Violation  
*D Angerame • M De Biasi • V Franco • L Generali*

## CLINICAL RESEARCH

- 123 24-Month Clinical Evaluation of Different Bulk-Fill Restorative Resins in Class II Restorations  
*T Guney • AR Yazici*
- 134 Perceptibility and Acceptability of Surface Gloss Variations in Dentistry  
*RS Rocha • TC Fagundes • TMF Caneppele • E Bresciani*

## LABORATORY RESEARCH

- 143 Volumetric Cuspal Deflection of Premolars Restored With Different Paste-like Bulk-fill Resin Composites Evaluated by Microcomputed Tomography  
*G Demirel • IH Baltacioglu • ME Kolsuz • M Ocak • B Bilecenoglu • K Orhan*
- 151 Clinical Evaluation of a Chairside Amalgam Separator to Meet Environmental Protection Agency Dental Wastewater Regulatory Compliance  
*DS Olivera • MT Morgan • SN Tewolde • EC Botts • FP Horvath • NJ Hamlin*
- 163 Influence of the Fabrication Technique on the Marginal and Internal Adaptation of Ceramic Onlays  
*CF Neto • GC Santos • MJMC Santos*
- 173 Dynamic Viscoelastic Characterization of Bulk-fill Resin-based Composites and Their Conventional Counterparts  
*AU Yap • AH Eweis • NA Yahya*
- 183 Influence of Application Time on Dentin Bond Performance in Different Etching Modes of Universal Adhesives  
*T Saito • T Takamizawa • R Ishii • A Tsujimoto • E Hirokane • WW Barkmeier • MA Latta • M Miyazaki*
- 196 The Effects of Aluminablasting on Bond Durability Between Universal Adhesives and Tooth Substrate  
*H Ouchi • T Takamizawa • K Tsubota • A Tsujimoto • A Imai • WW Barkmeier • MA Latta • M Miyazaki*
- 209 Effect of pH Cycling Followed by Simulated Toothbrushing on the Surface Roughness and Bacterial Adhesion of Bulk-fill Composite Resins  
*DC Somacal • FB Manfro • MSG Monteiro • SD Oliveira • HR Bittencourt • GA Borges • AM Spohr*
- 219 Influence of CAD/CAM Fabrication and Sintering Procedures on the Fracture Load of Full-Contour Monolithic Zirconia Crowns as a Function of Material Thickness  
*M Zimmermann • A Ender • A Mehl*

## DEPARTMENTS

- 227 Online Only Articles

## ONLINE ONLY ARTICLES

- E43 Fracture Resistance and Marginal Adaptation of Capped and Uncapped Bulk-fill Resin-based Materials  
*HN Al-Nahedh • Z Alawami*
- E57 Efficacy of a Light-cured Tetracaine-based Anesthetic Gel for Rubber Dam Clamp Placement: A Triple-blind Randomized Clinical Trial  
*LM Wambier • CC Gonzaga • ACR Chibinski • DS Wambier • PV Farago • AD Loguercio • A Reis*
- E66 Effect of Analgesic Drugs on Tooth Sensitivity Induced by In-office Dental Bleaching: A Systematic Review and Meta-analysis  
*RTF Costa • SLD Moraes • CAA Lemos • JR SoutoMaior • BC do E Vasconcelos • EP Pellizzer*
- E77 Efficacy and Adverse Effects of Whitening Dentifrices Compared With Other Products: A Systematic Review and Meta-analysis  
*A Devila • R Lasta • L Zanella • MA Dall Agnol • SA Rodrigues-Junior*
- E91 Effects of Finishing and Polishing Methods on the Surface Roughness and Surface Free Energy of Bulk-fill Resin Composites  
*R Ishii • T Takamizawa • A Tsujimoto • S Suzuki • A Imai • WW Barkmeier • MA Latta • M Miyazaki*