

OPERATIVE DENTISTRY

Sep/Oct 2020

Volume 45

Number 5

457–568



Downloaded from <https://prime-pdf-watermark.prime-prod.pubfactory.com/> at 2025-08-31 via free access

OPERATIVE
DENTISTRY

OPERATIVE DENTISTRY

Volume 45/Number 5
September/October 2020

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, or by contacting our subscription manager via email at 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, 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 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://jopdent.com/administration/policies-and-guidelines-of-operative-dentistry>

Permissions

For permission to reproduce material from Operative Dentistry please apply to Operative Dentistry at the Editorial Office address or online at www.jopdent.org.

Online Access

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

<https://meridian.allenpress.com/operative-dentistry>

Editorial Board

Reviewer names available at: <https://jopdent.com/administration/reviewer-recognition-2/>. We thank all our reviewers for their time and dedication to Operative Dentistry.

On The Cover

"Mounted Mongolian Eagle Hunter," Altai Valley, Mongolia. Photo provided by Lawrence Blank of Corrales, New Mexico, USA. Photo taken with a Canon 6D, 88mm f/5.6 1/1000 sec. ISO-200. © 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://jopdent.com/cover-photo-submission/>

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 E Eckert



Adapted Three-step Restorative Technique: Recovering Dental Substrate Compromised by Complex Erosive Wear in a Young Patient

CA Villavicencio-Espinoza • MC Giacomini • MH Narimatsu
AC Magalhães • MT Atta • L Wang

Clinical Relevance

This article presents the dental restoration of a young female patient complaining of erosive dental wear using a three-step restorative technique, an alternative approach with some novel adjustments.

SUMMARY

For successful tooth wear treatment, determining the etiological systemic and local factors is the main priority before deciding on effective and long-term preventive and/or therapeutic restorative approaches. In addition to professional intervention, achieving optimal outcomes requires patients to control their

diet and/or gastric issues, thus minimizing the wear process. However, continuous wear constitutes the most challenging scenario, mainly when it affects young patients' dentitions. This article describes the dental restoration of posterior teeth with reestablishment of occlusal vertical dimension before treating the anterior teeth, while educating the patient and providing medical monitoring. The three-step restorative technique seems to be proper-

Carlos Andres Villavicencio Espinoza, DDS, MS, PhD, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Brazil

Marina Ciccone Giacomini, DDS, MS, PhD, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Brazil

Mayara Hana Narimatsu, DDS, MS, PhD, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Brazil

Ana Carolina Magalhães, DDS, MS, PhD, associate professor, Department of Biological Sciences, Bauru School of Dentistry, University of São Paulo, Brazil

Maria Teresa Atta, DDS, MS, PhD, associate professor, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Brazil

*Linda Wang, DDS, MS, PhD, professor, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Brazil

*Corresponding author: Alameda Octávio Pinheiro Brisolla, 9-75, 17012-901, Bauru-SP, Brazil; e-mail: wang.linda@usp.br

<https://doi.org/10.2341/18-204-S>

ly applicable in cases of significant dental compromise due mainly to erosive wear and is based on direct procedures, which can assure a reliable and feasible approach.

INTRODUCTION

Recently, significant attention has gone toward addressing the prevention and/or treatment of events that cause tooth wear.¹⁻⁵ Extrinsic and intrinsic etiologic factors, such as unbalanced diet/unhealthy lifestyle or gastroesophageal reflux disease (GERD), have resulted in clinical oral challenges.⁴⁻⁹ They are thought to contribute to erosive tooth wear, a multifactorial condition characterized by the irreversible loss of tooth substance by acid exposure not involving bacteria.^{3,5,6,10}

In 2018, Resende and others⁵ proposed the term “biocorrosion” to define erosive lesions; however, since mechanical wear is also involved, the combination of chemical, biochemical, and mechanical processes still justifies the use of the term “erosion” to describe this complete phenomenon. In this context, frequent acid exposure allows for the partial demineralization of enamel or dentin, causing softening of the superficial surface, which is accelerated through the combined effect of the erosive and mechanical wear (abrasion and attrition) of the tooth surface.² Therefore, the severity and progression of dental erosion depend on the combination of these factors and result in erosive tooth wear, leading to surface loss that can be clinically detected.

The enamel surface is involved in the initial stage of erosive lesions. If this process is not stopped, widespread thinning of the enamel layer is observed, reaching the dentin and likely affecting the buccal, palatal, and occlusal dental surfaces. More complicated compromises, including hypersensitivity and occlusal disorders, can occur.^{3,11}

The choice of treatment depends on many factors, including location of the lesions, degree of involvement of the dental structure, and the age of the patient.^{5,6,12-14} However, gathering an accurate medical history to determine the main etiological reason for dental erosion and manifesting its control must be the first steps in successful treatment. When an accurate medical history is not possible, which occurs when treating patients with special needs, the idiopathic etiology is considered, since no specific monitoring can be performed. This scenario creates uncertainty in terms of treatment and its longevity.

When considering more extensive cases, prior planning and strategies may be essential to avoid sub-



Figure 1. Compromised dental wear of a 16-year-old patient as a consequence of dental erosion associated with bruxism. Intraoral view highlighting incisal compromise and loss of shiny aspect.

Figure 2. Lateral view indicating intense wear and loss of vertical dimension of the left side.

jecting the patient to compromised treatment.^{5,12-15} Additionally, these cases require special attention and the involvement of the patient. Direct procedures are always the preferable approach, as they offer a more conservative approach. However, when multiple teeth are affected, rationalization of the management is essential for successful treatment. One interesting strategy was introduced by Vailati and Belser^{14,15}; their technique is labeled the “three-step technique.” It was first described to be indicated for guiding the treatment of significant erosive lesions. This technique suggests combining laboratory (L) and clinical (C) steps into three phases: step 1 = esthetics (L, maxillary buccal wax up; C, assessment of the occlusal plane); step 2 = posterior support (L, posterior occlusal wax up; C, creation of posterior occlusion at an increased vertical dimension of occlusion [VDO]); and step 3 = anterior guidance (L, maxillary anterior palatal onlays; C, reestablishment of final anterior guidance).

The aim of this article is to report on the application of this full-mouth adhesive rehabilitation technique, which involves the use of composite resin to reestablish posterior teeth that have been compromised by erosion and that have a reduced vertical dimension. This article will then discuss the recovery of the extensively worn anterior dentition (palatal and buccal surfaces) in a young female patient presenting with constant dental erosion who had been monitored for four years.

DESCRIPTION OF TECHNIQUE

Medical and Clinical Reports

A 16-year-old female patient sought treatment at the Bauru School of Dentistry, complaining of severe esthetic problems. She reported unhappiness about her smile, especially due to the lack of esthetics of her anterior teeth (Figure 1), revealing anxiety and a strong desire to recover an esthetic smile.

Possible dietary causes of the erosion were investigated, since the patient presented with wear on the occlusal and buccal surfaces, commonly attributed to extrinsic factors. Even though the patient did not report high acidic consumption, the clinical evaluation revealed that the overall oral conditions made the patient more prone to erosive wear. Therefore, the next step was based on the analysis of tests regarding the patient's flow and consistency of saliva, which showed apparent normal parameters: unstimulated salivary flow: 0.25 mL/min; stimulated salivary flow: 1 mL/min; and an adequate buffer capacity. This assessment suggested no clinical compromise of the salivary protective properties. Additionally, the patient reported she was taking medication to maintain her thyroid hormone levels (Eutirox 88), which were abnormal due to previously diagnosed hypothyroidism. Eutirox is an international brand of levothyroxine, a synthetic form of the human hormone thyroxine.

The intrinsic causes of erosive wear were also investigated. The patient's parents reported that she did not present any signs suggesting bulimia or anorexia. For a more specific analysis, the patient was referred to a gastroenterologist for an evaluation of the existence of GERD. After some medical tests, including endoscopy and hiatus analyses, a limited esophageal sphincter contraction was observed, which was responsible for causing the reflux of small amounts of stomach acid into the oral environment. Additionally, the patient reported that she used to have a nap after lunch and usually turned to the left side, which likely explained the presence of more lesions on this side when compared to the right side (Figure 2). When analyzing her posterior teeth, occlusal and buccal enamel wear was evident. Other relevant data led to a diagnosis of bruxism, which, when combined with erosion accelerating the wear, contributed to the severity of the effects of bruxism. Excessive attrition due to bruxism, if not controlled, can require significant indirect and invasive approaches, especially when the periodontium is compromised.¹⁶⁻¹⁸

More detailed oral examination confirmed overall extensive enamel loss, decreased dimension of occlusion, and generalized dental sensitivity. A clinical evaluation and medical questionnaire confirmed increasing dental sensitivity, which was causing episodes of discomfort during drinking and eating. The patient was first referred for temporomandibular joint (TMJ) analysis, with no issues identified. This is essentially the first step, as the technique based on Vailati and Belser^{14,15} is not

recommended for patients who present with TMJ disorders.

Based on this lack of a TMJ diagnosis, the restorative treatment was started. According to the Vailati and Belser technique,^{14,15} it is necessary to initially determine the optimal position of the maxillary incisal edges of the planned final restorations to refer to the correct distribution of interocclusal space gained by the increase in VDO. Limitations will occur in cases involving more severe erosion/wear, which was not the case for the current patient. In situations involving severe wear, adjustments to this technique would be necessary.

Initial diagnosis evaluated the amount of dental wear, and the relationship of the mandibular/maxillary arches was analyzed. As a result, the thickness of restorative material on the occlusal surfaces varied, especially when considering that prior unsatisfactory restorations required replacement. In the present case, less than 1 mm was necessary to return the appropriate VDO.

The diagnosis was first performed for the anterior teeth (using a mockup) and then for the posterior teeth. However, the restorative sequence was switched: the posterior teeth were "arbitrarily" restored, taking the maxillary smile line into account and following the Curve of Spee. Restoring the posterior teeth was recommended to establish occlusal stability. As indicated by the authors, the composite resins can serve as "long-term provisional treatment" for adult patients. These temporary restorations can be helpful to diagnosis as well. Composite resin is more conservative than the use of ceramics and is also repairable. For young patients with the possibility of more frequent appointments, as in the current case, the selection of this material seemed to be appropriate.

When taking into account the likely etiological factors and overall clinical situation, all dental restorations were carefully planned. Even though the patient did not indicate that she consumed acidic food and beverages, the clinical characteristics suggested that they might be involved. As the patient was still an adolescent, the involvement of continuous facial development was considered. Photographs of the patient's face and smile from different perspectives and proportional measurements were taken for analysis, as a harmonious look depends on the balance between the face and smile.

A complementary smile analysis was performed by observing the position of the teeth in relation to the lips and gums. The patient showed discrete upper

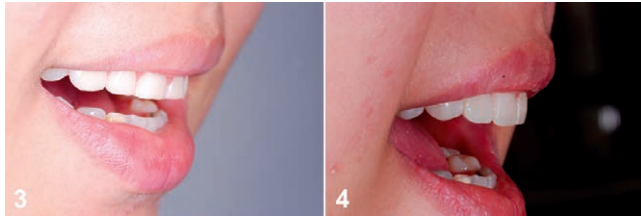


Figure 3. Lateral perspective revealing disproportionate measurements.

Figure 4. Lateral view of the bis-acrylic mockup performed for the evaluation of esthetic patterns from the face and smile of the patient.

teeth when smiling as a result of the compromised wear of the incisal faces of the anterior teeth (Figure 3). Therefore, her flat esthetic frontal plane suggested an older expression that did not correspond to the patient's actual age.

Dental prophylaxis was performed, as the patient was limited in cooperating with oral hygiene. Additionally, her gums presented signs related to an inflammatory process in the absence of plaque, indicating that her hormonal condition might be related to her condition as well.

The facial analysis provided evidence of a compromised vertical dimension. Also, TMJ was analyzed and it was not involved as already reported. Even though the patient was looking for a pleasant smile, recovering the functional characteristics of the posterior teeth was the first step in treating the patient. This was explained to the patient and her parents, who agreed with the presented treatment plan. For the anterior teeth, a combination of a full-mouth adhesive rehabilitation and the three-step technique was performed.

Restorative Procedures

Based on the restorative needs, treatment planning involved the application of the three-step restorative technique.¹²⁻¹⁵ As the patient was only 16 years old, composite resin was the selected material for reestablishing the posterior teeth. Additionally, the presence of enamel margins supported this choice, when considering effective bonding along the enamel margins and appropriate marginal sealing.^{1,4}

Impressions of both the maxillary and mandibular arches were made using addition silicone (Empress XT, 3M ESPE, St Paul, MN, USA) to obtain casts for completing the clinical analysis. A wax-up was performed on the maxillary anterior teeth for esthetic assessments and was based on the Digital Smile Design (DSD).^{19,20} The maxillary anterior wax-up served as the initial parameter for the

creation of the bis-acrylic resin mockups for the unprepared anterior teeth (Figure 4). The mockup also allowed for the preview of the static and dynamic aspects of future restorations.

After this first step, the posterior teeth were then diagnostically waxed-up. The initial analysis was performed for anterior teeth (using mockup) and then the posterior teeth in the treatment modality utilized in the current case study. However, the recommended sequence was switched. Therefore, the same casts were used to obtain wax-ups for the posterior teeth. This strategy was relevant for anticipating the height of the composite resin to be used to recover the vertical dimension of occlusion.

Based on the three-step technique, the wax-ups were used to replicate the anatomic characteristics of posterior teeth, allowing for the manufacture of a transparent resin matrix for fabricating and inserting the composite restorations for the posterior teeth. However, Vailati and Belser¹³ originally recommended the use of a translucent silicone. In the present case, a clear acetate matrix (Cristal-PET-G-0.75, Bioart, São Carlos, SP, Brazil) was used to restore the posterior teeth, which offers the advantage of allowing the joint insertion of the metal matrix between the teeth and allows for multiple restorations to be performed simultaneously (Figure 5).

The clear plastic matrix was cut along the equatorial line. After inserting the material and placing the matrix, the excessive restorative material was removed. The matrix position was checked based on the buccal and lingual equatorial lines of all involved teeth to ensure its correct position. It was essential to ensure that this matrix was in position before inserting the composite resin since the presence of any gap was considered unacceptable.

The surface of the lower first premolar and first molar were acid-etched with 37% phosphoric acid for 30 and 15 seconds for enamel and dentin, respectively. After washing and drying with absorbent paper, Adper ScotchBond Multipurpose primer and bond (3M ESPE) were applied according to the manufacturer's guidelines. Then the acetate matrix was filled with composite resin (Esthet X, Dentsply/Caulk, Milford, DE, USA). The teeth were separated by a customized metallic matrix and were light-activated for 40 seconds using an Optilight LD (Max Gnatus, Ribeirão Preto, SP, Brazil) at 600 mW/cm². The same procedure was used for the second premolar and the second molar. Figure 6 presents the final aspect immediately after the restorative

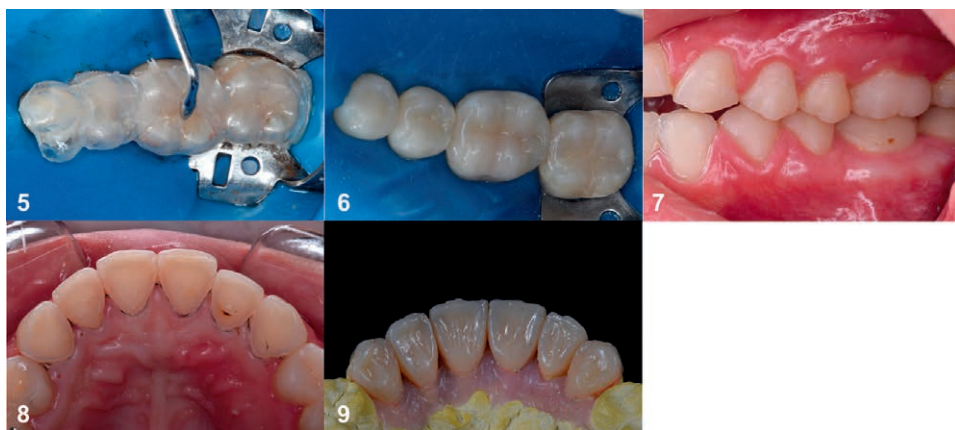


Figure 5. Prior placement of customized metallic matrix and use of an acetate matrix to restore these teeth using resin composite.

Figure 6. Immediate final view of the restorative procedures on posterior teeth.

Figure 7. Lateral view at the four-month follow-up of the left posterior restorations.

Figure 8. Use of retraction cord on the palatal surface of the maxillary anterior teeth prior to making an impression.

Figure 9. Final view of the palatal aspect of the composite resin veneers.

procedures were completed. After these restorations were cured, finishing diamonds and carbide burs were used to establish the buccal and lingual contours. This restorative sequence was performed in the mandibular right and left quadrants during the same session and in the maxillary right and left quadrants at the next session, recovering a mean height of 1 mm. After the completion of the restorative procedures a minor occlusal adjustment was made to stabilize the occlusion. The patient was monitored monthly to ensure occlusal stabilization for four months. After this time, the patient did not report any discomfort or pain (Figure 7).

Therefore, with a stable and functional posterior occlusion, the next step focused on the anterior teeth. A mixed technique involving indirect and direct composite resin restorations was executed for palatal and labial reconstruction of the maxillary anterior teeth (Figures 8 and 9). Using the wax-up of a dental model, indirect restorations were fabricated from composite resin involving the four maxillary incisors (Empress Direct, A3 dentin, A1 and A2 enamel, Ivoclar Vivadent, Schaan, Lichtenstein).

For tooth preparation, slight smoothing of the enamel was performed using a manual instrument, and no additional preparation was made, to include the lack of a bevel. A rubber dam was placed, and the restorations were carefully tested in their respective positions and individually luted to the palatal surfaces. The internal surface of the composite resin was sandblasted and cleaned using 37% phosphoric acid for 30 seconds, rinsed for the same amount of time, and dried with a stream of air. One coat of silane was applied (Monobond, Ivoclar Vivadent) and was gently evaporated using an air stream before

application of the adhesive system, Adper Scotchbond Multipurpose (3M ESPE), which was light-activated for 40 seconds. The dentin surface was cleaned and acid-etched for 15 seconds; Adper Scotchbond was used as already described. Then the dual-cure luting resin cement, All Cem Core, A2 shade (FGM, Joinville, SC, Brazil), was used to cement the restorations, positioning them on the palatal and incisal surfaces. This treatment involved all of the worn maxillary anterior teeth.

Excess cement was removed and minor occlusal/incisal adjustments were completed. In sequence, these restorations were well defined using a rubber dam. The external buccal surface of the anterior restorations was superficially removed using a 3131 diamond bur (KG Sorensen, Cotia, SP, Brazil), and a bevel was prepared (Figure 10). The buccal surfaces of all these restored teeth were removed using a 2135 bur. A 0.8-mm-thick palatal surface was maintained for each tooth. The removal of these buccal surfaces was performed to allow the necessary thickness for the layered resin technique to be utilized. Sandblasting was performed on the internal surfaces of the palatal restorations using aluminum oxide, followed by the application of the Scotchbond Multipurpose adhesive system; no silanization was necessary. These surfaces were restored using the incremental polychromatic composite layer direct technique, as shown in Figure 11 (Empress Direct, A2 dentin, A1 Enamel, Ivoclar Vivadent). All of these procedures were performed in the same day, which dispelled the need for any provisional restorations.

One week later, finishing and polishing were performed using multilaminated burs and abrasive rubber and polishing discs (TDV, Pomerode, SC,



Figure 10. Luted resin veneers with excess cement removed to allow adequate space for the direct incremental technique.

Figure 11. Direct restorative procedure for the palatal luted indirect surface using the stratified restorative technique.

Figure 12. View of the buccal surface of the maxillary anterior teeth, demonstrating the reestablishment of anatomic characteristics with a natural appearance and harmony with adjacent soft tissue.

Brazil). The final aspect of the anterior restorations reestablished the esthetic and functional aspects after one week (Figure 12). Finally, an occlusal splint guard was delivered to prevent wear and fracture of the restorations.

Follow-ups were made after one month, six months, and at three and four years. At the last two appointments continued erosion was detected on the cervical third of the buccal surfaces of the canines and premolars of the left side. On the right side, minor staining was detected. After this time, the patient was still very happy with the final result and did not complain about tooth sensitivity, although continuous erosive wear was still occur-

ring. The patient finally admitted that she consumed cola drinks at the three-year follow-up, which was in agreement with the clinical conditions. Most of the restorative procedures presented good performance, although the analyses indicated continuous progression of wear, especially on the cervical area of the left maxillary premolar, as shown in Figure 13a through d. After the four-year follow-up, some cervical and occlusal areas failed, clearly related to the continuous exposure to erosion, as shown in Figure 14a through d, even though the patient reported abstinence from consumption of cola drinks.

DISCUSSION

Erosion is considered a chronic event that is either a patient behavior-dependent issue or a result of a systemic disorder. When diagnosed, the patient is referred for evaluation and treatment, which does not necessarily yield the complete cessation of erosion. The frequency of appointments depends on severity.²¹ Clinical responses are very individual, as erosion is also considered to have a multifactorial etiology.

Clinically, correct diagnosis of the etiology of the lesions is essential for effective treatment. With moderate erosion, several preventive steps are indicated, such as a change in dietary or behavior patterns, the application of desensitizing fluoride therapy, and/or restorative treatment measures. In addition, restoration with glass ionomers or composite resin and composite or porcelain veneers has been examined in previous studies.^{3,5,6,11,21}

In the present case, a treatment plan was determined after making the patient and her parents aware of the situation and recommendations regarding reflux control and diet influence. All requested information was provided to the patient, and a referral was established with a physician to minimize the consequences of systemic oral erosion. As the patient was young, she drew special attention in terms of the desire to avoid continuous dental erosion. However, despite the risk of compromising the vertical dimension and causing sensitivity, the patient sought dental care to address her seemingly unpleasant smile. This type of scenario is becoming increasingly frequent and attests to the lack of dental health education.

As a result of the extensive treatment required for this patient, detailed treatment planning was required. The cooperation of the patient was essential for optimizing outcomes, especially when the main complaint (smile) was not primarily addressed.

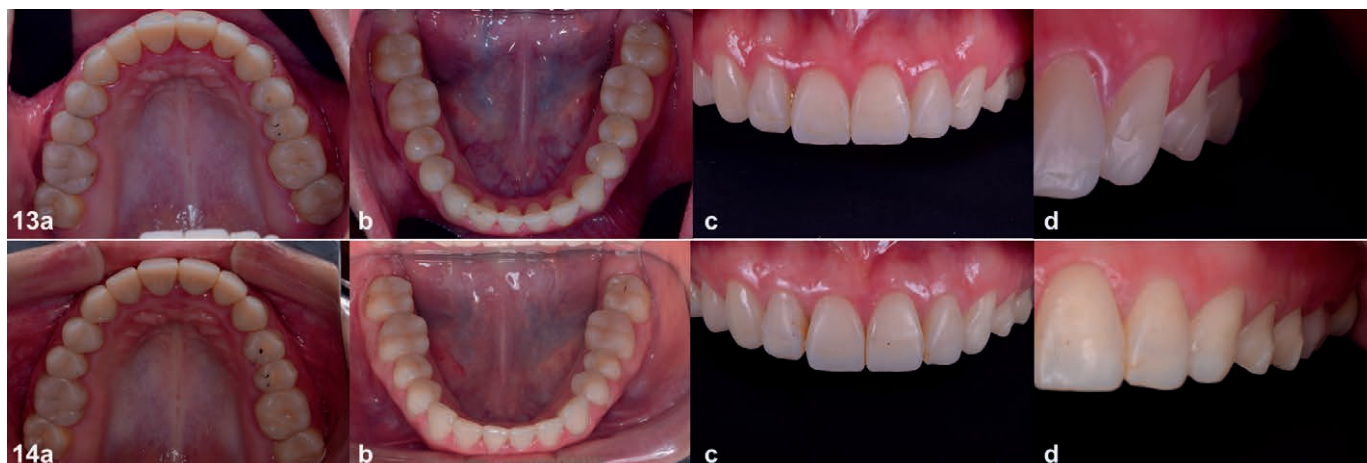


Figure 13. Case follow-up after three years. (a) Occlusal view of the maxillary teeth. (b) Occlusal view of the mandibular teeth. (c) View of the maxillary anterior teeth. (d) Lateral view of the left side of the maxillary premolars revealing continued wear of the cervical area of these teeth.

Figure 14. Case follow-up after four years. (a) Occlusal view of the maxillary teeth. (b) Occlusal view of the mandibular teeth. (c) View of the maxillary anterior teeth. (d) Lateral view of the left side of the maxillary premolars revealing continued wear of the cervical area of these teeth.

In complex cases, such as the present case, professionals argue frequently for the best treatment and the starting point, questioning “Starting from where?,” “What materials should be selected?,” “Is this a case for indirect restorative materials?,” etc. All of these points are of critical importance. When these factors are ignored, satisfactory restorative performance can be reduced. A more conservative treatment than what Vailati and Belser proposed in 2008¹⁴ consists of the combination of three laboratory and three clinical stages that are integrated and sequential, allowing for a logical and substantial approach. The main advantage of this technique is that it can help to achieve tooth rehabilitation by restoring the shape and function of a tooth with composite resin with minimal tooth preparation.

Coachman and Calamita¹⁹ described DSD, a technique that is helpful during the diagnostic waxing and mockup steps for achieving more predictability and an esthetically pleasing outcome as it systematically analyzes the relation between the patient’s lips, smile, and face. At first this analysis was performed solely on the buccal surfaces of teeth 5-12. After the mockup, however, diagnostic waxing in the posterior areas followed the original technique of Vailati and Carciofo.¹² An articulator was used to monitor the reestablishment of the VDO to recover the natural anatomy of the posterior teeth. At this stage, the final diagnostic waxing was completed, and composite resin was used to restore the posterior teeth to an adequate function.

This three-step technique reproduces the shape and position of the diagnostic wax using transparent

silicone.¹⁵ The current authors decided to use a clear plastic matrix vacuum molded to a waxed model. This is a rigid material that does not deform easily. The acetate matrix facilitates the joint insertion of a metal matrix between the teeth and allows multiple restorations to be performed simultaneously. On the other hand, when using a transparent silicon matrix and a metal matrix, the silicone can be easily deformed, causing deformation to the spatial arrangement of the waxed teeth and changes in the shape and strength of the interproximal contacts.

The main advantage of using this plastic and acetate matrix is that it allows one to recover the point of contact and the position of the teeth in the arch with few or no alterations. During the clinical occlusal analysis, minimal adjustments were made. On the other hand, the limitation of this technique is the substantial loss of the anatomical details that the diagnostic wax provided. During the finishing and final polishing, these anatomical details were made using finishing burs and a finishing and polishing system to recover the natural occlusal anatomy of the posterior teeth. The proximal faces were polished using EVA tips and a reciprocating diamond system, which allowed for removing contours and composite excess formed during the restorative process.

Once the VDO was recovered, the next step was to establish anterior esthetics with composite resin restorations using the same Vailati technique. According to the Vailati and Belser¹³⁻¹⁵ technique, it is necessary to first determine the optimal position of the maxillary incisal edges of the planned final restorations to determine the correct distribution of

interocclusal space gained by the increase in VDO. Limitations will occur in more severe cases of erosion, which was found in the current case. In these situations, adjustments of this technique are necessary.

During the initial diagnosis, the level of dental wear and the relationship of the mandibular and maxillary arches were analyzed. As a result, the thickness of the restorative material on the occlusal surfaces varied, considering that previously placed unsatisfactory restorations required replacement. In the present case, less than 1 mm was necessary to restore VDO. Additionally, VDO and TMJ health are of great relevance, and their relationship cannot be ignored. Two main reasons guide the recommendation for increasing VDO: 1) When major dental wear accompanies missing teeth, the VDO and TMJ positions can become unbalanced. In these cases, the VDO should be reestablished, especially in more complex cases of rehabilitation. 2) In the second scenario, a slight loss of VDO is presented or insufficient space for restorative material is available for restoring VDO. This situation corresponds to the present case. Therefore, the VDO analysis is essentially simultaneous with the analysis of TMJ, which was not compromised. For both situations, manipulating the VDO following recommended philosophies of occlusion is essentially arbitrary, especially considering the limitations of each case.²²⁻²⁷

Follow-ups in the current case revealed no negative impact on the TMJs after the posterior restoration and restoring VDO. After a four-month follow-up, the present patient did not present with any issue related to the TMJs. Additionally, she reported reduced dental sensitivity. Any minor adjustments of the restorative materials were performed during finishing procedures. The anterior teeth were restored only after the VDO was found to be stable.

An occlusal registration was made taking maximum intercuspation position (MIP) into account, as recommended in part 2 of this technique.¹⁵ The dilemma involving the decision of restoring VDO in centric relation (CR) and MIP was considered from the beginning. For this current patient, there was a discrepancy between these two parameters. When the teeth were positioned to CR, the overjet distance increased. According to Vailaiti and Belser's part 2 article,¹⁵ it is necessary to obtain correct anterior guidance. Another concern with this technique was related to the age of the present patient, who was still developing skeletally. Therefore, the VDO was

established using MIP, which was considered the most appropriate for these circumstances.

In this present case, the third laboratory stage began using indirect palatal veneers with composite for the six maxillary anterior teeth. The color selection was obtained via cross-polarized photography, and small portions of composite resin were placed on the buccal surfaces. The veneers were luted using direct incremental resin restorations. The stratification technique using a direct composite resin was successful for providing excellent mechanical and esthetic results. At this stage, the current authors avoided the use of indirect ceramic veneers, considering the need for minimally invasive dentistry. We preserved the enamel of the cervical two-thirds of the buccal faces of the teeth as much as possible.

After the sequential appointments for monitoring the patient, some changes were noticed, including the wear and tear of composite resins in specific and localized areas. The point is that even with well-planned and well-executed dental treatment, external factors, such as psychological, idiopathic, or even physiological aspects of the patient, can be a considerable determinant of long-term success. In this case report, the patient was found to have an etiological problem and chronic-based erosion. In the three-year follow-up, the patient finally admitted to the consumption of cola beverages, which she had stopped completely a few months prior to the three-year follow-up appointment.

Even with the presence of localized erosion, the main advantage of the use of composite resin is that it allowed for polishing, making repairs, and replacing an unsatisfactory restoration without the need for removing extra dental structure. After the four-year follow-up, the current authors detected erosive progression, especially around the buccal surfaces of the left maxillary premolars. This scenario was challenging, as the other restored areas performed well, to include minor adjustments (Figure 15). The patient reported that she had recently decided to refrain from the consumption of cola drinks, as she finally was aware of the impact of those progressive lesions.

The present authors conclude that there is a high chance of recurrence when dental erosion is not considered when planning, considering the type of material, and performing the dental treatment in patients affected by erosion, even when treatment occurs jointly with a psychologist and physician. An available strategy, such as full-mouth adhesive rehabilitation, is an excellent technique for allowing



Figure 15. Views after adjustments at the four-year follow-up. (a) Occlusal view of the maxillary teeth. (b) Occlusal view of the mandibular teeth. (c) View of the maxillary anterior teeth.

restorative procedures of complex cases. However, monitoring patients with a chronic disease is always mandatory for long-term clinical performance.

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 25 July 2019)

REFERENCES

1. Reis A, Higashi C, & Loguercio AD (2009) Re-anatomization of anterior eroded teeth by stratification with direct composite resin *Journal of Esthetic and Restorative Dentistry* **21**(5) 304-316.
2. Huysmans MC, Chew HP, & Ellwood RP (2011) Clinical studies of dental erosion and erosive wear *Caries Research* **45**(Supplement 1) 60-68.
3. Fradeani M, Barducci G, Bacherini L, & Brennan M (2012) Esthetic rehabilitation of a severely worn dentition with minimally invasive prosthetic procedures (MIPP) *International Journal of Periodontics and Restorative Dentistry* **32**(2) 135-147.
4. Giacomini MC, Scaffa P, Chaves LP, Vidal C, Machado TN, Honório HM, Tjäderhane L, & Wang L (2017) Role of proteolytic enzyme inhibitors on carious and eroded dentin associated with a universal bonding system *Operative Dentistry* **42**(6) E188-E196.
5. Resende TH, Reis KR, Schlichting LH, & Magne P (2018) Ultrathin CAD-CAM ceramic occlusal veneers and anterior bilaminar veneers for the treatment of moderate dental biocorrosion: A 1.5-year follow-up *Operative Dentistry* **43**(4) 337-346.
6. Magalhães AC, Wiegand A, Rios D, Honorio HM, & Buzalaf MA (2009) Insights into preventive measures for dental erosion *Journal of Applied Oral Science* **17**(2) 75-86.
7. Lussi A, Schlueter N, Rakhmatullina E, & Ganss C (2011) Dental erosion—An overview with emphasis on chemical and histopathological aspects *Caries Research* **45**(Supplement 1) 2-12.
8. Herbella FA & Patti MG (2010) Gastroesophageal reflux disease: From pathophysiology to treatment *World Journal of Gastroenterology* **16**(30) 3745-3749.
9. Bartlett D (2016) A personal perspective and update on erosive tooth wear—10 Years on: Part 2—Restorative management *British Dental Journal* **221**(4) 167-171.
10. ten Cate JM & Imfeld T (1996) Dental erosion, summary *European Journal of Oral Sciences* **104**(2(Pt 2)) 241-244.
11. Wang L, Magalhães AC, Francisconi-Dos-Rios LF, Calabria MP, Araújo D, Buzalaf M, Lauris J, & Pereira JC (2016) Treatment of dentin hypersensitivity using nano-hydroxyapatite pastes: A randomized three-month clinical trial *Operative Dentistry* **41**(4) E93-E101.
12. Vailati F & Carciofo S (2016) Treatment planning of adhesive additive rehabilitations: The progressive wax-up of the three-step technique *International Journal of Esthetic Dentistry* **11**(3) 356-377.
13. Vailati F & Belser UC (2010) Classification and treatment of the anterior maxillary dentition affected by dental erosion: The ACE classification *International Journal of Periodontics and Restorative Dentistry* **30**(6) 559-571.
14. Vailati F & Belser UC (2008) Full-mouth adhesive rehabilitation of a severely eroded dentition: The three-step technique Part 1 *European Journal of Esthetic Dentistry* **3**(1) 30-44.
15. Vailati F & Belser UC (2008) Full-mouth adhesive rehabilitation of a severely eroded dentition: The three-step technique. Part 2 *European Journal of Esthetic Dentistry* **3**(2) 128-146.
16. Bartlett DW (2016) A personal perspective and update on erosive tooth wear—10 Years on: Part 1—Diagnosis and prevention *Brazilian Dental Journal* **221**(3) 115-119.
17. Moazzez RV, Austin RS, Rojas-Serrano M, Carpenter G, Cotroneo E, Proctor G, Zaidel L, & Bartlett DW (2013) Comparison of the possible protective effect of the salivary pellicle of individuals with and without erosion *Caries Research* **48**(1) 57-62.
18. Moazzez R, Anggiansah A, & Bartlett DW (2005) The association of acidic reflux above the upper oesophageal

- sphincter with palatal tooth wear *Caries Research* **39**(6) 475-478.
19. Coachman C & Calamita M (2012) Digital smile design: A tool for treatment planning and communication in esthetic dentistry *Quintessence of Dental Technology* **35** 103-111.
 20. Meereis CT, de Souza GB, Albino LG, Ogliari FA, Piva E, & Lima GS (2016) Digital smile design for computer-assisted esthetic rehabilitation: Two-year follow-up *Operative Dentistry* **41**(1) E13-E22.
 21. Bartlett D, Ganss C, & Lussi A (2008) Basic Erosive Wear Examination (BEWE): A new scoring system for scientific and clinical needs *Clinical Oral Investigations* **12**(Supplement 1) S65-S68.
 22. Tryde G, Stoltze K, & Morimoto T (1977) Long term changes in the perception of comfortable mandibular occlusal positions *Journal of Oral Rehabilitation* **4**(1) 9-15.
 23. Hellsing G (1984) Functional adaptation to changes in vertical dimension *Journal of Prosthetic Dentistry* **52**(6) 867-870.
 24. Dawson P (1989) *Evaluation, Diagnosis, and Treatment of Occlusal* Mosby, St Louis, MO.
 25. Fernandes PR, de Vasconsellos HA, Okeson JP, Bastos RL, & Maia ML (2003) The anatomical relationship between the position of the auriculotemporal nerve and mandibular condyle *Cranio: The Journal of Craniomandibular Practice* **21**(3) 165-171.
 26. Serrano-Hernanz G, Futarmal Kothari S, Castrillón E, Álvarez-Méndez AM, Ardizzone-García I, & Svensson P (2019) Importance of standardized palpation of the human temporomandibular joint *Journal of Oral & Facial Pain Headache* **33**(2) 220-226.
 27. Okeson JP & de Leeuw R (2011) Differential diagnosis of temporomandibular disorders and other orofacial pain disorders *Dental Clinics of North America* **55**(1) 105-120.

Occlusal and Esthetic Enhancement: A Noninvasive Approach to an Old Dilemma

GS Lima • LGB Albino • CS dos Santos • MS Cenci • T Pereira-Cenci

Clinical Relevance

A clinical laboratory technique for occlusal and esthetic enhancement using an indirect restoration is presented. This technique is simple, low cost, and noninvasive compared with the preparation of ceramic veneers.

SUMMARY

This article describes an indirect composite restoration technique for diastema closure and tooth lengthening with a noninvasive approach using regularly available materials such as silicone, composite resin, and an adhesive system.

The procedure resulted in occlusal and functional improvement, with diastema closure, protrusive guide adjustment, and an increase in central incisor length. The procedure provided an adequate proportion of the central incisors with an esthetically natural appearance. It also resulted in simple, fast, and accurate manufacturing with a noninvasive esthetic indirect rehabilitation compared with more invasive preparation of ceramic veneers.

*Giana da Silveira Lima, DDS, MS, PhD, professor, Department of Restorative Dentistry, Graduate Program in Dentistry, School of Dentistry, Federal University of Pelotas, Rio Grande do Sul, Brazil

Luis Gustavo Barrote Albino, MS, PhD student, Graduate Program in Dentistry, Federal University of Pelotas, Pelotas/ Rio Grande do Sul/ Brazil

Cinthia Studzinski dos Santos, MS student, Graduate Program in Dentistry, Federal University of Pelotas, Pelotas/ Rio Grande do Sul/ Brazil

Maximiliano Sérgio Cenci, DDS, MS, PhD, professor, Department of Restorative Dentistry, Graduate Program in Dentistry, School of Dentistry, Federal University of Pelotas, Rio Grande do Sul, Brazil

Tatiana Pereira Cenci, DDS, MS, PhD, professor, Department of Restorative Dentistry, Graduate Program in Dentistry, School of Dentistry, Federal University of Pelotas, Rio Grande do Sul, Brazil

*Corresponding author: Room 504, 457 Gonçalves Chaves St, Rio Grande do Sul 96015-560, Brazil; e-mail: gianalima@gmail.com

<https://doi.org/10.2341/18-330-T>

INTRODUCTION

Diastema closure is a common clinical procedure that may be used to address inadequate tooth size, occlusal misalignments and undesirable esthetic features.¹⁻³ The most common treatment modality is tooth preparation and porcelain veneer placement or direct composite resin restoration.⁴⁻⁶ For porcelain veneer preparations, there is usually a need for the removal of sound tooth structure, while direct composite resin restorations are “no prep” in most clinical situations. Still, sometimes “no prep” may not always be the best choice because esthetics may eventually be compromised due to a color mismatch when substrate masking is required.⁷ Thus, the treatment plan has to consider the tooth shape and size, determine the need for substrate masking, and

indicate the most conservative treatment that properly matches function and esthetics.

In this case, the lengthening of the central incisors and a diastema closure were requested by the patient. They were also necessary for protrusive guide adjustment, because of the presence of posterior premature contacts that were impairing the patient's occlusion. Beyond that, in this clinical case, the diastema closure was done to prevent tooth movement after orthodontic treatment. The treatment plan for the patient consisted of two indirect composite resin veneers without any kind of tooth preparation. This was possible because there were adequate substrate color and space to perform the restoration. The present technique report describes an indirect composite restoration with a noninvasive approach using simple materials to establish occlusal improvement and esthetic appearance.

DESCRIPTION OF THE TECHNIQUE

The initial clinical situation was assessed and the amount of tooth structure to be restored was determined (Figure 1). A protrusive guide was assessed for planning the wax-up (Figure 2). One maxillary cast (Type IV Dental Die Stone GC FujiRock EP Golden Brown, GC America Inc, Alsip, IL, USA) was used in a semiaadjustable articulator for the wax-up and to obtain a silicone matrix guide (Honigum Heavy Body, DMG America, Ridgefield Park, NJ, USA; Figure 3A).

On a second cast (working cast), a 0.2-mm thick light wax layer relief was applied to the central incisors (Cera Kota, Kota Ind e Com Ltda, Cotia, SP, Brazil) to facilitate the removal of the restoration after its manufacture and prevent it from adhering to the model, as shown in Figure 3B. It is important to note that no dental preparation was performed, and what appears to be a gingival finish line in the maxillary right central incisor was, in fact, a variation of the normality of the enamel dental anatomy, coinciding with the anatomical neck of the tooth. First, the composite resin was applied to the restoration on the palatal area (Z350 XT WB White Body, 3M ESPE, St Paul, MN, USA; Figure 3C). Then, the same resin (A1) was added for dentin replacement followed by B1 resin for incisal buildup.

Characterization for a more natural aspect of restorations was achieved using the Resin Color Modifier and Opaquing Kit (Kerr Corporation, Orange, CA, USA), Kolor + Plus–White (Figure 4A) and Kolor + Plus–Ochre (Figure 4B) pigments. The

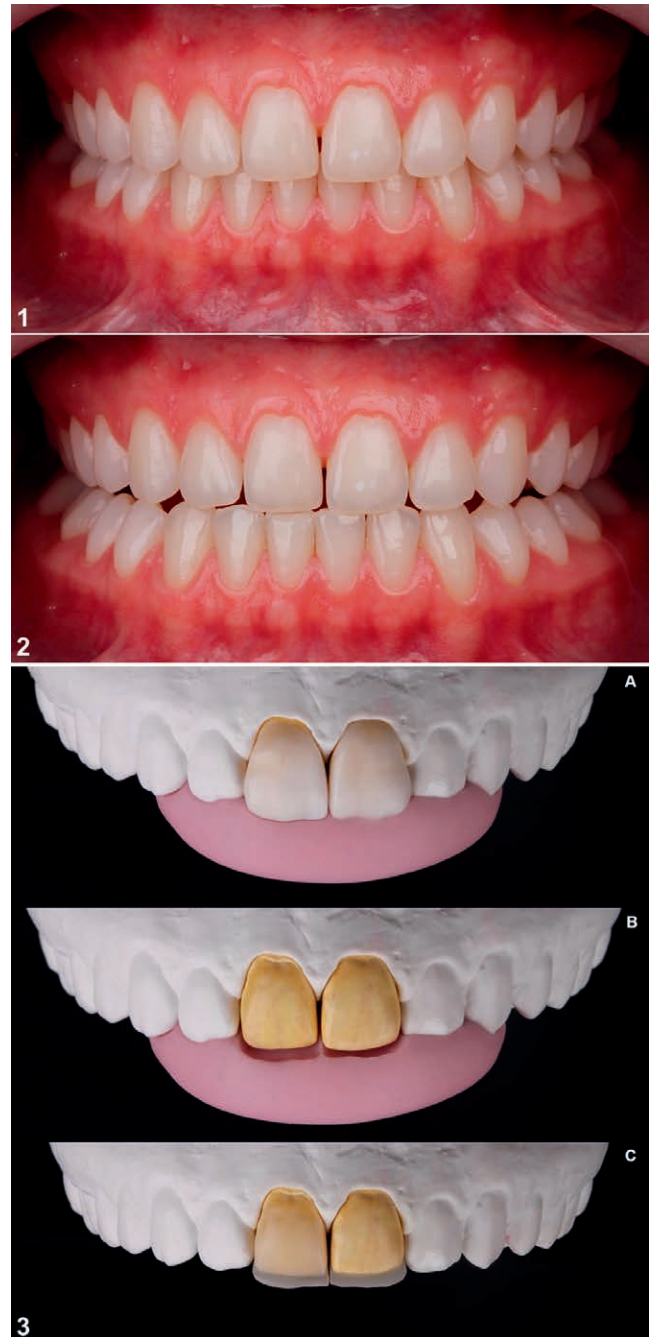


Figure 1. Intraoral view of the diastema and short central incisors of a 19-year-old woman.

Figure 2. Anterior guidance (protrusion). Note the group function with premature posterior contacts.

Figure 3. (A): Adapted silicone matrix on wax-up. (B): Wax relief area before indirect restoration. Notice a variation of the normality of the maxillary central incisors' enamel dental anatomy, with the anatomical neck of the tooth. No dental preparation has been performed. (C): Palatal reconstruction.

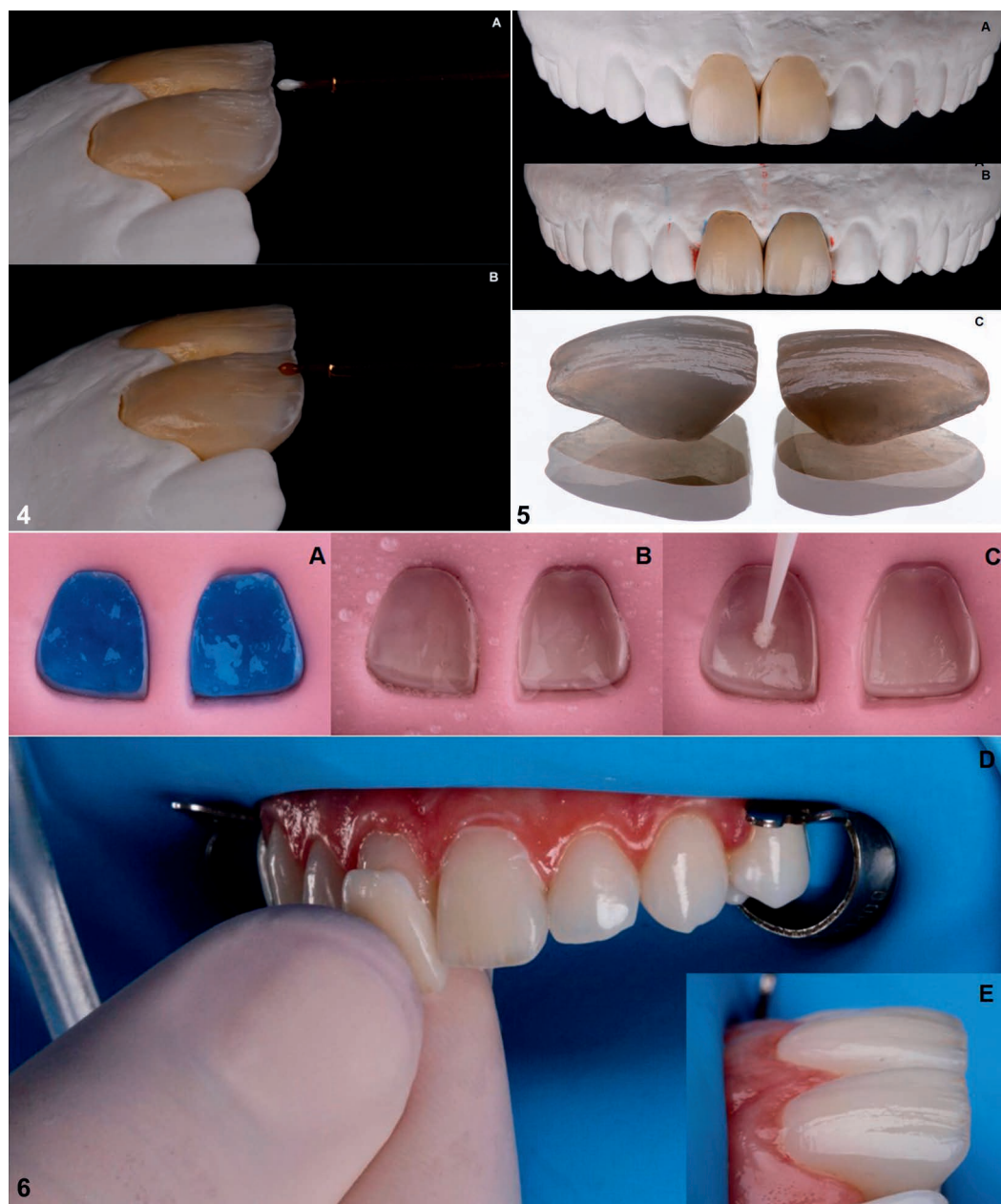


Figure 4. (A): Stratification technique, white. (B) Stratification technique, orange.

Figure 5. (A): Final view of the indirect restoration, finishing. (B): Final view of the indirect restoration, polishing. (C): Final view of the indirect restoration, surface texture.

Figure 6. Step-by-step indirect restoration preparation using a silicone matrix for better handling and buccal protection. (A): Acid etching. (B): Water spraying. (C): Silane application. (D): Cementation procedure. (E): Emergence profile of the indirect restoration.

last two layers were made with B0 resin (Renamel Microhybrid, Cosmedent, Chicago, IL, USA; Figure 5A). Abrasive ceramic rubbers were used for finishing (Gray Edenta, Labordental Ltda, São Paulo, SP, Brazil), and the texture was achieved with diamond burs (HF257SPE-023 and HF364CE-023, Cosmedent; Figure 5B,C).

The two veneers were removed from the cast using abundant boiling water, which melted the wax layer relief and released the veneers easily, and then their marginal adaptation was assessed in the patient. A try-in (NX3 Light Cure, Kerr Corporation) was used to test and choose the cement color. The veneers were placed in a silicone matrix to protect the buccal

face during the surface treatment before the cementation procedure. This preparation consisted of air abrading the inside of the veneers with aluminum oxide (20 μm), rinsing with water, etching with phosphoric acid (power etching 37%; BM4, Maringá, PR, Brazil; Figure 6A), rinsing with water (Figure 6B), and air drying. Silane (Silane Primer, Kerr Corporation) was applied, the excess was dried off (Figure 6C), and an adhesive layer was then applied (Adper Scotch Bond Multi-Purpose, 3M ESPE).

In the tooth substrate, a total etching technique was carried out using phosphoric acid for 30 seconds. The acid was subsequently removed with water and air dried, and the adhesive system was then applied (Adper Scotch Bond Multi-Purpose, 3M ESPE). The surface was gently air dried for removal of the solvent.

For the veneer adhesive procedures, a thin layer of a resin-based cement (NX3 Light Cure [clear], Kerr Corporation) was applied to the inside of each veneer before seating on the tooth (Figure 6D). The emergence profile was checked after cementation (Figure 6E).

An evaluation of the diastema closure, occlusion (Accufilm, Parkell Inc, Edgewood, NY, USA), and the increase of the central incisors' length (Figure 7) was performed as well as the assessment of the correct anterior guidance (protrusion), where space was created to adjust the previous posterior premature contacts (Figure 8). The restoration provided natural tooth color, texture, anatomic shape, and proportion of the central incisors.

The 2.5-year follow-up of the treatment was satisfactory, with adequate polishing of the restoration, contours, and color and without cracks or fractures. The patient's periodontal health is shown in Figure 9.

POTENTIAL PROBLEMS

The main potential problems of this technique may be related to the limitations of composite resin, such as short-term color mismatch, loss of brightness and polishing, or a restoration fracture.⁸ Another issue that should be considered is the need for an additional lab step and also a dental lab technician if the dentist does not have the skills to perform an indirect restoration.

SUMMARY OF ADVANTAGES AND DISADVANTAGES

The advantage of this technique is the possibility of using a low-cost combination of simple materials and

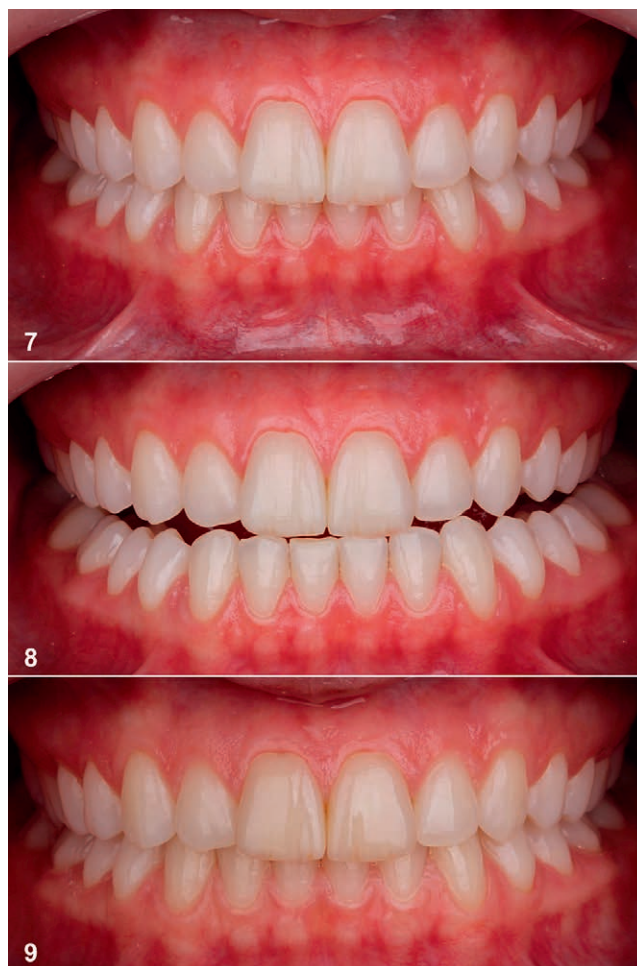


Figure 7. Intraoral view of esthetic restoration. Diastema closure and predominance of central incisors were achieved.

Figure 8. Intraoral view of corrected anterior guidance (protrusion).

Figure 9. Intraoral view of esthetic restoration after 2.5 years of follow-up.

techniques that may be manufactured by the dentist without the need for a dental laboratory. Indirect techniques allow for a preoperative design with a wax-up or digital wax-up and better management of the occlusion. As in this case, in which optimal form and esthetics are required, indirect techniques have advantages,⁹ such as allowing for the reproduction of the natural appearance of the teeth with less chair time and enabling intraoral repair using the same material and also the enhancement of resin mechanical and chemical properties (eg, additional polymerization with an optimized degree of conversion).^{9,10}

The optimal performance of the restorations depends on the proper polymerization of the resin components, featured by transforming monomers

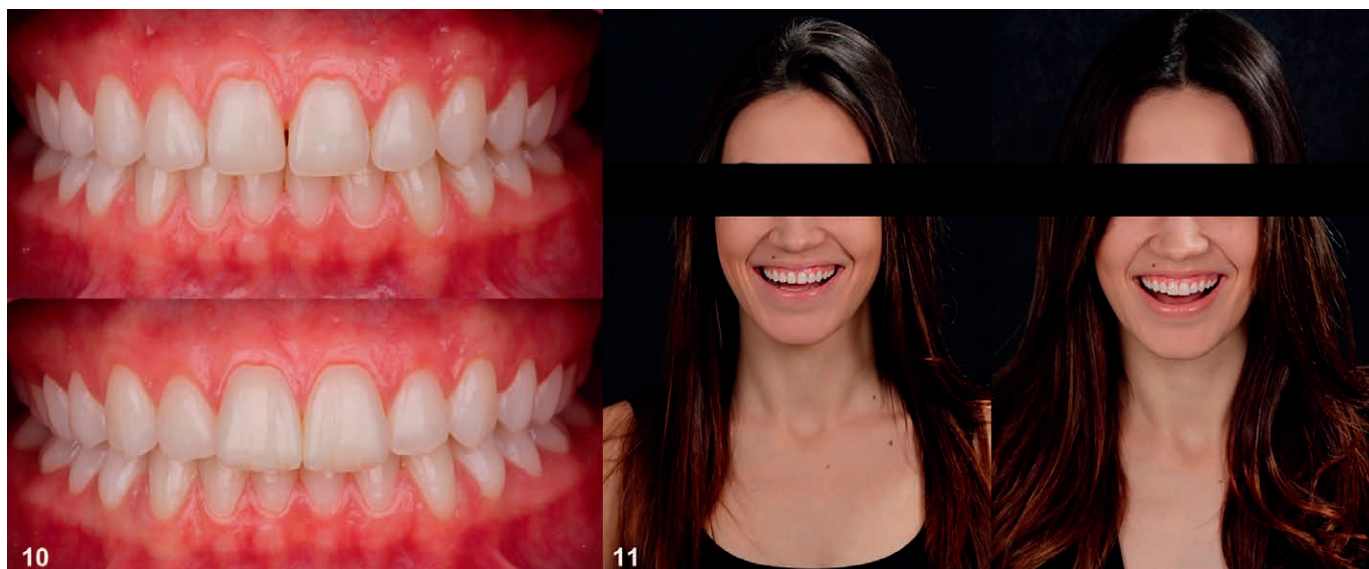


Figure 10. Before and after treatment intraoral view.

Figure 11. Before and after treatment facial view of the patient.

into polymers, and volumetric reduction of the material, which is known as polymerization shrinkage.^{11,12} With the indirect restorative procedure, the polymerization shrinkage stress at the adhesive interface will be restricted to the resin cement and not the entire restoration, contributing to a reduction in stress.¹³ However, there is a need for caution with the wax relief, as a thicker relief may result in a thicker layer of cement.

Although there is no reliable evidence to show a better approach of direct or indirect veneer restoration regarding the longevity of the restoration,¹⁴ studies show that anterior composite restorations have adequate clinical performance.^{15,16} A minimally invasive approach to the final restoration is generally preferred, and the removal of a sound tooth structure should be avoided when possible.⁹ No tooth preparation was performed on the tooth substrate to achieve the outcome. The minimally invasive approach is still controversial in the literature because of periodontal problems, due to overcontouring, possible pigmentation of the margins, and because long-term results are not available.¹⁷ However, some studies have shown that this approach is successful^{1,18,19} and has clinical advantages such as enamel preservation, absence of postoperative sensitivity, no need for provisional restoration, and, mainly, the possibility of treatment reversal. An underlying feature of this rehabilitation was the achievement of adequate protrusive guidance, which previously produced posterior prema-

ture contacts and impaired the occlusion. This feature can easily be adjusted by waxing up in a semiadjustable articulator.^{20,21} The 2.5-year follow-up showed that this technique performed satisfactorily, restoring function and esthetics properly. However, as with any other resin restoration, failures may occur and the patient needs to be regularly monitored for treatment maintenance.

Acknowledgements

The authors thank Francisco Mello and Osmar Gradnar for technical assistance. This study was conducted in a graduate program supported by CAPES, Brazil.

Regulatory Statement

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

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 15 October 2019)

REFERENCES

1. Strassler HE (2007) Minimally invasive porcelain veneers: indications for a conservative esthetic dentistry treatment modality *General Dentistry* **55**(7) 686-694.
2. Signore A, Kaitsas V, Tonoli A, Angiero F, Silvestrini-Biavati A, & Benedicenti S (2013) Sectional porcelain

- veneers for a maxillary midline diastema closure: a case report *Quintessence International* **44**(3) 201-206. <https://doi.org/10.3290/j.qi.a29058>
3. Morita RK, Hayashida MF, Pupo YM, Berger G, Reggiani RD, & Betiol EAG (2016) Minimally invasive laminate veneers: clinical aspects in treatment planning and cementation procedures *Case Reports in Dentistry* **2016** 1839793. <https://doi.org/10.1155/2016/1839793>
 4. Lempel E, Lovász BV, Meszarics R, Jeges S, Tóth Á, & Szalma J (2017) Direct resin composite restorations for fractured maxillary teeth and diastema closure: a 7 years retrospective evaluation of survival and influencing factors *Dental Materials* **33**(4) 467-476. <https://doi.org/10.1016/j.dental.2017.02.001>
 5. Wolff D, Kraus T, Schach C, Pritsch M, Mente J, Staehle HJ, & Ding P (2010) Recontouring teeth and closing diastemas with direct composite buildups: a clinical evaluation of survival and quality parameters *Journal of Dentistry* **38**(12) 1001-1009. <https://doi.org/10.1016/j.jdent.2010.08.017>
 6. Viswambaran M, Londhe SM, & Kumar V (2015) Conservative and esthetic management of diastema closure using porcelain laminate veneers *Medical Journal Armed Forces India* **71** 581-585. <https://doi.org/10.1016/j.mjafi.2014.08.014>
 7. Domingos PA dos S, Garcia PPNS, Oliveira ALBM de & Palma-Dibb RG (2011) Composite resin color stability: influence of light sources and immersion media *Journal of Applied Oral Science* **19**(3) 204-211. <https://doi.org/10.1590/S1678-77572011000300005>
 8. van De Sande FH, Moraes RR, Elias RV, Montagner AF, Rodolpho PA, Demarco FF, & Cenci MS (2018) Is composite repair suitable for anterior restorations? A long-term practice-based clinical study *Clinical Oral Investigations* **23**(6) 2795-2803. <https://doi.org/10.1007/s00784-018-2722-5>
 9. Opdam N, Frankenberger R, & Magne P (2016) From “direct versus indirect” toward an integrated restorative concept in the posterior dentition *Operative Dentistry* **41**(7) 27-34. <https://doi.org/10.2341/15-126-LIT>
 10. Angeletaki F, Gkogkos A, Papazoglou E, & Kloukos D (2016) Direct versus indirect inlay/onlay composite restorations in posterior teeth: a systematic review and meta-analysis *Journal of Dentistry* **53** 12-21. <https://doi.org/10.1016/j.jdent.2016.07.011>
 11. 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>
 12. Soares CJ, Faria-E-Silva AL, Rodrigues MP, Vilela ABF, Pfeifer CS, Tantbirojn D, & Versluis A (2017) Polymerization shrinkage stress of composite resins and resin cements—what do we need to know? *Brazilian Oral Research* **31**(1) 49-63. <https://doi.org/10.1590/1807-3107bor-2017.vol31.0062>
 13. Münchow EA, Meereis CTW, 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 technique protocol and photo-activation strategies *Journal of the Mechanical Behavior of Biomedical Materials* **82** 77-86. <https://doi.org/10.1016/j.jmbbm.2018.03.004>
 14. Wakiaga JM, Brunton P, Silikas N, Glenney AM, Gopakumar A, & Boyle L (2015) Direct versus indirect veneer restorations for intrinsic dental stains *Cochrane Database Systematic Review* **12** CD004347. <https://doi.org/10.1002/14651858.CD004347.pub3>
 15. Collares K, Opdam NJM, Laske M, Bronkhorst EM, Demarco FF, Correa MB, & Huysmans MCDNJM (2017) Longevity of anterior composite restorations in a general dental practice-based network *Journal of Dental Research* **96**(10) 1092-1099. <https://doi.org/10.1177/0022034517717681>
 16. Demarco FF, Collares K, Coelho-De-Souza FH, Correa MB, Cenci MS, Moraes RR, & Opdam NJM (2015) Anterior composite restorations: a systematic review on long-term survival and reasons for failure *Dental Materials* **31**(10) 1214-1224. <https://doi.org/10.1016/j.dental.2015.07.005>
 17. Zarone F, Leone R, Di Mauro MI, Ferrari M, & Sorrentino R (2018) No-preparation ceramic veneers: a systematic review *Journal of Osseointegration* **10**(1) 17-22.
 18. Imburgia M, Canale A, Cortellini D, Maneschi M, Martucci C, & Valenti M (2016) Minimally invasive vertical preparation design for ceramic veneers *International Journal of Esthetic Dentistry* **11**(4) 460-471.
 19. Farias-Neto A, Gomes EMDCF, Sánchez-Ayala A, Sánchez-Ayala A, & Vilanova LSR (2015) Esthetic rehabilitation of the smile with no-prep porcelain laminates and partial veneers *Case Reports in Dentistry* 1-6. <https://doi.org/10.1155/2015/452765>
 20. Türp JC, Greene CS, & Strub JR (2008) Dental occlusion: a critical reflection on past, present and future concepts *Journal of Oral Rehabilitation* **35**(6) 446-453. <https://doi.org/10.1111/j.1365-2842.2007.01820.x>
 21. Rinchuse DJ, Kandasamy S, & Sciote J (2007) A contemporary and evidence-based view of canine protected occlusion *American Journal of Orthodontics and Dentofacial Orthopedics* **132**(1) 90-102. <https://doi.org/10.1016/j.ajodo.2006.04.026>

Catechin-based Dentin Pretreatment and the Clinical Performance of a Universal Adhesive: A Two-year Randomized Clinical Trial

CAGA Costa • NLG Albuquerque • JS Mendonça • AD Loguercio • VPA Saboia • SL Santiago

Clinical Relevance

At 24 months, the dentin pretreatment with epigallocatechin-3-gallate did not impair the clinical performance of the adhesive Single Bond Universal regardless of the bonding strategy used.

SUMMARY

Purpose: To evaluate the two-year effect of dentin pretreatment with epigallocatechin-3-gallate (EGCG) on the clinical performance of restorations of noncarious cervical lesions (NCCLs) with Single Bond Universal, applied in two different modes (self-etch and etch-and-rinse).

Methods and Materials: In this randomized clinical trial, 33 volunteers were selected, and 156 NCCLs were assigned to four groups: ER,

etch-and-rinse; ER-EGCG, 0.1% EGCG dentin pretreatment + etch-and-rinse; SE, self-etch; and SE-EGCG, 0.1% EGCG dentin pretreatment + self-etch. The NCCLs were restored with a nanofilled resin composite and evaluated at baseline and at six, 12, 18, and 24 months using FDI criteria for retention, marginal staining, marginal adaptation, caries, and postoperative sensitivity. Two evaluators were blinded to the treatments performed, and impressions were taken for resin replicas to allow indirect observations. Statistical analyses were performed with Kruskal-Wallis and McNemar tests with a significance level of 5%.

Cecília Atem Gonçalves de Araújo Costa, DDS, MSc, PhD, Graduate Program in Dentistry, Faculty of Pharmacy, Dentistry, and Nursing, Federal University of Ceará, Fortaleza, Brazil

Nadine Luísa Guimarães Albuquerque, DDS, MSc, PhD, Graduate Program in Dentistry, Faculty of Pharmacy, Dentistry, and Nursing, Federal University of Ceará, Fortaleza, Brazil

Juliano Sartori Mendonça, DDS, MSc, PhD, Department of Restorative Dentistry, Faculty of Pharmacy, Dentistry, and Nursing, Federal University of Ceará, Fortaleza, Brazil

Alessandro Dourado Loguercio, DDS, MSc, PhD, Department of Restorative Dentistry, School of Dentistry, State University of Ponta Grossa, Ponta Grossa, Brazil

Vicente de Paulo Aragão Saboia, DDS, MSc, PhD, Department of Restorative Dentistry, Faculty of Pharmacy, Dentistry, and Nursing, Federal University of Ceará, Fortaleza, Brazil

*Sérgio Lima Santiago, DDS, MS, PhD, Department of Restorative Dentistry, Faculty of Pharmacy, Dentistry, and Nursing, Federal University of Ceará, Fortaleza, Brazil

*Corresponding author: Monsenhor Furtado s/n, Rodolfo Teófilo, Fortaleza, CE 60430-355, Brazil; e-mail: sergiosantiago@ufc.br

<https://doi.org/10.2341/19-088-C>

Results: Six restorations (one from ER, two from SE, one from ER-EGCG, and two from SE-EGCG) were lost at 24 months with no significant differences ($p>0.05$). The retention rates were 97.0% (ER and ER-EGCG), 94.1% (SE), and 94.2% (SE-EGCG). For marginal adaptation, a significant difference was detected between the baseline and 24 months for the SE group ($p=0.0313$). There were no statistical differences among all other evaluated criteria at 24 months, neither for each group at baseline nor for 24-month comparisons ($p>0.05$).

Conclusions: The pretreatment with EGCG provided no benefit in the clinical performance of the adhesive regardless of the bonding strategy used. In addition, it adds an additional required step to the restorative procedure.

INTRODUCTION

Randomized controlled trials are considered the ideal design for comparing different procedures in health care interventions¹ and the most reliable of evidence-based studies to dental practice.² Laboratory studies are widely used and provide faster results, but they do not take into account the complex oral environment.³ The current research in adhesive dentistry is aimed at improving the quality of the resin-dentin bonds and understanding their degradation mechanisms.⁴ Many strategies have been proposed to provide greater longevity of the union of the restorative materials to the dental structure, but few of them were evaluated under clinical conditions.⁵

Since proteolytic degradation of the hybrid layer has been described in the literature⁶ and associated with the host enzymes, matrix metalloproteinases (MMP), and cysteine cathepsins (CC), numerous investigations have been conducted with potential enzymatic inhibitors. Promising laboratory results have been achieved regarding the hybrid layer preservation and long-term durability.^{7,8} These enzymes are present in human dentin and saliva with the ability to degrade most extracellular matrix proteins, such as collagen type I, the organic component of the hybrid layer.^{9,10} Within the hybrid layer, denuded collagen fibrils become vulnerable to degradation by these host-derived proteases with collagenolytic activity.¹¹ Thus, the use of bioactive substances that inhibit the enzymes may avoid the degradation of exposed collagen fibrils at the adhesive interface.⁴

Chlorhexidine (CHX) is the “gold standard” antimicrobial agent for oral health and has demonstrated inhibition of MMP-2, MMP-8, MMP-9, and CC.^{12,13} Laboratory results have proven the benefits in the immediate and long-term bond effectiveness of CHX associated with adhesive systems. However, the use of chlorhexidine as an MMP inhibitor was tested under clinical trials (either as dentin pretreatment or incorporated in the adhesive),¹⁴⁻¹⁷ and the results (up to three-year follow-up)¹⁸ showed that there was no improvement in the clinical durability of adhesive restorations. These results also highlighted the lack of correlation between the results of the laboratory tests and the clinical reality to which the adhesive restorations are exposed.³

Other enzymatic inhibitors, mainly natural products, have aroused the interest of the scientific community. Epigallocatechin-3-gallate (EGCG) is the most abundant polyphenol found in green tea with a proven ability to inhibit the expression and action of MMP-2, MMP-9, and CC.¹⁹ Through laboratory studies, the use of EGCG has been recently associated with adhesive procedures and was effective in preserving the resin-dentin bond strength for up to six months when incorporated into the dentin adhesive²⁰ and up to six and 12 months when used as a dentin pretreatment with etch-and-rinse (ER) and self-etch (SE) adhesive systems, respectively.^{21,22} Unlike CHX, EGCG also has an additional cross-linking property with positive effects on the mechanical properties of collagen and its stabilization against proteolytic degradation.²³ It has been shown that galloyl radical monomeric catechins, such as EGCG, are more effective than other catechins without the radical in increasing the modulus of elasticity and reducing collagen biodegradation rates.²³

In this field, EGCG seems to be a promising bioactive substance that may maintain the integrity and stability of dentin collagen by synergistic effects, with protease inhibition and cross-linking ability in dentin. To the authors' knowledge, there are no randomized controlled trials evaluating the effect of EGCG as a dentin primer in the restorations of noncarious cervical lesions (NCCLs).

Thus, the objective of the present study was to evaluate the influence of dentin pretreatment with EGCG in restorations of noncarious cervical lesions performed with a universal adhesive system in the two adhesive strategies *in vivo*. The null hypotheses tested were that (1) there is no difference between the clinical performance of NCCL restorations

Table 1: Restorative Materials and Application Procedures According to the Manufacturer's Instructions

Material	Manufacturer	Composition	Lot Number	Application Procedure
Epigallocatechin-3-gallate	Sigma-Aldrich (St Louis, MO, USA)	≥80% (high-performance liquid chromatography), from green tea	SLBL 1959V	0.1% aqueous solution actively applied for 60 s with a microbrush before adhesive application
Filtek Z350 XT	3M ESPE (St Paul, MN, USA)	Treated silanized ceramics; silane-treated silica; urethane dimethacrylate; bisphenol A polyethylene glycol diether dimethacrylate; bisphenol A-glycidyl methacrylate; ceramics of zirconia; polyethylene glycol; dimethacrylate; triethylene glycol and dimethacrylate	190224, 147907	Up to three increments of composite resin, individually photopolymerized
Single Bond Universal	3M ESPE	Methacryloyloxydecyl dihydrogen phosphate; phosphate monomer; dimethacrylate resins; hydroxyethyl methacrylate; methacrylate-modified polyalkenoic acid copolymer; filler; ethanol; water; initiators; silane	582958	Etch-and-rinse strategy: total etching with 37% phosphoric acid (enamel 30 s, dentin 15 s) followed by rinsing with water and drying with air free of moisture and oil, without drying out. Adhesive application to tooth surface by scrubbing action (20 s), drying of the adhesive (5 s), and light curing (10 s) Self-etch strategy: adhesive application to tooth surface by scrubbing action (20 s), drying of the adhesive (5 s), and light curing (10 s)

bonded with the ER strategy associated or not with dentin pretreatment with EGCG and (2) there is no difference between the clinical performance of NCCL restorations bonded with the SE strategy associated or not with dentin pretreatment with EGCG.

METHODS AND MATERIALS

Study Design

This study was a blind, randomized clinical trial with an equal allocation rate among the four adhesive procedures under evaluation, and this report follows the Consolidated Standards of Reporting Trials statement.²⁴ In the control groups, the multimode adhesive was applied in the ER and SE strategies. In the experimental groups (ER-EGCG and SE-EGCG), a dentin pretreatment with a 0.1% w/v EGCG aqueous solution (Sigma-Aldrich, St Louis, MO, USA) was scrubbed on the teeth for 60 seconds, and then the adhesive system was used in the same ER (ER-EGCG) and SE (SE-EGCG) strategies. Table 1 summarizes the restorative materials and the application procedures used.

Patient Selection

Thirty-three volunteers (22 to 66 years old; 19 male and 14 female) were informed about the goals of the study and the risks and benefits to which they would

be exposed, were authorized to participate, and signed a written consent form. Inclusion criteria consisted of being over 18 years of age, having good oral hygiene and no periodontal disease or bruxism, and having at least four NCCLs and at least 20 teeth in occlusion. Exclusion criteria consisted of a previous cervical restorative procedure or caries lesions. All types of noncarious lesions (abrasion, erosion, and abfraction) were included, and all of them were nonretentive with no more than 50% of the margin in enamel and with the cervical margin in dentin.

Sample Size Calculation and Sample Randomization

The sample size was calculated based on the 96% retention rate of Single Bond Universal (3M ESPE, St Paul, MN, USA) in an 18-year clinical follow-up and considering a two-tailed test hypothesis.²⁵ At least 33 restorations per group were required to determine a 25% difference between groups with a significance level of 5% and a statistical power of 80%. A 20% increase in the sample size was set, considering possible loss during the study.

The assignment was done following one random list created using the website www.sealedenvelope.com, which indicated the control or experimental group for each tooth, without the patient's awareness.

Restorative Procedures

Each patient received at least one restoration per group for a total of 156 restorations. All NCCLs were classified according to cavity dimensions in millimeters (height, width, and depth), shape of the cavity (degree of the angle labeled as $<45^\circ$, 45° to 90° , 90° to 135° , or $>135^\circ$), degree of sclerotic dentin,²⁶ presence of incisal/occlusal wear facets, presence of preoperative sensitivity, and distribution among tooth types (Table 2).

Clinical photographs of the teeth prior to restoration were taken with a digital camera (Canon EOS 60D, Canon Macro EF 100-mm lens, Canon, Tokyo, Japan) in three positions: vestibular, proximal (mesial), and incisal/occlusal. Silicone rubber impressions with heavy- and light-body polyvinylsiloxane material (Adsil, Vigodent, Rio de Janeiro, Brazil) were obtained of each NCCL to make epoxy resin (MC130/FD154, Epoxyfiber, Rio de Janeiro, Brazil) replicas.²⁷

The same operator performed all restorations. Twenty-seven patients received four restorations, and six patients received eight restorations. No cavity preparation was carried out, enamel margins were not beveled, and no mechanical retention was placed. Local anesthesia with 1.8 mL of 2% lidocaine hydrochloride and phenylephrine 1:2500 (S.S. White 100, S.S.White, Petrópolis, Brazil), a labial retractor (Arc Flex, FGM, Joinville, Brazil), cotton rolls, and #00 retraction cord (Ultrapak, Ultradent, Indaiatuba, Brazil) were used for all cases. Prior to the restorative procedure, the NCCLs were cleaned with pumice and water (S.S.White), then rinsed and dried.

The adhesive system's application in the control groups (ER and SE) followed the ER and SE protocols suggested by their manufacturers (Table 1). The experimental groups (ER-EGCG and SE-EGCG) followed the same protocols but with a dentin pretreatment with a 0.1% EGCG aqueous solution (Sigma-Aldrich) scrubbed on the lesions for 60 seconds (Table 1). Increments of the resin composite Filtek Z350XT (3M ESPE) (up to three) were inserted and light cured individually for 20 seconds using the LED light-curing unit DB-685 (1100 mW/cm², Dabi Atlante, Ribeirão Preto, Brazil).

The initial finishing was performed with 12- and 30-fluted tungsten carbide burs (Microdont, São Paulo, Brazil) and F and FF diamond tips (KG Sorensen, São Paulo, Brazil). Polishing was performed with Sof-Lex Pop-On abrasive discs (3M ESPE). The initial baseline evaluation was per-

Table 2: *Noncarious Cervical Lesion (NCCL) Classification According to Shape, Cervico-Incisal Height, Degree of Sclerotic Dentin, Presence of Antagonist, Presence of Incisal/Occlusal Wear Facets, Presence of Preoperative Sensitivity, and Distribution Among Tooth Types*

Characteristics of NCCL	Number of Lesions			
	ER	ER-EGCG	SE	SE-EGCG
Shape (degree of angle)				
<45	1	2	1	3
45-90	7	10	5	4
90-135	20	20	24	19
>135	11	7	9	13
Cervico-incisal height (mm)				
<1.5	5	4	5	4
1.5-2.5	22	18	22	19
2.5-4.0	10	14	10	13
>4.0	2	3	2	3
Degree of sclerotic dentin				
1	17	20	23	22
2	19	14	12	12
3	3	5	4	5
4	0	0	0	0
Presence of incisal/occlusal wear facets				
Yes	32	31	26	33
No	7	8	13	6
Preoperative sensitivity (air dry)				
Yes	12	15	15	17
No	27	24	24	22
Preoperative sensitivity (spontaneous)				
Yes	7	7	6	6
No	32	32	33	33
Tooth distribution				
Anterior				
Incisors	10	9	10	8
Canines	6	3	7	2
Posterior				
Premolar	21	22	19	28
Molar	2	5	3	1
Arch distribution				
Maxillary	20	23	25	15
Mandibular	19	16	14	24
Abbreviations: ER, etch and rinse; ER-EGCG, etch and rinse + 0.1% EGCG pretreatment; SE, self-etch; SE-EGCG, self-etch + 0.1% EGCG pretreatment.				

formed one week later when another impression was taken of each restoration. New resin replicas from the restoration interface were obtained for observation under a scanning electron microscope (SEM) (Quanta FEG 450, FEI, Thermo Fisher Scientific, Hillsboro, OR, USA).

Clinical Evaluation

The study was blinded for the two independent, calibrated examiners who were responsible for the clinical evaluations. FDI World Dental Federation criteria^{28,29} were used to evaluate retention, the presence of fractures, marginal discoloration, marginal adaptation, caries, and postoperative sensitivity at baseline and after six, 12, 18, and 24 months. These variables were classified as follows: clinically very good, clinically good (scores 1+2), clinically sufficient/satisfactory (score 3), clinically unsatisfactory (score 4), and clinically poor (score 5).²⁹ The analysis was done independently and performed with a clinical mirror and dental explorer #5 (EXD56, Hufriedy, Rio de Janeiro, Brazil). In case of disagreement between the investigators, a consensus was reached by reexamination and discussion before the patient was dismissed.³⁰

The retention rates were calculated according to the following equation: cumulative failure percentage = $[(PF+NF)/(PF+RR)] \times 100\%$, where PF is the number of previous failures before the current assessment, NF is the number of new failures during the current evaluation, and RR is the number of restorations currently evaluated.²⁵

At all evaluation periods (baseline and six, 12, 18, and 24 months), impressions were taken from representative restorations from each group tested and each level score from marginal adaptation criteria.²⁷ All restorations were also photographed in two positions (vestibular and mesial).

Statistical Methods

The McNemar test was used for intragroup comparisons between the baseline and the other periods. The Kruskal-Wallis test was used for intergroup comparisons between each group in each period. The comparisons were performed for all criteria evaluated with a significance level of 5%. The Cohen kappa statistic was used to measure the agreement between examiners.

RESULTS

Figure 1 shows the patient's flow diagram. Thirty-eight out of 71 patients were not enrolled in the study because they did not fulfill the inclusion criteria. Thus, 33 subjects were selected, and the one hundred and fifty-six lesions were randomly assigned to one of the four experimental groups. Recall rates were 100% for baseline for six, 12, and 18 months. For 24 months, the recall rate was 96.9%,

with one patient not returning due to health problems. One patient was eliminated at 12 months and another at 18 months due to orthodontic treatment.

Table 3 presents data for retention/fractures, marginal staining, marginal adaptation, caries, and postoperative sensitivity at baseline and for all evaluation periods. The pooled (1+2) scores and score 3 present the total number of restorations classified as clinically acceptable. Scores 4 and 5 show the number of restorations considered clinically unsatisfactory. Figure 2 presents representative images of clinically satisfactory restorations from each group at baseline and at 24 months.

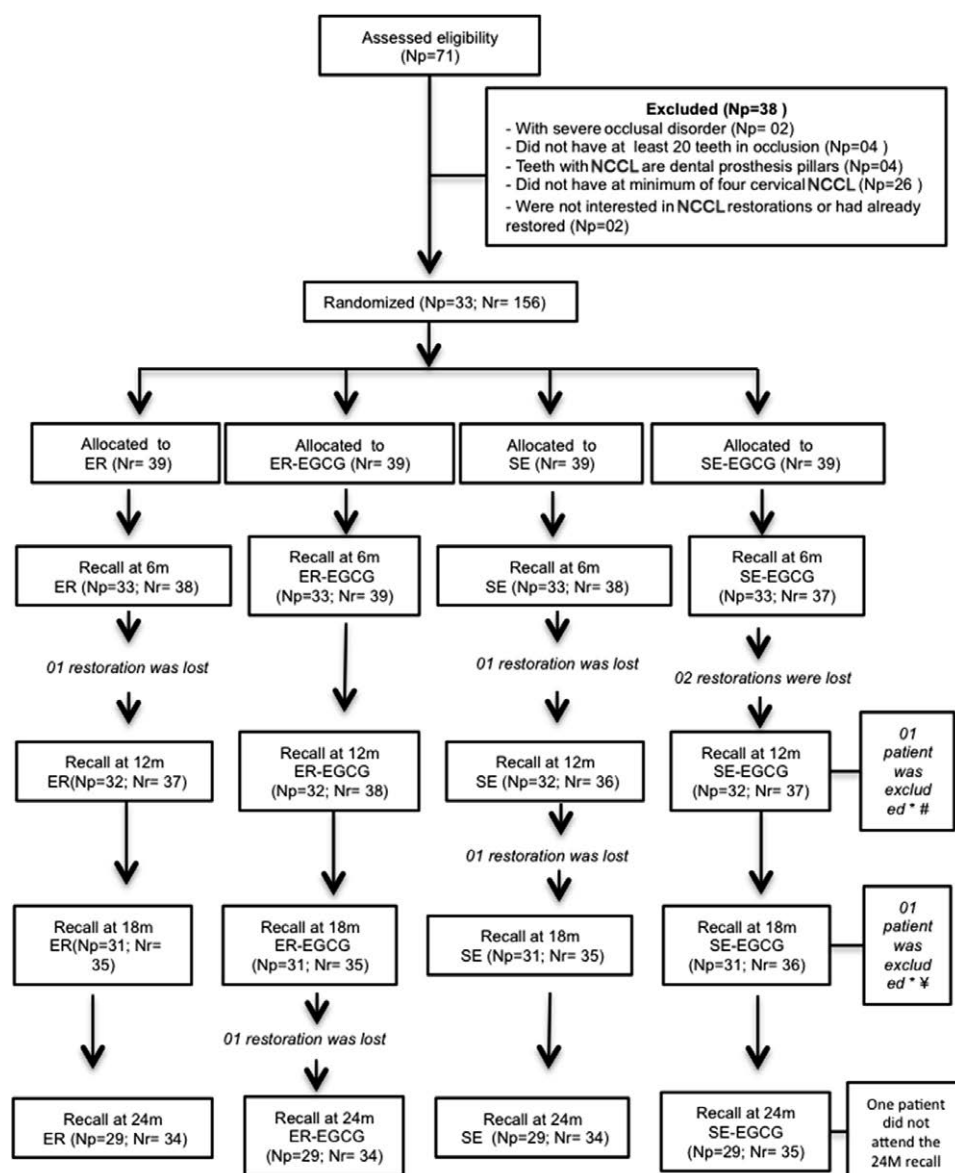
Four restorations were lost after six months, one restoration was lost at the 12-month recall, and one restoration was lost at the 18-month recall. Thus, a total of six restorations were lost by 24 months (one for ER, two for SE, one for ER-EGCG, and one for SE-EGCG). The 24-month retention rates were 97.1% for ER, 94.4% for SE, 97.1% for ER-EGCG, and 94.6% for SE-EGCG, with no statistical difference between any pair of groups at the 24-month recall and for each group when the baseline and 24-month results were compared ($p>0.05$; Table 3).

Two restorations had an acceptable material chip fracture (one for ER and one for SE), but it did not affect the marginal integrity score of 3. Six restorations had bulk fractures with partial loss for a score of 4 (two each for the SE, ER-EGCG, and SE-EGCG groups) (Figure 3).

Ten restorations were considered to have discrepancies in marginal adaptation at the 24-month recall. Half of them (one for ER and four for SE) were considered minor acceptable discrepancies, but another half (two for SE, one for ER-EGCG, and two for SE-EGCG) were considered unacceptable discrepancies, with severe material fracture (Figure 4). No significant difference was detected between any pair of groups at the 24-month recall ($p>0.05$), but a significant difference was detected when the baseline and 24-month data were compared within the SE group ($p=0.0313$) (Table 3).

Marginal staining was observed in seven restorations (one for SE, four for ER-EGCG, and two for SE-EGCG). Pronounced marginal staining was observed in only one restoration, which was in the SE-EGCG group. However, no significant difference was found between groups at 24 months and within each group when the baseline and 24-month findings were compared ($p>0.05$; Table 3).

Figure 1. Patient's flow diagram. Np, number of patients; Nr, number of restorations; NCCL, noncarious cervical lesion; ER, etch-and-rinse strategy; SE, self-etch strategy; EGCG, epigallocatechin-3-gallate; *, the patient was excluded due to orthodontic treatment; #, the excluded patient had already lost the restoration from the SE-EGCG group in the six-month recall; ¥, the excluded patient had eight noncarious cervical lesions restored and had already lost the restoration from the SE and SE-EGCG groups at the 12-month recall.



Three restorations had postoperative sensitivity one week after the restorative procedures (two for ER and one for SE). Two of them showed very intense sensitivity (score 4), but the sensitivity remained clinically acceptable at six, 12, and 18 months, and no restoration presented postoperative sensitivity at 24 months.

Finally, caries were not observed after 24 months of clinical service, and no tooth became nonvital as a result of the cervical restoration.

DISCUSSION

EGCG is the most abundant catechin found in green tea. It has recently been introduced into the dental field because of several promising benefits, such as

its antibacterial effect,³¹ protective effects for dental erosion,³² and effects in preserving the resin-dentin bond strength for up to six and 12 months associated with ER and SE adhesive systems, respectively, in laboratory studies.²⁰⁻²²

It has been suggested that EGCG could have a superior clinical application due to its positive effects on both mechanical properties and the stabilization of collagen against proteolytic degradation.²³ To the authors' knowledge, there are no clinical reports regarding the clinical performance of adhesive systems associated with EGCG. Thus, this randomized clinical trial evaluated a universal adhesive on different adhesive strategies, assessing the influence

Table 3: Number of Evaluated Restorations for Baseline and 6-, 12-, 18-, and 24-Mo Recalls According to the Experimental Groups^a

Criteria	Group	Baseline				6-Mo				12-Mo				18-Mo				24-Mo			
		Scores				Scores				Scores				Scores				Scores			
		1+2	3	4	5	1+2	3	4	5	1+2	3	4	5	1+2	3	4	5	1+2	3	4	5
Marginal staining	ER	39	—	—	—	37	1	—	—	36	1	—	—	35	—	—	—	34	—	—	—
	SE	39	—	—	—	38	—	—	—	36	—	—	—	35	—	—	—	33	1	—	—
	ER-EGCG	39	—	—	—	39	—	—	—	37	1	—	—	32	3	—	—	30	4	—	—
	SE-EGCG	39	—	—	—	37	—	—	—	36	1	—	—	35	1	—	—	33	1	1	—
Retention/fracture	ER	39	—	—	—	38	—	—	1	37	—	—	1	35	—	—	1	33	1	—	1
	SE	39	—	—	—	38	—	—	1	35	—	1	2	34	—	1	2	31	1	2	2
	ER-EGCG	39	—	—	—	39	—	—	—	37	—	1	—	34	—	1	1	32	—	2	1
	SE-EGCG	39	—	—	—	35	—	2	2	35	—	2	2	34	—	2	2	33	—	2	2
Marginal adaptation	ER	39	—	—	—	38	—	—	—	37	—	—	—	35	—	—	—	33	1	—	—
	SE	39 ^b	—	—	—	37	—	1	—	35	—	1	—	33	1	1	—	28 ^b	4	2	—
	ER-EGCG	39	—	—	—	39	—	—	—	37	—	1	—	34	—	1	—	33	—	1	—
	SE-EGCG	39	—	—	—	36	—	1	—	35	—	2	—	34	—	2	—	33	—	2	—
Caries	ER	39	—	—	—	38	—	—	—	37	—	—	—	35	—	—	—	34	—	—	—
	SE	39	—	—	—	38	—	—	—	36	—	—	—	35	—	—	—	34	—	—	—
	ER-EGCG	39	—	—	—	39	—	—	—	38	—	—	—	35	—	—	—	34	—	—	—
	SE-EGCG	39	—	—	—	37	—	—	—	37	—	—	—	36	—	—	—	35	—	—	—
Postoperative sensitivity	ER	37	1	1	—	38	—	—	—	36	1	—	—	34	1	—	—	34	—	—	—
	SE	38	—	1	—	38	—	—	—	34	2	—	—	35	—	—	—	34	—	—	—
	ER-EGCG	39	—	—	—	38	1	—	—	37	1	—	—	35	—	—	—	34	—	—	—
	SE-EGCG	39	—	—	—	36	1	—	—	37	—	—	—	36	—	—	—	35	—	—	—

Abbreviations: ER, etch and rinse; SE, self-etch; ER-EGCG, etch and rinse + 0.1% EGCG pretreatment; SE-EGCG, self-etch + 0.1% EGCG pretreatment.

^a Scores 1+2, clinically very good + clinically good; score 3, clinically sufficient/satisfactory; score 4, clinically unsatisfactory; score 5, clinically poor.^b Significant difference was detected when baseline and 24-mo data were compared within the SE group ($p=0.0313$).

of the dentin pretreatment with EGCG prior to the adhesive application.

After 24 months, there was no difference in the retention of NCCL restorations bonded with the ER or SE strategy, associated or not with dentin pretreatment with EGCG, which leads to failing to reject the null hypothesis.

In the current study, a 0.1% w/v EGCG aqueous solution was used as the dentin primer since it was the concentration with better laboratory results in preserving the bond strength over time.^{21,22} The one-minute application significantly improved the bond strength in research conducted by Zheng and Chen⁸ on MMP inhibitors. In the present clinical study, this time of application represented a feasible step to be reproduced in clinical practice.

The clinical efficacy of the four bonding procedures was evaluated by the retention of the restorations as a primary outcome.³ Retention in NCCLs cannot be associated with any other macromechanical retention, which can directly reflect the quality of the adhesive bonding.² In addition, the hypermineral-

ized dentin in NCCLs makes the bonding to this substrate very unpredictable and challenging for adhesive systems.³³

The high retention rates for all tested groups after 24 months showed that the bond effectiveness of the universal adhesive did not depend on the bonding strategy, in accordance with previous randomized clinical trials.^{25,34-37} This retention rate result may be explained by the Single Bond Universal (SU) (3M ESPE) chemical bond mechanism. SU contains 10-methacryloxydecyl dihydrogen phosphate (10-MDP), an acidic phosphate monomer capable of bonding to hydroxyapatite, producing adhesive interfaces with different chemical and morphological characteristics. It also contains a polyalkenoic acid copolymer.²⁵ These two processes involve micromechanical and chemical bonding to the enamel and dentin, which may have had a direct impact on the retention rate and clinical behavior of SU in the current study after 24 months.

The retention results also showed that the EGCG priming solution did not impair the bond effective-

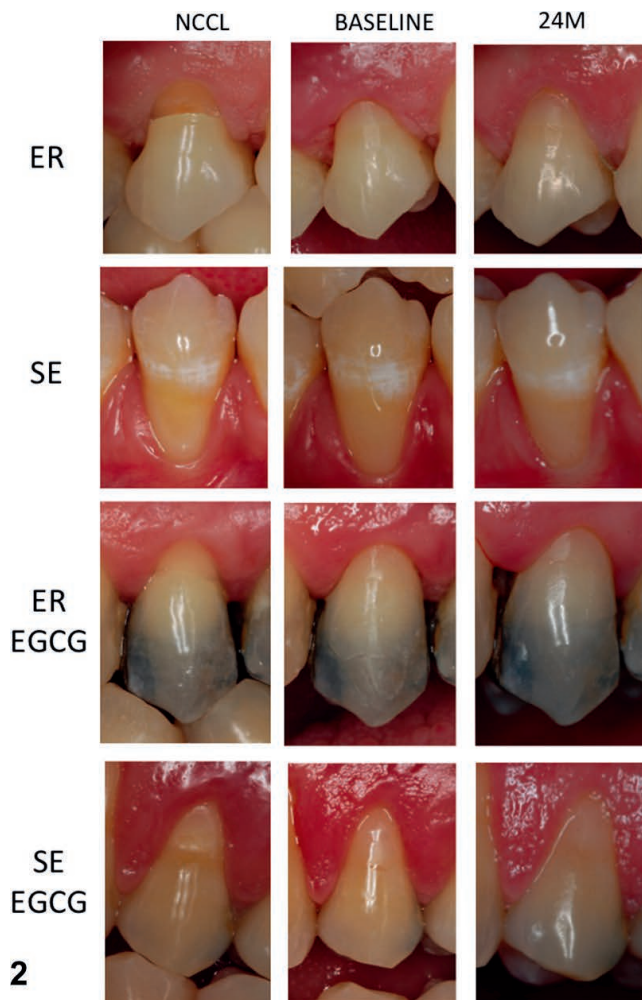
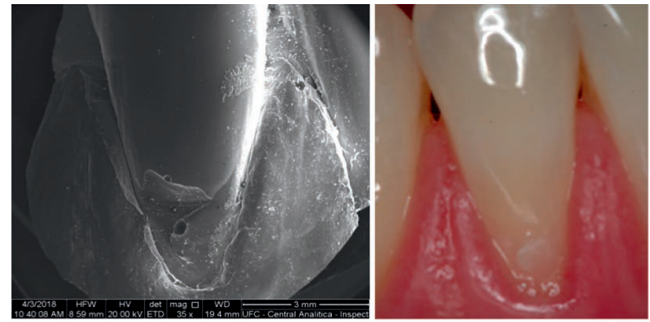


Figure 2. Noncarious cervical lesions from each group and clinically satisfactory restorations at baseline and 24 months.

ness and can be associated with both adhesive strategies as reported only by laboratory studies.^{21,22} Unlike chlorhexidine, which showed a negative association with a self-etching adhesive,³⁸ this result highlighted the versatile clinical application of EGCG as a dentin primer in NCCLs.

The scoring system used for the evaluation of the NCCL restorations was the FDI criteria.²⁹ Four specific secondary outcomes were selected from the total of 16 original parameters. The inclusion of “marginal staining” and “marginal adaptation” was an important measure since they are still shortcomings mainly of cervical restorations and it has been indicated that they may be predictors of future failures.³⁹ Also, a simplified clinical evaluation was chosen pooling scores 1 and 2 (equivalent to score A from the US Public Health Service criteria);³⁰ thus, the FDI criteria were expressed with only four scores: two for acceptable (1+2 and 3) and two for

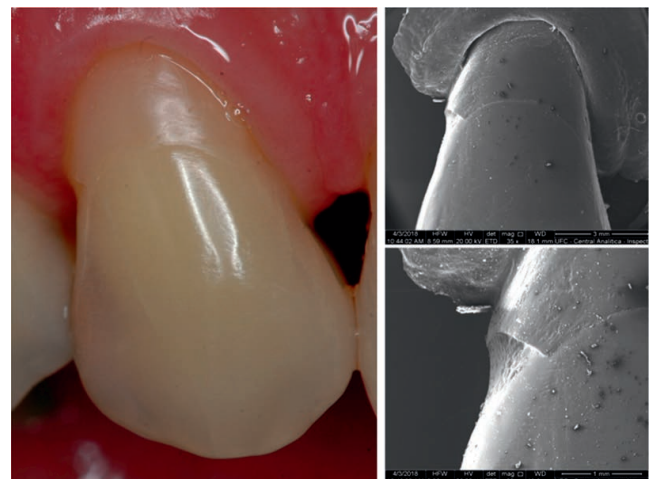


3

Figure 3. On the left, the scanning electron photomicrograph of resin epoxy replicas from the resin fracture. On the right, clinical photograph from the same resin restoration rated as clinically unsatisfactory but repairable (material fracture score 4) at the 24-month recall.

nonacceptable (4 and 5) for repairable and replacement, respectively.^{1,29}

For marginal adaptation, a significant difference was detected when baseline and 24-month data were compared within the SE group ($p=0.0313$). SU is considered an ultramild SE adhesive ($\text{pH}\sim 3.0$), and, although there is no consensus that pre-etching enamel improves the bond strength of universal adhesives,^{40,41} this result may be related to the limited micromechanical retention of the enamel surface conditioned by the SE mode. Surprisingly, the association of EGCG with the SE strategy did not jeopardize the marginal adaptation, and future evaluations could confirm a possible beneficial effect of EGCG priming on enamel.



4

Figure 4. On the left, a clinical photograph of resin restoration (self-etch group) rated as clinically unsatisfactory but repairable (marginal adaptation score 4) at the 24-month recall. On the right, scanning electron photomicrographs of resin epoxy replicas from the same restoration (superior right) and an approximated view (inferior right) from the resin fracture.

After 24 months, five restorations were rated as score 4 (two for SE, one for ER-EGCG, and two for SE-EGCG) with unacceptable discrepancies and severe material fractures. A prophylactic repair was indicated to recover the restoration integrity, avoiding premature replacement of the restoration.²⁹ These restorations can be viewed as “relative failures,” and this result may be associated with heavy occlusal or parafunctional forces since all teeth presented signs of occlusal wear (data not shown). A retrospective clinical study²⁷ showed that class V restorations of teeth with occlusal wear facets had a 6.65-fold increased risk of failure than those of teeth without wear facets. Clinical covariables shown in Table 2 could determine the bonding effectiveness, such as shape and size of the lesion, degree of sclerosis, presence of wear facets, patient age, and tooth type.

The restorations that were rated unsatisfactory were additionally investigated using digital photographs and epoxy resin replicas under SEM evaluation.²⁷ The replicas enabled indirect observation and were useful in analyzing changes in discrepancies and/or material fracture in restorations at different periods. This procedure was helpful in determining the need for timely repair or replacement.

Pronounced marginal staining was observed in only one restoration, which was in the SE-EGCG group, and it was associated with a color change along the whole of the restoration margin due to smoking habits and not to the marginal discrepancy.

A meta-analysis and trial sequential analysis⁴² evaluated randomized clinical trials comparing degradation inhibitory cavity pretreatment (CHX and quaternary ammonium) prior to the adhesive placement of the resin-based restorations. The authors concluded that there is insufficient evidence to recommend or refute degradation inhibitory cavity pretreatment prior to adhesively placing resin-based restorations. These findings corroborate the current results regarding EGCG since its effects (protease inhibition and cross-linking ability in dentin)²³ could not be proven by this trial. A longer follow-up could be required to validate these experimental strategies aiming to enhance the adhesive interface.

CONCLUSIONS

At 24 months, dentin pretreatment with EGCG did not provide any positive benefit in the clinical performance of the adhesive SU regardless of the bonding strategy used. In addition, including cate-

chin placement prebonding results in an additional required step in the restorative procedure.

Acknowledgements

This investigation was supported by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior–Brasil (CAPES) Finance Code 001 and (# PDSE 99999.006483/2015-01), from whom the first author received a scholarship. The authors also would like to thank the Central Analítica UFC/CT-INFRA/MCTI-SISNANO/Pró-Equipamentos CAPES for technical support.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee on Investigations involving human subjects of the Federal University of Ceará. The approval code issued for this study is 1.292.593.

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 22 October 2019)

REFERENCES

- Opdam NJM, Collares K, Hickel R, Bayne SC, Loomans BA, Cenci MS, Lynch CD, Correa MB, Demarco F, Schwendicke F, & Wilson NHF (2018) Clinical studies in restorative dentistry: New directions and new demands *Dental Materials* **34**(1) 1-12.
- 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.
- Van Meerbeek B, Pneumans M, Poitevin A, Mine A, Van Ende A, & Neves A (2010) Relationship between bond-strength tests and clinical outcomes *Dental Materials* **26**(2) 100-121.
- Tjäderhane L, Nascimento FD, Breschi L, Mazzoni A, Tersariol IL, Geraldeli S, Tezvergil-Mutluay A, Carrilho MR, Carvalho RM, Tay FR, & Pashley DH (2013) Optimizing dentin bond durability: Control of collagen degradation by matrix metalloproteinases and cysteine cathepsins *Dental Materials* **29**(1) 116-135.
- Reis A, Carrilho M, Breschi L, & Loguercio AD (2013) Overview of clinical alternatives to minimize the degradation of the resin-dentin bonds *Operative Dentistry* **38**(4) E1-E25.
- Pashley DH, Tay FR, Yiu C, Hashimoto M, Breschi L, Carvalho RM, & Ito S (2004) Collagen degradation by host-derived enzymes during aging *Journal of Dental Research* **83**(3) 216-221.
- Montagner AF, Sarkis-Onofre R, Pereira-Cenci T, & Cenci MS (2014) MMP inhibitors on dentin stability: A systematic review and meta-analysis *Journal of Dental Research* **93**(8) 733-743.

- month follow-up of cervical composite restorations placed with a new universal adhesive system: A randomized clinical trial *Operative Dentistry* **41**(5) 465-480.
36. Ruschel VC, Shibata S, Stolf SC, Chung Y, Baratieri LN, Heymann HO, & Walter R (2018) Eighteen-month clinical study of universal adhesives in noncarious cervical lesions *Operative Dentistry* **43**(3) 241-249.
37. Loguercio AD, de Paula EA, Hass V, Luque-Martinez I, Reis A, & Perdigão J (2015) A new universal simplified adhesive: 36-month randomized double-blind clinical trial *Journal of Dentistry* **43**(9) 1083-1092.
38. Costa CAGA, Passos VF, Neri JR, Mendonça JS, & Santiago SL (2019) Effect of metalloproteinase inhibitors on bond strength of a self-etching adhesive on erosively demineralized dentin *Journal of Adhesive Dentistry* **21**(4) 337-344.
39. Hayashi M & Wilson NHF (2003) Marginal deterioration as a predictor of failure of a posterior composite *European Journal of Oral Sciences* **111**(2) 155-162.
40. Rosa WL, Piva E, & Silva AF (2015) Bond strength of universal adhesives: A systematic review and meta-analysis *Journal of Dentistry* **43**(7) 765-776.
41. Kaczor K, Gerula-Szymańska A, Smektała T, Safranow K, Lewusz K, & Nowicka A (2018) Effects of different etching modes on the nanoleakage of universal adhesives: A systematic review and meta-analysis *Journal of Esthetic and Restorative Dentistry* **30**(4) 287-298.
42. Göstemeyer G & Schwendicke F. (2016) Inhibition of hybrid layer degradation by cavity pretreatment: Meta- and trial sequential analysis *Journal of Dentistry* **49** 14-21.

Literature Review

Is Conventional Resin Cement Adhesive Performance to Dentin Better Than Self-adhesive? A Systematic Review and Meta-Analysis of Laboratory Studies

LL Miotti • AC Follak • AF Montagner • RT Pozzobon • BL da Silveira • AH Susin

Clinical Relevance

Conventional multistep resin cements presented higher adhesive performance to dentin than simplified self-adhesive cements, which is an important criterion for indirect restoration prognosis.

SUMMARY

This study aimed to conduct a systematic review of the literature on laboratory studies assessing bonding performance to dentin of conventional and self-adhesive resin cements, in cementing indirect restorations. This review was reported according to the PRISMA Statement. Of a total of 518 studies, 36 were screened full text and reviewed according to exclusion criteria. Nineteen papers were included in the systematic review and meta-

analyses, according to the following inclusion criteria: studies that evaluated the bond strength to dentin of indirect restorations cemented with dual conventional or self-adhesive resin cements and those that presented bond strength data in MPa as an outcome. Statistical analyses were conducted using RevMan 5.1. Comparisons were performed with random effects models at 5% significance level. A global analysis comparing conventional and self-adhesive cements and three subgroup analyses comparing immediate and long-term

*Leonardo Lamberti Miotti, DDS, MS, PhD, Department of Restorative Dentistry, School of Dentistry, Federal University of Santa Maria, Santa Maria, Brazil

Andressa Cargnelutti Follak, DDS, MS, PhD, Department of Restorative Dentistry, School of Dentistry, Federal University of Santa Maria, Santa Maria, Brazil

Anelise Fernandes Montagner, DDS, MS, PhD, School of Dentistry, Federal University of Pelotas, Pelotas, Brazil

Roselaine Terezinha Pozzobon, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Federal University of Santa Maria, Santa Maria, Brazil

Bruno Lopes da Silveira, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Federal University of Santa Maria, Santa Maria, Brazil

Alexandre Henrique Susin, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Federal University of Santa Maria, Santa Maria, Brazil

*Corresponding author: Carlos Brenner Street, 130, Nossa Senhora de Lourdes, Santa Maria, RS 97050-100, Brazil; e-mail: llmiotti@gmail.com

<https://doi.org/10.2341/19-153-L>

results were performed. Global analysis showed a difference between groups, with conventional resin cements presenting higher bond strength results than self-adhesive resin cements, in immediate and long-term time periods (both $p=0.03$). Immediate and long-term bond strength results were different for self-adhesive cements, favoring immediate bond strength ($p=0.03$), but immediate and long-term bond strength results for conventional resin cements were not different ($p=0.06$). Medium or high risk of bias was found in all studies. Conventional multistep resin cements showed superior overall adhesive performance compared with simplified self-adhesive resin cements when used to cement indirect restorations to dentin.

INTRODUCTION

Adhesive dentistry has developed to the point of allowing restorative procedures to be performed with minimal tooth preparation, thereby contributing toward preserving the dental substrate. This is an important factor affecting restoration longevity.¹ Resinous luting agents have improved indirect restorative treatments, by promoting better retention and lower solubility, thereby reinforcing restoration and tooth structure.^{2,3} The first dual resin cements, called “conventional resin cements” or “regular cements,”⁴ were developed as systems, containing the resinous luting agent and a specific adhesive system or primer, used as an adhesion agent. The adhesive strategy for these conventional resin cements can use etch-and-rinse or self-etch approaches, depending on the adhesion agent.

The concept of self-adhesive luting agents was conceived in the early 2000s as a new class of resin cement capable of combining adhesive and cement in a single application, without requiring pretreatment of the dental substrate and the restoration surface.⁵ By mixing adhesive and cement in a single formulation, these cements promote simultaneous demineralization and substrate penetration. The simplified technique results in fewer operator-sensitive steps during the procedure compared with the traditional, conventional multistep resin cements.⁶ The luting agent class is considered a heterogeneous group of resin cements, with substantial differences in terms of composition, setting reaction, pH, and bonding performance.⁷

Dentin adhesion remains a concern regarding long-term retention. The adhesive interface can be considered the weakest link of the adhesion chain

and may jeopardize restorative longevity.⁸ Studies assessing dual resin cement adhesion to dentin are controversial. Some laboratory investigations assert that conventional multistep resinous luting agents promote higher and more stable adhesion to dentin,^{2,8-10} whereas others indicate that self-adhesive resin cements provide adequate bonding performance.¹¹⁻¹³ Hitz and others¹⁴ also stated that not all self-adhesive resin cements could be considered valid alternatives to conventional multistep resin cements, because chemical formulation is crucial to their performance.

Considering that opinions may diverge, the present study aimed to make a systematic review of the available scientific literature for laboratory studies that assessed the bonding performance of conventional and self-adhesive resin cements to unaltered dentin, when cementing indirect restorations. The null hypothesis was that there would be no difference in bonding strength between the two classes of resin cements tested, even regarding long-term bond strength.

METHODS

The present systematic review was conducted according to the Cochrane Handbook for systematic reviews of interventions¹⁵ and reported according to the PRISMA statement.¹⁶ A research question was defined to guide the literature search strategy and establish the variables analyzed in the meta-analysis: “Does the dual resinous cement type (conventional or self-adhesive) influence immediate and long-term bond strength to dentin when used to cement an indirect restoration to dentin?”

Search Strategy

The following electronic databases were used to search and identify studies that could be included in the present systematic review: MEDLINE (National Library of Medicine) via PubMed, Scopus, and ISI Web of Science (Core Collection). A computer search of databases and reference lists of the studies to be included were the search strategies used in this review. All the studies included were published before January 9, 2018. No publication year or language limit was applied.

The MEDLINE search via PubMed was performed with MeSH terms and keywords, as follows: “(((((((dentin[MeSH Terms]) OR dentin) OR dentins) OR dentine) OR dentines)) AND (((((bond strength*) OR microtensile bond strength) OR microshear bond strength) OR microtensile) OR microshear)) AND

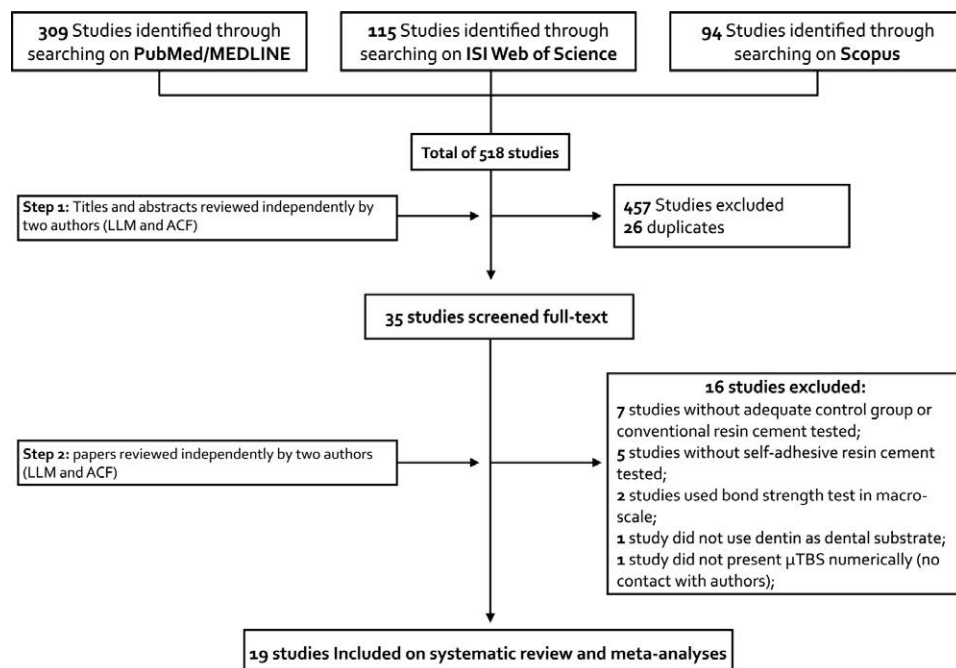


Figure 1. Flowchart of study selection according to PRISMA statement.

machine. Each parameter received the following score, based on its description in the article: 1, if the parameter was correctly described in the text; 0, if the information was not described in the text. The risk of bias classification was calculated according to the sum of 1 received by the study: 1-2 = high risk, 3-5 = medium risk, and 6-7 = low risk of bias.

Data Analysis

The pooled effect estimates for the meta-analysis were obtained by comparing the bond strength means with the conventional and the self-adhesive resin cements in the global analysis and in the three different subgroup analyses, according to use of an aging method before the bond strength test (immediate or long term). Each cement tested according to inclusion and exclusion criteria was considered an independent test group. Studies that reported immediate and long-term results were analyzed in all the three subgroups. No aging methods or time points were considered; the only consideration was if the specimens were submitted to an aging process. When called for, specific formulas were applied to combine multiple values into single sample sizes, mean values, and SDs for groups within a given study.¹⁵

Statistical heterogeneity between studies was evaluated by the Cochran Q test, with a threshold p value of 0.1 and inconsistency of I^2 , where values $>50\%$ were considered indicative of high heterogeneity.¹⁵ All analyses were performed with the

Review Manager (RevMan version 5.3 software 2014, Cochrane Collaboration, Copenhagen, Denmark), using the random effects method. The number of specimens was considered as the number of experimental units; $p \leq 0.05$ was considered statistically significant (Z-test). Forest plots were constructed to illustrate the meta-analysis calculation.

RESULTS

Search and Selection

Figure 1 shows a flowchart describing the study selection process, according to the PRISMA statement.¹⁶ The search strategy resulted in 518 potentially eligible studies, with 309 papers in MEDLINE via PubMed, 94 papers in Scopus, and 115 papers in ISI Web of Science. Of these, 457 were excluded for not meeting the inclusion criteria, and 26 were duplicates. The first screening resulted in 35 studies singled out for further full-text evaluation. According to the exclusion criteria, 16 studies were excluded: two presented a bond strength test on a macro scale^{19,20}; seven did not present an adequate control group or did not test a conventional resin cement²¹⁻²⁷; five studies did not assess a self-adhesive resin cement²⁸⁻³²; one study did not mention the dentin substrate³³; and one study did not present the microtensile bond strength (μ TBS) numerically.³⁴ After applying the exclusion criteria, 19 studies were included for systematic review and meta-analysis.

The meta-analysis included studies that were published between 2009 and 2016. Most papers used human molars. Only two studies used bovine incisors,^{4,13} and another two studies used premolars with inlay preparations.^{9,35} In the collection of included articles, the most frequently assessed conventional resin cements were RelyX ARC etch-and-rinse^{4,8-10,12,36-41} and Panavia F2.0 self-etch.^{7,11,35,38-44} The most commonly assessed self-adhesive resin cements were RelyX Unicem,^{7,10-13,35,36,38,41-44} followed by RelyX U100^{4,8,9,37,45} and G-cem.^{10,11,41,43,44}

The methodologic test used in all the 19 selected studies was μ TBS, with nontrimming beams. Most studies presented long-term bond strength results,^{7-9,11,12,35-40,42} with different aging methods: five studies^{9,35-37,40} performed mechanical cycling, with a number of cycles ranging from 50,000³⁶ up to 2,000,000,³⁷ loading from 50⁴⁰ to 100 N,³⁷ and frequency from 1³⁶ to 4 Hz^{9,37}; two studies stored specimens in artificial saliva^{8,36} and two stored them in distilled water,^{12,38} with a storage time ranging from 1¹² to 24 months³⁶; three studies performed thermocycling,^{7,11,42} all with the same parameters (5000 cycles, 5°C-55°C); and two studies used a simulated pulpal pressure aging design^{39,40} for periods of 7 days⁴⁰ and 3 months.³⁹ Two papers^{36,40} performed more than one aging method, but the specimens in both studies were submitted to only one aging method. The complete descriptive data per study in this collection is presented in Table 1.

Meta-analyses

The data from the 19 included studies were submitted to a meta-analysis calculation. The meta-analyses were conducted considering a global analysis (conventional vs self-adhesive resin cements; Figure 2) and three subgroup analyses: 1, conventional vs self-adhesive cements, considering only the after-aging results (Figure 3); 2, immediate vs aging bond strength of self-adhesive cements (Figure 4); and 3, immediate vs aging bond strength of conventional resin cements (Figure 5).

The global meta-analysis results are presented in Figure 2. In this analysis, heterogeneity (I^2 test) was 99%, and Cochran Q and Z ($p=0.03$) tests were <0.05 , thus corroborating the control group results (conventional resin cements). Considering the immediate and after-aging results pooled, the conventional resin cements showed better bond strength results than those of the self-adhesive resin cements. The first subgroup analysis (conventional vs self-adhesive after aging) presented Cochran Q and Z

($p=0.03$) tests <0.05 , with $I^2=99\%$, showing that conventional resin cements had better bond strength results than self-adhesive resin cements (Figure 3). Subgroup analyses 2 and 3, which analyzed both resin cement types separately, showed different results. Subgroup analysis 3 (Figure 4) showed a difference between immediate and long-term bond strength results for self-adhesive cements, favoring immediate bond strength, which showed higher bond strength values than the aged group ($p=0.03$, $I^2=99\%$). On the other hand, the subgroup analysis for conventional resin cements (Figure 5) indicated the Cochran Q test as <0.05 . However, the Z test was >0.05 ($p=0.06$), thus indicating no difference between the immediate and the aged groups ($I^2=98\%$).

Risk of Bias

Medium or high risk of bias was found in all the studies included (Table 2). In the present collection of articles, three took tooth dimensions into consideration,^{4,35,37} one had the same operator for the adhesive technique,⁷ two calculated the sample size,^{35,38} and two had a blinded operator for the adhesive test.^{4,7}

DISCUSSION

The present systematic review and meta-analysis established the pooled effect of data from laboratory studies that assessed the adhesion performance to dentin of two different resin cement types: conventional and self-adhesive. This study provides the first step to guide the decision-making process for selecting the most reliable adhesive strategy for cementing indirect coronal restorations. The overall results showed that the conventional adhesive approach, whereby the resin cement is applied in combination with an adhesive system or primer agent, tends to promote higher immediate- and long-term bond strength to dentin. The null hypothesis of the present study was rejected, because conventional multistep resin cements presented higher overall and long-term bond strength results.

The conventional class of resin cements was the first adhesive resinous luting agent to use an intermediate adhesion agent (adhesive system or specific primer) between the restorative material and the dental substrate. In the present collection of studies, the two most frequently assessed conventional resin cements were RelyX ARC (3M-ESPE, St Paul, MN, USA) and Panavia F2.0 (Kuraray Medical Inc, Okayama, Japan), etch-and-rinse and self-etch resin cements, respectively. Both systems were

Table 1: Descriptive Data From Studies Included in Meta-analyses

Paper	Type of Teeth	Restorative Material	Aging Method	Conventional Cements	Self-adhesive Cements
D'Arcangelo and others 2009 ⁴²	Human	Resin composite/ceramic	Thermocycling (5000 cycles, 5-55°C)	CoreXFlow (Dentsply DeTey, York, PA, USA) Enacem HF (Micerium, Aveno, GE, Italy) Panavia F2.0 (Kuraray, Chiyoda, Tokyo, Japan)	RelyX Unicem (3M ESPE, St. Paul, MN, USA)
Aguiar and others 2010 ⁴³	Human	Resin composite	No aging	Panavia F2.0 (Kuraray)	RelyX Unicem (3M ESPE) BisCem (Bisco Schaumburg, IL, USA) G-CEM (GC, Tokyo, Japan)
Sarr and others 2010 ⁴⁴	Human	Ceramic	No aging	Panavia F2.0 (Kuraray) Clearfil Esthetic cement (Kuraray) Calibra (Dentsply) Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein)	RelyX Unicem (3M ESPE) MaxCem (Kerr, Brea, CA, USA) MonoCem (Shofu, Kyoto, Japan) G-CEM (GC, Tokyo, Japan) Multilink Sprint (Ivoclar Vivadent)
Guarda and others 2010 ¹³	Bovine	Ceramic	No aging	Variolink II (Ivoclar Vivadent)	RelyX Unicem (3M ESPE)
De Angelis and others 2011 ⁷	Human	Resin composite/ceramic	Thermocycling (5000 cycles, 5-55°C)	Enacem HF (Micerium, Aveno) Panavia F2.0 (Kuraray)	RelyX Unicem (3M ESPE) MaxCem Elite (Kerr) iCEM Self-adhesive (Heareus Kulzer, Hanau, Germany)
Inukai and others 2012 ³⁵	Human	Resin Composite	Mechanical cycling (250,000 cycles, 100 N, 4 Hz)	Panavia F2.0 (Kuraray)	RelyX Unicem (3M ESPE) Clearfil SA cement (Kuraray)
Fuentes and others 2013 ⁴¹	Human	Resin composite	No aging	RelyX ARC (3M ESPE)	RelyX Unicem (3M ESPE) G-CEM (GC) MaxCem Elite (Kerr)
Suzuki and others 2013 ³⁸	Human	Resin composite	6-mo water storage	RelyX ARC (3M ESPE) Panavia F (Kuraray)	RelyX Unicem (3M ESPE)
Aguiar and others 2014 ³⁶¹	Human	Resin composite	Mechanical cycling (50,000 cycles, 80 N, 1 Hz) 12- and 24-mo storage in artificial saliva	RelyX ARC (3M ESPE) Clearfil esthetic cement (Kuraray)	RelyX Unicem (3M ESPE) Clearfil SA Cement (Kuraray)
Feitosa and others 2014 ³⁷	Human	Ceramic	Mechanical cycling (2,000,000 cycles, 100 N, 4 Hz)	RelyX ARC (3M ESPE)	RelyX U100 (3M ESPE)
Rigolin and others 2014 ⁴⁵	Human	Ceramic	No aging	Variolink II (Ivoclar Vivadent) Multilink (Ivoclar Vivadent)	RelyX U100 (3M ESPE)
Prochnow and others 2014 ⁹	Human	Resin composite	Mechanical cycling (1,000,000 cycles, 88 N, 4 Hz)	RelyX ARC (3M ESPE)	RelyX U100 (3M ESPE)
Vaz and others 2012 ¹²	Human	Resin Composite	1-mo storage in artificial saliva	RelyX ARC (3M ESPE) C&B cement (Bisco)	RelyX Unicem (3M ESPE)
Stape and others 2014 ⁸	Human	Resin composite	24-mo storage in artificial saliva	RelyX ARC (3M ESPE)	RelyX U100 (3M ESPE)
Giannini and others 2015 ¹¹	Human	Resin composite	Thermocycling (5000 cycles, 5-55°C)	Panavia F2.0 (Kuraray)	RelyX Unicem (3M ESPE) RelyX Unicem 2 (3M ESPE) Clearfil SA Cement (Kuraray) G-CEM (GC)
Skupien and others 2015 ⁴	Bovine	Resin composite	No aging	RelyX ARC (3M ESPE)	RelyX U100 (3M ESPE)

Table 1: Continued.					
Paper	Type of Teeth	Restorative Material	Aging Method	Conventional Cements	Self-adhesive Cements
Bacchi and others 2015 ³⁹	Human	Resin composite	3-mo simulated pulpal pressure	RelyX ARC (3M ESPE) Panavia F2.0 (Kuraray)	RelyX U200 (3M ESPE)
Bacchi and others 2015 ^{40a}	Human	Resin composite	7-day simulated pulpal pressure Mechanical cycling (200,000 cycles, 50 N, 2 Hz) Thermocycling (5000 cycles, 5-55°C)	RelyX ARC (3M ESPE) Panavia F2.0 (Kuraray)	RelyX U200 (3M ESPE)
Fuentes and others 2016 ¹⁰	Human	Resin composite	No aging	RelyX ARC (3M ESPE)	RelyX Unicem (3M ESPE) G-CEM (GC) Speedcem (Ivoclar Vivadent) MaxCem Elite (Kerr) Smartcem2 (Dentsply)
All studies used microtensile bond strength (μ TBS) as the bond strength test. ^a These studies used more than one aging method in the same specimens.					

tested with different adhesion agents, as follows: RelyX ARC was used together with Adper Single-bond^{4,8-10,12,36-41} (3M-ESPE) and Panavia 2.0 was used with ED primer (Kuraray Medical Inc), a self-etch primer included in the Panavia F2.0 system.^{7,11,35,38-40,42-44} Our review findings are corroborated by studies like that of Prochnow and others¹⁰ and Fuentes and others,⁴¹ in which RelyX ARC was the conventional resin cement, and that of Aguiar

and others⁴³ and Sarr and others,⁴⁴ in which Panavia F2.0 was tested. Despite the adhesive strategy, etch-and-rinse and self-etch conventional multistep dual resin cements seem to promote higher immediate- and long-term dentin bond strength than simplified self-adhesive cements.^{9,10,39,43,44}

The results for immediate- and long-term bond strength revealed that conventional multistep resin

Table 2: Risk of Bias Assessment of the Studies Included								
Study	Random	Caries	Materials	Teeth	Adhesive	Sample	Blinding	Risk
D'Arcangelo and others 2009 ⁴²	Y	Y	N	N	N*	N	N*	High
Aguiar and others 2010 ⁴³	Y	Y	Y	N	N*	N	N*	Medium
Sarr and others 2010 ⁴⁴	N	Y	Y	N	N*	N	N*	High
Guarda and others 2010 ¹³	Y	Y	Y	N	N*	N	N*	Medium
De Angelis and others 2011 ⁷	N	Y	N	N	Y	N	Y	Medium
Inukai and others 2012 ³⁵	Y	Y	N	Y	N*	Y	N*	Medium
Fuentes and others 2013 ⁴¹	Y	Y	Y	N	N*	N	N*	Medium
Suzuki and others 2013 ³⁸	N	Y	N	N	N*	Y	N*	High
Aguiar and others 2014 ³⁶	N	Y	Y	N	N*	N	N*	High
Feitosa and others 2014 ³⁷	Y	Y	N	Y	N*	N	N*	Medium
Rigolin and others 2014 ⁴⁵	Y	Y	Y	N	N*	N	N*	Medium
Prochnow and others 2014 ⁹	Y	Y	Y	N	N*	N	N*	Medium
Vaz and others 2012 ¹²	N	Y	Y	N	N*	N	N*	High
Stape and others 2014 ⁸	Y	Y	N	N	N*	N	N*	High
Giannini and others 2015 ¹¹	N	Y	Y	N	N*	N	N*	High
Skupien and others 2015 ⁴	Y	Y	Y	Y	N*	N	Y	Medium
Bacchi and others 2015 ³⁹	Y	Y	Y	N	N*	N	N*	Medium
Bacchi and others 2015 ⁴⁰	Y	Y	Y	N	N*	N	N*	Medium
Fuentes and others 2016 ¹⁰	Y	Y	Y	N	N*	N	N*	Medium
Abbreviations: N, no; Y, yes * not mentioned by authors								

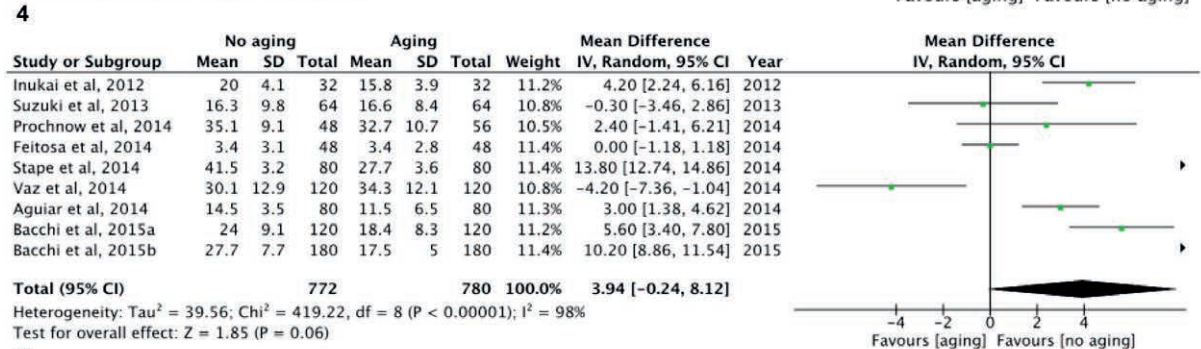
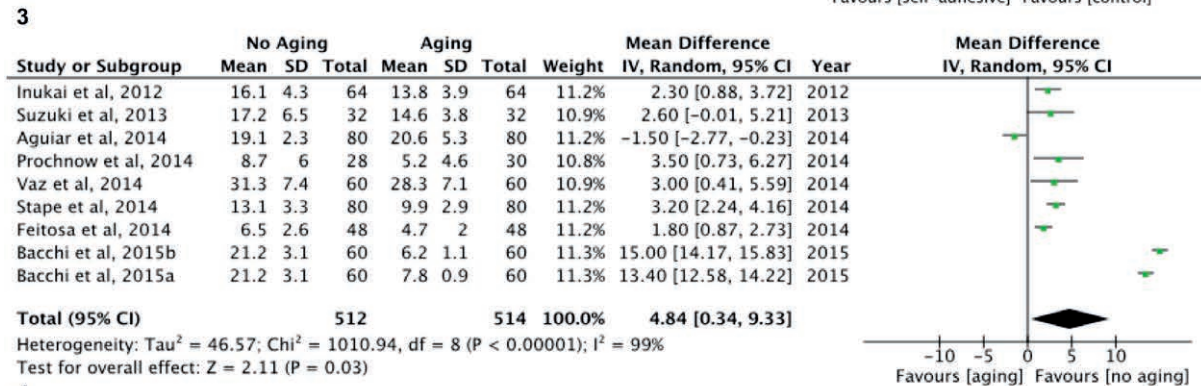
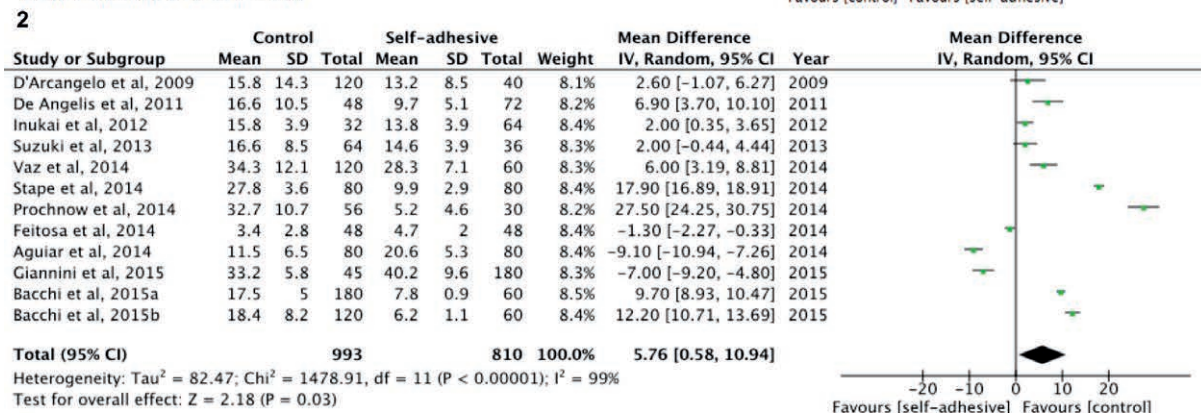
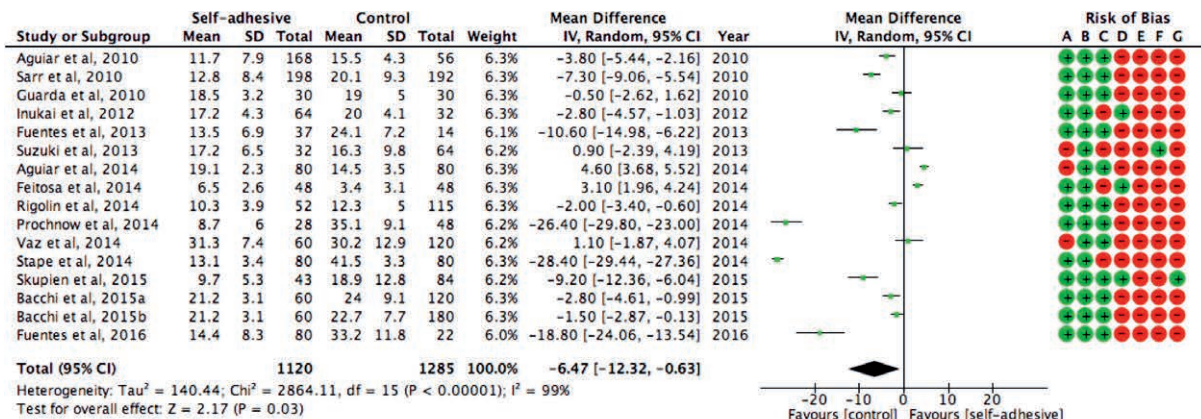


Figure 2. Global analysis comparing conventional (control) vs self-adhesive resin cements.

Figure 3. First subgroup analysis: conventional (control) vs self-adhesive resin cements after aging.

Figure 4. Second subgroup analysis: self-adhesive cement performance comparing immediate vs long-term results.

Figure 5. Third subgroup analysis: conventional resin cement performance comparing immediate vs long-term results.

cements did not show any significant difference between immediate and aged bond strength data, considering that the latter indicates a more stable long-term adhesion. Exposed dentinal collagen fibrils are subject to degradation mechanisms, like hydrolysis by water, mechanical fatigue, denaturation, or even a collagenolytic process derived from matrix metalloproteinases and cathepsins, inherently present in dentin.⁴⁶ The ability to envelop exposed collagen fibrils is directly dependent on priming steps, wetting characteristics of the bonding agent, and its chemical composition.¹² Although conventional resin cements are more technique sensitive, because they need a specific bond agent, these cements are more capable of interpenetrating the demineralized dentin substrate.

The mechanism of adhesion to dentin is an important issue that should be understood to explain the poorer performing self-adhesive cement in the present study collection. De Munck and others⁵ published one of the first studies assessing self-adhesive cements, described at the time as a new class of resin cement. The concept dealt with a resin-based cement that combines cement and adhesive in a single application, not requiring pretreatment of dental substrate or restorative materials.⁵ Its adhesive properties were said to be similar to those of self-etch adhesives, because these cements were based on acidic monomers capable of demineralizing and promoting micro-mechanical retention, and some of them were capable of chemical interaction with hydroxyapatite.⁵ Dentin hybridization requires hybrid layer formation by superficial demineralization, followed by resin monomer infiltration and polymerization, creating micro-mechanical retention. Without the etch-and-rinse process, the self-etch process does not remove the smear layer, but rather, dissolves and incorporates it into the hybrid layer.

Acidic monomers are responsible for priming and conditioning dental substrates simultaneously, and adhesion depends on them.⁴⁷ The demineralization capacity of self-etching systems is considered limited, specifically compared with etching acids like phosphoric acid.⁴⁸ De Munck and others⁵ assessed the interaction to dentin of RelyX Unicem (the most widely tested self-adhesive resin cement in the present meta-analyses) with ultra-structural transmission electron microscopy (TEM) and field-emission scanning electron microscopy (Fe-SEM). They observed that Unicem interacted only superficially with underlying dentin and did promote hybrid layer or tag formation. Furthermore, the

authors observed an irregular interaction zone. When the cement was applied over a fractured dentin piece with no smear layer, they observed hardly any cement-dentin interaction. The cement interaction with intratubular dentin was similar to that of intertubular dentin. The self-etch cement tested in the same study (Panavia F) formed what the authors called a “true hybrid layer.”⁵ Another issue concerning a self-adhesive resin cement is its pH-neutralization behavior. These cements have a low pH when mixed and manipulated, an important factor in the adhesion process. Cements with insufficient pH neutralization after self-curing can lead to a reduction in their mechanical properties, thus jeopardizing long-term adhesion.^{49,50} This behavior is considered material dependent, because it is directly related to cement composition and may diverge among different commercial brands.⁵⁰ In fact, the lack of interaction with the dental substrate, combined with reduced etching and demineralization capacity of acidic monomers and the lack of pH neutralization of some cements, could be the factors mainly responsible for the lower dentin bond strength values presented by the self-adhesives.

The results of this systematic review with meta-analyses do not refute the validity of using self-adhesive resin cements. When considering root canal glass-fiber post cementation, self-adhesive resin cements performed better than the conventional ones.^{17,51} Another point to be deliberated concerns self-adhesive cement interaction with restorative materials. Published laboratory studies assessing the interaction of self-adhesive cements with indirect restorative materials showed adequate bond strength to these substrates, with a brand-dependent distinction regarding chemical composition.^{50,52} Adhesion to dentin remains an issue of scientific assessment, because it is considered the weak link in the adhesion process. As for self-adhesive resin cements, further attention is needed by researchers and manufacturers to develop better material and clinical protocols.

Heterogeneity was considered high and statistically significant in all the meta-analyses researched ($I^2=98-99\%$). Methodologic discrepancies, mainly regarding the different aging methods, could represent the key factor responsible for this heterogeneity. Because of this methodologic variety, the studies could not be categorized according to aging methods, unless at least one aging method was used. Furthermore, greater methodologic rigor is needed to counter the neglected risk of bias parameters seen

in this review. Risk of bias assessment showed that most of the studies included had medium or high risk. This indicates the likelihood that these laboratory studies did not control all the variables that could influence the results, and this somewhat explains the high heterogeneity.

An important limitation of the present review is that it included only laboratory studies, with controlled environment and variables. The extracted data reveal only the adhesion to dentin factor in the retention of an indirect restoration to dentin, without considering other important parameters, like tooth preparation design and retention, restoration type and extension, tooth position technique sensitivity, and the operator,⁵³ who may influence ultimate survival of the restoration.

CONCLUSION

Despite the high heterogeneity and limitations of the present review of laboratory studies with meta-analyses, conventional multistep resin cements showed better overall adhesive performance to dentin compared with simplified self-adhesive resin cements. Laboratory results are an important factor to be considered in restoration longevity prognosis; however, other clinical aspects must be considered in assessing clinical success.

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 September 2019)

References

- Van Dijken JW & Hasselrot L (2010) A prospective 15-year evaluation of extensive dentin-enamel-bonded pressed ceramic coverages *Dental Materials* **26**(9) 929-939.
- Pavan S, Dos Santos PH, Berger S, & Bedran-Russo KB (2010) The effect of dentin pretreatment on the microtensile bond strength of self-adhesive resin cements *Journal of Prosthetic Dentistry* **104**(4) 258-264.
- Xiaoping L, Dongfeng R, & Silikas N (2014) Effect of etching time and resin bond on the flexural strength of IPS e.max Press glass ceramic *Dental Materials* **30**(12) 330-336.
- Skupien JA, Porto JAS, Munchow EA, Cenci MS, & Pereira-Cenci T (2015) Impairment of resin cement application on the bond strength of indirect composite restoration *Brazilian Oral Research* **29**(1) 1-7.
- De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts, & Van Meerbeek B (2004) Bonding of an auto-adhesive luting material to enamel and dentin *Dental Materials* **20**(10) 963-971.
- Cantoro A, Goracci C, Carvalho CA, Coniglio I, & Ferrari M (2009) Bonding potential of self-adhesive luting agents used at different temperatures to lute composite onlays *Journal of Dentistry* **37**(6) 454-461.
- De Angelis F, Minnoni A, Vitalone LM, Carluccio F, Vadini M, Paolantonio M, & D'Arcangelo C (2011) Bond strength evaluation of three self-adhesive luting systems used for cementing composite and porcelain *Operative Dentistry* **36**(6) 626-634.
- Stape THS, Menezes MS, Aguiar FHB, Quagliatto PS, Soares CJ, & Martins LRM (2014) Long-term effect of chlorhexidine on the dentin microtensile bond strength of conventional and self-adhesive resin cements: a two-year in vitro study *International Journal of Adhesion & Adhesives* **50**(April) 228-234.
- Prochnow EP, Amaral M, Bergoli CD, Silva TB, Saavedra G, & Valandro LF (2014) Microtensile bond strength between indirect composite resin inlays and dentin: effect of cementation strategy and mechanical aging *Journal of Adhesive Dentistry* **16**(4) 357-363.
- Fuentes MV, Escribano N, Baracco B, Romero M, & Ceballos L (2016) Effect of indirect composite treatment microtensile bond strength of self-adhesive resin cements *Journal of Clinical and Experimental Dentistry* **8**(1) 14-21.
- Giannini M, Takagaki T, Bacelar-Sá R, Vermelho PM, Ambrosano GMB, Sadr A, Nikaido T, & Tagami J (2015) Influence of resin coating on bond strength of self-adhesive resin dentin *Dental Materials Journal* **34**(6) 882-827.
- Vaz RR, Hipólito VD, D'Alpino PH, & Goes MF (2012) Bond strength and interfacial micromorphology of etch-and-rinse and self-adhesive resin cements to dentin *Journal of Prosthodontics* **21**(2) 101-111.
- Guarda GB, Gonçalves LS, Correr AB, Moraes RR, Sinhoreti MAC, & Correr-Sobrinho L (2010) Luting glass ceramic restorations using a self-adhesive resin cement under different dentin conditions *Journal of Applied Oral Science* **18**(3) 244-248.
- Hitz T, Stawarczyk B, Fischer J, Hammerle CHF, & Sailer I (2012) Are self-adhesive resin cements a valid alternative to conventional resin cements? A laboratory study of the long-term bond strength *Dental Materials* **28**(11) 1183-1190.
- Higgins JPT & Green S (2011) Cochrane handbook for systematic reviews of interventions, version 5.1.0; Retrieved online November 11, 2017 from www.cochrane-handbook.org
- Moher D, Liberati A, Tetzlaff J, & Altman DG (2009) Preferred reporting items for systematic reviews and meta-analyses: the PRISMA statement *PLoS Medicine* **6**(7) e1000097.
- Sarkis-Onofre R, Skupien JA, Cenci MS, Moraes RR, & Pereira-Cenci T (2014) The role of resin cement on bond strength of glass-fiber posts luted into root canals: a systematic review and meta-analysis of in vitro studies *Operative Dentistry* **39**(1) E31-44.

18. Moraes AP, Sarkis-Onofre R, Moraes RR, Cenci MS, Soares CJ, & Pereira-Cenci T (2015) Can silanization increase the retention of glass-fiber posts? A systematic review and meta-analysis of in vitro studies *Operative Dentistry* **40**(6) 567-580.
19. Furukawa K, Inai N, & Tagami J (2002) The effects of luting resin bond to dentin on the strength of dentin supported by indirect resin composite *Dental Materials* **18**(2) 136-142.
20. Turkmen C, Durkan M, Cimilli H, & Oksuz M (2011) Tensile bond strength of indirect composites luted with three new self-adhesive resin cements to dentin *Journal of Applied Oral Science: Revista FOB* **19**(4) 363-369.
21. Saker S, Alnazzawi A, & Ozcan M (2016) Adhesive strength of self-adhesive resins to lithium disilicate ceramic and dentin: effect of dentin chelating agents *Odontology* **104**(1) 53-59.
22. Vieira-Filho WS, Alonso RCB, González AHM, D'Alpino PHP, & Di Hipólito V (2017) Bond strength and chemical interaction of self-adhesive resin cements according to dentin region *International Journal of Adhesion and Adhesives* **73**(March) 22-27.
23. Hikita K, Van Meerbeek B, De Munck B, Ikeda T, Van Landuyt K, Maida T, Lambrechts P, & Peumans M (2007) Bonding effectiveness of adhesive luting agents to enamel and dentin *Dental Materials* **23**(1) 71-80.
24. Gotti VB, Feitosa VP, Salvatore S, Correr-Sobrinho L, & Correr AB (2014) Indirect resin composite restorations bonded to dentin using self-adhesive resin cements applied with an electric current-assisted method *American Journal of Dentistry* **27**(5) 233-236.
25. Di hipólito V, Azevedo LC, Piveta FB, Vieira-Filho WS, Anauate-Netto C, Alonso RCB & D'Alpino PHP (2014) Effect of dentinal surface preparation on the bonding of self-adhesive luting cements *Journal of Adhesion and Technology* **28**(19) 1907-1924.
26. Barcellos DC, Batista GR, da Silva MA, Rangel PM Torres CRG, & Fava M (2011) Evaluation of bond strength of self-adhesive cements to dentin with or without application of adhesive systems *Journal of Adhesive Dentistry* **13**(3) 261-265.
27. Broyles AC, Pavan S, & Bedran-Russo AK (2013) Effect of dentin surface modification on the microtensile bond strength of self-adhesive resin cements *Journal of Prosthodontics* **22**(1) 59-62.
28. Marocho SM, Ozcan M, Amaral R, Bottino MA, & Valandro LF (2013) Effect of resin cement type on the microtensile bond strength to lithium disilicate ceramic and dentin using different test assemblies *Journal of Adhesive Dentistry* **15**(4) 361-368.
29. Ariyoshi M, Nikaido T, Foxton RM, & Tagami J (2008) Microtensile bond strengths of composite cores to pulpal floor dentin with resin coating *Dental Materials Journal* **27**(3) 400-407.
30. Chung CWM, Yiu CKY, King NM, Hiraishi N, & Tay FR (2009) Effect of saliva contamination on bond strength of resin luting cements to dentin *Journal of Dentistry* **37**(12) 923-931.
31. Sultana S, Nikaido T, Matin K, Ogata M, Foxton RM, & Tagami J (2007) Effect of resin coating on dentin bonding of resin cement in Class II cavities *Dental Materials Journal* **26**(4) 506-513.
32. Takahashi R, Nikaido T, Ariyoshi M, Kitayama S, Sadr A, & Foxton RM (2010) Thin resin coating by dual-application of all-in-one adhesives improves dentin bond strength of resin cements for indirect restorations *Dental Materials Journal* **29**(5) 615-622.
33. Kim JY, Cho GY, Roh BD, & Shin Y (2016) Effect of curing mode on shear bond strength of self-adhesive cement to composite blocks *Materials* **9**(3) E210.
34. Escribano N & Macorra JC (2006) Microtensile bond strength of self-adhesive luting cements to ceramic *Journal of Adhesive Dentistry* **8**(5) 337-341.
35. Inukai T, Abe T, Ito Y, Pilecki P, Wilson RF, Watson TF, & Foxton RM (2012) Adhesion of indirect MOD resin composite inlays luted with self-adhesive and self-etching resin cements *Operative Dentistry* **37**(5) 474-484.
36. Aguiar TR, André CB, Correr-Sobrinho L, Arrais CAG, Ambrosano GMB, & Giannini M (2014) Effect of storage times and mechanical load cycling on dentin bond strength of conventional and self-adhesive resin luting cements *Journal of Prosthetic Dentistry* **111**(5) 404-410.
37. Feitosa SA, Corazza PH, Cesar PF, Bottino MA & Valandro LF (2014) Pressable feldspathic inlays in premolars: effect of cementation strategy and mechanical cycling on the adhesive bond between dentin and restoration *Journal of Adhesive Dentistry* **16**(2) 147-153.
38. Suzuki TY, Godas AG, Guedes AP, Catelan A, Pavan S, Briso AL, & Dos Santos PH (2013) Microtensile bond strength of resin cements to caries-affected dentin *Journal of Prosthetic Dentistry* **110**(1) 47-55.
39. Bacchi A, Abuna G, Babbar A, Sinhoreti MAC, & Feitosa VP (2015) Influence of 3-month simulated pulpal pressure on the microtensile bond strength of simplified resin luting systems *Journal of Adhesive Dentistry* **17**(3) 265-271.
40. Bacchi A, Abuna G, Consani RL, Sinhoreti MAC, Sauro S, & Feitosa VP (2015) Effects of simulated pulpal pressure, mechanical and thermocycling challenge on the microtensile bond strength of resin luting cements *International Journal of Adhesion & Adhesives* **60**(July) 69-74.
41. Fuentes MV, Ceballos L, & González-López (2013) Bond strength of self-adhesive resin cements to different treated indirect composites *Clinical Oral Investigations* **17**(3) 717-724.
42. D'Arcangelo C, De Angelis F, D'Amario M, Zazzeroni S, Ciampoli C, & Caputi S (2009) The influence of luting systems on the microtensile bond strength of dentin to indirect resin-based composite *Operative Dentistry* **34**(3) 328-336.
43. Aguiar TR, Di Francescantonio M, Ambrosano GMB, & Giannini M (2010) Effect of curing mode on bond strength of self-adhesive resin luting cements to dentin *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **93**(1) 122-127.
44. Sarr M, Mine A, De Munck J, Cardoso MV, Kane AW, Vreven J, Van Meerbeek B, & Van Landuyt KL (2010)

- Immediate bonding effectiveness of contemporary composite cements to dentin *Clinical Oral Investigations* **14**(5) 569-577.
45. Rigolin FJ, Miranda ME, Flório FM, & Basting RT (2014) Evaluation of bond strength between leucite-based and lithium disilicate-based ceramics to dentin after cementation with conventional and self-adhesive resin agents *Acta Odontológica Latinoamericana* **17**(1) 16-24.
46. Perdigão J, Reis A, & Loguercio AD (2013) Dentin adhesion and MMPs: a comprehensive review *Journal of Esthetic and Restorative Dentistry* **25**(4) 219-241.
47. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, & Van Landuyt KL (2011) State of the art of self-etch adhesives *Dental Materials* **27**(1) 17-28.
48. Mena-Serrano A, Kose C, De Paula EA, Tay LY, Reis A, Loguercio AD, & Perdigão J (2013) A new universal simplified adhesive: 6-month clinical evaluation *Journal of Esthetic and Restorative Dentistry* **25**(1) 55-69.
49. Zorzin J, Petschelt A, Ebert J, & Lohbauer U (2012) pH neutralization and influence on mechanical strength in self-adhesive resin luting agents *Dental Materials* **28**(6) 672-679.
50. Zorzin J, Belli R, Wagner A, Petschelt A, & Lohbauer U (2014) Self-adhesive resin cements: adhesive performance to indirect restorative ceramics *Journal of Adhesive Dentistry* **16**(6) 541-546.
51. Skupien JA, Sarkis-Onofre R, Cenci MS, Moraes RR, & Pereira-Cenci T (2015) A systematic review of factors associated with the retention of glass fiber posts *Brazilian Oral Research* **29**(1) 1-8.
52. Sabatini C, Patel M, & D'Silva E (2012) In vitro shear bond strength of three self-adhesive resin cements and a resin-modified glass ionomer cement to various prosthodontic substrates *Operative Dentistry* **38**(2) 186-196.
53. Frankenberger R, Reinelt C, Petschelt A, & Krämer N (2009) Operator vs. material influence on clinical outcome of bonded ceramic inlays *Dental Materials* **25**(8) 960-968.

Laboratory Research

Effect of Shortened Light-Curing Modes on Bulk-Fill Resin Composites

CS Sampaio • PG Pizarro • PJ Atria • R Hirata • M Giannini • E Mahn

Clinical Relevance

Shortened light curing does not affect volumetric polymerization shrinkage or cohesive tensile strength but negatively affects the shear bond strength of some bulk-fill resin composites. When performing shortened light curing, clinicians should be aware of the light output of their light-curing units.

SUMMARY

Purpose: To evaluate volumetric polymerization shrinkage (VPS), shear bond strength (SBS) to dentin, and cohesive tensile strength

(CTS) of bulk-fill resin composites (BFRCs) light activated by different modes.

Methods and Materials: Six groups were evaluated: Tetric EvoCeram bulk fill + high mode (10 seconds; TEC H10), Tetric EvoFlow bulk fill + high mode (TEF H10), experimental bulk fill + high mode (TEE H10), Tetric EvoCeram bulk fill + turbo mode (five seconds; TEC T5), Tetric EvoFlow bulk fill + turbo mode (TEF T5), and experimental bulk fill + turbo mode (TEE T5). Bluephase Style 20i and Adhese Universal Vivapen were used for all groups. All BFRC samples were built up on human molar bur-prepared occlusal cavities. VPS% and location were evaluated through micro-computed tomography. SBS and CTS tests were performed 24 hours after storage or after 5000 thermal cycles; fracture mode was analyzed for SBS.

Results: Both TEC H10 and TEE H10 presented lower VPS% than TEF H10. However, no significant differences were observed with the turbo-curing mode. No differences were observed for the same BFRC within curing modes. Occlusal shrinkage was mostly observed. Regarding SBS, thermal cycling (TC) affected all groups. Without TC, all groups showed higher SBS values for high mode than

*Camila S Sampaio, DDS, MS, PhD, assistant professor, Department of Biomaterials, College of Dentistry, Universidad de los Andes, Santiago, Chile

Paulina G Pizarro, DDS student, Department of Biomaterials, College of Dentistry, Universidad de los Andes, Santiago, Chile

Pablo J Atria, DDS, MS, assistant professor, Department of Biomaterials, College of Dentistry, Universidad de los Andes, Santiago, Chile

Ronaldo Hirata, DDS, MS, PhD, assistant professor, Department of Biomaterials and Biomimetics, New York University College of Dentistry, New York, NY, USA

Marcelo Giannini, DDS, MS, PhD, associate professor, Department of Restorative Dentistry, Piracicaba Dental School, Piracicaba, SP, Brazil

Eduardo Mahn, DDS, MS, PhD, professor, Department of Restorative Dentistry, College of Dentistry, Universidad de los Andes, Santiago, Chile

*Corresponding author: Monseñor Álvaro del Portillo, 12455, Las Condes, Santiago, Chile; e-mail: csampaio@miuandes.cl

<https://doi.org/10.2341/19-101-L>

turbo mode, while with TC, only TEC showed decreased SBS from high mode to turbo modes; modes of fracture were predominantly adhesive. For CTS, TC affected all groups except TEE H10. In general, no differences were observed between groups when comparing the curing modes.

Conclusions: Increased light output with a shortened curing time did not jeopardize the VPS and SBS properties of the BFRCs, although a decreased SBS was observed in some groups. TEE generally showed similar or improved values for the tested properties in a shortened light-curing time. The VPS was mostly affected by the materials tested, whereas the SBS was affected by the materials, curing modes, and TC. The CTS was not affected by the curing modes.

INTRODUCTION

In current practice, there is hardly a dental procedure that does not require the use of a polymeric resin material and light activation.¹ Resin composite restorations present good esthetic and mechanical properties as well as adhesion to the tooth structure using an adhesive system; however, the time spent on performing these procedures is a drawback.² For that reason, and as a matter of improving clinical efficiency, bulk-fill resin composites (BFRCs) were introduced in the market, and numerous *in vitro* studies have evaluated these restorative materials.³⁻¹⁷ BFRCs present modifications in their compositions, such as new monomers and changes in their concentrations, addition of new photoinitiators, increased translucency, and decreased or changes in their inorganic content. All of these modifications have allowed them to be applied in a 4- to 5-mm thicknesses, thus diminishing the time spent on the restorative procedure.¹⁶ Moreover, they are presented in two different formulations with high and low viscosity; the latter requires a capping layer of regular resin composite for finishing the restoration because of its weak mechanical properties,^{5,12} which compromises the time-saving benefit by adding an extra step.¹⁰

Most of the published literature suggests that BFRCs have good performance in terms of degree of conversion,^{13,14} polymerization shrinkage,^{3,15} and bonding properties.⁸ Although BFRCs are a faster procedure compared with the incremental technique, clinicians still aim to save more time. Thus, there has recently been a move toward an even faster light activation of BFRCs.¹⁸ Such time-saving procedures

should not be detrimental to the material's mechanical properties, because adequate light curing is required for resin composite restorations to reach their manufacturer's intended properties and ensure predictable long-term clinical success.¹ It seems logical that if a restoration does not receive sufficient incident irradiance, mostly for a 4-mm-depth BFRC restoration, it will not reach its ideal mechanical and bonding properties to the teeth,¹ which could lead to bulk fracture of the restoration, secondary caries due to adhesive failure, and breakdown of the margin.^{19,20}

The third-generation light-emitting diode (LED) light-curing units (LCUs), also called "polywave," provide a broad-spectrum LCU that can activate all current photoinitiators present in resin composites.¹⁸ The need for such LCUs is because different photoinitiators, apart from the conventional camphorquinone (light activated within the blue range, about 460-470 nm), are being used in formulations of resin composites. These are type I photoinitiators, and need another range of delivered light.¹⁸ Such photoinitiators (eg, Lucirin TPO and derivatives of dibenzoyl germanium such as Ivocerin) do not need additional co-initiators, as they decompose directly into 1 or more free radicals when sufficient energy at the correct wavelength is delivered.¹⁸

Ivocerin is most reactive at 408 nm but remains very sensitive to wavelengths of light between 400 and 430 nm. It is considered an "initiation booster" because of its ability to start the reaction with fewer photons, polymerize deeply, and regulate polymerization during curing of a material in bulk.²¹ Also, the greater ability of these materials to create free radicals per molecule unit can improve the light sensitivity of resin composites.²² Thus, depending on the photoinitiators used, even small changes in the emission spectrum (wavelength) can be relevant.¹⁸ Apart from the wavelength needed, the irradiance is also an important factor when light curing a resin composite.¹⁸ The introduction of LED LCUs with high power and short exposure has raised concerns regarding effectiveness, to ensure that the restoration is not undercured.^{7,22,23}

One of the most investigated properties of BFRCs is polymerization shrinkage,^{3,4,15} an unavoidable phenomenon that occurs because of the polymerization reaction of all resinous materials when the monomers are converted to polymers. Its undesirable clinical outcomes include shrinkage stress on the tooth-restoration interface, which can form gaps at the interface, and marginal infiltration.^{3,15} Volumetric polymerization shrinkage (VPS) can be observed

Table 1: Detailed Information of the Bulk-Fill Resin Composites Used in This Study

Material	Type, Shade	Manufacturer, Batch	Composition
Tetric Evoceram bulk fill	High-viscosity bulk-fill resin composite, IVA	Ivoclar Vivadent, W02598	Bis-GMA, UDMA, ytterbium trifluoride, dimethacrylate, barium glass, mixed oxide, prepolymer, additives, catalyst stabilizers 0.9 wt.%; pigments <0.1 wt.%; 79-81 wt% inorganic fillers. Photoinitiator present: Ivocerin
Tetric EvoFlow bulk fill	Low-viscosity bulk-fill resin composite, IVA	Ivoclar Vivadent, V41879	Bis-GMA, urethane dimethacrylate, decandiol dimethacrylate, 37.6 wt.% barium glass filler, ytterbium trifluoride, mixed oxide, highly dispersed silicon dioxide 41.1 wt.%, prepolymer 20.4 wt.%, additives, catalyst stabilizers 0.9 wt.%, pigments <0.1 wt.%; 62 wt.% inorganic fillers; photoinitiator present: Ivocerin
F-Composite 2 (experimental)	Experimental, IVA	Ivoclar Vivadent, W06912	Composition similar to its high-viscosity bulk-fill composite counterpart (Tetric Evoceram bulk fill), with a modification of double the amount of the Ivocerin

Abbreviations: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; UDMA, urethane dimethacrylate.

through a nondestructive, three-dimensional, high-resolution imaging technique test called micro-computed tomography (μ CT),^{3,4,15} which has been used for different purposes, such as evaluating cementation film thickness,²⁴ presence of bubbles with different cementation techniques,²⁵ and polymerization shrinkage vectors.²⁶

Mechanical properties^{5,6,12} play a fundamental role in the clinical performance of a restorative material. It can be evaluated through a great number of tests, such as degree of conversion, hardness, and tensile tests.^{8,14,27} As stated in a recent study, it is likely that most dental restorations fail by tensile stresses set up within their structure because of the loading mechanism of their complex geometries.²⁷

The bonding to the tooth structure is also an important parameter that needs attention when dealing with BFRCs, as the light beam from the LCU decreases with distance.⁸ Thus, the interface between the resin composite and tooth, which is where the adhesive system is located, is a critical spot, as it is the one farther away from the LCU's light tip. A reduction in the bond strength to dentin at the bottom of the cavity in class I restorations with BFRCs has been shown in the literature.¹⁷

Aiming to mimic the degradation of the oral environment, TC is an effective *in vitro* aging protocol, performed to create thermal strains at the bonding interface caused by thermal changes in water baths. Moreover, repetition of such baths were shown to weaken the bond between the resin matrix and filling material.²⁸

Thus, the aim of the present study is to determine the influence of different light-curing modes (high

mode and turbo mode) of a single LCU on the VPS, shear bond strength (SBS) to dentin, and cohesive tensile strength (CTS) of two commercial and one experimental BFRC. The hypotheses tested were the following: 1) different light-curing modes would affect the VPS, SBS, and CTS of different BFRCs, and 2) within a given BFRC, there would be significant differences regarding VPS, SBS, and CTS light activated with different curing modes.

METHODS AND MATERIALS

Material composition, manufacturers, and batch numbers are described in Table 1. Three different BFRCs were used: Tetric EvoCeram bulk fill (TEC; Ivoclar Vivadent, Schaan, Liechtenstein), Tetric EvoFlow bulk fill (TEF; Ivoclar Vivadent), and an experimental BFRC containing double the Ivocerin photoinitiator than its counterpart TEC (Tetric EvoCeram Experimental bulk fill [TEE]; Ivoclar Vivadent). All materials were light activated by the same LCU (Bluephase Style 20i, Ivoclar Vivadent) at two different curing modes: high mode for 10 seconds (H10; approximately 1200 mW/cm²) and turbo mode for five seconds (T5; approximately 2000 mW/cm²). Power outputs of the LCU were checked with a Bluephase Meter II (Ivoclar Vivadent) before and after all tests. All teeth used in the present study were obtained according to protocols approved by the New York University Ethical Committee.

Assessment of VPS and the Location

Thirty-six caries-free human third molars had their cusps flattened, and standardized boxed-shaped class I preparations (4-mm depth $\times$ 4-mm length $\times$ 4-mm width) were made with a diamond bur (AD20

Occlusal reduction bur, code 845-022; Strauss, Westport, CT, USA), which was replaced every five cavity preparations. Final cavity dimensions were checked with a digital caliper, and teeth were maintained in distilled water (25°C) until their use. Teeth were randomly divided into six groups (n=6 per group) according to BFRC and light-curing mode used: G1, TEC H10; G2, TEF H10; G3, TEE H10; G4, TEC T5; G5, TEF T5; and G6, TEE T5. All groups were light activated by the same LCU (Bluephase Style 20i) and used the same adhesive system (Adhese Universal Vivapen, Ivoclar Vivadent), which was light activated according to the same designated time as for the BFRCs. The adhesive system was used in a self-etching procedure, after cleaning the cavities with a pumice slurry. All cavities were filled in the bulk technique.

Each tooth was scanned three times using a μ CT apparatus (μ CT40, Scanco Medical, AG, Basserdorf, Switzerland), calibrated using a phantom standard at 70 kVp/BH 200 mgHA/cm. The operating condition for the μ CT device used was 70 kVp to 114 microamperes with a resolution of 16 μ m/slice. The average of the total number of slices was approximately 250, and the average scan time was 28 minutes, as previously described.^{3,15}

The first scan was performed after cavity preparation. This procedure was required for avoiding scattering and possible noise between the restorative material and the tooth structure but also to minimize possible artifacts in the threshold segmentation, because enamel, dentin, and restorative material present similar radiodensity.^{3,4,15} The second scan was performed after application of the adhesive system and resin composite and before the light-curing procedure (uncured scan); the third scan was done after light curing the samples (cured scan). During the scans, all samples were kept in a dark vial to prevent any contact with a light source for unwanted polymerization.

Acquired μ CT data were imported into a workstation and evaluated with Amira software (version 5.5.2, VSG, Burlington, MA, USA). All scans were superimposed with the Amira software tool called "superimposition," providing a perfect arrangement of all images. Registered μ CT data of uncured and cured samples were subtracted from the cavity data, isolating the restoration (uncured scan minus cavity preparation scan, and cured scan minus cavity preparation scan). This procedure enabled both uncured and cured resin composite volumes to be isolated and quantified, allowing the total VPS to be calculated as a percentage. Shrinkage observed on

the top and bottom/sides of the cavity were also evaluated; for that purpose, separation within the top and bottom/sides locations was evaluated with the Amira tool called "extract surface" and measured separately, also calculated as percentages. Location data were presented regarding the top surface, and the rest of the shrinkage was considered as bottom/sides.

Data regarding total VPS measurement and VPS in the different locations from the cavity (top and bottom/sides) were analyzed by a normality test followed by a two-way analysis of variance (ANOVA) and least significant difference (LSD) post hoc tests using 95% confidence intervals.

Assessment of SBS With and Without Thermal Cycling

One hundred twenty caries-free human third molars had their roots removed and crowns sectioned with a low-speed diamond disk saw under water cooling to expose mid-coronal dentin. Exposed surfaces were abraded in a semiautomatic polishing machine (LaboPol-6, Struers, Madrid, Spain) with 500-grit silicon carbide abrasive paper under water cooling, in order to standardize the smear layer. Groups were divided as previously described: G1, TEC H10; G2, TEF H10; G3, TEE H10; G4, TEC T5; G5, TEF T5; and G6, TEE T5. All groups were light activated by the same LCU (Bluephase Style 20i) and the same adhesive system (Adhese Universal Vivapen, Ivoclar Vivadent), which was light activated according to the same designated time as for the resin BFRCs. The adhesive system was used in a self-etching procedure, after cleaning the cavities with a pumice slurry.

The BFRC was built up in bulk through a disc with a central orifice ($\varnothing=3.0$ mm and 4-mm high), which was centered on the occlusal tooth surface to obtain the standardized resin composite bar for the SBS test. All resin blocks were prepared in a single increment.

After the restorative procedures, samples were subdivided in two subgroups (n=10): 1) storage in distilled water at 37°C for 24 hours or 2) 5000 thermal cycles in water baths for 30 seconds in each bath of 5°C and 55°C in a thermal cycling (TC) simulator machine.

After the designated storage period, each sample was placed in an SBS testing machine (Shear Bond Tester, Bisco Dental, Schaumburg, IL, USA), and SBS was measured at a cross-head speed of 1 mm/min. The maximum load of SBS of the composite

Table 2: Volumetric Polymerization Shrinkage (VPS) of Three Bulk-Fill Resin Composites Tested Under High- and Turbo-Curing Modes^a

Material	Curing Mode	
	High, 10 s	Turbo, 5 s
Volumetric polymerization shrinkage		
Tetric EvoCeram bulk fill (TEC)	2.6 (0.3) Ab	2.4 (0.2) Aa
Tetric EvoFlow bulk fill (TEF)	3.0 (0.4) Aa	2.8 (0.6) Aa
Experimental bulk fill (TEE)	2.7 (0.4) Ab	2.3 (0.4) Aa
Top shrinkage		
Tetric EvoCeram bulk fill (TEC)	88.8 (8.9) Aab	97.2 (7.4) Aa
Tetric EvoFlow bulk fill (TEF)	97.7 (9.2) Aa	98.7 (15.7) Aa
Experimental bulk fill (TEE)	80.5 (10.4) Ab	80.1 (9.2) Ab

^a Data represent the mean (SD) of the total VPS% and the shrinkage percentage located on the top (unbonded surface). Location data were presented regarding the top surface, as the rest of the shrinkage was considered as bottom/sides (bonded surfaces) shrinkage. Here, the letters (A, a, b) indicate statistical comparisons of different composites and curing modes ($p < 0.05$). Uppercase letters compare different curing modes within the same composite (in the horizontal direction), whereas the lowercase letters compare different composites within the same curing modes (in the vertical direction).

resin to dentin was recorded in newtons (N) and calculated in MPa, taking into account the cross-sectional area of the composite buildup. The SBS data were analyzed by normality test, followed by a three-way ANOVA and LSD post hoc tests using 95% confidence intervals.

After SBS testing, the fracture mode of each specimen was determined under a loupe (Panoramic Flip-up Adivista 2.5x; PeriOptix Inc, Lompoc, CA, USA) at 2.5× magnification and classified as 1 = adhesive fracture, 2 = cohesive fracture in dentin, 3 = cohesive fracture in resin composite, 4 = mixed fracture (adhesive + dentin), or 5 = mixed fracture (adhesive + resin composite).

Assessment of CTS With and Without TC

One hundred twenty beam-shaped samples with a cross-sectional area of approximately 1 mm² were constructed with a silicon matrix. Groups were divided as previously described for the SBS test: G1, TEC H10; G2, TEF H10; G3, TEE H10; G4, TEC T5; G5, TEF T5; and G6, TEE T5. After that, samples were subdivided into two subgroups (n=10): 1) storage in distilled water at 37°C for 24 hours or 2) 5000 thermal cycles in water baths for 30

seconds in each bath of 5°C and 55°C in a TC simulator machine.

After the designated storage period, each beam was individually fixed to a microtensile device with a cyanoacrylate-based glue (Zapit, Dental Ventures of America, Corona, CA, USA) and attached to a universal testing machine (Micro Tensile Tester, Bisco Dental). Specimens were tensile loaded until fracture at a cross-head speed of 1 mm/min. The CTS values were recorded in MPa. Data were analyzed by a normality test, followed by a three-way ANOVA and LSD post hoc tests using 95% confidence intervals.

RESULTS

Table 2 shows the VPS for the different BFRCs and curing modes and also the main location where it occurred. Only the top surface results are described in Table 2, as the rest of the shrinkage was considered as bottom/sides. Figure 1 shows the representative images obtained by the μ CT scans. No significant differences in VPS were observed between the high and turbo curing modes for any BFRC ($p > 0.05$). For turbo mode, no statistical differences were observed between the different BFRCs ($p > 0.05$), except for the high curing mode, in which TEF showed statistically higher VPS% than both TEC ($p = 0.032$) and TEE ($p = 0.036$). For all BFRCs and curing modes, most of the VPS occurred at the top, the unbonded portion, of the restoration ($p < 0.05$). Regarding the different curing modes within the same material, no statistical differences were observed in shrinkage location within the curing modes for any BFRC ($p > 0.05$). However, when the same curing mode was evaluated comparing different BFRCs, for turbo mode, TEE showed statistically less VPS% on top of the restoration than TEC ($p = 0.001$) and TEF ($p = 0.003$). For the high-curing mode, TEF presented statistically the same VPS% on the top of the restoration of TEC ($p = 0.379$) but was statistically different when compared with TEE ($p = 0.026$), whereas TEC and TEE showed statistically similar VPS% on top of the restoration ($p = 0.156$).

Table 3 shows the results from the SBS test for the different BFRCs at each curing mode, with and without TC. For all materials, the high-curing mode presented a higher SBS than turbo-curing mode, in which no TC was performed ($p \leq 0.05$). When TC was performed, only the values of TEC decreased statistically from high- to turbo-curing mode ($p = 0.018$), while for the other BFRCs, the values comparing both curing modes showed no significant

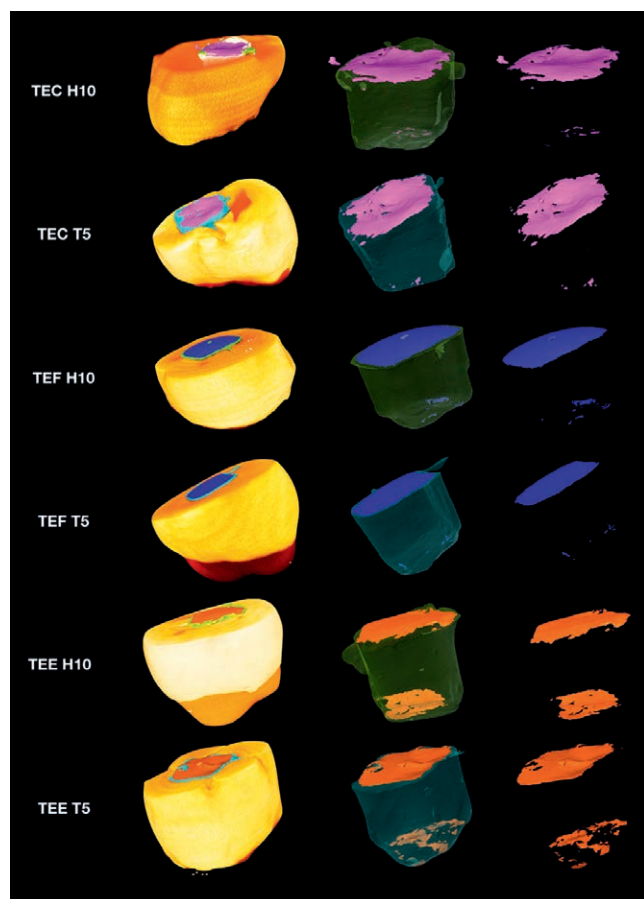


Figure 1. Micro-computed tomography (μ CT) scans of three bulk-fill resin composites tested under high (10 seconds) and turbo (five seconds) curing modes. The images show the location of shrinkage in pink (groups TEC H10 and T5), blue (groups TEF H10 and T5), and orange (groups TEE H10 and T5). Observe that the shrinkage was mostly frequent in the occlusal (unbonded surface) for all samples. Images from the left column show the restored teeth, the middle column shows the restoration and shrinkage digitally extracted from the tooth, and the right column shows the shrinkage only.

differences ($p>0.05$). When comparing the BFRCs with and without TC at the same curing mode, all groups showed a statistically decreased SBS when TC was performed ($p\leq 0.05$). When the different BFRCs were compared within the high-curing mode without TC, TEF and TEE showed a statistically similar SBS ($p=0.200$), and TEF showed an increased SBS when compared with TEC ($p=0.035$), a pattern that was repeated for turbo-curing mode without TC. When samples were subjected to TC and the different BFRCs were compared with the same curing mode, no statistical differences within the BFRCs were observed for the high-curing mode ($p>0.05$), while for the turbo-curing mode, the experimental TEE composite showed a statistically higher SBS than the other groups, TEC ($p=0.002$)

Table 3: Shear Bond Strength (SBS) of Three Bulk-Fill Resin Composites Tested Under High- and Turbo-Curing Modes^a

Material	Curing Mode	
	High, 10 s	Turbo, 5 s
Without TC		
Tetric EvoCeram bulk fill (TEC)	23.6 (3.6) Ab ^b	19.5 (4.3) Bb ^b
Tetric EvoFlow bulk fill (TEF)	27.3 (2.9) Aa ^b	23.7 (4.6) Ba ^b
Experimental bulk fill (TEE)	25.8 (6.6) Aab ^b	20.8 (3.4) Bab ^b
With TC		
Tetric EvoCeram bulk fill (TEC)	13.3 (3.4) Aa	8.3 (2.8) Bb
Tetric EvoFlow bulk fill (TEF)	12.7 (4.3) Aa	9.4 (2.6) Ab
Experimental bulk fill (TEE)	10.9 (4.6) Aa	14.7 (4.0) Aa

^a Data represent the mean (SD) with and without thermal cycling (TC). Here, the letters (A, B, a, b) indicate statistical comparisons of different composites and curing modes ($p<0.05$). The uppercase letters compare different curing modes within the same composite (in the horizontal direction), whereas the lowercase letters compare different composites within the same curing modes under the same thermal cycling condition (in the vertical direction).

^b Comparison of the same composite and curing mode with and without thermal cycling ($p<0.05$).

and TEF ($p=0.007$), with no statistical difference between the groups ($p=0.578$).

The SBS modes of fracture are presented in Table 4. This was predominantly adhesive for TEC H10s and TEC T5s, both with and without TC. Regarding TEF, without TC, for both high mode and turbo mode, the most predominant modes of fracture were adhesive and mixed (adhesive/resin). For samples subjected to TC, fractures were predominantly adhesive for both curing modes. When TEE was analyzed, the predominance of the fracture modes was adhesive for TEE H10s and TEE T5s both with and without TC.

Table 5 shows the results from the CTS test for the different BFRCs at each curing mode, with and without TC. When the same BFRC was compared in different light-curing modes, both with and without TC, no statistical differences were observed ($p>0.05$). When comparing the BFRCs with and without TC using the same curing mode, all materials showed a statistical decrease in CTS when TC was performed ($p<0.05$), except for TEE H10, for which the CTS values were statistically similar both with and without TC ($p=0.181$). When the different BFRCs were compared within the high-curing mode with or without TC, no statistical differences were observed within the BFRCs ($p>0.05$), while for the

Table 4: *Fracture Mode in the Shear Bond Strength (SBS) of Three Bulk-Fill Resin Composites Tested Under High (H10)–and Turbo (T5)—Curing Modes, With or Without Thermal Cycling (TC)*

Fracture Mode	Without TC						With TC					
	TEC H10	TEC T5	TEF H10	TEF T5	TEE H10	TEE T5	TEC H10	TEC T5	TEF H10	TEF T5	TEE H10	TEE T5
Adhesive	9	10	5	4	7	9	10	10	9	10	10	8
Cohesive in dentin	0	0	0	0	0	0	0	0	0	0	0	0
Cohesive in resin	0	0	0	0	0	0	0	0	0	0	0	0
Mixed (adhesive + dentin)	0	0	5	4	1	0	0	0	0	0	0	1
Mixed (adhesive + resin)	1	0	0	2	2	1	0	0	1	0	0	1

turbo-curing mode, TEF presented higher cohesive strength than TEE ($p=0.012$) when TC was not performed. However, it presented no significant difference compared with TEC ($p=0.100$). When TC was performed, again, no statistical differences were observed among BFRCs ($p>0.05$).

DISCUSSION

The first hypothesis tested was accepted, because different light-curing modes affected the VPS, SBS, and CTS of BFRCs. The second hypothesis tested was also accepted, since different BFRCs promoted significant differences regarding VPS, SBS, and CTS light activated with different curing modes.

Table 5: *Cohesive Tensile Strength (CTS) of Three Bulk-Fill Resin Composites Tested Under High- and Turbo-Curing Modes^a*

Material	Curing Mode	
	High, 10 s	Turbo, 5 s
Without TC		
Tetric EvoCeram bulk fill (TEC)	60.7 (7.0) Aa ^b	56.43 (12.2) Aab ^b
Tetric EvoFlow bulk fill (TEF)	61.3 (12.2) Aa ^b	63.57 (7.9) Aa ^b
Experimental bulk fill (TEE)	53.7 (9.5) Aa	52.35 (6.3) Ab ^b
With TC		
Tetric EvoCeram bulk fill (TEC)	47.7 (5.5) Aa	42.91 (8.2) Aa
Tetric EvoFlow bulk fill (TEF)	52.4 (11.3) Aa	51.08 (7.1) Aa
Experimental bulk fill (TEE)	47.3 (7.8) Aa	43.12 (18.1) Aa

^a Data represent the mean (SD) with and without thermal cycling (TC). Here, the letters (A, a, b) indicate statistical comparisons of different composites and curing modes ($p<0.05$). The uppercase letters compare different curing modes within the same composite (in the horizontal direction), whereas the lowercase letters compare different composites within the same curing modes under the same thermal cycling condition (in the vertical direction).

^b Comparison of thermal cycling (without vs with) in the same composite and curing mode ($p<0.05$).

The VPS percentages corroborate those of other studies,^{3,4,15} ranging from 2.3% to 3.0%. The low-viscosity TEF presented higher VPS than the other BFRCs tested for high-curing mode, TEC and TEE, an expected result, as TEF presents fewer inorganic fillers than its high-viscosity counterpart (62wt% and 79-81wt%, respectively). Previous reports showed that composites with lower filler content could result in higher VPS% than high-viscosity ones, because high-viscosity resin composites commonly present reduced monomer content.²⁹ However, no differences within the BFRCs were observed for the turbo mode, which can be explained by the faster polymerization using this light-activation mode, which could have reduced the pre-gel phase, forming short chains before the solid state is reached.^{29,30} BFRCs did not show a significant difference when the different curing modes were evaluated. This result is in accordance with a recent study⁴ that evaluated a single BFRC light activated with four different types of curing modes and observed no significant differences within them. However, the present study disagrees with the findings of other studies that showed that a light-curing protocol with lower irradiance and longer exposure time resulted in lower VPS, as slower polymerization forms longer chains with higher molecular weight compared with higher irradiance,⁵ a phenomenon not observed in the present study.

Regarding shrinkage location, for all groups, top shrinkage was detected more often than bottom/sides shrinkage, an expected result, as a higher VPS% has been demonstrated where there is no adhesive interface (unbonded surface), for instance, the occlusal surface.^{3,4,15} When the different BFRCs were compared, the experimental BFRC (TEE) showed a lower top (occlusal) VPS% than the other investigated materials and thus more VPS% in the bonded walls. This result could be related to the increased amount of photoinitiator in its composition, which could have resulted in a higher degree of polymerization in

deeper regions, thus promoting more VPS in the bottom/sides than the other BFRCs.^{3,15} Further studies should focus on comparing the VPS% and degree of conversion of resin composites with increased photoinitiators. Thus, according to the present study, with regard to VPS, it was observed that the parameter is mostly affected by the materials used rather than by the curing modes.

The SBS test demonstrated decreased values for all BFRCs with TC. The deterioration of the adhesive interface after thermal aging is in accordance with the literature.³¹ A recent study observed the deterioration of different universal adhesives, including the one used in this study, and found lower SBS and a less stable marginal sealing capability than in the immediate condition.³² This can be explained by the fact that TC produces hoop stress³³ as a result of the unequal thermal expansion of the different substrates compromised in the bonded interface, which could induce volumetric changes in the adhesive layers.³⁴ The SBS results also reflect the fracture modes observed in the present study, predominantly adhesive for all materials with TC, while without TC, TEF was the BFRC that resulted in more samples with a mixed type of fracture, which explains its higher SBS values compared with the other BFRCs. Without TC, all BFRCs tested showed a significant decrease in SBS when turbo mode was used compared with high mode. With TC, only TEC showed significantly decreased values for turbo mode compared with high mode, whereas TEF and TEE materials showed no statistical differences between high and turbo modes. This can be explained by the evidence that the flowable composite has higher translucency before polymerization, and the experimental composite, because of its increased amount of photoinitiator, could cause more stable adhesion with TC. It has been proven that the greater absorption of this photoinitiator also contributes to an increased depth of cure of resin materials.^{5,21}

When comparing the SBS of the different BFRCs within the same light-curing mode, for both high and turbo modes without TC, TEF and TEE showed the highest results, while TEC and TEE were not significantly different from each other. For the results with TC, the different BFRCs did not show significant between-group differences when using high mode, although for the turbo-curing mode, the experimental material TEE showed higher SBS values than those obtained for TEC and TEF. It was observed in a previous work from this group that the violet light loses its power with increased depths.²⁶ With a higher amount of Ivocerin in the experimental material TEE, deep regions (eg, 4 mm)

could have been more polymerized by both emitted lights (blue and violet) from the Bluephase Style 20i. The light activation of the experimental TEE with turbo mode resulted in a lack of significant differences between it and high mode. When this experimental material was evaluated with TC, higher SBS results were obtained using turbo mode compared with the other BFRCs studied.

The CTS values demonstrated no difference in light-curing mode for any of the BFRCs, both with or without TC. Although these results cannot be compared with the findings of any other study, as the CTS of BFRCs has not been investigated so far, they do agree with the VPS% results, as no differences were observed between the light-curing modes. However, significant differences were observed when TC aging was compared, as all BFRCs showed a decrease in CTS values, except for TEE H10. This result was expected, as it is known that thermal aging negatively affects the mechanical properties of resinous materials because of their expansion and shrinkage, which could weaken the bonds that connect the structure of the resin.³⁵ The lack of significant differences when TC was performed or not was observed only for the experimental BFRC; however, no significant differences were observed within the different materials with TC.

It has been shown that longer curing times at lower irradiances improve the mechanical properties of resin composites, and an increased volume of photoinitiator can also positively affect this parameter.²⁹ A significant difference among the different BFRCs was observed only for turbo mode without TC, in which the experimental BFRC TEE presented a lower CTS than the flowable BFRC did. This result was not expected, because this material has a greater amount of inorganic fillers than its flowable version. However, a possible explanation for this result is the increased translucency of the flowable material when compared with high-viscous materials before polymerization,⁵ which allows more light transmission through the material, possibly improving its mechanical properties. A limitation of this result is that the CTS test was performed in a 1-mm² beam sample. BFRCs are intended to be cured at up to 4-mm thickness; thus, the size of the sample could be related to the lack of statistically significant differences within the BFRCs.

The use of increased emitted irradiance in a reduced time has been suggested to save time during restorative procedures. However, results from the present study suggest that the use of a reduced curing time should be done with caution, mostly regarding the SBS of BFRCs. Moreover, clinically,

light exposure might be jeopardized by the hard access of the LCU in terms of the position of the teeth in the mouth (distance to target) and damaged or debris-contaminated light-curing tips, among others, which can also affect the amount of light delivered to the restorations.^{1,18} In addition, it was observed that LCUs in dental offices are mostly poorly maintained and can deliver inadequate light output.^{1,18} This is a limitation of the present study, as light could always be delivered from a favorable position, and maintenance of the LCU was performed accordingly. Future studies should focus on evaluating the use of experimental materials with increased amounts of photoinitiator and comparing shortened light-curing times for a greater diversity of composites.

CONCLUSIONS

Based on the limitations of the present study, the following conclusions can be made:

1. VPS is more greatly affected by the material tested than the light-curing mode. TEF showed higher VPS than the high-viscosity materials, TEC and TEE, in high-curing mode. Top shrinkage was more frequently observed than bottom/sides shrinkage.
2. SBS is affected by the material used, TC, and curing modes. Turbo mode showed lower SBS values than high mode, except for TEF and TEE with TC.
3. The CTS of the BFRCs studied was not affected by curing mode. However, TC reduced this property for all groups, except for TEE in high mode.
4. TEE generally showed similar or improved values for the tested properties in a shortened curing time compared with the other BFRCs, except for CTS without TC.

Acknowledgement

This work was partially supported by the Fondo Nacional de Desarrollo Científico y Tecnológico, Chile (FONDECYT project 11170920).

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of New York University Committee on Activities Involving Human Subjects.

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 28 August 2019)

REFERENCES

1. Price RB, Ferracane JL, & Shortall AC (2015) Light-curing units: a review of what we need to know *Journal of Dental Research* **94**(9) 1179-1186.
2. Ferracane JL (2011) Resin composite—state of the art *Dental Materials* **27**(1) 29-38.
3. 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.
4. Atria PJ, Sampaio CS, Cáceres E, Fernández J, Reis AF, Coelho PG, & Hirata R (2018) Micro-computed tomography evaluation of volumetric polymerization shrinkage and degree of conversion of composites cured by various light power outputs *Dental Materials Journal* **37**(1) 33-39.
5. Besegato JF, Jussiani EI, Andrello AC, Fernandes RV, Salomão FM, Vicentin BLS, Dezan-Garbellini CC, & Hoepfner MG (2019) Effect of light-curing protocols on the mechanical behavior of bulk-fill resin composites *Journal of the Mechanical Behavior of Biomedical Materials* **90** 381-387.
6. 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.
7. Daugherty MM, Lien W, Mansell MR, Risk DL, Savett DA, & Vandewalle KS (2018) Effect of high-intensity curing lights on the polymerization of bulk-fill composites *Dental Materials* **34**(10) 1531-1541.
8. 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.
9. Fronza BM, Rueggeberg FA, Braga RR, Mogilevych B, Soares LE, 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.
10. Ilie N (2017) Impact of light transmittance mode on polymerisation kinetics in bulk-fill resin-based composites *Journal of Dentistry* **63**(2017) 51-59.
11. Ilie N (2019) Sufficiency of curing in high-viscosity bulk-fill resin composites with enhanced opacity *Clinical Oral Investigations* **23**(2) 747-755.
12. 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.
13. 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.
14. Lima RBW, Troconis CCM, Moreno MBP, Murillo-Gómez F, & De Goes MF (2018) Depth of cure of bulk fill resin

- composites: a systematic review *Journal of Esthetic and Restorative Dentistry* **30**(6) 492-501.
15. 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.
 16. 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.
 17. Van Ende A, De Munck J, Van Landuyt, Poitevin A, Peumans M, & Van Meerbeek B (2013) Bulk-filling of high C-factor posterior cavities: effect on adhesion to cavity-bottom dentin *Dental Materials* **29**(3) 269-277.
 18. Price RBT (2017) Light curing in dentistry *Dental Clinics of North America* **61**(4) 751-778.
 19. Bernardo M, Luis H, Martin MD, Leroux BG, Rue T, Leitão J, & DeRouen TA (2007) Survival and reasons for failure of amalgam versus composite posterior restorations placed in a randomized clinical trial *Journal of the American Dental Association* **138**(6) 775-783.
 20. Opdam NJ, Bronkhorst EM, Roeters JM, & Loomans BA (2007) A retrospective clinical study on longevity of posterior composite and amalgam restorations *Dental Materials* **23**(1) 2-8.
 21. 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.
 22. Leprince JG, Hadis M, Shortall AC, Ferracane JL, Devaux J, Leloup G, & Palin WM (2011) Photoinitiator type and applicability of exposure reciprocity law in filled and unfilled photoactive resins *Dental Materials* **27**(2) 157-164.
 23. Kutuk ZB, Gurgan S, Hickel R, & Illie N (2017) Influence of extremely high irradiances on the micromechanical properties of a nano hybrid resin based composite *American Journal of Dentistry* **30**(1) 9-15.
 24. Sampaio CS, Barbosa JM, Cáceres E, Rigo LC, Coelho PG, Bonfante EA, & Hirata R (2017) Volumetric shrinkage and film thickness of cementation materials for veneers: an *in vitro* 3D microcomputed tomography analysis *Journal of Prosthetic Dentistry* **117**(6) 784-791.
 25. Cáceres EA, Sampaio CS, Atria PJ, Moura H, Giannini M, Coelho PG, & Hirata R (2018) Void and gap evaluation using microcomputed tomography of different fiber post cementation techniques *Journal of Prosthetic Dentistry* **119**(1) 103-107.
 26. Sampaio CS, Atria PJ, Rueggeberg FA, Yamaguchi S, Giannini M, Coelho PG, Hirata R, & Puppini-Rontani RM (2017) Effect of blue and violet light on polymerization shrinkage vectors of a CQ/TPO-containing composite *Dental Materials* **33**(7) 796-804.
 27. 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.
 28. Rinastiti M, Özcan M, Siswomihardjo W, & Busscher HJ (2011) Effects of surface conditioning on repair bond strengths of non-aged and aged microhybrid, nanohybrid, and nanofilled composite resins *Clinical Oral Investigations* **15**(5) 625-633.
 29. 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.
 30. Burdick JA, Lovestead TM, & Anseth KS (2003) Kinetic chain lengths in highly cross-linked networks formed by the photoinitiated polymerization of divinyl monomers: a gel permeation chromatography investigation *Biomacromolecules* **4**(1) 149-156.
 31. Sampaio CS, Rodrigues RV, Souza-Junior EJ, Freitas AZ, Ambrosano GM, Pascon FM, & Puppini-Rontani RM (2016) Effect of restorative system and thermal cycling on the tooth-restoration interface—OCT evaluation *Operative Dentistry* **41**(2) 162-170.
 32. Yao C, Yang H, Yu J, Zhang L, Zhu Y, & Huang C (2018) High bond durability of universal adhesives on glass ceramics facilitated by silane pretreatment *Operative Dentistry* **46**(6) 602-612.
 33. Crim GA, Swartz ML, & Phillips RW (1985) Comparison of four thermocycling techniques *Journal of Prosthetic Dentistry* **53**(1) 50-53.
 34. 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**(2016) 56-61.
 35. Pala K, Tekçe N, Tuncer S, Serim ME, & Demirci M (2016) Evaluation of the surface hardness, roughness, gloss and color of composites after different finishing/polishing treatments and thermocycling using a multi-technique approach *Dental Materials Journal* **35**(2) 278-289.

Fracture Resistance and Failure Mode of Custom-made Post-and-cores of Polyetheretherketone and Nano-ceramic Composite

KN Teixeira • TM Duque • HP Maia • TMSV Gonçalves

Clinical Relevance

Custom-made post-and-cores of polyetheretherketone (PEEK) and nano-ceramic composite showed good fracture resistance and failure mode. Clinicians should be aware that these materials might be an efficient alternative for post-and-cores, because they promote esthetics while reducing the risk of root fractures.

SUMMARY

Purpose: The purpose of this study was to evaluate the fracture resistance and failure mode of custom-made post-and-cores manufactured with different esthetic materials.

Methods and Materials: A total of 48 mandibular premolar extracted teeth were selected, endodontically treated, and prepared to receive the posts. Specimens were randomly divided into four groups (n=12/group): group 1, polyetheretherketone (PEEK, Degos; G1);

group 2, nano-ceramic composite (Lava Ultimate, 3M ESPE; G2); group 3, cast metal post (NiCr alloy, control; G3); group 4, fiberglass post (Whitepost, FGM, control; G4) customized with a nano-hybrid resin composite (Z250, 3M ESPE). Experimental post-and-cores (G1 and G2) were manufactured with a computer-aided design/computer-aided manufacturing system. All posts were cemented with self-adhesive resin cement (Rely X U200, 3M ESPE), and specimens were stored in distilled water at 37°C for 60 days. A universal testing machine was used to measure the fracture resistance (0.5 mm/min at an angle of 45° to the long axis of the tooth). Fractures were classified as repairable or catastrophic. One-way analysis of variance with Tukey post hoc and χ^2 tests for independence and Bonferroni adjustments were applied ($\alpha=0.05$).

Results: The fracture resistance values (mean $\pm$ SD, in newtons) were 379.4 $\pm$ 119.8 (G1), 506.4 $\pm$ 138 (G2), 939.6 $\pm$ 146.5 (G3), and 449.6 $\pm$ 66.5 (G4). Only G3 exhibited a significant difference ($p<0.05$). The χ^2 test showed an

*Karla Nunes Teixeira, PhD, Dentistry, Federal University of Santa Catarina, Florianopolis, Brazil

Thais Mageste Duque, PhD, Dentistry, Federal University of Santa Catarina, Florianopolis, Brazil

Hamilton Pires Maia, PhD, Dentistry, Federal University of Santa Catarina, Florianopolis, Brazil

Thais Marques Simek Vega Gonçalves, PhD, Dentistry, Federal University of Santa Catarina, Florianopolis, Brazil

Corresponding author: R Eng Agrônomo Andrei Cristian Ferreira, s/n, Florianopolis 88040-900, Brazil; e-mail: karlanunest@hotmail.com

<https://doi.org/10.2341/19-080-L>

association between failure mode and post-and-core material [$\chi^2(3) = 23.65; p < 0.001$]. After Bonferroni adjustment, only G3 presented a higher than expected incidence of catastrophic failures ($p < 0.001$). In the remaining groups, most failures were repairable and related to debonding.

Conclusions: Customized post-and-cores of PEEK and nano-ceramic composites exhibited good mechanical performance. Their fracture resistance was comparable to that observed for fiberglass customized posts, yet lower than that for cast metal posts. For PEEK post-and-cores, in particular, additional studies are needed to improve adhesiveness and reduce the risk of debonding.

INTRODUCTION

Fractures can develop as a result of endodontic treatment.^{1,2} In theory, medicaments and irrigants used during root canal treatment can lead to dehydration of the tooth hard tissue, thereby weakening both the compressive and tensile strengths of dentin structure.^{2,3} On the other hand, several studies⁴⁻⁷ have demonstrated that the moisture of dentin remains stable, and these findings do not support such theory. The amount of remnant hard tissue present also appears to be a crucial factor for the long-term performance of the root-filled teeth.^{7,8} In particular, a "ferrule effect" has been observed when at least 1.5 mm of tissue remains, and this is associated with increasing the longevity of post-endodontic restorations.^{9,10}

In cases where excessive loss of dental structure has occurred and a ferrule effect is not predicted, a post-and-core system can provide structural retention of filler materials and distribution of occlusal stress along the remaining tooth structure.^{9,10} In the past, only rigid posts were available, and most of them were metallic.¹¹ Although this kind of metal post-and-core is still popular, if the root is overloaded, a catastrophic failure may occur. The latter may involve a vertical or deep root fracture with this kind of post.¹¹ Failure appears to be related to the modulus of elasticity of the metal used, which is higher than that of the supporting dentin.¹² According to Newman and others,¹³ the load to failure of a stainless steel post-and-core is significantly stronger than the load to failure of composite post-and-cores. Alternatively, the failure mode and deflection of a fiber-reinforced composite post-and-core appear to indicate that the remaining tooth structure is protected from fracture.

Some additional materials have also been tested in clinical protocols to prevent catastrophic failures of endodontically treated roots.^{13,14} According to Lanza and others,¹⁵ an ideal material for a post-and-core system should be sufficiently elastic to accompany the natural flexural movements of the tooth. Post-and-core materials with biomechanical properties similar to the dentin could also be advantageous in reducing the risk of root fractures.^{9,15} Examples of these materials are the high-performance polymers, such as polyetheretherketone (PEEK) and polyetherketoneketone (PEKK), and nano-ceramic composites, such as the LAVA Ultimate (3M ESPE, Neuss, Germany) with 80% of ceramic nanoparticles.^{16,17} Authors emphasize that PEEK and PEKK are produced as several brands, and at this point of introduction, we are not specifying a manufacturer in particular; we are talking about the materials themselves.

A previous study revealed that PEKK (246 MPa) has a similar compression strength to dentin (297 MPa), yet a lower modulus of elasticity (5.1 GPa) compared with dentin (15 GPa).¹⁷ In addition, these materials are easily processed by milling in a computer-aided design/computer-aided manufacturing (CAD/CAM) system, allowing the fabrication of a single-piece post-and-core.^{18,19} This custom-made and industrially controlled process improves the mechanical behavior of the post-and-core system, resulting in an excellent adaptation of the post to the root canal walls.²⁰ It significantly reduces the thickness of the cement layer while increasing the frictional retention of the post, which improves the mechanical behavior of the post.²⁰

Previous studies have shown that PEEK and PEKK are biocompatible and present good mechanical strength, shock-absorbing ability, and both chemical and thermal resistance.^{18,19} However, only a few studies have evaluated PEEK and PEKK applied as post-core materials.^{16,17} According to an *in silico* analysis,¹⁶ PEKK posts showed a more favorable stress distribution compared with the conventional post-and-core materials, such as gold and fiberglass. On the other hand, a higher probability of debonding has been associated with PEKK posts because the inert surface of this material is dependent on the effectiveness of the surface treatment performed. Thus, use of such materials may potentially reduce the incidence of root fractures. However, additional experimental and clinical comparative studies are necessary to prove this theory.

Therefore, the aim of this laboratory study was to evaluate the fracture resistance and failure mode of experimental post-and-cores fabricated with PEEK and nano-ceramic composites by comparing them with conventional post-and-core materials (eg, metal and customized fiberglass). The null hypotheses tested were: 1) there is no difference between the post-and-cores (PEEK and nano-ceramic composite) in terms of fracture resistance and failure mode, and (2) there is no difference in the fracture resistance or failure mode of the post-and-core materials (eg, PEEK and nano-ceramic composite) compared with controls (eg, metal and customized fiberglass).

METHODS AND MATERIALS

Experimental Design

To test the fracture resistance and failure mode of different post-and-core materials, this laboratory experimental study was performed with a randomized and blinded design. To determine the number of specimens that would be required for each test group, a power analysis was calculated using G Power software (version 3.0.10, 2008). Considering four groups, one-way analysis of variance (ANOVA), an effect size of 0.97 and power of 95% ($\alpha=0.05$), a sample of 12 specimens per group was indicated to detect significant differences. Different post-and-core materials (PEEK, nano-ceramic composite [Lava Ultimate, 3M ESPE], nickel-chromium alloy, and fiberglass customized with nano-hybrid resin composite [Z250, 3M ESPE]) were tested.

Preparation of Specimens

A total of 48 human mandibular premolar teeth with similar anatomic root canals extracted for periodontal or orthodontic reasons were selected for this study. All specimens demonstrated fully developed apices and had no cracks, caries, restorations, abrasion, erosion, or fractures. Before extraction of teeth, all patients gave consent for their teeth to be used for research purposes. The donors were fully and irreversibly made anonymous.

The bucco-lingual and mesio-distal dimensions of the teeth were measured at the highest bulge with a digital caliper (Starrett, Itú, Brazil). Teeth were carefully cleaned with periodontal curettes (Duflex, SS White, Juiz de Fora, Brazil) and stored in 0.1% thymol solution at 4°C until used. The crowns were removed at the cement-enamel junction (CEJ) with a low-speed diamond saw (Isomet-1000, Buehler, Lake Bluff, IL, USA) under water cooling to give a uniform 14-mm measurement from the apex to the CEJ. Each

root was embedded in acrylic resin (Jet, Classico, Campo Limpo Paulista, Brazil), up to 2.0 mm short of the cervical portion, using a circular polyvinyl chloride cylinder (25 mm in diameter $\times$ 25 mm high). The set (tooth, matrix, and resin) remained immobile for 72 hours to ensure resin setting.

Endodontic treatment was then performed on all 48 teeth. Root canals were prepared by a single operator using a Wave one file (Dentsply Maillefer, Ballaigues, Switzerland) 1 mm short of the apex. Next, they were irrigated copiously with 1% sodium hypochlorite solution (Asfer Industrial Química, São Caetano do Sul, Brazil) throughout chemomechanical preparation and dried with absorbent paper points (Dentsply Maillefer). The root canals were filled with gutta-percha and AH Plus sealer (Dentsply Maillefer) using the cold lateral compaction technique. They were then immersed in distilled water and incubated at $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 36 hours, corresponding to three times the endodontic sealer setting time, as recommended by the manufacturer.

The post-and-core space was prepared using #2 and #3 drills (Largo, Dentsply Maillefer) to remove 10 mm of gutta-percha from each root canal.^{21,22} This procedure was performed without irrigation because the heat produced by the drill improved gutta-percha removal.²² Any residual gutta-percha on the walls of the post space was detected with radiographic imaging. To standardize the post space, a fiberglass system drill (Whitepost DC, FGM, Joinville, Brazil) (corresponding to 0.5 post) was used to finish the root canal preparation (10 mm in length).

After each root was prepared, the corresponding teeth were randomly assigned to four groups ($n=12$ /group) according to the material used to manufacture the post-and-core (Figure 1): group 1, PEEK (DEGOS, Germany; G1); group 2, nano-ceramic composite (Lava Ultimate, 3M ESPE; G2); group 3, cast metal post (nickel-chromium (NiCr) alloy (Talmax, Brazil) (control 1; G3); group 4, fiberglass (Whitepost, FGM) customized with a nano-hybrid resin composite (Z250, 3M ESPE, St. Paul, MN, USA) (control 2; G4).

In groups 1-3, a standard resin pattern for the post-and-cores was established with prefabricated resin pins (Pin Jet, Angelus, Brazil) and an acrylic resin (Pattern Resin LS, GC, Japan). Briefly, a distance of 10 mm was marked from the resin pin to the tip to guarantee that it would be completely inserted into the prepared root canal. After, the root canal space was then isolated with water-soluble gel



Figure 1. Representative customized post-and-core from each group. G1, specimen (PEEK post-and-core); G2, specimen (nano-ceramic composite [Lava Ultimate] post-and core); G3, specimen (metal cast post-and core); G4, specimen (fiberglass customized with nano-hybrid resin composite post-and core).

Figure 2. Loading tip position on the post-and-cores and for the load test setup.

(KY, Johnson & Johnson, Brazil), a resin pin covered with the acrylic resin was inserted into the canal space once more to capture the internal anatomy of the root canal. The pin remained in the canal space until the complete polymerization of the acrylic resin was achieved.

To standardize the size and shape of the core, a master post-and-core pattern was produced ($4 \times 4 \text{ mm}^2$), with acrylic resin (Pattern Resin LS, GC) in a standard root. This master pattern was positioned into the root and then the core was molded with silicone elastomeric impression material (Empress XT, 3M ESPE) To fabricate the cores for the remaining posts, a silicone mold was filled with acrylic resin (Pattern Resin LS, GC) and then positioned over each root containing an acrylic resin pin. The resin pin was responsible for obtaining an impression of the internal anatomy of each root canal. This procedure was intended to standardize the production of post-and-cores for all groups. The corresponding acrylic resin post-and-cores produced for each root were maintained in water at room temperature for 24 hours to allow the release of monomers.

A melting cast technique and NiCr alloy (Talmax) were used to transform the resin pattern into the specimens of group 3. For the specimens of groups 1 and 2, the post-and-core resin pattern was digitally scanned (Identica Hybrid, MEDIT, Korea) and modeled with a CAD/CAM system (Dental CAD v 2.2, Exocad, Germany). Milling of the PEEK (Degos) and nano-ceramic composite (Lava Ultimate) blocks was performed by using a CAM system (S1 CAD/CAM Milling 5-Axis, VHF, Germany).

In group 4, the 0.5 post of the fiberglass system (Whitepost DC, FGM) was tested in the root to verify its capacity to achieve an internal adaptation. Briefly, the root canal space was first lubricated with a hydrosoluble gel (KY Lubricating Gel, Johnson and Johnson) and then coated with a nano-hybrid resin composite (Z250, 3M ESPE). The post was subsequently inserted into the canal, according to Gomes and others.²³ After excess resin was removed, the set was light cured for 40 seconds at 800 mW/cm^2 with a LED light-curing unit (Valo Cordless; Ultradent Products, South Jordan, UT, USA). Following polymerization, the post was removed from the canal, thereby producing a customized fiberglass post. The core was also standardized by applying the same method described above to produce a silicon elastomeric impression mold. After the composite resin filling the mold was light cured, a standardized core shape was obtained. The goal of

this procedure was to produce similar cores for all groups, thereby reducing interference during the mechanical tests.²⁴

Each finished post-and-core received a surface treatment as recommended by the manufacturer's instructions. Briefly, the post-and-cores of groups 1 and 4 were sandblasted with aluminum oxide (Al_2O_3) (for five seconds at a 30-mm distance) and washed in an ultrasonic machine. A bonding agent (Rely X ceramic primer, 3M ESPE) was subsequently applied. The group 2 post surfaces were etched with 37% phosphoric acid, washed, and then cleaned with pure water (20 seconds). In group 4, the post surfaces were cleaned with 70% alcohol, followed by application of a bonding agent (Visio.link, Bredent, Germany). Excess of adhesive on each post was removed with air jets (10 seconds). All of the bonding agents were light cured for 40 seconds at 800 mW/cm^2 with a LED light-curing unit (Valo Cordless, Ultradent).

Before post-and-core cementation, the smear layer of each prepared root canal space was removed with 3 mL 17% ethylenediaminetetraacetic acid (EDTA) solution for three minutes. After the space was flushed with 5 mL saline solution, the canals were dried with absorbent paper points (Dentsply Maillefer). A bonding agent (Adper Scotchbond Multipurpose Plus, 3M ESPE) was then applied before a self-adhesive dual-polymerized cement (RelyX U200, 3M ESPE) was applied into the root canal. The post-and-core was subsequently inserted. To standardize the force applied during cementation, a special device of the universal testing machine (4444, Instron Corporation, Norwood, MA, USA) was set to apply a constant load of 50 N for six minutes. After the excess cement was removed, an LED light-curing unit (Valo Cordless, Ultradent) (800 mW/cm^2) was applied, according to manufacturer's instructions. The specimens were stored in distilled water at 37°C for 60 days (aging simulation).

Fracture Resistance and Fracture Mode Analyses

The universal testing machine (4444, Instron Corporation) was used to evaluate fracture resistance. An increasing oblique compressive load (45°) was applied directly on the buccal edge of the core using a knife-shaped device with a round tip (2 mm diameter). The load was applied at a crosshead speed of 0.5 mm/min until fracture. The maximum failure load was recorded in newtons.

Fracture analysis was visually performed under a magnifying glass (Kozo Optical and Electronical

Instruments Co Ltd, Nanjing, China) providing $3.5\times$ magnification. For the cases involving catastrophic failure, the root fragment was removed from the acrylic resin with a Kelly hemostatic forceps to better observe the fracture present. A fracture was then classified as repairable when the fracture line was above the simulated bone level or catastrophic when a vertical root fracture was observed or if the fracture line was below the simulated bone level.

Statistical Analysis

After testing for normal distribution using the Shapiro-Wilk test, load to failure between groups was compared with one-way ANOVA. Furthermore, Tukey's honestly significant difference (HSD) post hoc test was performed for multiple comparisons. Failure mode was also analyzed with the χ^2 test and Bonferroni adjustments. The α level was set at 0.05 for all statistical testing.

RESULTS

Results for the fracture resistance test are shown in Table 1. A significant difference was observed among the materials tested in regard to fracture resistance. The cast metal post group (G3) exhibited significantly higher values of fracture resistance compared with the other groups (G1, G2, and G4; $p < 0.0001$). The PEEK (G1) and nano-ceramic composite (Lava Ultimate, 3M ESPE) (G2) groups did not differ from each other or from G4, in terms of fracture resistance.

Figure 1 illustrates finished posts of each group and an example of the customized PEEK post-and-core, cemented to the root, before and after fracture resistance analysis. Figure 2 illustrates the percentage of repairable and catastrophic failures observed in each group. Most failures observed were non-catastrophic with repairable failures, and they affected 62.5% of all of the specimens examined.

The χ^2 test identified an association between failure mode and post-and-core material [$\chi^2(3) = 23.65$; $p < 0.001$]. After Bonferroni adjustment, only G3 presented a higher than expected incidence of catastrophic failures ($p < 0.001$). In the remaining groups, the differences were not significant ($p > 0.05$).

In particular, all repairable failures observed at G1 were related to the post-core debonding, whereas in groups 2 and 4, these failures were more often linked to core chipping or fracture. The unique repairable failure observed in G3 was found to be related to post debonding.

Table 1: Mean Values $\pm$ SD for Fracture Resistance (N) According to Each Group

Groups	Post-and-core Composition	n	Mean $\pm$ SD ^a	95% CI for Mean	
				Lower Bound	Upper Bound
G1	PEEK (Degos)	12	379.4 $\pm$ 119.8 A	303.3	455.5
G2	Nano-ceramic composite (Lava Ultimate)	12	506.4 $\pm$ 138 A	418.7	594.1
G3	NiCr cast (Talmax)	12	939.6 $\pm$ 146.5 B	846.5	1032.7
G4	Fiberglass customized nano-hybrid resin composite (Z250)	12	449.6 $\pm$ 66.5 A	407.4	491.9

^a Means followed by different letters show significant difference (one-way ANOVA and Tukey HSD)

DISCUSSION

To avoid catastrophic failures, several materials have been tested as post-and-core systems, especially those presenting biomechanical properties similar to that of the dentin. Moreover, the use of CAD/CAM techniques provides an industrially controlled milling process with standard and homogeneous material blocks. As a result, the biomechanical behavior of such materials is improved. Although previous studies^{20,25-27} evaluated CAD/CAM post-and-core systems, to the best of the authors' knowledge, none of them analyzed the use of PEEK as a CAD/CAM post-and-core material.

Data support the acceptance of the first null hypothesis because similar mechanical behavior was observed when comparing fracture resistance and failure mode of the two experimental materials, namely PEEK and nano-ceramic composite (Lava Ultimate; groups 1 and 2). Literature regarding the mechanical behavior of PEEK post-and-cores milled in a CAD/CAM system is scarce, making it difficult to compare results. However, a previous *in silico* study¹⁶ revealed that, in comparison to dentin, PEEK had similar flexural strength (PEEK, 200 MPa; dentin, 212.9 MPa) but lower elastic modulus (PEEK, 5.1 GPa; dentin, 18.6 GPa). As a consequence, PEEK induces lower stress in the post-and-core components, mainly because of the flexibility of this material.¹⁶ In contrast, when the PEEK post is overloaded, stress is concentrated on the adjacent cement and core.¹⁶ This may explain the higher incidence of post-cement debonding we observed in group 1. Beyond that, in most debonding cases, the remaining cement was left inside the root canal. It is known that PEEK has an inert surface, making the bonding procedure a challenging step.¹⁹ In the present study, all of the PEEK posts were conditioned as recommended by the manufacturer. However, it appears that the surface treatment applied was not sufficient to prevent failures at the adhesive interface between PEEK and the resin cement. Moreover, fracture resistance of the PEEK group

was comparable to that of G2 and G4. Thus, further studies are necessary to evaluate the long-term clinical performance of such posts and to improve the bonding between posts and adhesive cements.

Fracture resistance observed in the nano-ceramic composite group (506.4 $\pm$ 138 N) was lower than previously documented in the literature.²⁰ According to Falcão Spina and others,²⁰ a fracture resistance of 621.3 $\pm$ 100.3 N was detected when nano-ceramic composite post-and-cores were analyzed. These contradictory results might be attributed to differences in methodology, but a similar mechanical behavior was evident in both studies. It is known that the elastic modulus of nano-ceramic composite (13 GPa) is similar to that of dentin (18 GPa).²⁸ The high number of nanoparticles (80%) and the homogeneity of the CAD/CAM blocks would also provide a better biomechanical force distribution in function. It might explain the high values of fracture resistance presented in G2 and the higher incidence of root fracture compared with G1 (PEEK) and G4 (fiberglass customized with a nano-hybrid resin composite).

It is important to emphasize that the values of fracture resistance observed in both present and previous²⁰ studies were higher than those corresponding to a normal adult occlusal force, which varies from 190-290 N (in the anterior teeth) to 200-360 N (in the posterior region).²⁹ Biofeedback of the periodontal receptors also acts to inhibit excessive muscle activity, avoiding damage to the tooth.³⁰ It significantly reduces the chance of catastrophic failure of the post-and-core systems in a real clinical situation.

A second null hypothesis was also raised, claiming no differences in fracture resistance or failure mode between the customized post-and-core materials (G1 and G2) and controls (G3 and G4). Based on these results, this hypothesis was rejected, in part, because significantly higher values of fracture resistance and a greater incidence of catastrophic failures were observed in the cast metal post-and-

core group (G3, control 1; $p < 0.0001$). This result was predicted, because the modulus of elasticity of the NiCr alloy (200 GPa) is much higher than that of dentin (18 GPa) and the other post materials (PEEK, 5.1 GPa; nano-ceramic composite, 13 GPa; customized fiberglass post, 53.8 GPa).^{16,20,31} Therefore, under excessive loading, the cast metal post produces a slow-growing crack that causes successive adhesive failure at the post–cement–root dentin interface.³² It leads to the mobility of the post within the root, consequently acting like a wedge.³² Thus, the energy accumulated in the inner post is transferred to the dentin, causing root fracture.³² This might explain the fracture behavior observed in this group, where most failures were catastrophic (91.6%). The exclusive repairable failure, observed in G3 (cast metal post), was related to debonding, which agrees with the theory described.

When we compared the G1 (PEEK) and G2 (nano-ceramic composite) experimental groups to the second control group, G4 (fiberglass post customized with nano-hybrid resin composite), the second null hypothesis was accepted, because no differences were observed. Although one study³³ reported higher values of fracture resistance for fiberglass post-and-cores, the results of the present study are consistent with other previous studies.^{20,32} There are a few possible reasons for these discrepancies. First, in the study with higher values,³³ all the fiberglass post-and-cores received a cemented ceramic crown, and this may have improved the mechanical behavior of the post-and-core system. Second, the materials of all three groups (G1, G2, and G3) present similar moduli of elasticity, which are similar to that of dentin. As a result, load distribution is improved concomitant with a reduced risk of catastrophic failures. Third, all post-and-cores of G1-G3 were customized, thereby increasing adaptation of the posts to the root walls and reducing thickness of the resin cement. This customization was achieved with CAD/CAM techniques or with a fiberglass post relining technique performed with a nano-hybrid resin composite, which captured the anatomy of the root canal. Thus, good post adaptation was found to increase frictional retention, thereby resulting in a better performance of the post-and-core systems.³⁴ This characteristic, in addition to the modulus of elasticity involved, may have contributed to a reduction in the incidence of nonrepairable fractures of such posts compared with the metal cast post of G3. Similar mechanical behavior has been reported in previous studies.^{20,35}

Nowadays, esthetics is important to both patients and clinicians. In this sense, new esthetic materials, such as PEEK, nano-ceramic composite, and fiberglass, seem to be good options for clinical practice. In addition, the use of CAD/CAM techniques produces a well-adapted post unit, with color compatibility, good optimal properties and mechanical resistance, and lower incidence of nonrepairable root fracture. Furthermore, blocks are also available in different shades, improving the esthetic outcomes, especially important for anterior restorations.

The results of the present study appear promising, although they should be interpreted with caution due to the limitations inherent to a laboratory study. For example, a single crown was not cemented to the different post-and-core systems tested. It is known that the use of crowns may influence the bonding performance of the post-and-cores because crowns can exert an additional protective factor to avoid catastrophic failures.²⁰ The aim of the present study was to evaluate the mechanical behavior of four different materials for post-and-core. Use of cemented crowns could potentially represent an external influence on force distribution, thereby interfering with the results obtained. For this reason, we applied a mechanical test directly to the cemented post-and-cores in the present study. Second, only single-rooted lower premolars were used, and a load was applied at a 45° angle. These parameters were selected because they present the worst case scenario regarding fracture resistance of endodontically treated teeth, which is the most suitable method for evaluating the behavior of post-and-core.³⁶ In addition, to achieve maximum standardization and reduce possible interferences during the mechanical test, the coronal part (core) of all posts was assembled based on a master pattern core, as described in the methods. Therefore, core sizes and dimensions were similar in all groups, as shown in Figure 1.

Finally, in the present laboratory study, static force was used to evaluate fracture resistance, and specimens were only stored in distilled water at 37°C for 60 days without thermocycling. Although the results might be useful in predicting clinical function, it is suggested that the effect of other factors, such as thermocycling and other dynamic fatigue tests or surrogate models, must be tested to more realistically evaluate the clinical applicability of the PEEK and nano-ceramic composite post-and-core systems. Another point of concern is related to the bonding strength of such experimental post-and-cores. Further studies should investigate the so-

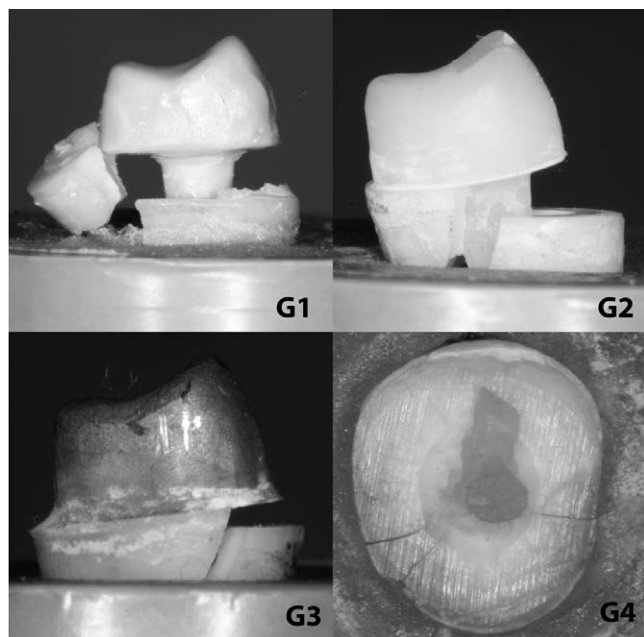


Figure 3. Catastrophic failures observed. G1, specimen (PEEK post-and-core); G2, specimen (nano-ceramic composite [Lava Ultimate] post-and core); G3, specimen (metal cast post-and core); G4, specimen (fiberglass customized with nano-hybrid resin composite post-and core).

called ideal bond strength, which must be strong enough to ensure the long-term clinical success of the restoration.

CONCLUSIONS

Within the limitations of this laboratory study, the following conclusions were drawn regarding the use of new materials for post-and-core systems. Customized post-and-cores manufactured with PEEK and a nano-ceramic composite exhibited good mechanical performance. Moreover, the fracture resistance of these post-and-cores was comparable to that of fiberglass posts customized with a nano-hybrid resin composite and lower than that of cast metal posts. The high incidence of debonding observed for PEEK post-and-cores requires further study, especially regarding the post surface conditioning. Due to the lower incidence of catastrophic failures and mechanical properties comparable to those of fiberglass customized posts, these esthetic materials should be considered as alternatives, especially when produced with CAD/CAM milling systems.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee

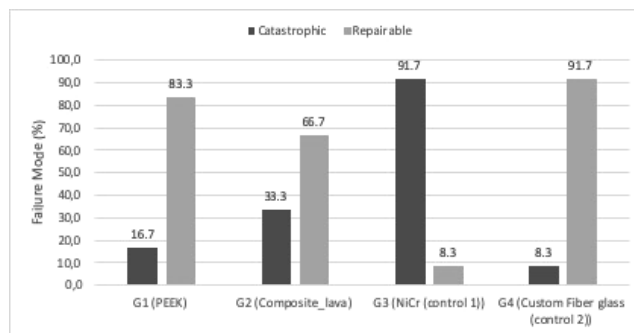


Figure 4. Failure modes (catastrophic or repairable) observed in each post-and-core group (n=12/group).

guidelines and policies of the Research Ethics Committee of the Federal University of Santa Catarina. The approval code issued for this study is: CAAE 49116315.0.0000.0121.

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 18 October 2019)

REFERENCES

- Dietschi D, Duc O, Krejci I, & Sadan A (2008) Biomechanical considerations for the restoration of endodontically treated teeth: a systematic review of the literature, part II (evaluation of fatigue behavior, interfaces, and in vivo studies) *Quintessence International* **39**(2) 117-129. 2008/06/19.
- Mannocci F & Cowie J (2014) Restoration of endodontically treated teeth *British Dental Journal* **216**(6) 341-346.
- Grigoratos D, Knowles J, Ng YL, & Gulabivala K (2001) Effect of exposing dentine to sodium hypochlorite and calcium hydroxide on its flexural strength and elastic modulus *International Endodontic Journal* **34**(2) 113-119.
- Papa J, Cain C, & Messer HH (1994) Moisture content of vital vs endodontically treated teeth *Endodontics & Dental Traumatology* **10**(2) 91-93.
- Huang TJ, Schilder H, & Nathanson D (1992) Effects of moisture content and endodontic treatment on some mechanical properties of human dentin *Journal of Endodontics* **18**(5) 209-215.
- Sedgley CM & Messer HH (1992) Are endodontically treated teeth more brittle *Journal of Endodontics* **18**(7) 332-335.
- Olusile AO & Oginni A (2004) Restoration of endodontically treated teeth: a review *The Nigerian Postgraduate Medical Journal* **11**(1) 50-57.

8. Goodacre CJ (2004) Five factors to be considered when restoring endodontically treated teeth *Practical Procedures & Aesthetic Dentistry* **16(6)** 457-462.
9. Dietschi D, Duc O, Krejci I, & Sadan A (2007) Biomechanical considerations for the restoration of endodontically treated teeth: a systematic review of the literature: part 1. Composition and micro- and macro-structure alterations *Quintessence International* **38(9)** 733-743.
10. Juloski J, Radovic I, Goracci C, Vulicevic ZR, & Ferrari M (2012) Ferrule effect: a literature review *Journal of Endodontics* **38(1)** 11-19.
11. Vire DE (1991) Failure of endodontically treated teeth: classification and evaluation *Journal of Endodontics* **17(7)** 338-342.
12. Gbadebo OS, Ajayi DM, Oyekunle OO, & Shaba PO (2014) Randomized clinical study comparing metallic and glass fiber post in restoration of endodontically treated teeth *Indian Journal of Dental Research* **25(1)** 58-63.
13. Newman MP, Yaman P, Dennison J, Rafter M, & Billy E (2003) Fracture resistance of endodontically treated teeth restored with composite posts *Journal of Prosthetic Dentistry* **89(4)** 360-367.
14. Ferrari M, Cagidiaco MC, Grandini S, De Sanctis M, & Goracci C (2007) Post placement affects survival of endodontically treated premolars *Journal of Dental Research* **86(8)** 729-734.
15. Lanza A, Aversa R, Rengo S, Apicella D, & Apicella A (2005) 3D FEA of cemented steel, glass and carbon posts in a maxillary incisor *Dental Materials* **21(8)** 709-715.
16. Lee KS, Shin JH, Kim JE, Kim JH, Lee WC, Shin SW, & Lee JY (2017) Biomechanical evaluation of a tooth restored with high performance polymer PEKK post-core system: a 3D finite element analysis *BioMed Research International* **2017** 1373127.
17. Song CH, Choi JW, Jeon YC, Jeong CM, Lee SH, Kang ES, Yun MJ, & Huh JB (2018) Comparison of the microtensile bond strength of a polyetherketoneketone (PEKK) tooth post cemented with various surface treatments and various resin cements *Materials (Basel)* **11(6)** pii E916.
18. Najeib S, Zafar MS, Khurshid Z, & Siddigui F (2016) Applications of polyetheretherketone (PEEK) in oral implantology and prosthodontics *Journal of Prosthodontic Research* **60(1)** 12-19.
19. Wiesli MG & Ozcan M (2015) High-performance polymers and their potential application as medical and oral implant materials: a review *Implant Dentistry* **24(4)** 448-457.
20. Falcao Spina DR, Goulart da Costa R, Farias IC, da Cunha LG, Ritter AV, Gonzaga CC, & Correr GM (2017) CAD/CAM post-and-core using different esthetic materials: fracture resistance and bond strengths *American Journal of Dentistry* **30(6)** 299-304.
21. Tsintsadze N, Garcia M, Grandini S, Goracci C, & Ferrari M (2015) Effect of Reciproc endodontic treatment with three different post space preparation instruments on fiber post retention *American Journal of Dentistry* **28(5)** 251-254.
22. Coniglio I, Magni E, Goracci C, Radovic I, Carvalho CA, Grandini S, & Ferrari M (2008) Post space cleaning using a new nickel titanium endodontic drill combined with different cleaning regimens *Journal of Endodontics* **34(1)** 83-86.
23. Gomes GM, Gomes OM, Gomes JC, Loguercio AD, Calixto AL, & Reis A (2014) Evaluation of different restorative techniques for filling flared root canals: fracture resistance and bond strength after mechanical fatigue *Journal of Adhesive Dentistry* **16(3)** 267-276.
24. Naas HM, Dashti MH, Hashemian R, & Hifeda NY (2014) A technique to ensure the reproducibility of a cast post and core *Journal of Prosthetic Dentistry* **112(6)** 1585-1587.
25. de Andrade GS, Tribst JP, Dal Piva AO, Bottino MA, Borges AL, Valandro LF, & Özcan M (2019) A study on stress distribution to cement layer and root dentin for post and cores made of CAD/CAM materials with different elasticity modulus in the absence of ferrule *Journal of Clinical and Experimental Dentistry* **11(1)** e1-e8.
26. Bilgin MS, Erdem A, Dilber E, & Ersoy I (2016) Comparison of fracture resistance between cast, CAD/CAM milling, and direct metal laser sintering metal post systems *Journal of Prosthodontic Research* **60(1)** 23-28.
27. Guth JF, Edelhoff D, Goldberg J, & Magne P (2016) CAD/CAM polymer vs direct composite resin core buildups for endodontically treated molars without ferrule *Operative Dentistry* **41(1)** 53-63.
28. Boschian Pest L, Cavalli G, Bertani P, Gagliani M (2002) Adhesive post-endodontic restorations with fiber posts: push-out tests and SEM observations *Dental Materials* **18(8)** 596-602.
29. Steiner M, Mitsias ME, Ludwig K, & Kern M (2009) In vitro evaluation of a mechanical testing chewing simulator *Dental Materials* **25(4)** 494-499.
30. Trulsson M (2006) Sensory-motor function of human periodontal mechanoreceptors *Journal of Oral Rehabilitation* **33(4)** 262-273.
31. Durmus G & Oyar P (2014) Effects of post core materials on stress distribution in the restoration of mandibular second premolars: a finite element analysis *Journal of Prosthetic Dentistry* **112(3)** 547-554.
32. Barcellos RR, Correia DP, Farina AP, Mesquita MF, Ferraz CC, & Cecchin D (2013) Fracture resistance of endodontically treated teeth restored with intra-radicular post: the effects of post system and dentine thickness *Journal of Biomechanics* **46(15)** 2572-2577.
33. Habibzadeh S, Rajati HR, Hajmiragha H, Esmailzadeh S, & Kharazifard M (2017) Fracture resistances of zirconia, cast Ni-Cr, and fiber-glass composite posts under all-ceramic crowns in endodontically treated premolars *Journal of Advanced Prosthodontics* **9(3)** 170-175.
34. Marcos RM, Kinder GR, Alfredo E, Quaranta T, Correr GM, Cunha LF, & Gonzaga CC (2016) Influence of the resin cement thickness on the push-out bond strength of glass fiber posts *Brazilian Dental Journal* **27(5)** 592-598.

35. Costa RG, De Moraes EC, Campos EA, Michel MD, Gonzaga CC, & Correr GM (2012) Customized fiber glass posts. Fatigue and fracture resistance *American Journal of Dentistry* **25**(1) 35-38.
36. Zicari F, Van Meerbeek B, Scotti R, & Naert I (2013) Effect of ferrule and post placement on fracture resistance of endodontically treated teeth after fatigue loading *Journal of Dentistry* **41**(3) 207-215.

Effect of Methacryloyloxydecyl Dihydrogen Phosphate—Containing Silane and Adhesive Used Alone or in Combination on the Bond Strength and Chemical Interaction With Zirconia Ceramics Under Thermal Aging

MF Gutierrez • J Perdigão • P Malaquias • AM Cardenas
F Siqueira • V Hass • A Reis • AD Loguercio

Clinical Relevance

Achieving durable bonding to zirconia is fundamental for the application of a methacryloyloxydecyl dihydrogen phosphate (MDP)—containing silane solution or an MDP-containing silane solution associated with an MDP-containing universal adhesive.

SUMMARY

Objectives: To evaluate the effect of a methacryloyloxydecyl dihydrogen phosphate (MDP)—

Mario Felipe Gutierrez, DDS, MSc, PhD, professor, Institute for Research in Dental Sciences, Faculty of Dentistry, University of Chile, Chile; Faculty of Dentistry, Universidad Finis Terrae, Chile.

Jorge Perdigão, DMD, MS, PhD, professor, Department of Restorative Sciences, University of Minnesota, Minneapolis, MN, USA

Pâmela Malaquias, PhD candidate, Department of Restorative Dentistry, School of Dentistry, State University of Ponta Grossa, Paraná, Brazil

Andres Millan Cardenas, Department of Postgraduate Program in Dentistry, CEUMA University, São Luis, MA, Brazil

Fabiana Siqueira, Department of Postgraduate Program in Dentistry, CEUMA University, São Luis, MA, Brazil

containing silane coupling agent and universal adhesive, used alone or in combination, on the microshear bond strength (μ SBS) to zirconia after 24 hours of water storage (24h) and after 10,000 thermocycles (TC), complemented with

Viviane Hass, Department of Postgraduate Program in Dentistry, CEUMA University, São Luis, MA, Brazil

Alessandra Reis, DDS, MSc, PhD, professor, Department of Restorative Dentistry, School of Dentistry, State University of Ponta Grossa, Paraná, Brazil

*Alessandro D. Loguercio, DDS, MSc, PhD, professor, Department of Restorative Dentistry, School of Dentistry, State University of Ponta Grossa, Paraná, Brazil

*Corresponding author: Rua Carlos Cavalcanti, 4748 Bloco M, Sala 64-A, Uvaranas, Ponta Grossa, Paraná, Brazil 84030-900; e-mail: aloguercio@hotmail.com

<https://doi.org/10.2341/18-093-L>

chemical analysis of the surface to establish the presence of MDP on the surface of the zirconia after bonding procedures.

Methods and Materials: Thirty computer-aided design/computed-aided manufacturing blocks of zirconia were cut into four sections (6×6×6 mm) and sintered. Zirconia sections (n=96) were assigned to 24 groups according to three factors: 1) silane (no silane, Monobond S [MBS], Monobond P [MB⁺]), 2) adhesive + resin cement (no adhesive + Enforce [ENF], no adhesive + RelyX Ultimate [REX], Prime&Bond Elect + Enforce [PBE/ENF], Scotchbond Universal + RelyX Ultimate [SBU/REX]), and 3) thermocycling (no thermocycling [24h], 10,000 thermocycles [TC]). Upon silane/adhesive application, cylinder-shaped matrices were filled with resin cement and light cured. Specimens were tested in μ SBS (1.0 mm/min) after 24h or TC. The μ SBS data were analyzed using two-way ANOVA and Tukey's post hoc test ($\alpha=0.05$). In addition, micro-Raman spectroscopy was used to analyze the zirconia surface for immediate chemical interaction analysis (n=24).

Results: For the 24h condition, PBE/ENF resulted in lower mean μ SBS than both groups with silane without PBE (MBS and MB⁺ groups; $p<0.001$). SBU alone or MB⁺ alone and MB⁺ associated with SBU showed the highest mean μ SBS ($p<0.001$). For the TC condition, all groups showed a significant decrease in mean μ SBS compared with those of 24h ($p<0.001$), with the exception of MB⁺ associated to SBU ($p>0.05$). However, the application of MB⁺ alone or MB⁺ associated to SBU resulted in higher mean μ SBS ($p<0.001$) after TC than the remaining TC groups. In terms of chemical interaction, only the SBU groups, alone or combined with both of the silane agents, were associated with the methacrylate groups after rinsing.

Conclusions: The results of the current study support the use of an MDP-containing silane solution or an MDP-containing silane solution associated with an MDP-containing universal adhesive for bonding to air-abraded zirconia, as a more stable bonding after thermocycling.

INTRODUCTION

Zirconia-based ceramics (zirconia) have been used as biomaterials for orthopedic and dental prostheses, holding a unique place among oxide ceramics

because of their excellent biomechanical properties, such as flexural strength, resistance to crack propagation, biocompatibility, adequate esthetics, and stability.¹⁻⁴ These superior mechanical properties compared with those of other all-ceramic materials make zirconia an excellent material for crowns, bridges, and frameworks in the anterior and posterior region.^{5,6} Most of the zirconia that is currently used in dentistry is tetragonal zirconia polycrystals stabilized with 3 mol% of yttria (3Y-TZP).^{1,4}

One of the major limitations of the use of zirconia is the difficulty of adhering to this material.⁷ Different luting cements have been suggested for zirconia restorations, including glass-ionomer cements, conventional resin cements, and self-adhesive resin cements. Nevertheless, resin cements are generally preferred for cementation of zirconia to improve retention and to obtain sealed restorations.⁸ Resin bonding to glass-matrix ceramics is achieved by etching the surface with hydrofluoric acid to partially dissolve the glass phase and create porosities for micromechanical attachment. A silane coupling agent is then applied to provide chemical adhesion to silica.^{9,10} As zirconia does not contain a glass phase, hydrofluoric acid etching is less effective on zirconia than on glass-matrix ceramics, when applied under the same conditions.^{2,11} The lack of silica in zirconia may also preclude chemical bonding with silane, making conventional silane less effective for zirconia bonding.^{2,12,13} Consequently, creating predictable and durable bonding to zirconia is challenging, and the most effective clinical protocol is still under discussion.¹⁴⁻¹⁷

Although no correlation has been found between surface roughness and tensile bond strength, indicating the importance of chemical bonding to zirconia,¹⁸ air-abrasion methods have been used to increase the surface area for micro-mechanical interlocking, including alumina air abrasion and tribochemical silica coating.¹⁹⁻²³ In addition, specific primers have been recently introduced to enhance chemical bonding to zirconia. The latest examples are phosphate monomer-based primers (eg, Monobond Plus, Ivoclar Vivadent, Schaan, Liechtenstein), which promote chemical adhesion between resin materials and the hydroxyl groups present on the zirconia surface.^{12,14,24,25}

Monobond Plus contains methacryloyloxydecyl dihydrogen phosphate (MDP) in an alcohol solution of a silane (3-methacryloxypropyl-trimethoxysilane, and sulfide methacrylate). The idea behind this universal primer is to make the clinical bonding

procedure easier than having to apply a separate silane and a separate adhesive. However, despite promising results, only a few studies have focused on the efficacy of this specific primer to bond to zirconia.²⁵⁻²⁹

Concomitantly with the introduction of adhesion-promoting primers for zirconia, a novel family of adhesive systems, known as “universal” adhesives, has been developed.³⁰⁻³³ Most universal adhesives contain functional monomers, such as MDP. For enamel and dentin, universal adhesives are recommended as etch-and-rinse (two-step) or self-etch (one-step) adhesives. More recently, the indication for universal adhesives has been extended to other substrates, including resin composite, glass-matrix ceramics, oxide-based ceramics, and metal alloys, without the need for additional primers.^{21,34,35}

Several studies have reported that the application of universal adhesives provides higher bond strengths to zirconia than the application of zirconia primers.^{14,23,34,36} Nevertheless, two studies^{37,38} found no differences between a zirconia primer and a universal adhesive. Therefore, there is still controversy in the literature as to whether universal adhesives can replace zirconia primers.

Thus, the aim of this study was to evaluate the effect of silane with or without MDP and the effect of universal adhesives with or without MDP on the bonding ability of resin cements to zirconia, after 24 hours of water storage and after 10,000 thermocycles (TC), using microshear bond strength (μ SBS) testing. In addition, chemical analysis of the surface by micro-Raman spectroscopy was performed to establish the presence of MDP on the surface of the zirconia after bonding procedures as a complementary evaluation. The null hypotheses tested were 1) the use of a silane and universal adhesive combined will not influence the long-term bonding to zirconia when compared with the use of separate silane and separate universal adhesive, 2) an MDP-containing silane will not influence the long-term bonding to zirconia when compared with a silane without MDP, and 3) a universal adhesive containing MDP will not influence the long-term bonding to zirconia when compared with an MDP-free universal adhesive.

METHODS AND MATERIALS

Specimen Preparation

A total of 30 computer-aided design/computed-aided manufacturing blocks (12×12×6 mm) of yttria-tetragonal zirconia polycrystal (zirconia; Ceramill, Amann Girrbach AG, Koblach, Austria) were used.

All blocks were cut into four rectangular sections (6×6×6 mm) (n=120) using a diamond disc on slow speed (Isomet, Buehler, LakeBluff, IL, USA) under water cooling. After ultrasonically cleaning in distilled water for 15 minutes, the zirconia sections were sintered in a furnace (Ceramill Therm, Amann Girrbach AG) using a universal program of 8°C per minute from 200°C to 1450°C, after two hours at a fixed temperature of 1450°C, and the correct cooling time.

Experimental Design

Ninety-six zirconia sections were randomly assigned (<http://www.sealedenvelope.com>) into 24 experimental conditions to evaluate the μ SBS (n=4) according to the following independent variables: 1) silane (no silane, Monobond S [MDP-free silane; MBS], Monobond Plus [MDP-containing silane; MB⁺], 2) universal adhesive + resin cement (no adhesive + Enforce [ENF], no adhesive + RelyX Ultimate [REX], Prime&Bond Elect [silane-free and MDP-free universal adhesive] + Enforce [PBE/ENF], Scotchbond Universal Adhesive [silane- and MDP-containing universal adhesive] + RelyX Ultimate [SBU/REX]), and 3) thermocycling (no thermocycling [24h] or after 10,000 TC). To evaluate the chemical interaction of silane and universal adhesives with zirconia surfaces by micro-Raman spectroscopy in the immediate time period, 24 zirconia sections were randomly assigned (<http://www.sealedenvelope.com>) into eight experimental conditions (n=3) (silane vs universal adhesive + resin cement). The composition, application mode, and batch numbers are described in Table 1.

Microshear Bond Strength (μ SBS)

All zirconia sections were mounted in a polyvinyl chloride (PVC) ring filled with acrylic resin (Auto-Clear, DentBras, Pirassununga, São Paulo, Brazil) displaying the zirconia surface with a height of 3 mm on the top of the cylinder. The sections were sandblasted with <50 μ m Al₂O₃ particles (2.8 bar, seven seconds), ultrasonically cleaned in distilled water for 180 seconds, washed in 96% ethanol, and dried with oil-free air. The silane and universal adhesives were then applied according to the respective manufacturer's instructions (Table 1). A single operator performed all bonding procedures.

Subsequently, seven polyethylene transparent Tygon tubes (Tygon Medical Tubing Formulations 54-HL, Saint Gobain Performance Plastics, Akron, OH, USA), with an internal diameter of 0.8 mm and

Table 1: *Materials Used, Batch Numbers, Compositions, and Application Mode*

Material	Manufacturer (Batch Number)	Composition	Application Mode
Monobond S (MBS)	Ivoclar Vivadent (R26558)	1% 3-methacryloxypropyl trimethoxysilane, ethanol/water-based solvent, acetic acid	1. Apply with a brush and let react for 60 s. 2. Apply strong stream of air to ensure solvent evaporation.
Monobond Plus (MB ⁺)	Ivoclar Vivadent (S31153)	Ethanol, 3-trimethoxysilylpropyl methacrylate, 10-MDP, sulfide methacrylate	1. Apply with a brush and let react for 60 s. 2. Apply strong stream of air to ensure solvent evaporation.
Prime & Bond Elect (PBE)	Dentsply Sirona (130811)	Adhesive: mono-, di-, and trimethacrylate resins; PENTA diketone; organic phosphine oxide; stabilizers; cetylamine hydrofluoride; acetone; water; self-curing activator: mono- and di-methacrylate resins, catalyst, photoinitiators, stabilizers, acetone, water	1. Mix one drop each of adhesive and self-curing activator. 2. Apply a generous amount of adhesive/activator to thoroughly wet all surfaces and leave undisturbed for 20 s. 3. Gently dry with clean air for at least 5 s. 4. Light cure adhesive/activator for 10 s.
Scotchbond Universal Adhesive (SBU)	3M Oral Care (523652)	10-MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	1. Apply the adhesive and leave undisturbed for 20 s. 2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent is evaporated completely.
Enforce (ENF)	Dentsply Sirona (108813H)	Base: bis-GMA, TEG-DMA, camphorquinone, EDAB, BHT; DHEPT catalyst: bis-GMA, BHT, EDAB, TEG-DMA, benzoylperoxide	1. Mix the two pastes at a 1:1 ratio. 2. Light cure for 30 s.
RelyX Ultimate (REX)	3M Oral Care (564178)	Base: silane-treated glass powder, 2-propenoic acid, 2-methyl-, 1,1-[1-(hydroxymethyl)-1,2-ethanediyl] ester, reaction products with 2-hydroxy-1,3-propanediyl DMA and phosphorus oxide, TEGDMA, silane-treated silica, oxide glass chemicals, sodium persulfate, tert-butyl peroxy-3,5,5-trimethylhexanoate, copper (II) acetate monohydrate; catalyst: silane-treated glass powder, substituted DMA, 1,12-dodecane DMA, silane-treated silica, 1-benzyl-5-phenyl-barbic-acid, calcium salt, sodium p-toluenesulfonate, 2-propenoic acid, 2-methyl-, [(3-methoxypropyl) imino]di-2,1-ethanediyl ester, calcium hydroxide, titanium dioxide	1. Mix the two pastes in the automix syringe using a standard mixing tip. 2. Light cure for 20 s.
Abbreviations: BHT, butylhydroxytoluene; Bis-GMA, bisphenol-A glycidyl dimethacrylate; DHEPT, N,N-di-(2-hydroxyethyl)-4-toluidine; EDAB, ethyl 4-dimethylamine b; HEMA, hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; PENTA, dipentaerythritol penta acrylate monophosphate; TEGDMA, triethylene glycol dimethacrylate.			

a height of 0.5 mm, were positioned over the zirconia surface in each specimen. The resin cement (Table 1) was carefully packed inside each tube, and a clear Mylar matrix strip was placed over the filled Tygon tube and pressed gently into place. The resin cement was light cured for 20 seconds using a light-emitting diode light-curing unit set at 1200 mW/cm² (Radii-cal, SDI Limited, Bayswater, Victoria, Australia). A radiometer (Demetron L.E.D. Radiometer, Kerr Sybron Dental Specialties, Middleton, WI, USA) was used to check the light intensity every five specimens. These procedures were carried out under magnifying loupes.³⁹

After bonding procedures, all zirconia sections were stored in distilled water for 24 hours at 37°C. After storage, the Tygon tubes from all zirconia sections were carefully removed with a blade to expose the cement cylinder specimen. Each specimen was examined under a stereomicroscope at 10× magnification. The cement specimen was discarded if there was evidence of porosity or gaps at the interface. Half of the groups were tested after 24 hours of storage in water, and the other half were tested after 10,000 TC between water baths held at 5°C and 55°C, with a dwell time of one minute.²²

Each PVC ring with the zirconia section was attached to a shear-testing fixture (Odeme Biotech-

nology, Joaçaba, SC, Brazil) and tested in a universal testing machine (Kratos IKCL 3-USB, Kratos Equipamentos Industriais Ltda, Cotia, São Paulo, Brazil). Then, the set zirconia section fixture was placed in the universal testing machine, and a thin orthodontic wire (0.2-mm diameter) was looped around the base of each resin cement cylinder. The setup was kept aligned (resin cement-zirconia interface, the wire loop, and the center of the load cell) to ensure the correct orientation of the shear forces.⁴⁰ The cross-head speed was set at 1 mm/min until failure.

The μ SBS (MPa) was calculated by dividing the load at failure by the surface area (mm^2). After testing, the specimens were examined under an optical microscope (SZH-131, Olympus Ltd, Tokyo, Japan) at 100 $\times$ magnification to define the location of the bond failure. The failure mode was classified as adhesive/mixed (failure at the resin cement-zirconia interface, which may include cohesive failure of the neighboring substrates), cohesive in resin cement (failure exclusively within resin cement), or cohesive in zirconia (failure exclusively within zirconia). Also, premature failures (PFs) before the test were recorded. A PF was considered as a specimen that showed porosities or other defects when examined under an optical microscope at 50 $\times$ or a specimen that showed spontaneous fracture during testing.

Chemical Interaction Analysis for Micro-Raman Spectroscopy

Two zirconia sections for each experimental treatment group were analyzed immediately. A micro-Raman spectrometer (Horiba Scientific, Tokyo, Nagoya, Japan) was first calibrated for zero and then for coefficient values using a silicon specimen. Zirconia sections were analyzed using the following Raman parameters: 20 mW HeNe laser with 632.8 nm wavelength (green laser at 532 nm), with a spectral resolution at 1 cm^{-1} , accumulation time of 30 seconds, with two coadditions, and magnification of 20 $\times$ (Olympus UK, London, UK) in a range of 100–1800 cm^{-1} . First, spectra were taken of silanes, uncured universal adhesives, and the zirconia surface separately. Then, each silane and each universal adhesive was applied according to the respective manufacturer's instructions (Table 1). The zirconia sections were subsequently rinsed for one minute in distilled water (30 seconds) and absolute ethanol (30 seconds), as described by Yoshihara and others,⁴¹ to remove the components that remained on the zirconia surface without

chemically interacting with the substrate. The spectra were obtained in triplicate, and a comparison was carried out by spectra subtraction and thus analyzed qualitatively.

Statistical Analysis

Before submitting the data for analysis using the appropriate statistical test, the Kolmogorov-Smirnov test was performed to assess whether the data followed a normal distribution, and the Bartlett's test for equality of variances was performed to determine if the assumption of equal variances was valid.⁴² After observing the normality of the data distribution and the equality of the variances, the μ SBS (MPa) data were subjected to a parametric statistical analysis. All premature failures were excluded from the statistical analysis. The μ SBS data were analyzed using two-way analysis of variance (silane vs thermocycling) for each combination of adhesive + resin cement. Tukey's post hoc test was used at $\alpha = 0.05$.

RESULTS

Microshear Bond Strength (μ SBS)

No specimens were discarded because of porosities or other defects. Most of the specimens showed adhesive/mixed failures (Table 2). However, there was an increased number of PFs for most of the groups after TC (Table 2).

Enforce resulted in the lowest 24h mean μ SBS ($p < 0.0001$; Table 3) when neither PBE adhesive nor silane was used. The use of PBE without any silane, or PBE associated with MBS, showed statistically similar and intermediary 24h mean μ SBS ($p > 0.05$; Table 3). The use of MBS alone or MB⁺ associated with PBE resulted in significantly higher 24h mean μ SBS compared with the use of the adhesive PBE without any silane ($p < 0.0001$; Table 3). The application of MB⁺ without any adhesive showed the highest 24h mean μ SBS when compared with all other groups ($p < 0.0001$; Table 3). After TC, all groups showed a significant decrease in mean μ SBS ($p < 0.0001$; Table 3). For five of the six groups, the percentage reduction in mean μ SBS ranged from 73% to 91%. The only exception was the MB⁺ silane group, which showed higher TC mean μ SBS than the other five groups, with only a 32% reduction in mean μ SBS ($p < 0.001$; Table 3) compared with the respective 24h mean μ SBS.

RelyX Ultimate resulted in the lowest 24h mean μ SBS ($p < 0.0001$; Table 3) when neither SBU adhesive nor silane was used. The use of MBS alone

Table 2: Number (%) of Specimens According to Fracture Mode

Resin Cement	Fracture Mode							
	24h				TC			
	A/M	CC	CZ	PF	A/M	CC	CZ	PF
Enforce^a								
No adhesive/no silane	28 (100)	0 (0)	0 (0)	0 (0)	16 (52)	0 (0)	0 (0)	12 (48)
Adhesive	28 (100)	0 (0)	0 (0)	0 (0)	21 (72)	0 (0)	0 (0)	7 (28)
Silane without 10-MDP (MBS)	27 (96)	1 (4)	0 (0)	0 (0)	22 (76)	0 (0)	0 (0)	6 (24)
Silane without 10-MDP (MBS) + adhesive	28 (100)	0 (0)	0 (0)	0 (0)	18 (60)	0 (0)	0 (0)	10 (40)
Silane with 10-MDP (MB ⁺)	27 (96)	1 (4)	0 (0)	0 (0)	25 (88)	3 (12)	0 (0)	0 (0)
Silane with 10-MDP (MB ⁺) + adhesive	28 (100)	0 (0)	0 (0)	0 (0)	20 (68)	2 (8)	0 (0)	6 (24)
RelyX Ultimate^b								
No adhesive/no silane	28 (100)	0 (0)	0 (0)	0 (0)	19 (64)	0 (0)	0 (0)	9 (36)
Adhesive	28 (100)	0 (0)	0 (0)	0 (0)	22 (76)	0 (0)	0 (0)	6 (24)
Silane without 10-MDP (MBS)	27 (96)	1 (4)	0 (0)	0 (0)	24 (84)	0 (0)	0 (0)	4 (16)
Silane without 10-MDP (MBS) + adhesive	27 (96)	1 (4)	0 (0)	0 (0)	27 (96)	0 (0)	0 (0)	1 (4)
Silane with 10-MDP (MB ⁺)	27 (96)	1 (4)	0 (0)	0 (0)	24 (84)	2 (8)	0 (0)	2 (8)
Silane with 10-MDP (MB ⁺) + adhesive	28 (100)	0 (0)	0 (0)	0 (0)	24 (84)	3 (12)	0 (0)	1 (4)

Abbreviations: A/M, adhesive/mixed (failure at the cement-zirconia interface, which may include cohesive failure of the neighboring substrates); CC, cohesive in cement (failure exclusively within cement); CZ, cohesive zirconia (failure exclusively within zirconia); 24h, 24 hours of water storage; TC, after thermocycling; MB⁺, Monobond Plus (Ivoclar Vivadent); MBS, Monobond S (Ivoclar Vivadent); 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; PF, premature failures (failure before the test).

^a Prime&Bond Elect was used with Enforce (Dentsply Sirona).

^b Scotchbond Universal Adhesive was used with RelyX Ultimate (3M Oral Care).

or MBS associated with SBU showed significantly higher 24h mean μ SBS only when compared with groups with no silane/no adhesive ($p < 0.0001$; Table 3). RelyX Ultimate resulted in a significantly higher 24h mean μ SBS ($p < 0.001$; Table 3) when the SBU adhesive or MB⁺ was used alone, as well as when MB⁺ was associated with SBU. After TC, most groups showed a significant decrease in mean μ SBS ($p < 0.0001$; Table 3). However, the application of resin cement with no silane/no adhesive or associated to SBU, as well as the application of MBS alone, resulted in the lowest mean μ SBS after TC, while the

application of MB⁺ alone or associated with SBU showed a similar higher mean μ SBS after TC than the other four groups ($p > 0.05$; Table 3).

Chemical Interaction Analysis for Micro-Raman Spectroscopy

Representative Raman spectra obtained with zirconia treated according to the experimental conditions, as well as after rinsing, are shown in Figure 1. The zirconia specimens were identified by the characteristic peaks of the ZrO₂ groups (267 cm⁻¹, 335 cm⁻¹, 476 cm⁻¹, 637 cm⁻¹),^{43,44} which were identified in all

Table 3: Mean of Bond Strengths (MPa) for Enforce and RelyX Ultimate to Zirconia^a

	No Adhesive/ No Silane	Adhesive ^b	Silane Without MDP (MBS)	Silane Without MDP (MBS) + Adhesive	Silane With MDP (MB ⁺)	Silane With MDP (MB ⁺) + Adhesive
24h Enforce	5.6 ± 1.3 dA	22.9 ± 2.1 cA	29.9 ± 2.8 bA	27.9 ± 7.2 bcA	36.3 ± 3.3 aA	30.4 ± 0.3 bA
TC Enforce	1.5 ± 0.1 cB	3.5 ± 0.3 bcB	5.9 ± 2.7 bB	2.6 ± 0.8 bcB	24.6 ± 3.2 aB	5.5 ± 3.0 bB
% reduction of BS	73	85	80	91	32	82
24h RelyX Ultimate	17.2 ± 1.9 eA	29.0 ± 1.1 bA	26.7 ± 2.7 cA	21.2 ± 4.8 dA	31.5 ± 4.3 abA	27.5 ± 1.6 bcA
TC RelyX Ultimate	7.7 ± 2.3 cB	11.6 ± 1.3 cB	10.4 ± 1.6 cB	15.1 ± 2.9 bB	26.5 ± 1.7 aB	26.2 ± 5.3 aA
% reduction of BS	55	60	61	28	16	5

Abbreviations: 24h, 24 hours of water storage; BS, bond strength; MB⁺, Monobond Plus (Ivoclar Vivadent); MBS, Monobond S (Ivoclar Vivadent); MDP, methacryloyloxydecyl dihydrogen phosphate; TC, after thermocycling.

^a Comparisons are valid only within resin cement. Different lowercase letters represent statistically significant differences within the same line ($p < 0.05$; horizontal comparisons). Different uppercase letters represent statistically significant differences within the same column ($p < 0.05$; vertical comparisons). Two-way analysis of variance and Tukey's test ($\alpha = 0.05$).

^b Prime&Bond Elect was used for Enforce (Dentsply Sirona), and Scotchbond Universal was used for RelyX Ultimate (3M Oral Care).

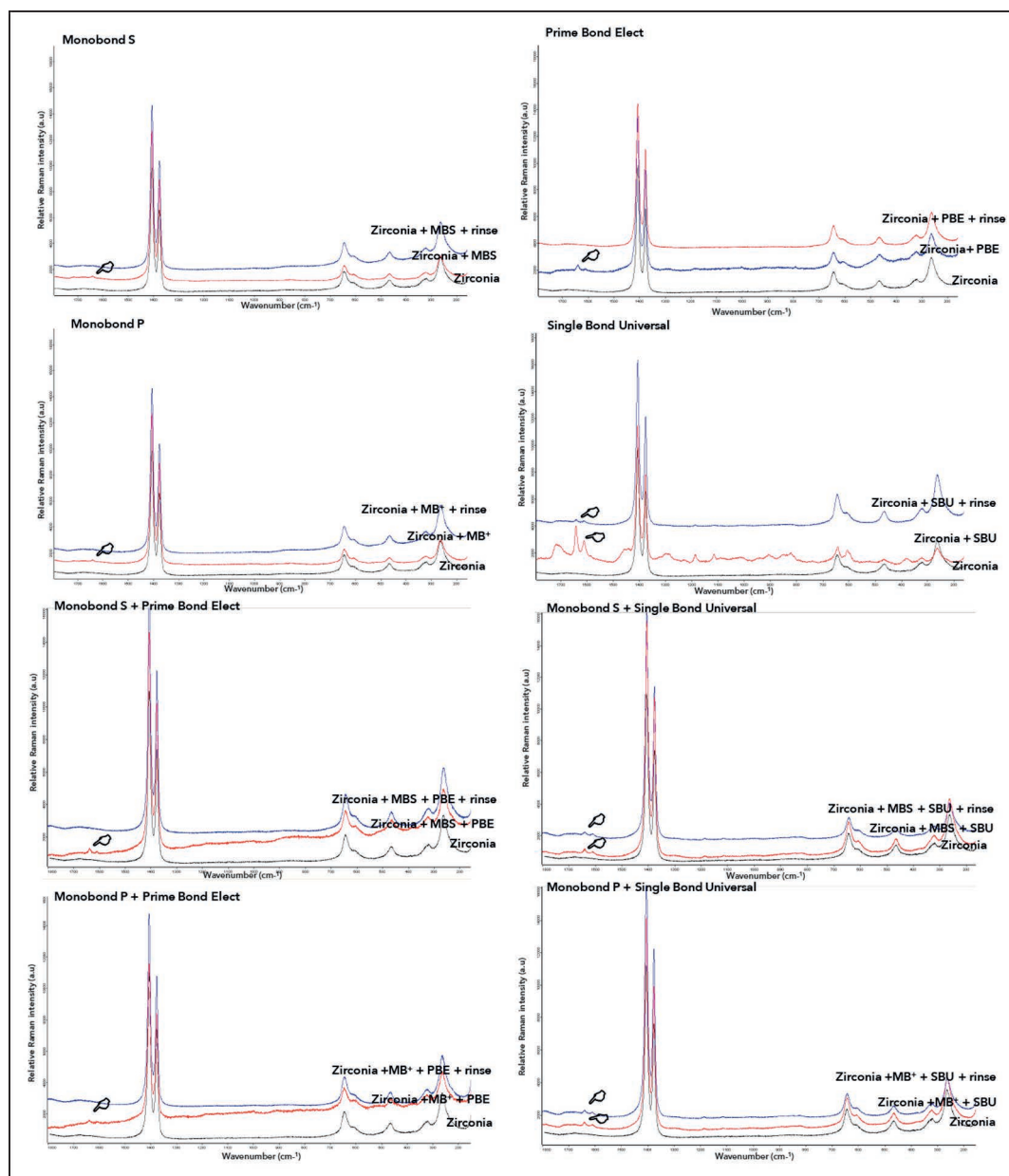


Figure 1. Representative Raman spectra for zirconia, adhesives (PBE and SBU), and silane coupling agents (MBS and MB⁺) combined with the adhesives and after rinsing, suggesting the chemical interaction/adsorption capacity. For all groups, the first spectrum (black) is representative only of the zirconia surface, in which it is possible to identify the ZrO₂ groups (267 cm⁻¹, 335 cm⁻¹, 476 cm⁻¹, 637 cm⁻¹, 1380 cm⁻¹, and 1420 cm⁻¹ peaks). The second spectrum (red) is representative of treatment of the ceramic surface (adhesive/silane coupling agent and adhesive); methacrylate peaks are evident (1610 cm⁻¹ C=C aromatic, 1640 cm⁻¹ C=C aliphatic, 1728 cm⁻¹ C=O carbonyl; black pointers on the top). The third spectrum (blue) is the result after rinsing. In this case, only the SBU groups (alone or combined with both the silane agents) are associated with the methacrylate groups (black pointer), suggesting that the monomer is kept on the zirconia surface, regardless of the presence of MDP in the silane agent.

spectra. The methacrylate peaks in the adhesives or silanes, as well as the combination of them, were characterized (1610 cm⁻¹ [C=C aromatic], 1640 cm⁻¹ [C=C aliphatic]) and identified in all spectra after treatment. However, in terms of chemical interaction, only the SBU groups, alone or combined with both of the silane agents, were associated with the methacrylate groups after rinsing, suggesting that

the monomer is kept on the zirconia surface, regardless of the presence of MDP in the silane agent.

DISCUSSION

Strong and durable adhesion to dental zirconia has eluded researchers for more than 10 years. System-

atic reviews have reported that, while mechanical and chemical pretreatment improve *in vitro* bonding to zirconia, adhesive monomers may be needed for durable chemical bonding.¹⁵⁻¹⁷ Nevertheless, MDP-based resin cements may result in improved bonding effectiveness compared to other cement types.¹⁷

While Nagaoka and others⁴⁵ reported increasing bond strengths to zirconia for higher concentrations of MDP up to 1 wt%, a recent article compared experimental primers with different concentrations of MDP (3% to 15%) with a primer without MDP. The immediate bond strengths to polished zirconia showed that the presence of MDP was more important for bonding to zirconia than the concentrations evaluated.⁴⁶

Also, Yoshida and others¹² reported that 10,000 TC significantly reduced the bond strengths for concentrations of MDP up to 1 wt%. These results after TC cannot be directly compared with our results, as the concentration of MDP in current commercial MDP-containing zirconia primers is higher than 1 wt%.⁴⁵ More recently, Go and others⁴⁷ confirmed that an increased MDP concentration did not lead to an additional enhancement in bond strength. These authors showed that the presence of MDP in a luting cement or in a primer was more important for bond strength to zirconia than the application of MDP-containing primer association with MDP-based resin cements. Another study used air-abraded zirconia²⁴ and reported that two MDP-containing primers were able to maintain bonding durability after water storage with TC as compared with when an MDP-free primer or no primer was used.

MB⁺ associated with SBU was the only group in our study for which there was not a significant reduction in mean bond strengths after TC; therefore, the first null hypothesis was rejected. Coincidentally, this was the only group for which MDP was present both in the silane and in the adhesive. MDP, either included in the composition of primers or luting cements, provides chemical bonding between methacrylate-based materials and the zirconium oxide layer present on the surface of 3Y-TZP (zirconia).^{12,45,48} While a study using secondary ion mass spectrometry suggested a chemical bond between zirconia and an MDP-containing primer,⁴⁹ the X-ray photoelectron spectroscopy results obtained by Xie and others³⁷ provided irrefutable evidence of chemical bonding, revealing that ZrO₂-MDP was well formed, with a high ratio of ZrO. More recently, Nagaoka and others⁴⁵ reported a novel chemical-bonding mechanism involving hydrogen-

bonding interactions between neighboring adsorbed phosphate groups from the MDP molecule, which might simultaneously interact with zirconia via ionic bonding.

According to Yoshida and others,¹² MDP may also react with zirconia through hydroxyl groups present on both the MDP molecule and the zirconia surface. This was also discussed by Piascik and others,⁵⁰ who reported that when the zirconia surface reacts with a phosphate ester-containing monomer (such as MDP) in the adhesive, hydroxyl groups on the ester can form hydrogen bonds with surface hydroxyl groups.⁴⁹

The pH of the solution has been reported to influence the structure of silanes.^{12,51} When MB⁺ is applied to surfaces with hydroxyl groups, the silane component is hydrolyzed in the presence of the acidic monomer MDP.¹² While silane bonds to hydroxyl groups on glass-matrix ceramic surfaces, these same hydroxyl groups are sparsely distributed on the zirconium oxide layer,¹⁸ occupying 5.4% of the entire surface. This might be enough to guarantee high 24h mean μ SBS to zirconia, as MB⁺ resulted in the highest 24h mean μ SBS for both resin cements (Table 3).

However, the application of a second coating of silane, included in the composition of SBU adhesive, might be able to react with the residual hydroxyl groups provided by the silane from the previously applied MB⁺, providing some kind of shielding effect to the MDP-containing silane. In addition, the methacrylate groups in SBU, including MDP, may copolymerize with the methacrylate groups of both silanes (including the methacrylate radical in 3-methacryloyloxypropyl trimethoxysilane of MBS and MB⁺), a reaction previously described²¹ for the interaction between this same silane molecule and a conventional resin cement. Nonetheless, this mechanism would not be solely responsible for this chemical reaction, as PBE, an MDP-free adhesive, resulted in a significant decrease (82%) in mean μ SBS after TC when it was applied over MB⁺ (Table 3). Actually, PBE may be too hydrophilic, as it does not contain 10-MDP. This monomer is quite hydrophobic because of its long carbonyl chain.⁵² The hydrophilicity of PBE might be responsible for water degradation of the adhesive interface after TC.

On the other hand, the interaction between the MB⁺ molecule and SBU may be significantly improved with the chemical bonding provided by the MDP molecule, making these reactions responsible for the statistically similar mean μ SBS of MB⁺

associated to SBU before and after TC or for the smaller reduction in mean μ SBS when MDP was not included in the silane (MBS associated to SBU). In fact, our Raman results suggest the chemical interaction of SBU with the zirconia surface independent of the application of a silane. The inclusion of MDP in the composition of SBU might explain the identification of a Raman peak after rinsing, which is compatible with a monomer. Future studies will need to be carried out to evaluate whether this interaction occurs when different MDP-containing universal adhesives are applied to the zirconia surface.

Notwithstanding, some authors have suggested that the acidity (pH=3.1) of MB⁺ is responsible for the decrease in bond strengths to zirconia⁵³ and subsequent hydrolysis of the silane component in acidic solution. It seems that the application of a less acidic MDP-containing silane did not jeopardize the bond strength to zirconia.⁴⁷ In fact, MDP bonds more efficiently to zirconia in alkaline conditions.⁵⁴

However, the formation of polysiloxane structures after the application of the silane-containing adhesive (SBU) on the zirconia surface previously treated with MB⁺ may be another potential mechanism to explain the stronger bonds of MB⁺ associated with SBU after TC compared with the application of MB⁺ or SBU separately. In fact, when MB⁺ was applied without adhesive followed by RelyX Ultimate or Enforce (Table 3), the mean μ SBSs were statistically lower after TC, corresponding to a decrease of 16% and 32%, respectively. When SBU or PBE was applied without any silane, the reduction in mean μ SBS after TC was extremely abrupt (60% and 85%, respectively; Table 3), which was identical to the reduction observed with the application of an MDP-free silane (MBS) without adhesive (61% and 80%, respectively, for RelyX Ultimate or Enforce; Table 3). Therefore, the second hypothesis was rejected.

This difference may suggest that an MDP-containing silane solution, MB⁺, is more crucial to adhesion to zirconia than the MDP-containing adhesive, SBU. The combination of an MDP-free silane (MBS) with SBU still resulted in a statistically lower mean μ SBS after TC, although the reduction was not as pronounced as that observed for the groups in which silane and adhesive were applied separately (28% for the former when compared with 60%-61% for the silane and adhesive alone). Likewise, when Enforce was used, the application of MB⁺ alone resulted in the lowest reduction in mean μ SBS after TC (32%) in comparison with all the remaining Enforce groups,

which displayed a 73% higher reduction in mean μ SBS.

Interestingly, the application of MB⁺ associated with PBE resulted in a significant decrease in mean μ SBS after TC (82%) when compared with MB⁺ alone (32%). The application of PBE, an MDP-free adhesive, might have protected the silane layer, especially when the MDP-containing silane (MB⁺) was used. However, the application of PBE deteriorated the 24h mean μ SBS to zirconia, with greater deterioration, after TC. This result indicates the rejection of the third hypothesis.

PBE is an acetone-based adhesive, while SBU is an ethanol-based adhesive. As acetone evaporates faster than ethanol,⁵² the application of only one coat of PBE may have been insufficient to promote a complete protection on the silane layer as previously reported for bonding to dentin.^{55,56} Also, PBE is more acidic than SBU. While the pH of PBE is 2.5, SBU has a pH range of 2.7 to 3.0.^{31,56,57} The higher acidity of PBE could decrease the pH of the silane solution and boost the decrease in bond strength to zirconia, as previously shown.⁵⁴ Future studies need to be carried out to evaluate the effect of universal adhesives with different pH on the interaction with zirconia.

In fact, this reduction of bond strength also occurred when the MDP-free silane MBS was used. An extreme reduction of 91% in mean μ SBS after TC occurred for the combination of MDP-free silane and PBE, as well as for the MDP-containing silane associated to PBE (82%; Table 3), suggesting that the use of PBE itself may not boost chemical bonding to zirconia intermediated by a silane. Actually, the absence of MDP, presence of acetone, and lower pH also help to explain the lower mean μ SBS after TC for PBE, as well as the higher percentage of premature failures when compared with SBU. Although there was a very similar fracture pattern between different groups at 24h and after TC, PBE had a 27.3% rate of PFs compared with 15.3% for SBU.

It is worth mentioning that, in all groups of the present study, the adhesive + resin cement from the same manufacturer were compared. This approach was followed because, during a luting procedure, the clinician usually applies adhesive + resin cement from the same manufacturer. A closer view of the results shows that in several groups, the luting procedure realized with Enforce resulted in lower 24h and TC mean μ SBS than RelyX Ultimate, mainly after TC. This means not only that the

silane/adhesive materials could be responsible for the degradation of the adhesive interface after TC but also that the resistance of the degradation of each resin cement could also be responsible. This may be a result of the physical properties of Enforce, which have resulted in statistically lower mechanical properties than RelyX ARC, a luting cement considered to be the predecessor of RelyX Ultimate.^{58,59} Resin-based materials with lower mechanical properties have been associated with weaker bond strengths.⁶⁰ Silva and others⁶¹ showed that Enforce showed greater sorption and solubility when in contact with water in comparison with Relyx ARC. This higher hydrophilicity of Enforce may influence its mechanical properties⁶² and, in the end, affect the bond strength of all groups cemented with Enforce when submitted to TC. Future studies need to be done to evaluate the hypothesis tested in the present study but while also comparing the adhesive + resin cement of different manufacturers.

Long-term water storage and thermal fatigue are challenging conditions often used to test the durability of bonding to different substrates.^{24,63-65} In the present study, we used 10,000 TC because this method has been estimated to correspond to approximately 1 year of thermal fatigue in the mouth.⁶³ As dental restorations are expected to last much longer than 1 year in the clinical environment, laboratory studies with longer thermocycling regimens are needed to confirm the results of the present study.

CONCLUSION

The results of the current study indicate that the use of an MDP-containing silane solution or an MDP-containing silane solution associated with an MDP-containing universal adhesive for bonding to air-abraded zirconia results in more stable bonding after thermocycling. The use of a silane solution or adhesive without MDP results in less stable bonding to air-abraded zirconia, mainly after thermocycling.

Acknowledgements

This study was partially supported by the National Council for Scientific and Technological Development (CNPq) under grants 305588/2014-1 and 304105/2013-9 as well as Araucária Foundation.

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 20 September 2019)

REFERENCES

- Denry I & Kelly JR (2008) State of the art of zirconia for dental applications *Dental Materials* **24**(3) 299-307.
- Thompson JY, Stoner BR, Piascik JR, & Smith R (2011) Adhesion/cementation to zirconia and other non-silicate ceramics: where are we now? *Dental Materials* **27**(1) 71-82.
- Tinschert J, Zvez D, Marx R, & Anusavice KJ (2000) Structural reliability of alumina-, feldspar-, leucite-, mica- and zirconia-based ceramics *Journal of Dentistry* **28**(7) 529-535.
- Hannink R, Kelly P, & Muddle B (2000) Transformation toughening in zirconia-containing ceramics *Journal of the American Ceramic Society* **83**(3) 461-487.
- Wendler M, Belli R, Petschelt A, Mevec D, Harrer W, Lube T, Danzer R, & Lohbauer U (2017) Chairside CAD/CAM materials. Part 2: flexural strength testing *Dental Materials* **33**(1) 99-109.
- Kelly JR & Benetti P (2011) Ceramic materials in dentistry: historical evolution and current practice *Australian Dental Journal* **56**(Supplement 1) 84-96.
- Magne P, Paranhos MP, & Burnett LH, Jr. (2010) New zirconia primer improves bond strength of resin-based cements *Dental Materials* **26**(4) 345-352.
- Campos F, Valandro LF, Feitosa SA, Kleverlaan CJ, Feilzer AJ, de Jager N, & Bottino MA (2017) Adhesive cementation promotes higher fatigue resistance to zirconia crowns *Operative Dentistry* **42**(2) 215-224.
- Calamia JR (1983) Etched porcelain facial veneers: a new treatment modality based on scientific and clinical evidence *New York Journal of Dentistry* **53**(6) 255-259.
- Tian T, Tsoi JK, Matinlinna JP, & Burrow MF (2014) Aspects of bonding between resin luting cements and glass ceramic materials *Dental Materials* **30**(7) e147-162.
- Sriamporn T, Thamrongananskul N, Busabok C, Poolthong S, Uo M, & Tagami J (2014) Dental zirconia can be etched by hydrofluoric acid *Dental Materials Journal* **33**(1) 79-85.
- Yoshida K, Tsuo Y, & Atsuta M (2006) Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler *Journal of Biomedical Materials Research Part B, Applied Biomaterials* **77**(1) 28-33.
- Matinlinna JP, Heikkinen T, Ozcan M, Lassila LV, & Vallittu PK (2006) Evaluation of resin adhesion to zirconia ceramic using some organosilanes *Dental Materials* **22**(9) 824-831.
- Amaral M, Belli R, Cesar PF, Valandro LF, Petschelt A, & Lohbauer U (2014) The potential of novel primers and universal adhesives to bond to zirconia *Journal of Dentistry* **42**(1) 90-98.
- Tzanakakis EG, Tzoutzas IG, & Koidis PT (2016) Is there a potential for durable adhesion to zirconia restorations? A systematic review *Journal of Prosthetic Dentistry* **115**(1) 9-19.

16. Inokoshi M, De Munck J, Minakuchi S, & Van Meerbeek B (2014) Meta-analysis of bonding effectiveness to zirconia ceramics *Journal of Dental Research* **93**(4) 329-334.
17. Ozcan M & Bernasconi M (2015) Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis *Journal of Adhesive Dentistry* **17**(1) 7-26.
18. Lohbauer U, Zipperle M, Rischka K, Petschelt A, & Muller FA (2008) Hydroxylation of dental zirconia surfaces: characterization and bonding potential *Journal of Biomedical Materials Research Part B, Applied Biomaterials* **87**(2) 461-467.
19. Attia A & Kern M (2011) Effect of cleaning methods after reduced-pressure air abrasion on bonding to zirconia ceramic *Journal of Adhesive Dentistry* **13**(6) 561-567.
20. Blatz MB, Phark JH, Ozer F, Mante FK, Saleh N, Bergler M, & Sadan A (2010) *In vitro* comparative bond strength of contemporary self-adhesive resin cements to zirconium oxide ceramic with and without air-particle abrasion *Clinical Oral Investigations* **14**(2) 187-192.
21. Bomicke W, Schurz A, Krisam J, Rammelsberg P, & Rues S (2016) Durability of resin-zirconia bonds produced using methods available in dental practice *Journal of Adhesive Dentistry* **18**(1) 17-27.
22. Inokoshi M, Kameyama A, De Munck J, Minakuchi S, & Van Meerbeek B (2013) Durable bonding to mechanically and/or chemically pre-treated dental zirconia *Journal of Dentistry* **41**(2) 170-179.
23. Pereira Lde L, Campos F, Dal Piva AM, Gondim LD, Souza RO, & Ozcan M (2015) Can application of universal primers alone be a substitute for airborne-particle abrasion to improve adhesion of resin cement to zirconia? *Journal of Adhesive Dentistry* **17**(2) 169-174.
24. Yang B, Barloi A, & Kern M (2010) Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin *Dental Materials* **26**(1) 44-50.
25. Azimian F, Klosa K, & Kern M (2012) Evaluation of a new universal primer for ceramics and alloys *Journal of Adhesive Dentistry* **14**(3) 275-282.
26. Passia N, Mitsias M, Lehmann F, & Kern M (2016) Bond strength of a new generation of universal bonding systems to zirconia ceramic *Journal of the Mechanical Behavior of Biomedical Materials* **62** 268-274.
27. Attia A & Kern M (2011) Long-term resin bonding to zirconia ceramic with a new universal primer *Journal of Prosthetic Dentistry* **106**(5) 319-327.
28. Sakrana AA & Ozcan M (2017) Effect of chemical etching solutions versus air abrasion on the adhesion of self-adhesive resin cement to IPS e.max ZirCAD with and without aging *International Journal of Esthetic Dentistry* **12**(1) 72-85.
29. Koizumi H, Nakayama D, Komine F, Blatz MB, & Matsumura H (2012) Bonding of resin-based luting cements to zirconia with and without the use of ceramic priming agents *Journal of Adhesive Dentistry* **14**(4) 385-392.
30. Perdigao J, Kose C, Mena-Serrano AP, De Paula EA, Tay LY, Reis A, & Loguercio AD (2014) A new universal simplified adhesive: 18-month clinical evaluation *Operative Dentistry* **39**(2) 113-127.
31. Munoz MA, Luque I, Hass V, Reis A, Loguercio AD, & Bombarda NH (2013) Immediate bonding properties of universal adhesives to dentine *Journal of Dentistry* **41**(5) 404-411.
32. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Ende A, Van Meerbeek B, & De Munck J (2012) Bonding effectiveness of a new 'multi-mode' adhesive to enamel and dentine *Journal of Dentistry* **40**(6) 475-484.
33. Perdigao J, Sezinando A, & Monteiro PC (2012) Laboratory bonding ability of a multi-purpose dentin adhesive *American Journal of Dentistry* **25**(3) 153-158.
34. Seabra B, Arantes-Oliveira S, & Portugal J (2014) Influence of multimode universal adhesives and zirconia primer application techniques on zirconia repair *Journal of Prosthetic Dentistry* **112**(2) 182-187.
35. Kim JH, Chae SY, Lee Y, Han GJ, & Cho BH (2015) Effects of multipurpose, universal adhesives on resin bonding to zirconia ceramic *Operative Dentistry* **40**(1) 55-62.
36. Zhao L, Jian YT, Wang XD, & Zhao K (2016) Bond strength of primer/cement systems to zirconia subjected to artificial aging *Journal of Prosthetic Dentistry* **116**(5) 790-796.
37. Xie H, Li Q, Zhang F, Lu Y, Tay FR, Qian M, & Chen C (2016) Comparison of resin bonding improvements to zirconia between one-bottle universal adhesives and tribochemical silica coating, which is better? *Dental Materials* **32**(3) 403-411.
38. Lopes GC, Spohr AM, & De Souza GM (2016) Different strategies to bond Bis-GMA-based resin cement to zirconia *Journal Adhesive Dentistry* **18**(3) 239-246.
39. Muñoz MA, Baggio R, Emilio Mendes YB, Gomes GM, Luque-Martinez I, Loguercio AD, & Reis A (2014) The effect of the loading method and cross-head speed on resin-dentin microshear bond strength *International Journal of Adhesion and Adhesives* **50**(Supplement C) 136-141.
40. Shimada Y, Yamaguchi S, & Tagami J (2002) Micro-shear bond strength of dual-cured resin cement to glass ceramics *Dental Materials* **18**(5) 380-388.
41. Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Torii Y, Osaka A, Suzuki K, Minagi S, Van Meerbeek B, & Van Landuyt KL (2011) Self-etch monomer-calcium salt deposition on dentin *Journal of Dental Research* **90**(5) 602-606.
42. Montgomery DC (2005) *Design and Analysis of Experiments 6th edition* John Wiley & Sons, Hoboken, NJ.
43. Durand JC, Jacquot B, Salehi H, Fages M, Margerit J, & Cuisinier FJ (2012) Confocal Raman microscopic analysis of the zirconia/feldspathic ceramic interface *Dental Materials* **28**(6) 661-671.
44. Ramos CM, Tabata AS, Cesar PF, Rubo JH, Fracisconi PA, & Sanches Borges AF (2015) Application of micro-Raman spectroscopy to the study of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) phase transformation *Applied Spectroscopy* **69**(7) 810-814.

45. Nagaoka N, Yoshihara K, Feitosa VP, Tamada Y, Irie M, Yoshida Y, Van Meerbeek B, & Hayakawa S (2017) Chemical interaction mechanism of 10-MDP with zirconia *Scientific Reports* **7** 45563.
46. Llerena-Icochea AE, Costa RM, Borges A, Bombonatti J, & Furuse AY (2017) Bonding polycrystalline zirconia with 10-MDP-containing adhesives *Operative Dentistry* **42**(3) 335-341.
47. Go EJ, Shin Y, & Park JW (2019) Evaluation of the microshear bond strength of MDP-containing and non-MDP-containing self-adhesive resin cement on zirconia *Operative Dentistry* **44**(4) 379-385.
48. Wolfart M, Lehmann F, Wolfart S, & Kern M (2007) Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods *Dental Materials* **23**(1) 45-50.
49. Chen L, Suh BI, Brown D, & Chen X (2012) Bonding of primed zirconia ceramics: evidence of chemical bonding and improved bond strengths *American Journal of Dentistry* **25**(2) 103-108.
50. Piascik JR, Swift EJ, Braswell K, & Stoner BR (2012) Surface fluorination of zirconia: adhesive bond strength comparison to commercial primers *Dental Materials* **28**(6) 604-608.
51. Miller JD, Hoh K-P, & Ishida H (1984) Studies of the simulation of silane coupling agent structures on particulate fillers: the pH effect *Polymer Composites* **5**(1) 18-28.
52. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, & Van Meerbeek B (2007) Systematic review of the chemical composition of contemporary dental adhesives *Biomaterials* **28**(26) 3757-3785.
53. Suh B & Chen L (2010) Zirconia-primer-2: Evaluation of silane priming function of zirconia/silane primers **89**(Special Issue A) Abstract 1330.
54. Xie H, Tay FR, Zhang F, Lu Y, Shen S, & Chen C (2015) Coupling of 10-methacryloyloxydecyl dihydrogenphosphate to tetragonal zirconia: effect of pH reaction conditions on coordinate bonding *Dental Materials* **31**(10) e218-e225.
55. Luque-Martinez IV, Perdigao J, Munoz MA, Sezinando A, Reis A, & Loguercio AD (2014) Effects of solvent evaporation time on immediate adhesive properties of universal adhesives to dentin *Dental Materials* **30**(10) 1126-1135.
56. Gutierrez MF, Sutil E, Malaquias P, de Paris Matos T, de Souza LM, Reis A, Perdigao J, & Loguercio AD (2017) Effect of self-curing activators and curing protocols on adhesive properties of universal adhesives bonded to dual-cured composites *Dental Materials* **33**(7) 775-787.
57. Toledano M, Sauro S, Cabello I, Watson T, & Osorio R (2013) A Zn-doped etch-and-rinse adhesive may improve the mechanical properties and the integrity at the bonded-dentin interface *Dental Materials* **29**(8) e142-e152.
58. Faria-e-Silva AL, Piva E, Lima GS, Boaro LC, Braga RR, & Martins LR (2012) Effect of immediate and delayed light activation on the mechanical properties and degree of conversion in dual-cured resin cements *Journal of Oral Science* **54**(3) 261-266.
59. Fonseca RG, Cruz CA, & Adabo GL (2004) The influence of chemical activation on hardness of dual-curing resin cements *Brazilian oral Research* **18**(3) 228-232.
60. Andrade AM, Moura SK, Reis A, Loguercio AD, Garcia EJ, & Grande RH (2010) Evaluating resin-enamel bonds by microshear and microtensile bond strength tests: effects of composite resin *Journal of Applied Oral Science* **18**(6) 591-598.
61. Silva EM, Noronha-Filho JD, Amaral CM, Poskus LT, & Guimaraes JG (2013) Long-term degradation of resin-based cements in substances present in the oral environment: influence of activation mode *Journal of Applied Oral Science* **21**(3) 271-277.
62. Ferracane JL (2006) Hygroscopic and hydrolytic effects in dental polymer networks *Dental Materials* **22**(3) 211-222.
63. Gale MS & Darvell BW (1999) Thermal cycling procedures for laboratory testing of dental restorations *Journal of Dentistry* **27**(2) 89-99.
64. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, & Van Meerbeek B (2005) A critical review of the durability of adhesion to tooth tissue: methods and results *Journal of Dental Research* **84**(2) 118-132.
65. Armstrong S, Breschi L, Ozcan M, Pfefferkorn F, Ferrari M, & Van Meerbeek B (2017) Academy of Dental Materials guidance on *in vitro* testing of dental composite bonding effectiveness to dentin/enamel using microtensile bond strength (muTBS) approach *Dental Materials* **33**(2) 133-143.

Influence of Material and Surface Roughness of Resin Composite Cements on Fibroblast Behavior

N Rohr • N Bertschinger • J Fischer • A Filippi • NU Zitzmann

Clinical Relevance

A well-polished cement surface increases the viability and spreading of gingival fibroblasts. The tested resin composite cements did not reveal any cytotoxic effects.

SUMMARY

Objective: This *in vitro* study aimed to investigate the effect of cement type and roughness on the viability and cell morphology of human gingival fibroblasts (HGF-1).

Methods and Materials: Discs of three adhesive (Panavia V5 [PV5], Multilink Automix [MLA], RelyX Ultimate [RUL] and three self-adhesive (Panavia SA plus [PSA], SpeedCem plus [SCP],

RelyX Unicem [RUN]) resin composite cements were prepared with three different roughnesses using silica paper grit P180, P400, or P2500. The cement specimens were characterized by surface roughness and energy-dispersive X-ray spectroscopic mapping. A viability assay was performed after 24 hours of incubation of HGF-1 cells on cement specimens. Cell morphology was examined with scanning electron microscopy.

Results: The roughness of the specimens did not differ significantly among the different resin composite cements. Mean Ra values for the three surface treatments were $1.62 \pm 0.34 \mu\text{m}$ for P180, $0.79 \pm 0.20 \mu\text{m}$ for P400, and $0.17 \pm 0.08 \mu\text{m}$ for P2500. HGF-1 viability was significantly influenced by the cement material and the specimens' roughness, with the highest viability for $\text{PSA} \geq \text{RUN} = \text{MLA} \geq \text{SCP} = \text{PV5} > \text{RUL}$ ($p < 0.05$) and for $\text{P2500} = \text{P400} > \text{P180}$ ($p < 0.001$). Cell morphology did not vary among the materials but was affected by the surface roughness.

Conclusion: The composition of resin composite cements significantly affects the cell viability of HGF-1. Smooth resin composite cement surfaces with an Ra of $0.2\text{--}0.8 \mu\text{m}$ accelerate flat cell spreading and formation of filopodia.

*†Nadja Rohr, Dr Med Dent, Biomaterials and Technology, Clinic for Reconstructive Dentistry, University Center for Dental Medicine Basel, Basel, Switzerland

†Nadine Bertschinger, Med Dent, Clinic for Reconstructive Dentistry, University Center for Dental Medicine Basel, Basel, Switzerland

Jens Fischer, Prof Dr Dr, Biomaterials and Technology, Clinic for Reconstructive Dentistry, University Center for Dental Medicine Basel, Basel, Switzerland

Andreas Filippi, Prof Dr, Department of Oral Surgery, University Center for Dental Medicine Basel, Basel, Switzerland

Nicola Ursula Zitzmann, Prof Dr PhD, Clinic for Reconstructive Dentistry, University Center for Dental Medicine Basel, Basel, Switzerland

*Corresponding author: Mattenstrasse 40, CH-4058 Basel, Switzerland; e-mail: nadja.rohr@unibas.ch

†Authors contributed equally to this work.

<https://doi.org/10.2341/19-113-L>

INTRODUCTION

Most dental restorations are fixed with cement material, and the selection of cement depends on the type of restoration, the restorative material, and the tooth or implant abutment surface contacting the restoration. Zinc phosphate cement has been used for decades for fixation of restorations, with exact fit providing thin cement layers of a minimum of 20 μm .¹ However, in restorations with larger cement margins, zinc oxide phosphate cement is more susceptible to hydrolytic degradation compared with resinous cements.² In laboratory and clinical situations, the cement margin width is reported to vary between 20 and 373 μm .³⁻⁷ The introduction of resin composite cements provided materials that could adhere to etched enamel and to a lower degree also to dentin surfaces.⁸ In addition, adhesion to the restoration was achieved, for instance, after etching and silanization of feldspar or lithium-disilicate ceramics. These adhesive characteristics of resin composite cements enabled the design of nonretentive restorations, even nonprep restorations, and facilitated a shift toward noninvasive preparation designs.⁹ After inserting a reconstruction with resin composite cement, excess cement is initially removed with either foam pellets or by light curing for two seconds to achieve an initial polymerization followed by removal with a hand instrument of the consequently slightly hardened cement. Especially in the subgingival and interdental area, excess cement is difficult to eliminate, and residual cement is often found on parts of the reconstruction or surrounding tissue, acting as a potential cause of inflammation.^{10,11} Afterward, the cement margin can be smoothed with fine burs and rubber polishers to remove the oxygen inhibition layer containing unreacted monomer and to prevent extensive biofilm formation.¹²

For implant reconstructions, screw retention is generally preferred because of the retrievability in cases with complications or need for restoration renewal.¹³ Direct intraoral cementation can be indicated for compensation of divergent implant angulations or when zirconia implants are used, which are mainly one-piece designs.^{14,15} Even with screw-retained implant restorations, resin composite cements are used for the fixation of computer-aided design/computer-aided manufacturing full-ceramic fixed-dental prostheses, which are extra-orally cemented on titanium-bonding bases (Ti-Base) and then screwed onto the intraosseous implant part.¹⁶

Around teeth and implants, the junctional epithelium and the gingival fibroblasts in the connective tissue compartment provide a seal between the oral cavity and the bone by their hemidesmosomes, thus preventing bacteria and bacterial toxins from migrating along the interface between soft tissue and reconstruction. The cement and restorative materials, which are in contact with soft tissues, should therefore promote fibroblast cell adhesion and growth while also impeding extensive biofilm formation.¹⁷ *In vitro* tests with human gingival fibroblasts (HGF-1) cells are commonly performed to evaluate the biocompatibility of transgingival implant components.¹⁸⁻²¹ On dental cements, the highest viability of fibroblasts was found on noneugenol zinc oxide, followed by zinc phosphate, zinc oxide, and resin composite cement.²² Furthermore, fibroblast adhesion to resin composite cement was found to be lower than to dentin and to titanium but higher than to zirconia.²³ Previously, it was demonstrated that the bacterial adhesion on dental cement is significantly influenced by the material composition^{12,22,24} and the surface roughness.²² Biofilm formation was significantly lower on resin composite cement specimens having a surface roughness with an Ra value of 0.1 μm compared to those with an Ra value of 1.2 μm .¹² However, it is currently unknown if the composition and roughness of resin composite cements affect the behavior of the fibroblasts, as was found for bacterial biofilm formation. Hence, the purpose of this comparative experimental *in vitro* study was to evaluate whether the material composition and surface roughness of composite cements influence the viability and morphology of gingival fibroblasts. The null hypothesis was that the viability of HGFs does not differ among resin composite cements of different composition and roughness.

METHODS AND MATERIALS

The viability and morphology of HGF-1 cells were investigated on three adhesive (Panavia V5 [PV5], Multilink Automix [MLA], RelyX Ultimate [RUL]) and three self-adhesive (Panavia SA plus [PSA], SpeedCem plus [SCP], RelyX Unicem 2 Automix [RUN]) resin composite cements with three different surface roughnesses. Cell viability assays and scanning electron microscopy (SEM) were performed after 24 hours of cell cultivation on the cement specimens. The cement materials were chosen in accordance with a previous study of biofilm formation⁷ and are listed in Table 1.

Table 1: *Composition of Resin Composite Cement Materials*

Code	Name	Manufacturer	Type	Monomers	Fillers	Initiators
PV5	Panavia V5	Kuraray	Adhesive resin composite cement	Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate Paste B: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate	38 vol% • Silanated barium glass filler • Silanated fluoroaluminosilicate glass filler • Colloidal silica • Silanated aluminum oxide filler Particle size: 0.01-12µm	dl-Camphorquinone
PSA	Panavia SA plus	Kuraray	Self-adhesive resin composite cement	Paste A: 10-MDP, Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, HEMA Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate	40 vol% • Silanated barium glass filler • Silanated colloidal silica Particle size: 0.02-20 µm	dl-Camphorquinone
MLA	Multilink Automix	Ivoclar Vivadent	Adhesive resin composite cement	Base paste: Bis-GMA, HEMA, 2-dimethylaminoethyl methacrylate Catalyst paste: ethoxylated bisphenol A dimethacrylate, UDMA, HEMA	40 vol% • Barium glass • Ytterbium trifluoride • Spheroid mixed oxide Particle size: 0.25-3.0 µm	Dibenzoyl peroxide
SCP	SpeedCem Plus	Ivoclar Vivadent	Self-adhesive resin composite cement	Base paste: UDMA, TEGDMA, polyethylene glycol dimethacrylate Catalyst paste: polyethylene glycol dimethacrylate, TEGDMA, methacrylated phosphoric acid ester, UDMA 1,10-decandiol dimethacrylate	40 vol% • Barium glass • Ytterbium trifluoride • Silicium dioxide Particle size: 0.1-7 µm	Dibenzoyl peroxide
RUL	RelyX Ultimate	3M ESPE	Adhesive resin composite cement	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers Catalyst paste: methacrylate monomers	43 vol% • Silanated fillers • Alkaline (basic) fillers Particle size: 13 µm	Sodium toluene-4-sulphinate, disodium peroxodisulphate, Tert-butyl 3,5,5-trimethylperoxyhexanoate
RUN	RelyX Unicem 2 Automix	3M ESPE	Self-adhesive resin composite cement	Base paste: phosphoric acid modified methacrylate monomers, bifunctional methacrylate Catalyst paste: methacrylate monomers	43 vol% • Alkaline (basic) fillers • Silanated fillers Particle size: 12.5 µm	Sodium toluene-4-sulphinate, sodium persulfate, tert-butyl 3,5,5-trimethylperoxyhexanoate

Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol A-glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Cement Specimens

Discs of each cement were produced using a ring-shaped Teflon mold with an inner diameter of 13 mm and a thickness of 1 mm. The dimension of the specimens was chosen to allow them to be fitted into a 24-well plate, where cells were later cultivated on the specimens. The mold was placed on Mylar foil located on a glass object slide. The cement was filled into the mold, which was again covered with a Mylar foil and a glass slide and fixed with clamps from two sides. The Mylar foil was used to obtain an even specimen surface and to simulate the clinical situation, in which the initial oxygen inhibition

layer would be removed by polishing the cement margin.

The cement was light cured from both sides for 60 seconds each (Elipar DeepCure-S, 3M ESPE, Seefeld, Germany) and then stored in an incubator (CTS T-4025, CTS Clima Temperatur Systeme GmbH, Hechingen, Germany) at 37°C for 15 minutes according to ISO 4049 to await further polymerization. Cement specimens were then removed from the molds, and the surfaces were wet polished (Minitech 265, Presi, Hagen, Germany) with silica carbide paper (Presi). Three different surface structures were created to imitate the following clinical situa-

tions: 1) polishing the cement margin with a rough red proxoshape diamond bur (silica carbide paper P180), 2) polishing with a coarse polisher (P400), and 3) polishing with a fine polisher (P2500).¹²

After grinding, the specimens were cleaned with distilled water in an ultrasonic bath (TPC-15, Telsonic, Bronschhofen, Switzerland) for four minutes, followed by four minutes in 70% ethanol and another four minutes in distilled water; air dried; and stored in sterile 24-well plates. This cleaning process was required to avoid contamination of cement specimens used for cell experiments.

To determine the composition of the cement materials, one specimen per cement was polished with P1200/P4000 and observed with SEM followed by an energy-dispersive X-ray (EDX) spectroscopy mapping (FEI Nova NanoSEM230, Thermo Fisher Scientific, Waltham, MA, USA) at 15 kV and 5000× magnification over 12 hours. The surface roughness of three specimens of each cement type and roughness was measured three times using a tactile profilometer (T1000/TKK50, Hommelwerke, Schwenningen, Germany). Measurements were taken over a distance of 4.8 mm (T1E, tip 5 µm 90°, 1.6 mN, Hommel-Etamic/Jenoptik, Jena, Germany). Ra values were determined as the arithmetic average of the absolute values of the profile heights (peaks/valleys) over the evaluation length.

Cell Cultivation

Human primary gingival fibroblast cells (HGF-1, ATCC American Type Culture Collection, Manassas, VA, USA) were cultivated in Dulbecco's Modified Eagle Medium (DMEM high glucose, Sigma-Aldrich, Darmstadt, Germany) with 1% L-glutamine (Gibco, Thermo Fisher Scientific), 1% sodium-pyruvate (Gibco, Thermo Fisher Scientific), 1% penicillin-streptomycin (Sigma-Aldrich), and 1% amphotericin B solution (Sigma-Aldrich) as well as 10% fetal calf serum (bioswisstec, Schaffhausen, Switzerland) at 37°C with 5% CO₂. The cultures were kept for an average of three weeks in the incubator (B5060 EK/CO₂, Heraeus, Hanau, Germany) until confluence, while the culture medium was changed every second day. Cells were then washed twice with phosphate-buffered saline (PBS) solution (Gibco, Thermo Fisher Scientific) without calcium and magnesium and detached using 0.5% trypsin/0.2% EDTA solution (Sigma-Aldrich). In 24-well plates, 10⁴ cells in a drop of 140 µL cell culture medium were seeded per specimen surface. To prevent cells from growing on well bottoms, the drops were carefully placed on each specimen. After one hour in the incubator, an

additional 200 µL of cell culture medium was added, and cells were cultivated for another 23 hours in the incubator. Polystyrene discs (Thermanox, Faust Laborbedarf, Schaffhausen, Switzerland) served as the control.

Cell Viability Assay

To evaluate the effect of cement material and roughness on gingival fibroblasts, a WST-1 cell viability assay (cell proliferation reagent WST-1, Sigma Aldrich) was performed after 24 hours. In this assay, the stable water-soluble tetrazolium salt WST-1 is converted to a soluble formazan by a cellular mechanism, and the amount of formazan directly correlates to the number of metabolically active cells. Cells were seeded on all cements with three different roughnesses in triplicates, meaning three specimens per cement and roughness were seeded, and the experiments were repeated three times (n=9 per group; 162 specimens in total).

Polystyrene discs with a diameter of 13 mm served as control (n=9). In addition, one specimen per group and experiment was not seeded with cells and served as a blank (n=3 per group). After 24 hours, specimens were rinsed once with PBS, and 300 µL of WST-1 solution mixed with DMEM (1:3) was added to each well and incubated for two hours. Afterward, 200 µL of the supernatant was transferred to a 96-well plate, and the optical density (OD) was recorded at 490 nm with a microplate reader (RT-2100C Microplate Reader, Versamax, Molecular Devices LLC, San Jose, CA, USA). A cell viability of 100% was attributed to the cells grown on polystyrene control discs. The relative cell viability was calculated using the following equation:

Relative cell viability =

$$(OD_{\text{specimen}} - OD_{\text{blank specimen}}) / (OD_{\text{control}} - OD_{\text{blank control}}).$$

Cell Morphology

Cell morphology after 24 hours was visualized on one additional specimen per group for three repetitions (n=3 per group) using SEM (FEI Nova NanoSEM230, Thermo Fisher Scientific) at 15 kV. Cells on the specimens were rinsed with PBS, fixed with 2.5% glutaraldehyde (Sigma-Aldrich) for 20 minutes at room temperature, rinsed with PBS and distilled water, dehydrated with ethanol (30%, 50%, 70%, 80%, 90%, 96%, abs), critical point dried (Autosamdri-815, Tousimis Research Corporation, Rockville, MD, USA), and gold sputtered.

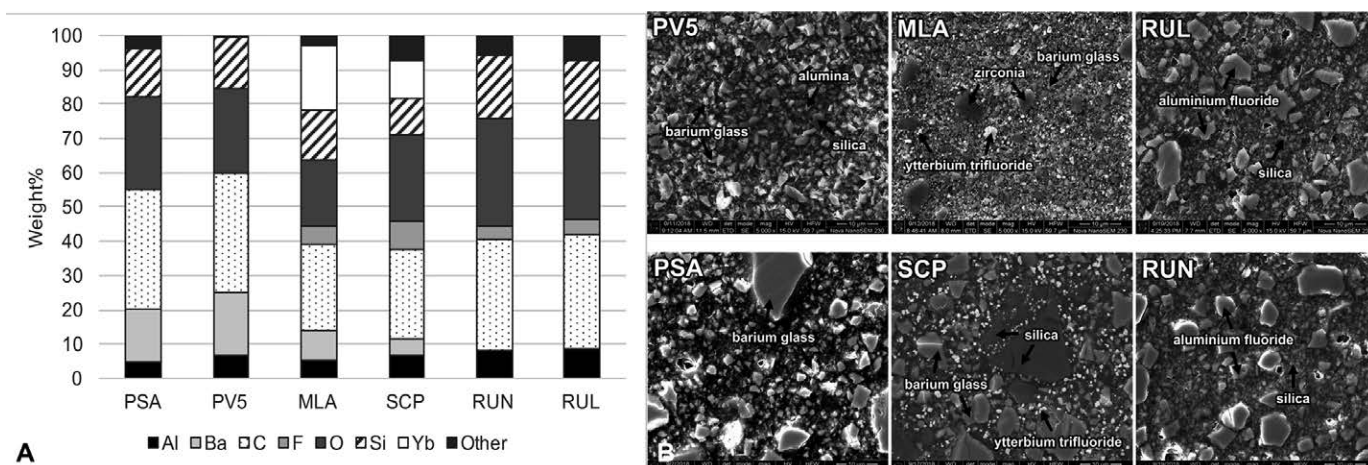


Figure 1. (A): EDX analysis of elements present in weight% at resin composite cement surfaces. (B): SEM images of resin composite cement surfaces. Ceramic fillers determined with EDX mapping are indicated with black arrows. PV5, Panavia V5; PSA, Panavia SA plus; MLA, Multilink Automix; SCP, SpeedCem plus; RUL, RelyX Ultimate; RUN, RelyX Unicem 2 Automix.

Statistical Analysis

Values of surface roughness and cell viability were tested for normal distribution using Shapiro-Wilk test. Two-way analysis of variance was applied afterward to determine the effect of roughness and material. Differences within subgroups were evaluated with post hoc Fisher least significant difference test. The level of significance was set to $\alpha = 0.05$.

RESULTS

Cement Characterization

EDX mapping of the cements provided the weight% of the elements present on the surfaces of the specimens (Figure 1A). Based on the distribution of the elements displayed in the corresponding SEM images, the filler composition was determined (Figure 1B).

There was no statistical difference in Ra values between the different cement types when polished to the same roughness ($p=0.204$). The three different polishing approaches resulted in significantly different Ra values, with $Ra = 1.62 \pm 0.34 \mu\text{m}$ for P180, which was significantly higher than $Ra = 0.79 \pm 0.20 \mu\text{m}$ for P400 ($p<0.001$), and significantly higher than $Ra = 0.17 \pm 0.08 \mu\text{m}$ for P2500 ($p<0.001$).

Cell viability

Cement material ($p=0.002$) and surface roughness ($p<0.001$) significantly affected the viability of HGF-1 cells. Cell viability was significantly highest for $PSA \geq RUN = MLA \geq SCP = PV5 > RUL$ ($p<0.05$). Surface treatment with silica carbide paper grit 180

resulted in significantly lower cell viability ($p<0.001$) than treatment with grit 400 and 2500, which did not differ significantly ($p=0.606$). Mean cell viability varied between $73\% \pm 6\%$ for RUL (P180) and $92\% \pm 13\%$ for PSA (P400) and was smallest for P180 for all cements (Table 2). The correlation between cell viability and the surface roughness of the specimens on which the cells were cultured is displayed in Figure 2.

Cell Morphology

SEM images of cells on the specimens revealed no visible differences in cell morphology or growth pattern of the fibroblasts between the different cements with the same surface roughness. On rough specimens (P180 $Ra=1.62 \mu\text{m}$), fibroblast cells aligned themselves parallel to the direction of the grinding grooves and displayed an elongated morphology, while on smooth surfaces (P2500 $Ra=0.17 \mu\text{m}$), cells spread planar and without a specific orientation (Figure 3). On surfaces treated with silica paper grit P400 ($Ra=0.79 \mu\text{m}$), both cell morphologies were observed. Filopodia were formed by cells on all surfaces but most prominently by cells on surfaces treated with silica paper grit P2500.

DISCUSSION

This *in vitro* study investigated the influence of different resin composite cements with varying surface roughnesses on the viability of gingival fibroblasts. The results demonstrated that cell viability was significantly higher on surfaces with a roughness Ra value of between 0.2 and 0.8 μm ;

Table 2: *Relative Cell Viability (%) of HGF-1 on Resin Composite Cements PV5, PSA, MLA, SCP, RUL, and RUN With Surface Pretreatment Grits P180, P400, and P2500^a*

%	PV5	PSA	MLA	SCP	RUL	RUN
P180	78 ± 11 ^{A,B,a}	78 ± 11 ^{A,B,a}	80 ± 9 ^{A,a}	76 ± 9 ^{A,B,a}	73 ± 6 ^{B,a}	78 ± 6 ^{A,B,a}
P400	83 ± 14 ^{A,a,b}	92 ± 13 ^{B,b}	88 ± 12 ^{B,b}	88 ± 8 ^{B,b}	78 ± 12 ^{A,a}	90 ± 8 ^{B,b}
P2500	87 ± 15 ^{A,b}	91 ± 21 ^{A,b}	87 ± 11 ^{A,b}	85 ± 6 ^{A,b}	80 ± 8 ^{B,a}	88 ± 10 ^{A,b}

^a Data are mean ± standard deviation. Statistically significant differences (p<0.05) between groups determined with post hoc Fisher least significant difference test are indicated with superscript letters (uppercase horizontal comparison, lowercase vertical comparison). PV5: Panavia V5; PSA: Panavia SA plus; MLA: Multilink Automix; SCP: SpeedCem plus; RUL: RelyX Ultimate; RUN: RelyX Unicem 2 Automix^a

hence, the null hypothesis was rejected. On these smoother surfaces, cells spread flat, and the formation of filopodia was observed.

Based on the composition given by the manufacturers in combination with the mapping images determined with SEM/EDX, the respective inorganic fillers of the resin composite cements could be identified. The characterization of the specimens' filler contents and morphologies revealed that similar inorganic fillers were selected for the adhesive and self-adhesive cement by the same manufacturer, especially for RUL and RUN, which also presented identical surface structures. For PV5 barium glass, alumina and silica could be identified, while PSA was mainly composed of irregular-sized barium glass fillers. Barium glass and additionally ytterbium trifluoride were present in MLA and SCP. To MLA, zirconia fillers were added, while SCP contained chunks of silica fillers. Both RUN and RUL contained silica particles and aluminum fluoride fillers. The weight% of the analyzed elements corresponded to the fillers visible in the SEM images. Neither the type of cement, adhesive or self-adhesive, nor the cement composition correlated with cell viability among the investigated cements.

Using EDX analysis, only inorganic compositions could be determined, and organic compositions are displayed as contents of carbon and partially oxygen.

In addition, elements present in very small amounts, such as copper, which may have affected cell behavior, could not be captured because of the limited sensitivity of the detector and the presence of heavier elements that potentially covered the signal from lighter elements. For the organic components, it was reported that monomer release induces toxic effects, especially for cements that are not sufficiently polymerized.²⁵ The *in vitro* cytotoxicity of monomers to HGFs has been reported to be significantly highest for bisphenol A-glycidyl methacrylate (PV5, PSA, MLA) > urethane dimethacrylate (MLA, SCP) > triethylene glycol dimethacrylate (PV5, PSA, SCP) > 2-hydroxyethyl methacrylate (PSA, MLA).^{26,27} However, in the present study, the contents of those monomers in the cements as indicated by the manufacturers were not correlated with cell viability. The manufacturer of the RUL and RUN cements did not specify their monomer contents. It was demonstrated that effective polymerization of methacrylate-based materials improves their biocompatibility, physical properties, and clinical performance.²⁸ Measurements of the polymerization degree of the cement specimens and consequently an estimation of potential monomers leaking from the surfaces would have allowed further interpretation of the results.

On all cements—except RUL on the roughest surface—fibroblast viability values of HGF-1 were greater than 75% compared with the control cells grown on polystyrene, which is categorized as noncytotoxic according to ISO standard 10993-5.

The effect of surface roughness on the behavior of HGF-1 is controversial.²⁹⁻³² A surface roughness with an Ra value between 0.15 and 0.25 μm has been proposed to provide optimal conditions for growth of HGF-1 cells.³¹ The surface roughness of the specimens in the present study was chosen in accordance with the roughness relevant in a clinical situation by

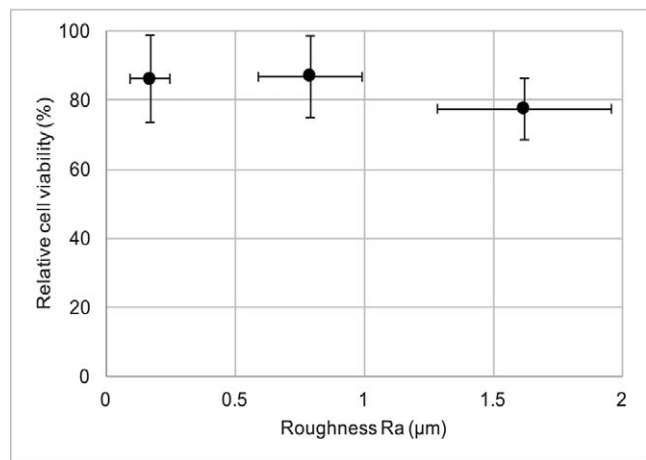


Figure 2. Correlation between surface roughness and relative cell viability for HGF-1 on all cements.

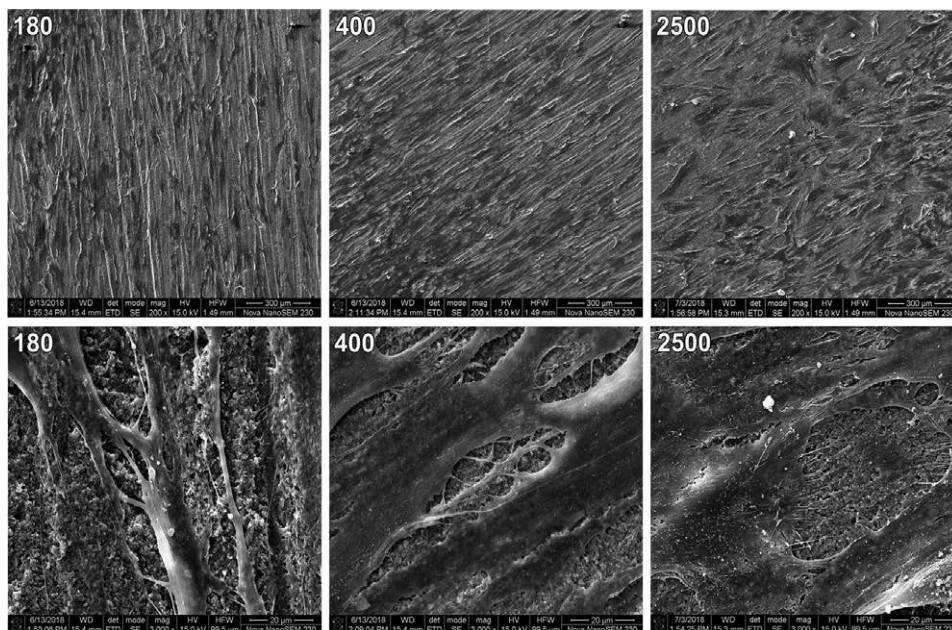


Figure 3. SEM images of HGF-1 cells on a randomly selected cement (MLA: Multilink Automix) surface with pretreatment of grit P180, P400, or P2500 (first row 200 $\times$, second row 3000 $\times$).

polishing the cement margin with a diamond bur ($R_a=1.6\text{ }\mu\text{m}$) or a coarse ($R_a=0.8\text{ }\mu\text{m}$) or fine intraoral polisher ($R_a=0.2\text{ }\mu\text{m}$).¹² Since the viability of HGF-1 cells on cement surfaces with an R_a value of $1.6\text{ }\mu\text{m}$ was significantly lower than on smoother surfaces, the cement margin should be finished with rubber polishers, which agrees with previous findings for biofilm formation on resin composite cements.¹²

Interestingly, it has been proposed that cell attachment is influenced by surface roughness between $R_a\text{ }0.5\text{ }\mu\text{m}$ and $0.2\text{ }\mu\text{m}$ and that wettability plays a major role in the surface roughness at an R_a value of $0.2\text{ }\mu\text{m}$ or less.³³ It can be speculated that the cement's microstructure, defined by the filler size and distribution, which are not considered within the R_a measurements, may have also affected the cell viability. Since the morphology of RUL and RUN was very similar while different cell viability was observed, it is more likely that the difference between those cements is due to the chemical composition of the surface.

HGFs spread more widely, and the formation of filopodia was increased, on smooth surfaces with an R_a value $<0.8\text{ }\mu\text{m}$ than on the rough specimens with an R_a value of $1.6\text{ }\mu\text{m}$. The behavior of HGFs on rough compared with smooth surfaces has been described in several studies on titanium.^{17,34,35} The current study confirms this behavior on all tested cements. HGFs were oriented on rougher surfaces along grooves, which is referred to as contact guidance.^{17,34,35} Cells on smooth surfaces were

randomly distributed and not oriented. The cell size of HGF-1 ($15\text{ to }150\text{ }\mu\text{m}$)^{36,37} was within the range of the width of the cement margin ($20\text{ to }373\text{ }\mu\text{m}$).³⁻⁷ During cementation in a clinical situation, cement is often smeared over larger areas of the reconstruction and is difficult to be fully retrieved. Therefore, the surface structure and cytotoxic potential of cements are important factors influencing the adaption of gingival tissue around a tooth or an implant abutment.

The present results on how the inorganic composition and surface roughness potentially affect cell behavior highlights the complexity of material influencing cell behavior. Numerous other factors such as surface wettability, aging, and the formation of an oxygen inhibition layer, as well as the interaction with saliva and blood at the cement surfaces, may also affect the behavior of fibroblasts in a clinical situation and should be evaluated in further studies.

CONCLUSIONS

This study is the first to provide *in vitro* data on how the surface roughness and composition of resin composite cements affect HGF cell viability. A smooth surface increases viability after 24 hours. Hence, clinicians should finish the surface of the cement margin with rubber polishers to obtain an optimal substrate for fibroblast growth. With respect to viability, all tested resin composite cements displayed no cytotoxic reactions and can be recommended for clinical use.

Acknowledgements

The authors are grateful to Fredy Schmidli for performing the SEM imaging and to Martha Imholz for the cell laboratory support at the University of Basel.

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 Center for Dental Medicine, University of Basel, Switzerland.

Conflict of Interest

In addition to his function as the head of the Division of Dental Materials and Engineering at the University of Basel, Switzerland, Jens Fischer is the manager of research and development at VITA Zahnfabrik, Bad Säckingen, Germany. All other authors declare no conflict of interest.

(Accepted 31 August 2019)

REFERENCES

- White SN & Yu Z (1992) Film thickness of new adhesive luting agents *Journal of Prosthetic Dentistry* **67**(6) 782-785.
- Fraga RC, Luca-Fraga LR, & Pimenta LA (2000) Physical properties of resinous cements: an *in vitro* study *Journal of Oral Rehabilitation* **27**(12) 1064-1067.
- Bindl A & Mörmann WH (2003) Clinical and SEM evaluation of all-ceramic chair-side CAD/CAM-generated partial crowns *European Journal of Oral Sciences* **111**(2) 163-169.
- Rojpaibool T & Leevailoj C (2017) Fracture resistance of lithium disilicate ceramics bonded to enamel or dentin using different resin cement types and film thicknesses *Journal of Prosthodontics* **26**(2) 141-149.
- Arena A, Baldissara P, Ciocca L, Scotti R, & Monaco C (2019) Influence of preparation design and spacing parameters on the risk of chipping of crowns made with Cerec Bluecam before cementation *Journal of Prosthodontic Research* **63**(1) 100-104.
- Zaugg LK, Zehnder I, Rohr N, Fischer J, & Zitzmann NU (2018) The effects of crown venting or pre-cementing of CAD/CAM-constructed all-ceramic crowns luted on YTZ implants on marginal cement excess *Clinical Oral Implants Research* **29**(1) 82-90.
- Kokubo Y, Tsumita M, Kano T, Sakurai S, & Fukushima S (2011) Clinical marginal and internal gaps of zirconia all-ceramic crowns *Journal of Prosthodontic Research* **55**(1) 40-43.
- Rohr N & Fischer J (2017) Tooth surface treatment strategies for adhesive cementation *Journal of Advanced Prosthodontics* **9**(2) 85-92.
- Edelhoff D & Özcan M (2007) To what extent does the longevity of fixed dental prostheses depend on the function of the cement? Working group 4 materials: cementation. *Clinical Oral Implants Research* **18**(Supplement 3) 193-204.
- Sancho-Puchades M, Crameri D, Özcan M, Sailer I, Jung RE, Hämmerle CHF, & Thoma DS (2017) The influence of the emergence profile on the amount of undetected cement excess after delivery of cement-retained implant reconstructions *Clinical Oral Implants Research* **28**(12) 1515-1522.
- Anami LC, Pereira CA, Guerra E, Assunção e Souza RO, Jorge AO, & Bottino MA (2012) Morphology and bacterial colonisation of tooth/ceramic restoration interface after different cement excess removal techniques *Journal of Dentistry* **40**(9) 742-749.
- 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 Sciences* **125**(4) 294-302.
- Staubli N, Walter C, Schmidt JC, Weiger R, & Zitzmann NU (2017) Excess cement and the risk of peri-implant disease—a systematic review *Clinical Oral Implants Research* **28**(10) 1278-1290.
- Pieralli S, Kohal RJ, Jung RE, Vach K, & Spies BC (2017) Clinical outcomes of zirconia dental implants: a systematic review *Journal of Dental Research* **96**(1) 38-46.
- Balmer M, Spies BC, Vach K, Kohal RJ, Hämmerle CHF, & Jung RE (2018) Three-year analysis of zirconia implants used for single-tooth replacement and three-unit fixed dental prostheses: a prospective multicenter study *Clinical Oral Implants Research* **29**(3) 290-299.
- Lopes ACO, Machado CM, Bonjardim LR, Bergamo ETP, Ramalho IS, Witek L, Coelho PG, & Bonfante EA (2019) The effect of CAD/CAM crown material and cement type on retention to implant abutments *Journal of Prosthodontics* **28**(2) 552-556.
- Ferraris S, Truffa Giachet F, Miola M, Bertone E, Varesano A, Vineis C, Cochis A, Sorrentino R, Rimondini L, & Spriano S (2017) Nanogrooves and keratin nanofibers on titanium surfaces aimed at driving gingival fibroblasts alignment and proliferation without increasing bacterial adhesion *Materials Science and Engineering C Materials for Biological Applications* **76** 1-12.
- Yang Y, Zhou J, Liu X, Zheng M, Yang J, & Tan J (2015) Ultraviolet light-treated zirconia with different roughness affects function of human gingival fibroblasts *in vitro*: the potential surface modification developed from implant to abutment *Journal of Biomedical Materials Research—Part B: Applied Biomaterials* **103**(1) 116-124.
- Raffaelli L, Rossi Iommetti P, Piccioni E, Toesca A, Serini S, Resci F, Missori M, De Spirito M, Manicone PF, & Calviello G (2008) Growth, viability, adhesion potential, and fibronectin expression in fibroblasts cultured on zirconia or feldspathic ceramics *in vitro* *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **86**(4) 959-968.
- Neunzehn J, Lüttenberg B, & Wiesmann HP (2018) Investigation of biomaterials by human epithelial gingiva cells: an *in vitro* study *Head & Face Medicine* **8** 35.
- Grenade C, De Pauw-Gillet MC, Gailly P, Vanheusden A, & Mainjot A (2016) Biocompatibility of polymer-infiltrated-ceramic-network (PICN) materials with human gingival fibroblasts (HGFs) *Dental Materials* **39**(2) 1152-1164.

22. Rodriguez LC, Saba JN, Chung KH, Wadhvani C, & Rodrigues DC (2017) *In vitro* effects of dental cements on hard and soft tissues associated with dental implants *Journal of Prosthetic Dentistry* **118**(1) 31-35.
23. Mehl C, Kern M, Schütte AM, Kadem LF, & Selhuber-Unkel C (2016) Adhesion of living cells to abutment materials, dentin, and adhesive luting cement with different surface qualities *Dental Materials* **32**(12) 1524-1535.
24. Raval NC, Wadhvani CP, Jain S, & Darveau RP (2015) The interaction of implant luting cements and oral bacteria linked to peri-implant disease: an *in vitro* analysis of planktonic and biofilm growth—a preliminary study *Clinical Implant Dentistry and Related Research* **17**(6) 1029-1035.
25. Lin-Gibson S, Sung L, Forster AM, Hu H, Cheng Y, & Lin NJ (2009) Effects of filler type and content on mechanical properties of photopolymerizable composites measured across two-dimensional combinatorial arrays *Acta Biomaterialia* **5**(6) 2084-2094.
26. Reichl FX, Esters M, Simon S, Seiss M, Kehe K, Kleinsasser N, Folwaczny M, Glas J, & Hickel R (2006) Cell death effects of resin-based dental material compounds and mercurials in human gingival fibroblasts *Archives of Toxicology* **80**(6) 370-377.
27. Gupta SK, Saxena P, Pant VA, & Pant AB (2012) Release and toxicity of dental resin composite. *Toxicology International* **19**(3) 225-234.
28. Ergun G, Egilmez F, & Yilmaz S (2011) Effect of reduced exposure times on the cytotoxicity of resin luting cements cured by high-power LED *Journal of Applied Oral Sciences* **19**(3) 286-292.
29. Nothdurft FP, Fontana D, Ruppenthal S, May A, Aktas C, Mehraein Y, Lipp P, & Kaestner L (2015) Differential behavior of fibroblasts and epithelial cells on structured implant abutment materials: a comparison of materials and surface topographies *Clinical Implant Dentistry and Related Research* **17**(6) 1237-1249.
30. Rutkunas V, Bukelskiene V, Sabaliauskas V, Balciunas E, Malinauskas M, & Baltriukiene D (2015) Assessment of human gingival fibroblast interaction with dental implant abutment materials *Journal of Materials Science: Materials in Medicine* **26**(4) 169.
31. Gómez-Florit M, Ramis JM, Xing R, Taxt-Lamolle S, Haugen HJ, Lyngstadaas SP, & Monjo M (2014) Differential response of human gingival fibroblasts to titanium- and titanium-zirconium-modified surfaces *Journal of Periodontal Research* **49**(4) 425-436.
32. Mustafa K, Silva Lopez B, Hultenby K, Wennerberg A, & Arvidson K (1998) Attachment and proliferation of human oral fibroblasts to titanium surfaces blasted with TiO₂ particles: a scanning electron microscopic and histomorphometric analysis *Clinical Oral Implants Research* **9**(3) 195-207.
33. Kim YS, Shin SY, Moon SK, & Yang SM (2015) Surface properties correlated with the human gingival fibroblasts attachment on various materials for implant abutments: a multiple regression analysis *Acta Odontologica Scandinavica* **73**(1) 38-47.
34. Guillem-Marti J, Delgado L, Godoy-Gallardo M, Pegueroles M, Herrero M, & Gil FJ (2013) Fibroblast adhesion and activation onto micro-machined titanium surfaces *Clinical Oral Implants Research* **24**(7) 770-780.
35. Pae A, Lee H, Kim HS, Kwon YD, & Woo YH (2009) Attachment and growth behaviour of human gingival fibroblasts on titanium and zirconia ceramic surfaces *Biomedical Materials* **4**(2) 025005.
36. Qin Z, Worthen CA, & Quan T (2017) Cell-size-dependent upregulation of HGF expression in dermal fibroblasts: impact on human skin connective tissue aging *Journal of Dermatological Science* **88**(3) 289-297.
37. Herráez-Galindo C, Rizo-Gorrita M, Luna-Oliva I, Serera-Figallo MÁ, Castillo-Oyagüe R, & Torres-Lagares D (2019) *In vitro* comparative study of fibroblastic behaviour on polymethacrylate (PMMA) and lithium disilicate polymer surfaces *Polymers* **11**(4) E744.

Simulated Cuspal Deflection and Flexural Properties of Bulk-Fill and Conventional Flowable Resin Composites

Y Shimatani • A Tsujimoto • WW Barkmeier • NG Fischer
Y Nagura • T Takamizawa • MA Latta • M Miyazaki

Clinical Relevance

Some bulk-fill flowable resin composites produce less cuspal deflection than a conventional incrementally filled flowable resin composites.

SUMMARY

Objective: This study investigated simulated cuspal deflection and flexural properties of bulk-fill and conventional flowable resin composites.

Methods and Materials: Five bulk-fill and six conventional flowable resin composites were evaluated. Aluminium blocks with a mesio-occlusal-distal cavity were prepared and randomly divided into groups for each of the different measurement techniques and were further subdivided according to the type of flowable resin composite. The simulated cuspal deflection caused by the polymerization of

resin composite within an aluminium block was measured using a highly accurate submicron digimatic micrometer or a confocal laser scanning microscope (CLSM). In addition, the flexural properties of tested resin composites were measured to investigate the relation between cuspal deflection and flexural properties, and the resin composites were observed using scanning electron microscopy.

Results: Simulated cuspal deflection of some bulk-fill flowable resin composites was found to be significantly lower than or similar to those for conventional counterparts, regardless of the measurement method. There were

Yusuke Shimatani, DDS, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

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

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

Nicholas G Fischer, BS, University of Minnesota, Minneapolis, MN, USA

Yuko Nagura, DDS, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

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

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: tsujimoto.akimasa@nihon-u.ac.jp

<https://doi.org/10.2341/18-160-L>

statistically significant differences in flexural properties depending on the material, regardless of the type of flowable resin composite. Pearson correlation analysis did not reveal a statistically significant relationship between flexural properties and cuspal deflection.

Conclusion: These results indicate that some bulk-fill flowable resin composites exhibit lower cuspal deflection with the bulk-filling technique than is shown by conventional flowable resin composites using the incremental filling technique. Simulated cuspal deflection can be measured using either a micrometer or CLSM, but this experiment failed to show a significant relationship between cuspal deflection and flexural properties of flowable resin composites.

INTRODUCTION

Resin composites have come to be considered the first-choice material for direct posterior restorations because of the improvements in their mechanical properties.¹ Heintze and others² reported that approximately 800 million resin composite restorations were placed worldwide in 2015, with approximately 80% of them placed in posterior teeth and 20% in anterior teeth based on the quantity of restorative materials sold. Alvanforoush and others³ reported that the overall clinical failure rates of resin composite restorations in posterior teeth were similar between 1995 and 2005 (10.59%) and between 2006 and 2016 (13.13%). In addition, the fracture rates of resin composite showed a notable increase depending on the increase in the number of cases of larger resin composite restorations in posterior teeth (1995-2005, 28.84%; 2006-2016, 39.07%) and it might be important to consider their mechanical properties when planning larger restorations. Based on the restoration failure rates, it is estimated that at least 32 million resin composite restorations that were placed in posterior teeth in 2015 will need to be repaired or replaced due to failure by 2025,⁴ suggesting the importance of continuously improving the physical properties of resin composites. Therefore, manufacturers have continued to develop resin composites with enhanced physical properties.

During the formation of a highly crosslinked polymer, adequate light polymerization of resin monomers is thought to be essential to attain superior physical properties in resin composites.⁵ However, volumetric shrinkage in the range of 1.5% to 5%⁶ accompanies the light polymerization of resin com-

posites because of the decrease in the distance between monomer chains owing to the conversion of weak electrostatic forces into covalent bonds. The volumetric shrinkage leads to the development of polymerization shrinkage stress as the resin composite is bonded to the tooth structures of the cavity.⁷ Thus, polymerization shrinkage stress of resin composites may lead to internal and marginal gaps, microleakage, and microcracking of the tooth structure due to cuspal deflection. While studying the cause of resin composite failure, these issues associated with polymerization shrinkage should be considered.⁸ Some researchers have reported that resin composites with higher mechanical properties typically demonstrate higher polymerization shrinkage stress,⁹⁻¹¹ which suggests that modifying the formulation of resin composites to obtain higher mechanical properties and to avoid fracture may increase the risk of problems related to polymerization shrinkage stress. While the mechanisms of polymerization shrinkage stress development within resin composite restorations are quite complex, the generation, measurement, and characterization of polymerization shrinkage stress have been researched extensively for 50 years, beginning with studies by Bowen¹² and Bowen and others,¹³ and they proliferated following the work of Davidson and others¹⁴ and Feilzer and others.¹⁵ At present, many research laboratories are investigating the best methods for measuring the polymerization shrinkage stress of resin composites.

One of the methods for analyzing polymerization shrinkage stress of resin composites is measuring the simulated cuspal deflection using aluminium blocks with linear variable differential transformers (LVDT) developed by Park and others.¹⁶ The advantage of this measurement method is that the cuspal deflection during polymerization of resin composites can be measured in real time. However, LVDT measurements are not widely used, and a custom-built apparatus to hold the LVDT and the specimens is required to perform these measurements. Consequently, most of the studies based on simulated cuspal deflection using LVDT have been conducted at a single research institute.¹⁶⁻¹⁸ Therefore, to find an alternative to the LVDT method, the measurement of simulated cuspal deflection resulting from the polymerization of resin composite bonded to a precisely prepared mesio-occlusal-distal (MOD) cavity within an aluminium block using a novel highly accurate submicron digimatic micrometer (MDH-25M, Mitutoyo, Tokyo, Japan) or a confocal laser scanning microscope (CLSM; VK-9710, Keyence, Tokyo, Japan) was investigated in this study. Neither the microme-

ter nor the CLSM method of cuspal deflection with an aluminium block has been used previously for this purpose; these methods may allow for more accessible measurement processes. An aluminium block (modulus of elasticity, 68.5 GPa) has been used in previous studies¹⁶⁻¹⁸ to simulate cuspal deflection with polymerization of resin composites, as the mechanical properties are within the range of those for enamel (modulus of elasticity, 84.1 GPa) and dentin (modulus of elasticity, 18.5 GPa).

A previous study reported that the cuspal deflection for the incremental filling technique was considerably lower than that for the bulk-filling technique and that there was no significant difference between the horizontal and oblique incremental filling techniques.¹⁶ Recently, using bulk-fill flowable resin composites has expedited the restoration process by enabling increments of up to 4 mm in thickness to be light polymerized, thereby avoiding the time-consuming incremental filling technique.¹⁹ Manufacturers claim that the polymerization shrinkage stress of bulk-fill flowable resin composites may be reduced using advanced technology for the treatment of filler particles, monomer synthesis, and development of modulators to retard the polymerization rate. Therefore, using bulk-fill flowable resin composites reduces cuspal deflection even with the bulk-filling technique, when compared with that of conventional flowable resin composites using the incremental filling technique. However, few independent studies have compared the cuspal deflection between bulk-fill and conventional flowable resin composites using different filling techniques.

This study aimed to evaluate methods for measuring the polymerization shrinkage stress of bulk-fill and conventional flowable resin composites by measuring simulated cuspal deflection and to compare these values with those of flexural properties. The null hypotheses to be tested were 1) there would be no differences in simulated cuspal deflection between bulk-fill and conventional flowable resin composites, 2) there would be no differences in the cuspal deflection of resin composites measured using different methods, and 3) there would be no relationship between simulated cuspal deflection and flexural properties for any measurement method.

METHODS AND MATERIALS

Study Materials

Five bulk-fill flowable resin composites were evaluated: 1) Beautifil Bulk Flowable (BF; Shofu, Kyoto, Japan), 2) Bulk Base (BB; Sun Medical, Shiga,

Japan), 3) Filtek Fill and Core (FF; 3M Oral Care, St Paul, MN, USA), 4) SDR (SD; Dentsply Sirona, York, PA, USA), and 5) X-tra base (XB; VOCO GmbH, Cuxhaven, Germany). We also evaluated six conventional flowable resin composites: 1) Clearfil Majesty ES Flow (CE; Kuraray Noritake Dental Inc, Tokyo, Japan), 2) Clearfil Majesty LV (CL; Kuraray Noritake Dental), 3) Estelite Universal Flow (EU; Tokuyama Dental, Tokyo, Japan), 4) G-aenial Universal Injectable (GI; GC, Tokyo, Japan), 5) Filtek Supreme Ultra Flowable (FS; 3M Oral Care), and 6) UniFil LoFlo Plus (UF; GC). The tested materials are listed in Table 1 with their associated lot numbers and main components.

Simulated Cuspal Deflection Measurement

Aluminum blocks (10 [W] × 8 [L] × 15 [D] mm) with an MOD cavity (4 [W] × 8 [L] × 4 [D] mm) were fabricated using a milling machine, creating two remaining cusps. The inside of the cavity was air abraded with 50 µm Al₂O₃ powder for 10 seconds to create microroughness for improved adhesion. The air pressure was set to 0.2 MPa, and the distance between the orifice and metal surface was approximately 10 mm (Jet Blast II, J. Morita Mfg Corp, Osaka, Japan). Universal adhesive (Scotchbond Universal Adhesive, 3M Oral Care) was applied prior to placing the low-viscosity bulk-fill and conventional flowable resin composites according to the manufacturer's instructions. The adhesive was light cured for 10 seconds at a standardized distance of 1 mm using a quartz-tungsten-halogen (QTH) curing unit (OptiLux 501, Demetron/Kerr, Danbury, CT, USA). The power density (>700 mW/cm²) of the QTH curing unit was confirmed using a dental radiometer (model 100, Demetron) prior to specimen preparation.

The aluminium blocks were randomly divided into four groups for different measurement techniques (micrometer vs CSLM) and were further subdivided according to the type of resin composite (bulk-fill vs conventional flowable resin composite; Figure 1).

Group 1 (Micrometer × Bulk-Fill Flowable Resin Composite)—Bulk-fill flowable resin composites were placed in bulk and were light cured from the three exposed surfaces for 40 seconds each. Simulated cuspal deflection was calculated from the difference in the distance between the centers of the two remaining cusps prior to resin composite placement and 10 minutes after polymerization, as measured by a micrometer.

Group 2 (Micrometer × Conventional Flowable Resin Composite)—Conventional flowable resin com-

Table 1: Bulk-Fill and Conventional Flowable Resin Composites Used in This Study

Resin Composite (Shade)	Type of Flowable Resin Composite (Code)	Resin Matrix Composition	Inorganic Filler Composition	Manufacturer (Lot No.)
Beautifil Bulk Flowable (Dentin)	Bulk-fill (BF)	Bis-GMA, Bis-MPEPP, TEGDMA, UDMA	Fluoro-silicate glass	Shofu, Kyoto, Japan (031719)
Bulk Base (Universal)	Bulk-fill (BB)	Bis-MPEPP, UDMA	Barium-silicate glass, Strontium-fluoro-alumino-silicate glass	Sun Medical, Shiga, Japan (RG12)
Filtek Fill and Core Flow (A3)	Bulk-fill (FF)	Bis-GMA, UDMA	Inorganic fillers	3M Oral Care, St. Paul, MN, USA (N863610)
SDR (Universal)	Bulk-fill (SD)	Bis-EMA, modified TEGDMA, UDMA	Barium-fluoro-alumino-silicate glass, Strontium-fluoro-alumino-silicate glass	Dentsply Sirona, York, PA, USA (1508033)
X-tra base (Universal)	Bulk-fill (XB)	Aliphatic dimethacrylate, Bis-EMA	Inorganic fillers	Voco GmbH, Cuxhaven, Germany
Clearfil Majesty ES Flow (A3)	Conventional (CE)	Hydrophobic aromatic dimethacrylate, TEGDMA	Silanated barium glass filler, Silanated silica filler	Kuraray Noritake Dental, Tokyo, Japan (BA0207)
Clearfil Majesty LV (A3)	Conventional (CL)	Hydrophobic aromatic dimethacrylate, TEGDMA	Silanated barium glass filler, Silanated colloidal silica	Kuraray Noritake Dental, Tokyo, Japan (850029)
Estelite Universal Flow (A3)	Conventional (EU)	Bis-GMA, Bis-MPEPP, TEGDMA, UDMA	Silica-zirconia filler	Tokuyama Dental, Tokyo, Japan (019957)
Filtek Supreme Ultra Flow (A3)	Conventional (FS)	Bis-GMA, substituted dimethacrylate, TEGDMA	Silane treated ceramic, Silane treated silica, Ytterbium fluoride	3M Oral Care, St Paul, MN, USA (N838182)
G-aenial Universal Injectable (A2)	Conventional (GI)	Bis-EMA, Dimethacrylate, UDMA	Strontium glass	GC, Tokyo, Japan (1702081)
UniFil LoFlow Plus (A2)	Conventional (UF)	Dimethacrylate, UDMA	Fluoro-alumino-silicate glass	GC, Tokyo, Japan (1702161)

Abbreviations: Bis-EMA, ethoxylated bisphenol-A-dimethacrylate; Bis-GMA, bisphenol-A-glycidyl dimethacrylate; Bis-MPEPP, bisphenol A polyethoxy methacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

posites were placed in two horizontal consecutive layers (2 mm each). Each increment was light cured from the three exposed surfaces for 40 seconds each to ensure that an identical curing time was maintained. Simulated cuspal deflection was measured in the same manner as in group 1.

Group 3 (CLSM × Bulk-Fill Flowable Resin Composite)—Bulk-fill flowable resin composites were placed using the same method as in group 1. Simulated cuspal deflection was calculated from the distance between the center of the two remaining cusps prior to resin composite placement and 10 minutes after polymerization, as measured by a CLSM with built-in analysis software (VK-Analyzer, Keyence).

Group 4 (CLSM × Conventional Flowable Resin Composite)—Conventional flowable resin composites were placed in the same manner as in group 2. Simulated cuspal deflection was measured in the same manner as in group 3.

Flexural Properties Measurement

A Teflon split mold (2.0 [W] × 25 [L] × 2.0 [D] mm) was used to prepare the specimens, which minimized

the stresses applied to the specimens during their retrieval. A condenser was used to place the resin composites into the mold. The top side of the mold was covered with a matrix strip, and the resin composites were pressed with a glass slide under a load of 5 N. The exit window of the QTH curing unit was placed against the glass plate at the center of the specimen, which was light cured for 40 seconds. Next, the exit window was moved to the section next to the center in such a way that the previous section was overlapped by approximately one-half. Light curing was performed by sequentially curing overlapping regions until the entire sample surface had been light cured. The hardened specimens were carefully removed from the mold after light curing, and silicon carbide (SiC) papers (No. 600, Struers, Cleveland, OH, USA) were used to polish the specimens to obtain smooth and flat surfaces. Fifteen specimens for each resin composite were prepared under ambient laboratory conditions of 23°C ± 2°C and 50% ± 10% relative humidity. Specimen dimensions were measured using a high-accuracy submicron digimatic micrometer, and the accepted specimen size was 2.0 ± 0.020 mm in width and height and 25 ± 0.025 mm in length. The specimens

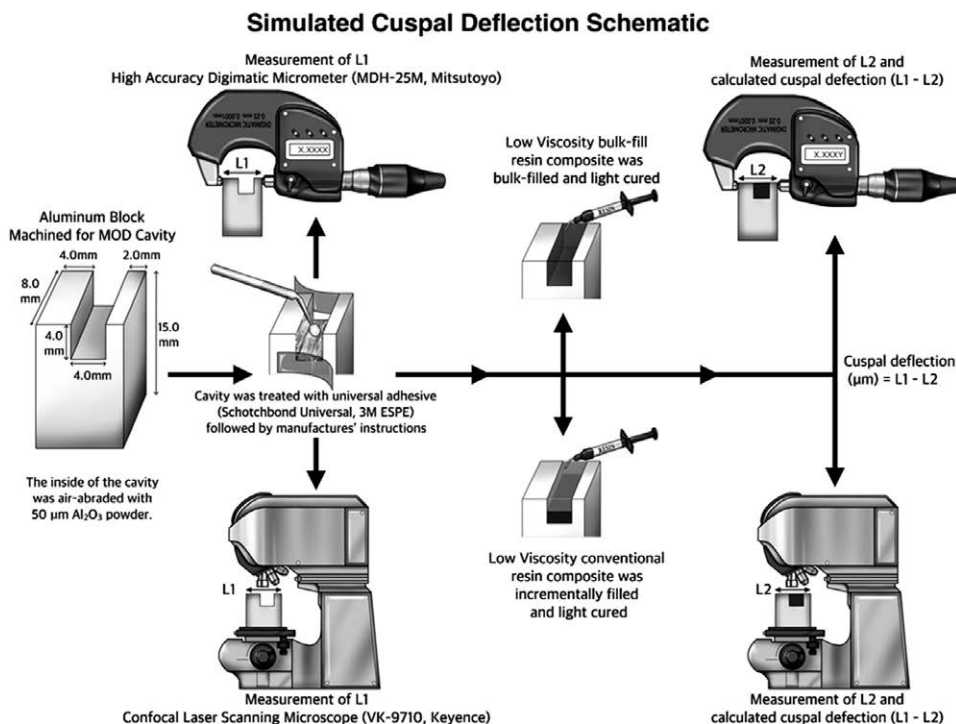


Figure 1. Schematic drawing of the experimental setup for simulated cuspal deflection of flowable resin composites with a micrometer or CLSM.

were immersed in distilled water in an incubator (IC802, Yamato Scientific, Tokyo, Japan) at 37°C for 24 hours.

The specimens for each resin composite underwent a three-point bending test on a universal testing machine (5500R, Instron, Norwood, MA, USA) at a crosshead speed of 1.0 mm/min until specimen fracture occurred as outlined in ISO 4049. The stress-strain curve was used to determine the flexural strength in MPa and flexural modulus in GPa using a custom software package (Bluehill 2 version 2.5, Instron) linked directly to the testing machine.

Flexural strength (σ) was calculated as follows:

$$\sigma = 3PD/2bd^2,$$

where P = maximum load at the fracture point, D = distance between the supports (20 mm), b = specimen width, and d = specimen height.

The flexural modulus (E) was calculated as follows:

$$E = P_1D/4bd^3\delta,$$

where P_1 = the load at an intersection point within the elastic region of the stress-strain curve and δ = specimen deformation at P_1 .

Scanning Electron Microscopy Observation

The polished surfaces of the resin composites underwent ultrastructural observation conducted through scanning electron microscopy (SEM). A Teflon mold with a diameter and height of 10.0 and 2.0 mm, respectively, was used to form the specimens of the resin composites. The mold was placed on a glass slide covered with a matrix strip, and the resin composites were placed into the mold using a condenser instrument. The top side of the mold was covered with a matrix strip, and the resin composites were pressed with a glass slide under a load of 5 N. The exit window of the QTH curing unit was placed against the glass slide, and the resin composite was light cured for 40 seconds. After light curing, the hardened specimens were removed from the mold and polished with SiC papers (No. 600) to obtain smooth and flat surfaces. Three specimens for each resin composite were prepared under ambient laboratory conditions at 23°C $\pm$ 2°C and 50% $\pm$ 10% relative humidity. The specimens were immersed in distilled water in an incubator at 37°C for 24 hours. After storage in the incubator, the specimen surfaces were prepared and polished using a gradually increasing sequence (No. 320, 600, 1200, 2000, and 4000) of SiC papers in a grinder/polisher (Minitech 333, Presi, Eybens, France). Finally, the surfaces were polished with

Table 2: Simulated Cuspal Deflection of Bulk-Fill and Conventional Flowable Resin Composites^a

Rank Order	Resin Composite	Type of Flowable Resin Composite	Simulated Cuspal Deflection, μm	
			Micrometer	CLSM
1	SDR (Dentsply)	Bulk-fill	7.2 (3.5) ^{a,A}	7.6 (1.5) ^{a,A}
2	Filtek Fill and Core Flow (3M Oral Care)	Bulk-fill	8.1 (1.6) ^{a,A}	8.2 (1.4) ^{a,A}
3	Bulk Base (Sun Medical)	Bulk-fill	10.9 (0.7) ^{b,A}	11.1 (1.0) ^{b,A}
4	Clearfil Majesty ES Flow (Kuraray Noritake Dental)	Conventional	15.3 (2.6) ^{c,A}	15.5 (0.6) ^{c,A}
5	UniFil LoFlow Plus (GC)	Conventional	16.0 (3.8) ^{c,A}	16.7 (0.9) ^{c,A}
6	Clearfil Majesty LV (Kuraray Noritake Dental)	Conventional	16.7 (2.1) ^{c,A}	17.0 (1.1) ^{c,A}
7	X-tra base (Voco GmbH)	Bulk-fill	17.2 (1.5) ^{c,A}	16.8 (1.0) ^{c,A}
8	G-aenial Universal Injectable (GC)	Conventional	19.8 (2.8) ^{d,A}	19.6 (0.8) ^{d,A}
9	Filtek Supreme Ultra Flow (3M Oral Care)	Conventional	20.1 (0.4) ^{d,A}	20.6 (1.0) ^{d,A}
10	Beautifil Bulk Flowable (Shofu)	Bulk-fill	20.2 (0.6) ^{d,A}	19.9 (0.6) ^{d,A}
11	Estelite Universal Flow (Tokuyama Dental)	Conventional	20.3 (2.3) ^{d,A}	20.3 (0.9) ^{d,A}

^a Values in parentheses are standard deviations (n=5). The same lowercase letter in the same vertical column indicates no significant difference ($p > 0.05$). The same uppercase letter within individual rows indicates no significant difference ($p > 0.05$).

a soft cloth using 1.0- μm grit diamond paste (DP-Paste, Struers, Ballerup, Denmark). SEM specimens of the resin composites were dehydrated by first immersing them in ascending concentrations of aqueous *tert*-butanol (50%, 75%, and 95% for 20 minutes each and 100% for 2 hours) and then transferring them from the final 100% bath to a critical-point dryer (model ID-3, Elionix, Tokyo, Japan) for 30 minutes. To enhance the filler visibility, the polished surfaces were etched for 30 seconds with an argon ion-beam (Type EIS-200ER, Elionix) directed perpendicular to the surface at an accelerating voltage and ion current density of 1.0 kV and 0.4 mA/cm², respectively. Next, the surfaces were coated with a thin film of gold in a vacuum evaporator (Quick Coater SC-701, Sanyu Electron, Tokyo, Japan) and observed using field-emission SEM (ERA-8800FE, Elionix) with an operating voltage of 10 kV.

Statistical Analysis

Two-way analysis of variance (ANOVA) was used to analyze cuspal deflection data using the factors 1) type of resin composite and 2) cuspal deflection measurement, followed by Tukey's post hoc honestly significant difference (HSD) test with a significance level (α) of 0.05. Flexural strength and modulus data were analyzed using one-way ANOVA along with the Tukey HSD test with a significance level of 0.05. In addition, we conducted Pearson correlation analysis between cuspal deflection, using both a micrometer and CLSM, and flexural strength and modulus.

RESULTS

Simulated Cuspal Deflection

Results for the simulated cuspal deflection of bulk-fill and conventional flowable resin composites using both the micrometer and CLSM are shown in Table 2. Simulated cuspal deflection of bulk-fill flowable resin composites was material dependent, ranging from 7.2 to 20.2 μm for the micrometer and 7.6 to 19.9 μm for CLSM. In the bulk-fill flowable resin composites, SD, FF, and BB showed significantly lower cuspal deflection than did XB and BF. On the other hand, cuspal deflection of conventional flowable resin composites was also material dependent, ranging from 15.3 to 20.3 μm for the micrometer and 15.5 to 20.6 μm for CLSM. Simulated cuspal deflection of the conventional flowable resin composites was significantly higher than that for SD, FB, and BB, and most cuspal deflections were similar to those for BF and XB. A statistically significant relationship between cuspal deflection measured with the micrometer and CLSM ($R=0.98$, $p<0.001$) was seen in Pearson correlation analysis.

Flexural Properties

The results for the flexural strength and modulus of bulk-fill and conventional flowable resin composites are shown in Table 3. The flexural strength of the bulk-fill flowable resin composites ranged from 68.9 to 116.1 MPa, and the flexural modulus ranged from 2.0 to 5.3 GPa. For the conventional flowable resin composites, the flexural strength ranged from 79.9 to 132.8 MPa, and flexural modulus ranged from 3.3 to 7.4 GPa. There were statistically significant differ-

Table 3: Flexural Properties of Bulk-Fill and Conventional Flowable Resin Composites ^a				
Rank Order	Resin Composite	Type of Flowable Resin Composite	Flexural Strength, MPa	Flexural Modulus, GPa
1	Clearfil Majesty ES Flow (Kuraray Noritake Dental)	Conventional	132.8 (9.2) ^a	7.4 (0.5) ^a
2	G-ænial Universal Injectable (GC)	Conventional	128.8 (8.5) ^a	6.5 (0.4) ^b
3	Filtek Fill and Core Flow (3M Oral Care)	Bulk-fill	116.1 (6.7) ^b	5.3 (0.4) ^c
4	Filtek Supreme Ultra Flow (3M Oral Care)	Conventional	113.9 (7.2) ^b	5.2 (0.4) ^c
5	Estelite Universal Flow (Tokuyama Dental)	Conventional	110.6 (7.1) ^{b,c}	5.8 (0.4) ^c
6	X-tra base (Voco GmbH)	Bulk-fill	110.4 (6.8) ^{b,c}	5.0 (0.3) ^c
7	SDR (Dentsply)	Bulk-fill	105.7 (6.9) ^c	7.2 (0.5) ^a
8	Clearfil Majesty ES Flow (Kuraray Noritake Dental)	Conventional	104.7 (7.3) ^c	6.3 (0.5) ^b
9	Beautifil Bulk Flowable (Shofu)	Bulk-fill	102.1 (6.9) ^c	6.3 (0.6) ^b
10	UniFil LoFlow Plus (GC)	Conventional	79.9 (6.1) ^d	3.3 (0.3) ^d
11	Bulk Base (Sun Medical)	Bulk-fill	68.9 (5.4) ^e	2.0 (0.2) ^e

^a Values in parentheses are standard deviations (n=15). The same lowercase letter in the same vertical column indicates no significant difference (p>0.05).

ences in flexural strength and elastic modulus depending on the material, regardless of the type of resin composite. The rank order was different for flexural properties and for simulated cuspal deflection. In addition, no statistically significant relationship was revealed between flexural strength and cuspal deflection on Pearson correlation analysis ($R=0.32$, $p=0.344$ for the micrometer; $R=0.33$,

$p=0.334$ for CLSM) or between flexural modulus and cuspal deflection ($R=0.22$, $p=0.761$ for the micrometer; $R=0.21$, $p=0.755$ for CLSM).

SEM Observation

Representative SEM micrographs of bulk-fill and conventional flowable resin composites are shown in

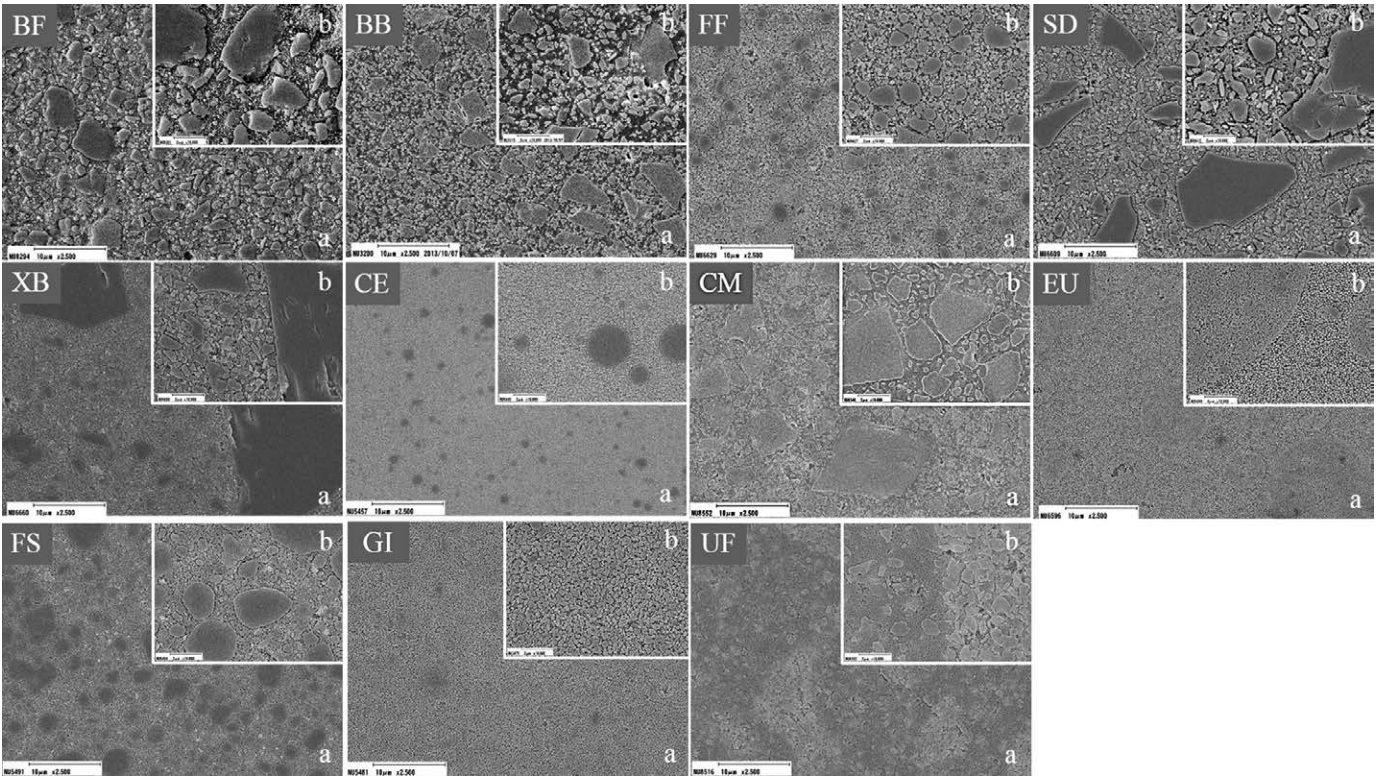


Figure 2. Representative SEM images of the surfaces of bulk-fill and conventional flowable resin composites at (a) 2500 $\times$ and (b) 10,000 $\times$ magnifications. BB, Bulk Base; BF, Beautifil Bulk Flowable; CE, Clearfil Majesty ES Flow; CL, Clearfil Majesty LV; EU, Estelite Universal Flow; FF, Filtek Fill and Core Flow; FS, Filtek Supreme Ultra Flowable; GI, G-ænial Universal Injectable; SD, SDR; UF, UniFil LoFlo Plus; XB, X-tra base.

Figure 2. The resin composites were composed of a wide variety of fillers, and filler particle size and shape were material dependent. In the bulk-fill flowable resin composites, BF, BB, SD, and XB showed a wide size range ($<1\text{-}5\text{ }\mu\text{m}$ for BF and BB, $<1\text{-}20\text{ }\mu\text{m}$ for SD, and $<1\text{-}30\text{ }\mu\text{m}$ for XB) of irregular-shaped fillers, and FF showed relatively uniform, small-sized ($<1\text{-}2\text{ }\mu\text{m}$) irregular-shaped fillers. In the conventional flowable resin composites, CE showed a wide size range ($<1\text{-}10\text{ }\mu\text{m}$) of irregular-shaped fillers, and CM showed nonuniform, small-sized ($<1\text{ }\mu\text{m}$) irregular-shaped fillers and small-sized ($1\text{-}4\text{ }\mu\text{m}$) spherical-shaped fillers. Nonuniform, small-sized ($<1\text{ }\mu\text{m}$) spherical-shaped fillers for EU- and irregular-sized UF-shaped fillers were observed, and some fillers were aggregated. FS showed a wide size range ($<1\text{-}7\text{ }\mu\text{m}$) of irregular-shaped fillers, and GI showed uniform, small-sized ($<1\text{ }\mu\text{m}$) irregular-shaped fillers.

DISCUSSION

Some of the bulk-fill flowable resin composites (SD, FF, and BB) used in this study showed significantly lower simulated cuspal deflection than their conventional counterparts, and the cuspal deflection of XB and BF was similar to that for most conventional flowable resin composites, regardless of the measurement system. Thus, the first null hypothesis that there would be no differences in simulated cuspal deflection between bulk-fill and conventional flowable resin composites was partially rejected. A previous study reported that the cuspal deflection of resin composites using the bulk-filling technique was significantly higher than that with the incremental filling technique regardless of type of resin composite, implying that the bulk-filling technique led to significantly more cuspal deflection than did the incremental filling technique in those experiments.¹⁶ In the present study, the cuspal deflection of bulk-fill and conventional flowable resin composite was investigated using the filling technique specified in the manufacturers' instructions. Simulated cuspal deflection of bulk-fill flowable resin composites using the bulk-filling technique was lower than or similar to that of conventional flowable resin composites with the incremental filling technique.

The cuspal deflection of the tested resin composites was material dependent regardless of filling technique, which suggests that the cuspal deflection of resin composites is primarily influenced by their composition rather than the filling technique. In the SEM observations, a wide variation of filler type was seen in the resin composites, but there was no clear

relationship between filler particle size, shape, and cuspal deflection. Although previous studies reported an effect of filler particle size and shape on shrinkage stress,²⁰ a systematic review of the polymerization shrinkage stress of resin composites found that modification of the resin matrix had the largest impact on minimizing stress development.²¹ According to the manufacturers, SD, FB, and BB showed significantly lower cuspal deflection than did BF and XB because the resin matrix contains high-molecular-weight polymerization modulators. There was a lower ratio of functional groups for making double bonds through polymerization to molecular weight in comparison with a typical resin matrix, which is purported to reduce polymerization shrinkage.²² Thus, the results of the present study for cuspal deflection of resin composites appeared to have been mainly influenced by the modifications of the resin matrix.

In the present study, a novel micrometer or CLSM cuspal deflection measurement method was used to measure the cuspal deflection of resin composites as a more accessible replacement for the LVDT method. There was no significant difference between the cuspal deflection measured using the micrometer and using the CLSM, and Pearson correlation analysis revealed a statistically significant relationship ($R=0.98$, $p<0.001$) between values measured with the micrometer and those measured using CLSM. Hence, the second null hypothesis that there would be no difference in the cuspal deflection of resin composites measured with different methods was not rejected.

One of the concerns with using the micrometer was the possible influence of instrument-related stress exerted on the aluminium block, while errors arising from the process of combining the scanned micrographs were a concern with CLSM; both of these factors could potentially influence the measured values. Simulated cuspal deflection measured with the micrometer ranged from $7.2\text{ to }20.3\text{ }\mu\text{m}$ and ranged from $7.0\text{ to }20.6\text{ }\mu\text{m}$ for CLSM. A previous study¹⁶⁻¹⁸ reported that cuspal deflection measured using an aluminium block with LVDT was approximately $5\text{-}30\text{ }\mu\text{m}$, although differences in the wall thickness of aluminium and size of the trench made direct comparison difficult.¹⁸ If the micrometer did apply stress during the measurement period, it would be expected to cause a greater deformation of the block during the prepolymerization measurement, especially prior to resin composite filling, which would lead to a lower cuspal deflection. However, this overestimation was not observed, suggesting that stress from the micrometer was not

a significant factor. On the other hand, stitching individual micrographs together to form a complete three-dimensional rendering from the CLSM could bias the values in either direction, but no such deviations were observed; thus, the original concerns did not appear to be justified.

During the experiment, a serious problem was noted with CLSM: it could not measure the cuspal deflection precisely after 10 minutes of polymerization because of the time required (5-8 minutes) for scanning. In this study, the measurement was conducted 10 minutes after polymerization based on a previous study; thus, the scanning duration is thought to have a minimal influence. However, the values of cuspal deflection slightly increased over a longer time period.¹⁶⁻¹⁸ There is a small possibility that the cuspal deflection measured with CLSM will be higher than that with micrometer, but this was not observed in the present study. Since there was no significant difference in the cuspal deflection of resin composites measured using different methods (micrometer vs CLSM), investigators may rely on these methods for measuring cuspal deflection. In comparison with the LVDT method, the micrometer may be more accessible and easier, and CLSM may allow for more automation in the process used to measure cuspal deflection. Overall, micrometer and CLSM measurement methods of cuspal deflection of the aluminium block may be effective for evaluating the polymerization shrinkage stress of resin composite restorations. Further research is needed to determine the best experimental setup for measuring cuspal deflection as an indicator of polymerization shrinkage stress.

The flexural strength and modulus of resin composites were material dependent, but the rank order of the results was different from that of cuspal deflection. Pearson correlation analysis did not show any statistically significant relationship between flexural properties and cuspal deflection ($R=0.32$, $p=0.344$ for flexural strength and simulated cuspal deflection; $R=0.22$, $p=0.761$ for flexural modulus and simulated cuspal deflection). Therefore, the third null hypothesis that there would be no relationship between cuspal deflection and flexural properties for any measurement method was not rejected. The results of the correlation analysis do not directly support the results of earlier studies, but they are not decisive evidence against a connection.

CONCLUSION

The results of this study indicate that the simulated cuspal deflection of bulk-fill and conventional

flowable resin composites can be measured using either a micrometer or CLSM. These measurements showed that some of the bulk-fill flowable resin composites used in this study had superior cuspal deflection using bulk-filling techniques in comparison with conventional flowable resin composites using incremental filling techniques. However, this experiment did not show a statistically significant relationship between the flexural properties and simulated cuspal deflection of resin composites. Further research is necessary to clarify these relationships.

Acknowledgements

The authors thank Dr Masao Irie of Okayama University and Mr Kazuyoshi Shimizu of 3M Oral Care for comments that greatly improved the article.

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 17 September 2018)

REFERENCES

1. Lynch CD, Opdam NJ, Hickel R, Brunton PA, Gurgan S, Kakaboura A, Shearer AC, Vanherle G, & Wilson NH; Academy of Operative Dentistry European Section (2014) Guidance on posterior resin composites: Academy of Operative Dentistry-European Section *Journal of Dentistry* **42**(4) 377-383.
2. Heintze SD, Ilie N, Hickel R, Reis A, Loguercio A, & Rousson V (2017) Laboratory mechanical parameters of composite resins and their relation to fractures and wear in clinical trials—a systematic review *Dental Materials* **33**(3) e101-e114.
3. Alvanforoush N, Palamara J, Wong RH, & Burrow MF (2017) Comparison between published clinical success of direct resin composite restorations in vital posterior teeth in 1995-2005 and 2006-2016 periods *Australian Dental Journal* **62**(2) 132-145.
4. Tsujimoto A, Barkmeier WW, Fischer NG, Nojiri K, Nagura K, Takamizawa T, Latta MA, & Miyazaki M (2018) Wear of resin composites: current insights into underlying mechanisms, evaluation methods and influential factors *Japanese Dental Science Review* **54**(2) 76-87.
5. Cramer NB, Stansbury JW, & Bowman CN (2011) Recent advances and developments in composite dental restorative materials *Journal of Dental Research* **90**(4) 402-416.
6. 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.

7. Kaisarly D & Gezawi ME (2016) Polymerization shrinkage assessment of dental resin composites: a literature review *Odontology* **104**(3) 257-270.
8. Ilie N & Hickel R (2011) Resin composite restorative materials *Australian Dental Journal* **56**(Supplement 1) 59-66.
9. Condon JR & Ferracane JL (2000) Assessing the effect of composite formulation on polymerization stress *Journal of the American Dental Association* **131**(4) 497-503.
10. Ferracane JL (2005) Developing a more complete understanding of stresses produced in dental composites during polymerization *Dental Materials* **21**(1) 36-42.
11. Pfeifer CS, Ferracane JL, Sakaguchi RL, & Braga RR (2008) Factors affecting photopolymerization stress in dental composites *Journal of Dental Research* **87**(11) 1043-1047.
12. Bowen RL (1967) Adhesive bonding of various materials to hard tooth tissues. VI. Forces developing in direct-filling materials during hardening *Journal of the American Dental Association* **74**(3) 439-445.
13. Bowen RL, Nemoto K, & Rapson JE (1983) Adhesive bonding of various materials to hard tooth tissues: forces developing in composite materials during hardening *Journal of the American Dental Association* **106**(4) 475-477.
14. Davidson CL & de Gee AJ (1984) Relaxation of polymerization contraction stresses by flow in dental composites *Journal of Dental Research* **63**(2) 146-148.
15. Feilzer AJ, De Gee AJ, & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66**(11) 1636-1639.
16. Park J, Chang J, Ferracane J, & Lee IB (2008) How should composite be layered to reduce shrinkage stress: incremental or bulk filling? *Dental Materials* **24**(11) 1501-1505.
17. Kwon Y, Ferracane J, & Lee IB (2012) Effect of layering methods, composite type, and flowable liner on the polymerization shrinkage stress of light cured composites *Dental Materials* **28**(7) 801-809.
18. Kim YJ, Kim R, Ferracane JL, & Lee IB (2016) Influence of the compliance and layering method on the wall deflection of simulated cavities in bulk-fill composite restoration *Operative Dentistry* **41**(6) e183-e194.
19. Tsujimoto A, Barkmeier WW, Takamizawa T, Latta MA, & Miyazaki M (2017) Depth of cure, flexural properties and volumetric shrinkage of low and high viscosity bulk-fill giomers and resin composites *Dental Materials Journal* **36**(2) 205-213.
20. Satterthwaite JD, Maisuria A, Vogel K, & Watts DC (2012) Effect of resin-composite filler particle size and shape on shrinkage-stress *Dental Materials* **28**(6) 609-614.
21. 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 the Mechanical Behavior of Biomedical Materials* **82** 268-281.
22. 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.

Effect of the Photo-initiator System Contained in Universal Adhesives on Radicular Dentin Bonding

RF Carvalho • AFM Cardenas • CN Carvalho • JJ de Souza • JRO Bauer
FSF Siqueira • A Armas-Vega • AD Loguercio • V Hass

Clinical Relevance

The photo-initiator system based on an advanced polymerization system may be an alternative that can be used to overcome the disadvantages of radicular dentin, especially for the apical third.

SUMMARY

Objectives: The purpose of this study was to evaluate the effects of universal adhesives with different photo-initiator systems applied in etch-and-rinse (ER) and self-etch (SE) modes on dentin interaction (push-out bond strength [PBS], nanoleakage [NL], and degree of conversion [DC] within the hybrid layer) in the

different root thirds after fiber post cementation.

Methods and Materials: Roots of endodontically prepared human premolars were randomly divided into six groups according to one of three adhesive systems (Scotchbond Universal [SBU], Ambar Universal [AMB], and Ambar Universal APS [AMB-APS]) and two adhesive strategies (ER and SE) for each system. Posts were cemented, and PBS was tested at 0.5 mm/min. The NL was evaluated by scanning elec-

Roberta Furtado Carvalho, DDS, MSc, PhD student, Department of Postgraduate Program in Dentistry, CEUMA University, São Luis, Maranhão, Brazil

Andres Felipe Millan Cardenas, DDS, MSc, PhD, professor, Department of Postgraduate Program in Dentistry, CEUMA University, São Luis, Maranhão, Brazil

Ceci Nunes Carvalho, DDS, MSc, PhD, professor, Department of Postgraduate Program in Dentistry, CEUMA University, São Luis, Maranhão, Brazil

Jullian Josnei de Souza, DDS, MSc, PhD student, Department of Restorative Dentistry, State University of Ponta Grossa, Ponta Grossa, Paraná, Brazil

José Roberto de Oliveira Bauer, DDS, MSc, PhD, associate professor, School of Dentistry, Federal University of Maranhão, São Luís, Maranhão, Brazil

Fabiana Suelen Figuerêdo de Siqueira, DDS, MSc, PhD, professor, Department of Postgraduate Program in Dentistry, CEUMA University, São Luis, Maranhão, Brazil

Ana Armas-Vega, DDS, MSc, PhD, professor, Escuela de la Salud Eugenio Espejo, Universidad UTE, Quito, Ecuador

*Alessandro Dourado Loguercio, DDS, MSc, PhD, professor, Department of Restorative Dentistry, State University of Ponta Grossa, Ponta Grossa, Paraná, Brazil. Escuela de la Salud Eugenio Espejo, Universidad UTE, Quito, Ecuador

Viviane Hass, DDS, MSc, PhD, professor, Postgraduate Program in Dentistry, UNOPAR–University North Parana, Londrina, PR, Brazil, and postdoctoral fellow, School of Dentistry, University of Missouri–Kansas City, Kansas City, MO, USA

*Corresponding author: Avenida Carlos Cavalcanti, 4748–Uvaranas, Ponta Grossa, Paraná, Brazil, CEP: 84030-900; e-mail: aloguercio@hotmail.com

<https://doi.org/10.2341/19-146-L>

tron microscopy. DC was measured using micro-Raman spectroscopy. The data were analyzed by three-way analysis of variance and Tukey tests ($\alpha=0.05$).

Results: AMB-APS showed similar performance in all root thirds ($p>0.05$) and higher values of DC, especially in the apical third ($p<0.0001$). AMB and SBU showed the lowest values in the apical third ($p<0.0001$).

Conclusions: The APS photo-initiator system contained in universal adhesives is a feasible alternative for improving radicular bonding procedure.

INTRODUCTION

Fiber post cementation has become a very popular alternative with clinicians for endodontically treated teeth. The success of cementation depends on the strong bond to tooth substrates,^{1,2} and a suitable dentin bonding is needed for infiltration of resin monomers on etched substrates and subsequent polymerization.^{3,4} However, in radicular dentin, there are several limitations, such as typical histological morphology in the apical third⁵ and a high C-factor within the root canal⁶ impairing the bonding on this substrate.⁷ Furthermore, the polymerization process inside the root canal, which is a prerequisite for adequate bonding, is impaired because of the difficulty in controlling humidity and solvent evaporation of the adhesive system.^{8,9} In addition, lower bonding performance can be caused by the reduced radiant exposure achieved in the apical third,¹⁰ making radicular dentin bonding a challenge.

Some authors have proposed alternatives for increasing the success of fiber post cementation into radicular dentin by reducing the required light intensity for the polymerization of the material inside of the radicular dentin.^{11,12} However, the most commonly employed photo-initiator systems for the polymerization of resin-based materials are based on a camphorquinone (CQ)/amine system. Basically, a photosensitizer molecule (CQ) can absorb light directly, and a co-initiator (typically a tertiary amine) interacts with the activated photo-initiator to generate a reactive free radical. These reactive free-radical species attack monomer molecules and initiate polymerization.¹³ However, CQ and amines are hydrophobic, which are antagonistic to the adhesive solutions. These solutions contain hydrophilic components required for the interaction with tooth substrates, promoting the resin infiltration on the wet dentin surface.¹⁴ This problem

encourages research efforts to identify alternative co-initiators as substitutes for amines to improve the photo-initiator systems without impairing the polymerization reaction and the bonding ability of dental adhesives.¹⁵⁻¹⁷

Furthermore, it was previously demonstrated that in the hydrophilic-rich phase of simplified adhesives, photo-initiator systems (CQ/amines) are present in limited concentrations in the hydrophobic phase. This effect could compromise the photo-polymerization of the hydrophilic-rich phase and result in loosely cross-linked regions, facilitating diffusion of the oral fluid into the resin-dentin interface.¹⁸ Therefore, more-hydrophilic photo-initiator systems added into adhesive systems could be an interesting alternative for improving the polymerization on the apical third and, consequently, the bonding performance in radicular dentin. However, to the best of our knowledge, this approach has not been previously evaluated.

Thus, this study investigated the use of three universal adhesives with different photo-initiator systems (applied with etch-and-rinse [ER] and self-etch [SE] strategies) on the interaction with radicular dentin (push-out bond strength [PBS], nano-leakage [NL] evaluation, and degree of conversion [DC] within the hybrid layer). The different root thirds were also evaluated. The null hypotheses tested were that the evaluated bonding characteristics of post-to-radicular dentin are not affected by 1) adhesive strategies (ER and SE), 2) different universal adhesives, or 3) different root thirds.

METHODS AND MATERIALS

Sample Preparation

After approval by the local ethics committee (protocol 2.408.873), 66 caries-free human mandibular premolars (root length of about 14 mm measured from the cemento-enamel junction), extracted within the past 6 months, were selected. The dental crowns were removed by a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). A single operator, a specialist in endodontics and experienced in all the techniques used in this study, performed the root canal preparation and obturation according to Vilas-Boas and others.¹⁹ Root canals were prepared 1-mm shorter than the apical foramen with Reciproc R40 (VDW, München, Germany) and irrigation using 1% sodium hypochlorite (10 mL in total). Subsequently, the smear layer was removed using 5 mL of 17% EDTA for three minutes and flushed with 10 mL of distilled water. Canals were dried with absorbent

Table 1: *Universally Used Adhesive Systems (Manufacturer/Batch Number), Detailed Composition, and Application Mode, According to the Adhesive Strategy^a*

Adhesive System (Manufacturer/ Batch No.)	Composition	Strategies	
		Self-etching	Total Etching
AMB-APS (FGM, Joinville, SC, Brazil/070817)	10-MDP, methacrylic monomers, photo-initiator APS, CQ, silica nanoparticles, ethanol, co-initiators, and stabilizers	1. Apply two layers with a micro brush for 20 s (10 s each layer) 2. Evaporate the adhesive solvent by using gentle air for 10 s 3. Light-cure for 10 s to 1200 mW/cm ²	1. Phosphoric acid etching in enamel for 15 s 2. Rinsing with water spray to remove the acid 3. Evaporation of the adhesive solvent, using gentle air 4. Application of the adhesive system to the self-etching mode
AMB (FGM, Joinville, SC, Brazil/200416)	10-MDP, hydrophilic methacrylic monomers, ethanol, silanized silicon dioxide, CQ, co-initiators, and stabilizers	1. Apply two layers with a micro brush for 20 s (10 s each layer) 2. Evaporate the adhesive solvent, using a gentle air for 10 s 3. Light-cure for 10 s to 1200 mW/cm ²	1. Phosphoric acid etching in enamel for 15 s 2. Rinsing with water spray to remove the acid 3. Evaporation of the adhesive solvent using gentle air 4. Application of the adhesive system to the self-etching mode
SBU (3M Oral Care, St Paul, MN, USA/ 1630600505)	10-MDP, HEMA, bis-GMA, decamethylene dimethacrylate, ethanol, silane-treated silica, water, copolymer of acrylic and itaconic acid, CQ, dimethylaminobenzoate(–4)	1. Apply the adhesive actively for 20 s and, if necessary, reapply the adhesive system 2. Air dry for 5 s until the adhesive does not move and the solvent evaporates completely 3. Light-cure for 10 s to 1200 mW/cm ²	1. Phosphoric acid etching in enamel for 15 s 2. Rinsing with water spray to remove the acid 3. Evaporation of the adhesive solvent using gentle air 4. Application of the adhesive system to the self-etching mode

Abbreviations: bis-GMA: bisphenol A-glycidyl methacrylate; CQ: camphorquinone; HEMA: 2-hydroxyethyl methacrylate; 10-MDP: 10-methacroyloxydecyl dihydrogen phosphate.
^a All adhesive systems were applied according to the manufacturers' instructions.

paper points (VDW). Then, the canals were filled with AH Plus (Dentsply Ind Com Ltd, Petropolis, RJ, Brazil) using the single-cone technique (R40, Reciproc). The cervical opening was sealed (glass-ionomer cement Maxxion R, FGM, Joinville, SC, Brazil) and the teeth were stored at 37°C in 100% humidity for seven days.

Fiber Post Cementation

The filling material was removed from the coronal 10 mm of the canal (leaving 4 mm of gutta-percha in the apical third of the root) using #3 Largo burs (Dentsply Maillefer, Ballaigues, Switzerland). The post space was prepared using a #2 bur (White Post DC, FGM) with a 10-mm length, followed by irrigation (10 mL of distilled water) and dried with absorbent paper points. The specimens were randomly divided into six groups (n=12) according to one of three adhesive systems (Scotchbond Universal [SBU], 3M Oral Care, St Paul, MN, USA; Ambar Universal [AMB], FGM; and Ambar Universal APS [AMB-APS], FGM) and two adhesive strategies (ER and SE) for each system. Product information and modes of application are detailed in Table 1.

Before cementation, glass fiber posts were horizontally sectioned in the coronal region with a water-cooled diamond cutting instrument to reduce the post length to 13 mm. While 10 mm was cemented inside the root canal, the coronal 3 mm served as a guide to standardize the distance of the light-curing device from the cervical root area. All posts were cleaned with gauze immersed in 70% alcohol for five seconds.

After the adhesive application, a dual-cure resin cement (All Cem, shade A2, FGM) was inserted with a Centrix syringe (NOVA DFL, Rio de Janeiro, RJ, Brazil); the posts (DC 2, FGM) were inserted immediately and light polymerized for 40 seconds (1200 mW/cm², Valo, Ultradent Products, Lake City, UT, USA).

Specimen Preparation and Measurement

After storage in water at 37°C for seven days, the specimens were sectioned perpendicular to the long axis into six 1-mm serial slices under water cooling (Isomet 1000, Buehler). Both sides of each slice were photographed at 40× magnification to measure the coronal and apical diameters of the posts to calculate

the individual bonding areas (Image J, National Institutes of Health [NIH], Bethesda, MD, USA).

Push-out Bond Strengths

The PBS test ($n=8$ teeth) was performed by measuring the bond strength of the fiber post to dentin. The cervical side of each test specimen was placed in contact with a support (Odeme, Joaçaba, SC, Brazil), which was coupled to the base of a universal test machine (Instron, 3342, Canton, MA, USA). Loading was performed at a crosshead speed of 0.5 mm/min^{-1} until the post was completely dislodged from the root slice. The maximum value obtained in kilogram-force was used to calculate the bond strength in megapascals (MPa) using the following formula: $A = \pi (R + r) \sqrt{h^2 + (R - r)^2}$, where π is the constant 3.14; r_1 and r_2 are the smaller and larger post space radii, respectively; and h is the height of the section in millimeters. The debonded specimens were observed under $40\times$ magnification with a stereomicroscope loupe (SZ61, Olympus America Inc, Center Valley, PA, USA) to categorize the failure mode into six types: 1) adhesive at the post-cement interface, 2) adhesive at the cement-dentin interface, 3) adhesive mixed at the post-cement-dentin interface, 4) cohesive into dentin, 5) cohesive into cement, and 6) cohesive into post.

Nanoleakage Evaluation

For NL evaluation ($n=2$ teeth), the slices were immersed in 50 wt% ammoniacal silver nitrate solution for 48 hours and photo developed for eight hours under indirect fluorescent light. After polishing with a wet 600-, 1000-, 1200-, 1500-, 2000-, 2500-, and 4000-silicon carbide paper, each slab was ultrasonically cleaned, mounted, and sputter-coated in a vacuum evaporator (SCD 050, Balzers Union, Balzers, Liechtenstein), and the entire surface was examined using a scanning electron microscope (VEGA 3 TESCAM, Shimadzu, Tokyo, Japan). First, the slices were examined at a magnification of $600\times$ to identify the center region, and then several micrographs were taken at a magnification of $1000\times$. The relative percentage of NL at the bonded interface was measured with the software Image J (NIH).

In Situ DC Within the Hybrid Layer

For the *in situ* DC determination ($n=2$ teeth), two bonded slices obtained by the sectioning that had not been used in the PBS were wet polished using 1500- and 2000-grit SiC papers and analyzed through

micro-Raman spectroscopy (HORIBA Scientific, Kyoto, Japan) with a 638-nm diode laser, $100\times$ objective, 600 lines/mm grating centered between 800 and 1800 cm^{-1} , 100 mW of power, and accumulation time of 30 seconds with five coadditions. The spectra were obtained at the dentin-adhesive interface at three random sites (per slice) within the intertubular-infiltrated dentin. The spectra of uncured adhesives were taken as references, and DC was calculated according to Hass and others.²⁰ The ratio of double bonds of uncured and after curing (monomer to polymer) in the adhesive was calculated according to the following formula: $\text{DC}(\%) = (1 - [R_{\text{cured}}/R_{\text{uncured}}]) \times 100$, where R is the ratio of aliphatic and aromatic peak intensities at 1639 cm^{-1} and 1609 cm^{-1} in cured and uncured adhesives.

Statistical Analysis

The data of PBS, NL, and DC of all slices from the same tooth were averaged for statistical purposes. After evaluating the normality (Kolmogorov-Smirnov) and the equality of variances (Bartlett) tests, the data were evaluated by three-way analysis of variance (adhesive system vs adhesive strategy vs root third) and the Tukey test ($\alpha=0.05$). All of the analyses were performed using the software SPSS (Statistical Package for the Social Science) version 17.0 (SPSS Inc, Chicago, IL, USA). In the case of NL results, the representative images were taken only after the data were collected.

RESULTS

In most specimens, the adhesive (at the cementum-dentin interface) and adhesive-mixed were detected in all experimental groups (data not shown). The obtained results for all bonding properties are shown in Table 2. For all properties, three-way analysis of variance (ANOVA) revealed a statistically significant effect for the cross-product interaction ($p<0.0001$). For all bonding properties, there was no significant difference between adhesive strategies for all of the evaluated adhesives ($p>0.05$).

Push-out Bond Strengths

Considering the PBS, for the adhesives SBU and AMB, the cervical third showed higher values than those of the apical third in both strategies ($p<0.0001$). The exception was AMB-APS, which showed no significant difference when radicular thirds were compared in both adhesive strategies ($p>0.05$). Significant and lower PBS values were

Table 2: Means and Standard Deviations of Values Obtained by the Push-out Bond Strength (PBS), Nanoleakage (NL), and In Situ Degree of Conversion (DC) for All Experimental Conditions ^a						
Experimental Condition	PBS, MPa		NL, %		DC, %	
	ER	SE	ER	SE	ER	SE
AMB-APS						
Cervical	13.6 ± 4.5 AB	11.9 ± 6.2 AB	7.3 ± 1.3 a	7.2 ± 1.1 a	73.9 ± 2.5 ^a	78.6 ± 3.9 ^a
Middle	12.1 ± 5.7 AB	10.9 ± 3.6 BC	8.0 ± 1.7 ab	6.6 ± 1.6 a	65.2 ± 5.0 ^b	65.4 ± 6.2 ^b
Apical	13.6 ± 4.7 A	12.2 ± 3.4 AB	7.9 ± 1.1 ab	6.1 ± 1.1 a	63.3 ± 5.5 ^b	63.2 ± 5.5 ^b
AMB						
Cervical	15.9 ± 2.7 A	13.9 ± 5.5 A	7.1 ± 1.7 a	6.9 ± 0.9 a	71.0 ± 6.2 ^a	73.3 ± 5.6 ^a
Middle	12.3 ± 6.1 AB	10.6 ± 2.8 BC	9.9 ± 1.1 b	7.8 ± 2.2 ab	56.9 ± 6.5 ^c	56.4 ± 3.8 ^c
Apical	8.2 ± 3.3 BC	9.2 ± 4.0 BC	11.3 ± 1.6 c	11.1 ± 2.7 c	45.6 ± 7.8 ^d	52.9 ± 3.8 ^{cd}
SBU						
Cervical	13.4 ± 4.7 AB	13.6 ± 5.6 AB	8.3 ± 1.1 ab	7.7 ± 1.2 a	75.4 ± 2.3 ^a	74.1 ± 2.7 ^a
Middle	10.44 ± 4.6 BC	10.9 ± 4.4 BC	9.7 ± 1.0 bc	11.1 ± 1.2 c	51.6 ± 4.5 ^c	59.3 ± 6.5 ^{bc}
Apical	7.5 ± 2.8 C	7.3 ± 2.4 C	11.5 ± 1.1 c	12.2 ± 1.6 c	41.9 ± 1.9 ^d	43.1 ± 5.1 ^d
^a Different letters indicate a statistically significant difference for the comparisons inside each property (three-way ANOVA and Tukey test; p<0.0001). Different uppercase letters are valid for comparisons for PBS, lowercase letters for NL, and superscript lowercase letters for DC.						

observed for SBU and AMB in comparison with AMB-APS in the apical third ($p<0.0001$).

Nanoleakage Evaluation

Considering the NL evaluation (Table 2), the demonstrative scanning electron micrographs of each experimental group are shown in Figure 1. According to the three-way ANOVA (Table 2), for AMB and SBU, the cervical third showed lower NL values than those of the apical third ($p<0.0001$). However, the AMB-APS did not show any significant

difference when thirds were compared in both adhesive strategies ($p>0.05$). Moreover, significant and higher NL values for SBU and AMB were observed in comparison with AMB-APS, especially in the apical third ($p<0.0001$).

In Situ DC Within the Hybrid Layer

For the *in situ* DC analysis (Table 2), the middle and apical thirds showed lower *in situ* DC values in comparison with the cervical radicular third for all of the evaluated adhesives ($p<0.0001$). However, AMB-

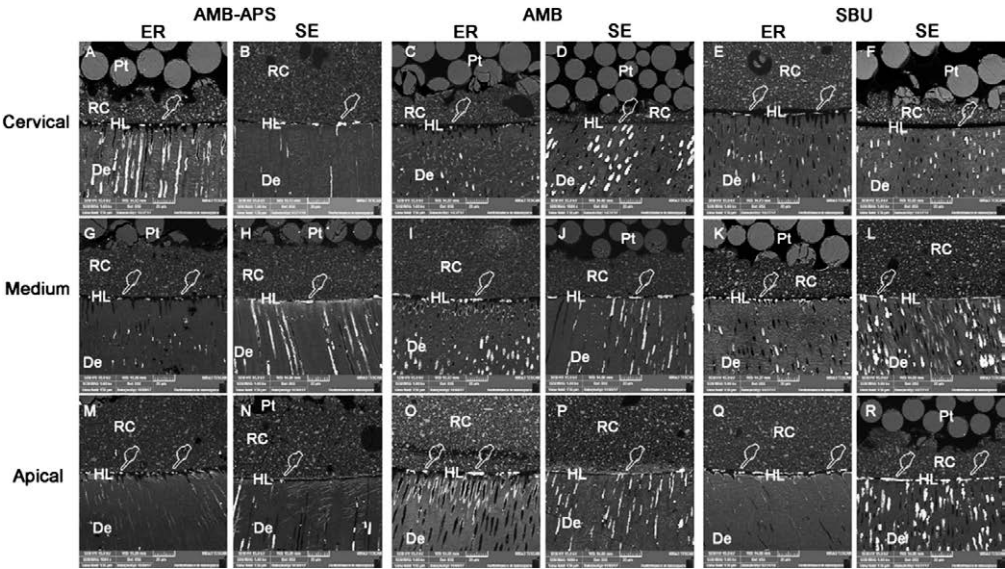


Figure 1. Representative scanning electron photomicrographs (1000x) of fiber post to radicular dentin interfaces for all experimental groups. Higher silver nitrate uptake (hand indicator) can be observed on the apical third for AMB (figures O and P) and SBU (figures Q and R), in both adhesive strategies. In general mode, the AMB-APS showed the lowest silver nitrate uptake on fiber post-radicular dentin interfaces, especially on the apical third (figures M and N). Pt, post; RC, resin cement; HL, hybrid layer; De, radicular dentin.

APS did not show any significant difference when middle and apical thirds were compared ($p>0.05$). Significantly higher *in situ* DC values were observed for AMB-APS in comparison with SBU and AMB, especially in the apical third ($p<0.0001$).

DISCUSSION

This study found different performance among the tested adhesives and root thirds. However, there was no difference between the adhesive strategies, leading us to accept the first null hypothesis.

Although mechanical interlocking remains the primary bonding mechanism in immediate bonding, chemical adhesion has become a key to overcome the long-term degradation of dentin-resin interfaces.²¹ The phosphate-acid monomer (10-MDP) in all of the adhesives promotes chemical bonds to hydroxyapatite, forming hydrolytically stable calcium salts.^{21,22} Considering the universal application concept behind these adhesives, bond strength should not be compromised by the application mode. Therefore, similar results between the adhesive strategies should be expected.²³ Our results confirm that both mechanisms (mechanical and chemical interaction) were able to promote efficient bonding to the tooth substrate, which is the reason for the similar performance between the strategies.

Regarding root thirds, the cervical third presented higher bond strength and *in situ* DC values as well as lower silver nitrate uptake when compared with the apical third for two of the three adhesives evaluated, leading to partial rejection of the second null hypothesis. This performance could be attributed to the better access or visibility for humidity control, suitable solvent evaporation prior to polymerization, and higher radiant exposure by the proximity of the light-curing source.^{8,10,24,25} In contrast, the lowest values in the apical third could be explained by the contrary phenomena. The higher water content may cause phase separation of methacrylate adhesives during the photopolymerization, limiting the infiltration of bisphenol A-glycidyl methacrylate and consequently inhibiting the formation of an impervious, structurally integrated resin-dentin interface.²⁶ In fact, this could be confirmed by the highest NL and lower *in situ* DC values in the apical third. The water and arrested organic solvents within the adhesive layer prevent the approximation between reactive pendant species, making cross-linking reactions inside the hybrid layer more difficult.^{27,28} Thus, the polymer backbone may have had its free space greatly augmented in a level directly related to the amount

of organic solvent present during polymerization.²⁹ Consequently, the properties of the adhesive itself³⁰ and the final bond strength to the dentin substrate are affected.^{31,32}

Furthermore, incompletely polymerized adhesive layers contribute to the creation of porous hybrid layers with reduced sealing ability,^{33,34} contributing to higher silver nitrate uptake in the apical thirds. In addition, a lower radiant exposure was delivered to the apical third during the polymerization of the adhesive, and hence, fewer free radicals required for the polymerization process were promoted by the amine co-initiator.³⁵ All of these events certainly contributed to the decrease in *in situ* DC values^{10,36} and, consequently, to the reduced bond strength and higher NL in the apical third, explaining the difference among the root thirds.

However, a closer view of all the evaluated universal adhesives showed several different results regarding the evaluated bonding properties, leading to partial rejection of the third null hypothesis. The SBU and AMB systems presented an intense reduction of adhesive properties on the apical third when compared with AMB-APS. The former contains CQ as the photosensitizer molecule and a tertiary amine as co-initiator (CQ/amine system) in the composition, whereas the AMB-APS contains the APS photo-initiator system (Advanced Polymerization System, FGM).

The entire photo-initiated polymerization process is sensitive to initiator and co-initiator systems, types of monomers, and the reaction medium.¹⁴ Universal systems have acidic and hydrophilic monomers required for SE ability.³⁷ The 2-hydroxyethyl methacrylate (HEMA) contained in adhesive systems induced a notable reduction of photopolymerization and photoreactivity of systems based on CQ/amines,^{14,38} such as SBU and AMB. Furthermore, the type of co-initiator affects the rate of polymerization and the final conversion level of hydrophilic adhesives in the presence of water,¹⁴ as these effects were more intense in the apical third. Thereby, since SBU and AMB contain CQ/tertiary amine-based photo-initiator, which has limited polymerization ability in HEMA and water presence, the polymerization of the adhesive system was even more affected on the intrinsically wet apical third, resulting in the poorest bonding properties. This can be confirmed by the Raman results, which demonstrated a significant reduction of DC.

An earlier investigation quantified the components of methacrylate-based adhesive in the hydrophilic-rich phase. This study showed that the

hydrophobic cross-linker and the widely used photo-initiator system, such as CQ and tertiary amines such as ethyl 4-(dimethylamino) benzoate, were present in limited concentrations. This could compromise the photo-polymerization of the hydrophilic-rich phase and result in loosely cross-linked regions, facilitating diffusion of the oral fluid into the resin-dentin interface.¹⁸ Certainly, it would be related to a lower DC and bonding properties promoted by the CQ/amines-based adhesive on radicular dentin. The hydrophobic characteristics of the photosensitizer CQ and the aromatic co-initiator amines could undergo a higher incompatibility because of the humidity of the apical third in addition to the hydrophilic characteristics of adhesive systems. Consequently, the ability of photopolymerization promoted by CQ/amine-based systems was reduced.³⁸ This effect was the possible reason for the lower performance in the apical thirds for SBU and AMB in comparison with AMB-APS.

As previously mentioned, AMB-APS contained the APS photo-initiator system in its composition. According to the manufacturer, the APS photo-initiator system reduces the amount of CQ, which is balanced by the combination of several photo- and co-initiators acting synergistically. It would contribute to the reduction of incompatibility in the hydrophilic-rich phase promoted by the hydrophobicity of CQ/amines. In fact, a high *in situ* DC was shown in the apical third of the AMB-APS, demonstrating higher polymerization ability even in a wetter environment.

An indirect advantage of adhesives containing APS was recently shown by Oliveira and others.³⁹ One of the main disadvantages of CQ is its inherently yellowish-brown color, which significantly influences the color of the adhesive resin.⁴⁰ Nevertheless, the yellow color partially fades after curing, and the remaining yellow color may possibly cause problems in color matching.⁴¹ Because there is a diminished amount of CQ in the APS system, it is possible to produce an adhesive system (AMB-APS) with higher color stability in comparison with CQ-based adhesives, especially when the final color of thin ceramic veneers is evaluated.³⁹

Unfortunately, the exact composition of each universal adhesive is proprietary information. However, as previously shown, the addition of a third and more-hydrophilic component in the hydrophobic photo-initiator system seems an interesting approach.^{14,42} In an earlier study, visible-light photo-initiator systems were investigated using formulations with the combination of hydrophobic (CQ) and

several hydrophilic photosensitizers and hydrophilic co-initiators.⁴² The authors showed that the addition of a third and more-hydrophilic component to a photo-initiator system reduced the detrimental effects of nanoscale phase separation. This means that, if active radicals are produced by both hydrophobic and hydrophilic initiators, these different phases can be polymerized effectively, consequently improving polymerization of both hydrophilic and hydrophobic domains.⁴² Furthermore, the addition of other more hydrophilic co-initiators to a photo-initiator system increased the photopolymerization and photo-reactivity of hydrophilic monomers because of the ability to keep the energy level more stable during the excited state of CQ.¹⁴

Another hypothesis is that an APS photo-initiator system can keep this energy level more stable during excitation of CQ, as previously shown by Ilie and others.⁴³ According to these authors, this higher stability of the CQ molecule is an interesting form to recycle the CQ molecule and then potentialize its action.⁴³ Therefore, the outcome would be an increase in polymerization. The addition of hydrophilic components would become more compatible with wet substrates, as shown in the present study, not only in terms of higher *in situ* DC values and bond strength in the apical third but also in the reduction of silver nitrate uptake. Future studies need to be done to prove these hypotheses related to the APS photo-initiator system when associated with different adhesive substrates, and long-term studies should be conducted to evaluate the bonding performance of the post to radicular dentin.

CONCLUSIONS

The use of the APS photo-initiator system contained in universal adhesive systems showed an improvement in bonding properties (higher PBS, lower silver nitrate uptake, and higher *in situ* DC values), mainly in the apical third. This approach seems to be an interesting alternative to overcome the challenges of the radicular bonding procedure.

Acknowledgements

This study was performed by Roberta Carvalho as partial fulfillment of the MS degree at UniCEUMA, São Luiz, MA, Brazil. This study was partially supported by the National Council for Scientific and Technological Development (CNPq) under grant 308286/2019-7.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee

guidelines and policies of the Ceuma University. The approval code issued for this study is 2.408.873.

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 16 January 2020)

REFERENCES

- Dietschi D, Duc O, Krejci I, & Sadan A (2008) Biomechanical considerations for the restoration of endodontically treated teeth: a systematic review of the literature, part II (evaluation of fatigue behavior, interfaces, and *in vivo* studies) *Quintessence International* **39**(2) 117-129.
- Goracci C & Ferrari M (2011) Current perspectives on post systems: a literature review *Australian Dental Journal* **56**(Supplement 1) 77-83.
- 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.
- Nakabayashi N, Kojima K, & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials Research* **16**(3) 265-273.
- Mjor IA, Smith MR, Ferrari M, & Mannocci F (2001) The structure of dentine in the apical region of human teeth *International Endodontic Journal* **34**(5) 346-353.
- Tay FR, Loushine RJ, Lambrechts P, Weller RN, & Pashley DH (2005) Geometric factors affecting dentin bonding in root canals: a theoretical modeling approach *Journal of Endodontics* **31**(8) 584-589.
- Breschi L, Mazzoni A, De Stefano Dorigo E, & Ferrari M (2009) Adhesion to intraradicular dentin: a review *Journal of Adhesion Science and Technology* **23**(7-8) 1053-1083.
- Cuadros-Sanchez J, Szesz A, Hass V, Patzlaff RT, Reis A, & Loguercio AD (2014) Effects of sonic application of adhesive systems on bonding fiber posts to root canals *Journal of Endodontics* **40**(8) 1201-1205.
- Hayashi M & Ebisu S (2008) Key factors in achieving firm adhesion in post-core restorations *Japanese Dental Science Review* **44**(1) 22-28.
- Szesz A, Cuadros-Sanchez J, Hass V, da Cruz GK, Arrais CA, Reis A, & Loguercio AD (2015) Influence of delivered radiant exposure values on bonding of fiber posts to root canals *Journal of Adhesive Dentistry* **17**(2) 181-188.
- Pereira RD, Valdivia AD, Bicalho AA, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2015) Effect of photoactivation timing on the mechanical properties of resin cements and bond strength of fiberglass post to root dentin *Operative Dentistry* **40**(5) E206-E221.
- Boing TF, Gomes GM, Gomes JC, Reis A, & Gomes OM (2017) Is the bonding of self-adhesive cement sensitive to root region and curing mode? *Journal of Applied Oral Sciences* **25**(1) 2-9.
- Ogunyinka A, Palin WM, Shortall AC, & Marquis PM (2007) Photoinitiation chemistry affects light transmission and degree of conversion of curing experimental dental resin composites *Dental Materials* **23**(7) 807-813.
- Wang Y, Spencer P, Yao X, & Ye Q (2006) Effect of coinitiator and water on the photoreactivity and photopolymerization of HEMA/camphorquinone-based reactant mixtures *Journal of Biomedical Materials Research A* **78**(4) 721-728.
- Park YJ, Chae KH, & Rawls HR (1999) Development of a new photoinitiation system for dental light-cure composite resins *Dental Materials* **15**(2) 120-127.
- Wang K, Yang D, Xiao M, Chen X, Lu F, & Nie J (2009) Sesamin as a co-initiator for unfilled dental restorations *Acta Biomaterialia* **5**(7) 2508-2517.
- Lima GDS, Moreira AG, Meereis CTW, Lima GB, Leal FB, Moraes RR, Ogliari FA, Petzhold CL, & Piva E (2018) Long-term bonding efficacy of adhesives containing benzodioxoles as alternative co-initiators *Brazilian Oral Research* **32** e104.
- Ye Q, Park J, Parthasarathy R, Pamatmat F, Misra A, Laurence JS, Marangos O, & Spencer P (2012) Quantitative analysis of aqueous phase composition of model dentin adhesives experiencing phase separation *Journal of Biomedical Materials Research B Applied Biomaterials* **100**(4) 1086-1092.
- Vilas-Boas DA, Graziotin-Soares R, Ardenghi DM, Bauer J, de Souza PO, de Miranda Candeiro GT, Maia-Filho EM, & Carvalho CN (2018) Effect of different endodontic sealers and time of cementation on push-out bond strength of fiber posts *Clinical Oral Investigations* **22**(3) 1403-1409.
- Hass V, Luque-Martinez I, Sabino NB, Loguercio AD, & Reis A (2012) Prolonged exposure times of one-step self-etch adhesives on adhesive properties and durability of dentine bonds *Journal of Dentistry* **40**(12) 1090-1102.
- Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, & Van Landuyt KL (2011) State of the art of self-etch adhesives *Dental Materials* **27**(1) 17-28.
- Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Torii Y, Osaka A, Suzuki K, Minagi S, Van Meerbeek B, & Van Landuyt KL (2011) Self-etch monomer-calcium salt deposition on dentin *Journal of Dental Research* **90**(5) 602-606.
- 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.
- dos Santos Alves Morgan LF, Peixoto RT, de Castro Albuquerque R, Santos Correa MF, de Abreu Poletto LT, & Pinotti MB (2008) Light transmission through a translucent fiber post *Journal of Endodontics* **34**(3) 299-302.
- Goracci C, Corciolani G, Vichi A, & Ferrari M (2008) Light-transmitting ability of marketed fiber posts *Journal of Dental Research* **87**(12) 1122-1126.

26. Spencer P & Wang Y (2002) Adhesive phase separation at the dentin interface under wet bonding conditions *Journal of Biomedical Materials Research* **62(3)** 447-456.
27. Paul SJ, Leach M, Rueggeberg FA, & Pashley DH (1999) Effect of water content on the physical properties of model dentine primer and bonding resins *Journal of Dentistry* **27(3)** 209-214.
28. Ye Q, Spencer P, Wang Y, & Misra A (2007) Relationship of solvent to the photopolymerization process, properties, and structure in model dentin adhesives *Journal of Biomedical Materials Research A* **80(2)** 342-350.
29. Cho BH & Dickens SH (2004) Effects of the acetone content of single solution dentin bonding agents on the adhesive layer thickness and the microtensile bond strength *Dental Materials* **20(2)** 107-115.
30. Loguercio AD, Loeblein F, Cherobin T, Ogliari F, Piva E, & Reis A (2009) Effect of solvent removal on adhesive properties of simplified etch-and-rinse systems and on bond strengths to dry and wet dentin *Journal of Adhesive Dentistry* **11(3)** 213-219.
31. Hass V, Folkuenig MS, Reis A, & Loguercio AD (2011) Influence of adhesive properties on resin-dentin bond strength of one-step self-etching adhesives *Journal of Adhesive Dentistry* **13(5)** 417-424.
32. Takahashi A, Inoue S, Kawamoto C, Ominato R, Tanaka T, Sato Y, Pereira PN, & Sano H (2002) *In vivo* long-term durability of the bond to dentin using two adhesive systems *Journal of Adhesive Dentistry* **4(2)** 151-159.
33. Hass V, Dobrovolski M, Zander-Grande C, Martins GC, Gordillo LA, Rodrigues Accorinte Mde L, Gomes OM, Loguercio AD, & Reis A (2013) Correlation between degree of conversion, resin-dentin bond strength and nanoleakage of simplified etch-and-rinse adhesives *Dental Materials* **29(9)** 921-928.
34. Tay FR, Pashley DH, Suh BI, Carvalho RM, & Itthagarun A (2002) Single-step adhesives are permeable membranes *Journal of Dentistry* **30(7-8)** 371-382.
35. Peutzfeldt A (1997) Resin composites in dentistry: the monomer systems *European Journal of Oral Sciences* **105(2)** 97-116.
36. Konerding KL, Heyder M, Kranz S, Guellmar A, Voelpel A, Watts DC, Jandt KD, & Sigusch BW (2016) Study of energy transfer by different light curing units into a class III restoration as a function of tilt angle and distance, using a MARC patient simulator (PS) *Dental Materials* **32(5)** 676-686.
37. Perdigao J & Swift EJ Jr (2015) Universal adhesives *Journal of Esthetic and Restorative Dentistry* **27(6)** 331-334.
38. Guo X, Peng Z, Spencer P, & Wang Y (2009) Effect of initiator on photopolymerization of acidic, aqueous dental model adhesives *Journal of Biomedical Materials Research A* **90(4)** 1120-1127.
39. Oliveira OF Jr, Kunz PVM, Baratto Filho F, Correr GM, Cunha LFD, & Gonzaga CC (2019) Influence of pre-curing different adhesives on the color stability of cemented thin ceramic veneers *Brazilian Dental Journal* **30(3)** 259-265.
40. Janda R, Roulet JF, Kaminsky M, Steffin G, & Latta M (2004) Color stability of resin matrix restorative materials as a function of the method of light activation *European Journal of Oral Sciences* **112(3)** 280-285.
41. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, & Van Meerbeek B (2007) Systematic review of the chemical composition of contemporary dental adhesives *Biomaterials* **28(26)** 3757-3785.
42. Ye Q, Park J, Topp E, & Spencer P (2009) Effect of photoinitiators on the *in vitro* performance of a dentin adhesive exposed to simulated oral environment *Dental Materials* **25(4)** 452-458.
43. Ilie N, Kreppel I, & Durner J (2014) Effect of radical amplified photopolymerization (RAP) in resin-based composites *Clinical Oral Investigations* **18(4)** 1081-1088.

Degree of Conversion Contributes to Dentin Bonding Durability of Contemporary Universal Adhesives

A Tichy • K Hosaka • A Abdou • M Nakajima • J Tagami

Clinical Relevance

The degree of conversion of contemporary universal adhesives positively correlates with the bond strength to dentin. The correlation is more marked after thermocycling, suggesting that a high degree of conversion is required for long-term dentin bonding durability.

SUMMARY

Purpose: The objectives of this study were to evaluate the micro-tensile bond strength (μ TBS) of five contemporary universal adhesives to dentin after 24 hours and thermocycling (TC), to measure their degrees of conversion (DC) and to test the correlation between μ TBS and DC.

*Antonin Tichy, DDS, Cariology and Operative Dentistry, Tokyo Medical and Dental University, Tokyo, Japan; Institute of Dental Medicine, First Faculty of Medicine of the Charles University, Prague, Czech Republic

Keiichi Hosaka, DDS, PhD, Cariology and Operative Dentistry, Tokyo Medical and Dental University, Tokyo, Japan

Ahmed Abdou, MSc, Biomaterials, Faculty of Dentistry, Modern University for Technology and Information, Cairo, Egypt

Masatoshi Nakajima, DDS, PhD, Cariology and Operative Dentistry, Tokyo Medical and Dental University, Tokyo, Japan

Junji Tagami, DDS, PhD, Cariology and Operative Dentistry, Tokyo Medical and Dental University, Tokyo, Japan

*Corresponding author: Karlovo namesti 32, Prague 121 11, Czech Republic; e-mail: antonin.tichy@lf1.cuni.cz

<https://doi.org/10.2341/19-165-L>

Methods and Materials: Four commercially available universal adhesives, Prime&Bond universal (PBU), Ecosite Bond (EB), G-Premio Bond (GPB), and Clearfil Universal Bond Quick (UBQ), and one experimental adhesive, UBQ without an amide monomer (UBQ-A), were used in this study. For the μ TBS test, midcoronal dentin of 50 human molars was exposed, ground using 600-grit SiC paper, and the adhesives were applied according to the manufacturers' instructions. After resin-composite buildup and 24-hour water storage, one-half of the specimens were subjected to 15,000 thermal cycles. The specimens were sectioned into beams and stressed in tension at a cross-head speed of 1 mm/min until failure. The DC of adhesives applied to dentin was evaluated using attenuated total reflectance Fourier-transform infrared spectroscopy immediately after light-curing. All data were statistically analyzed at a significance level of 0.05.

Results: The highest μ TBSs were obtained with UBQ, UBQ-A, and PBU, which were not significantly different from each other both after 24 hours and TC. The μ TBS of GPB was lower compared with the aforementioned adhesives, but significantly only after TC, and the lowest

μ TBSs were obtained with EB. TC did not affect the μ TBSs of UBQ, UBQ-A, and PBU significantly, but a significant decrease was observed with GPB and EB. The highest DC was obtained with PBU and UBQ, followed by 2-hydroxyethyl methacrylate-rich adhesives UBQ-A and EB, which exhibited significantly lower DCs. The DC of GPB could not be determined because the reference peak at 1608 cm^{-1} was not detected in its spectra. A significant positive correlation was shown between μ TBS and DC after 24 hours ($r=0.716$) and TC ($r=0.856$).

Conclusion: μ TBS and DC were positively correlated, more markedly after TC, which suggests that DC may be an important factor for bond durability.

INTRODUCTION

The range of use of contemporary one-bottle one-step self-etch adhesives has become very wide. The so-called universal adhesives are applicable to various substrates in both self-etch and etch-and-rinse modes and offer dentists a choice in almost any clinical situation. However, all one-bottle adhesives are very complex blends of hydrophilic and hydrophobic monomers, water, and solvents. Consequently, drawbacks including inferior durability, excessive water sorption, increased nanoleakage, phase separation (in 2-hydroxyethyl methacrylate [HEMA]-poor or HEMA-free adhesives), and reduced shelf life are frequently reported.¹

Given the great simplicity and variability of the use of universal adhesives, focus has been placed on overcoming these drawbacks. However, the inevitable hydrophilicity resulting from the presence of hydrophilic monomers and water remains a problem,^{2,3} because water diffusion into the adhesive layer after polymerization may lead to hydrolytic degradation and thus lower durability.^{4,5} The increased water sorption is mainly associated with the presence of HEMA,^{6,7} but it cannot be simply eliminated, because of HEMA's positive effect on the wetting properties of the adhesives and the ability to prevent phase separation.⁸ However, it is also reported that HEMA has negative effects on the mechanical properties, bond strengths, and polymerization of the adhesives.⁸⁻¹⁰ Hence, some newly marketed adhesives partially or completely replace HEMA with acrylamide monomers, which are claimed to reduce the aforementioned drawbacks. Recent studies report that the acrylamides exhibit lower water sorption, higher bond strength,¹¹ and

favorable durability while preventing phase separation.¹² Acrylamide monomers are also more hydrolytically stable, allowing for a longer shelf life.¹³

Besides HEMA, the polymerization of one-bottle adhesives may be adversely affected by remaining solvent^{14,15} and water,¹⁶ because their complete evaporation is clinically difficult.¹⁷ As a consequence, one-step adhesives are associated with a lower degree of conversion (DC) compared with multistep adhesives, which usually include a solvent-free resin as the final step. The lower DC may result in higher permeability,^{18,19} lower durability, and impaired mechanical properties^{17,20,21} of the simplified adhesives. Therefore, HEMA-free adhesives were introduced, and solvents such as isopropanol or tert-butanol²² and more efficient photoinitiators²³⁻²⁵ have been incorporated in some of the products to improve DC.

Four recent commercially available universal adhesives and one experimental product were included in this study. The adhesives differed in the content of HEMA, the incorporation of acrylamide monomers, solvent type, and photoinitiating systems. Their micro-tensile bond strength (μ TBS) to dentin was evaluated both after 24 hours and after thermocycling (TC), and the immediate DC was measured using attenuated total reflectance Fourier-transform infrared spectroscopy (ATR-FTIR). The main objective of this study was to investigate whether μ TBS is correlated with DC. The null hypothesis was that there is no significant correlation between the μ TBS and the DC of universal adhesives.

METHODS AND MATERIALS

Universal adhesives, Prime&Bond universal (PBU; Dentsply DeTrey GmbH, Konstanz, Germany), Eco-site Bond (EB; DMG CPF GmbH, Hamburg, Germany), G-Premio Bond (GPB; GC Corp, Tokyo, Japan), and Clearfil Universal Bond Quick (UBQ; Kuraray Noritake Dental Inc, Tokyo, Japan), and an experimental version of UBQ without the amide monomer, which was replaced with HEMA (UBQ-A; Kuraray Noritake Dental Inc), were used in this study. Table 1 presents a detailed description of the adhesives' composition and application procedures.

Micro-tensile Bond Strength Test

Specimen Preparation—Fifty sound human third molars were used for μ TBS testing. The teeth were stored in distilled water at 4°C and used within six months of extraction. Before bonding, midcoronal

Table 1: Composition, Batch Number, and Application Procedure of Adhesive Systems

Adhesive System (Manufacturer)	Composition (Batch Number)	pH	Application
Prime&Bond Universal (Dentsply DeTrey GmbH)	10-MDP, bisacrylamide monomers, PENTA, isopropanol, water, initiator, stabilizer (1703000839)	2.5	1. Apply to the entire dentin surface and agitate for 20 seconds 2. Air dry for six seconds with mild air pressure (0.2 MPa) 3. Light cure for 10 seconds
Ecosite Bond (DMG CPF GmbH)	10-MDP, dental resins, HEMA, ethanol, water, additives, catalysts (788410)	4.5	1. Apply to the entire dentin surface, rub for 10 seconds and leave for 20 seconds 2. Air dry for five seconds with mild air pressure (0.2 MPa) 3. Light cure for 10 seconds
G-Premio Bond (GC Corp)	4-MET, 10-MDP, MEPS, methacrylate monomer, acetone, water, TPO, silica (1806201)	1.5	1. Apply to the entire dentin surface and leave for 10 seconds 2. Air dry for five seconds with maximum air pressure (0.35 MPa) 3. Light cure for 10 seconds
Clearfil Universal Bond Quick (Kuraray Noritake Dental Inc)	10-MDP, Bis-GMA, HEMA, hydrophilic amide monomer, colloidal silica, ethanol, water, silane coupling agent, sodium fluoride, CQ (041807)	2.3	1. Apply to the entire dentin surface 2. Air-dry immediately for six seconds with mild air pressure (0.2 MPa) 3. Light cure for 10 seconds
Clearfil Universal Bond Quick without amide monomer (Kuraray Noritake Dental Inc)	10-MDP, Bis-GMA, HEMA, colloidal silica, ethanol, water, silane coupling agent, sodium fluoride, CQ (171117)	2.3	1. Apply to the entire dentin surface 2. Air-dry immediately for six seconds with mild air pressure (0.2 MPa) 3. Light cure for 10 seconds
Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; PENTA, dipentaerythritol pentacrylate monophosphate; HEMA, 2-hydroxyethyl methacrylate; 4-MET, 4-methacryloxyethyl trimellitic acid; MEPS, methacryloyloxyalkyl thiophosphate methylmethacrylate; TPO, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide; Bis-GMA, bisphenol-A-glycidyl methacrylate; CQ, camphorquinone.			

dentin surfaces were exposed using a low-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) under water cooling and ground using a wet 600-grit SiC paper (DCCS, Sankyo Fuji Star, Saitama, Japan) to produce a standardized smear layer.

Adhesive Application—For each of the five adhesives, 10 teeth were randomly selected. First, the dentin surfaces were gently air-blown to remove excess water. Then, the adhesives were applied using a disposable microbrush and air-blown according to the respective manufacturer's instructions (Table 1). The adhesives were light-cured from a distance of approximately 2 mm for 10 seconds using an LED light curing unit (10 seconds, 1000 mWcm⁻², Valo, Ultradent Products Inc, South Jordan, UT, USA). Next, the teeth were built up using resin composite Clearfil AP-X (shade A2, Kuraray Noritake Dental Inc) to a thickness of approximately 5 mm. The buildups were constructed in three increments less than 2 mm in thickness, and each of them was light-cured for 20 seconds (1000 mWcm⁻², Valo, Ultradent Products Inc). All the procedures were performed under standard room conditions, 23°C, and a constant relative humidity. The specimens were then stored in distilled water at 37°C for 24 hours.

Thermocycling—After 24-hour water storage, one-half of the specimens of each group (n=5) were subjected to 15,000 thermal cycles in distilled water at temperatures of 5°C and 55°C. The dwell time was 30 seconds and the transfer time was five seconds.

μTBS Testing—The specimens were sectioned perpendicular to the bonding layer into 12-16 non-trimmed beams using an Isomet saw (Isomet 1000, Buehler) under water cooling. The beams were approximately 1 × 1 × 8 mm in size, and their dimensions were checked using a digital caliper Mitutoyo CD15 (Mitutoyo, Kawasaki, Japan) for precise bonding area calculation. Using command-set glue (Model Repair II Blue, Dentsply-Sankin, Tokyo, Japan), the beams were attached to a flat μTBS jig and stressed at a crosshead speed of 1 mm/min until fracture, using an EZ Test testing machine (Shimadzu Corp, Kyoto, Japan). The μTBS in MPa was obtained by dividing the recorded force at failure by the calculated bonded area of the specimens.

Scanning Electron Microscopy (SEM) Analysis—The fractured specimens were mounted on brass stubs, dried, sputter-coated with gold, and observed using a scanning electron microscope (JSM-IT100, JEOL Ltd, Tokyo, Japan). Three different failure modes were distinguished: 1) adhesive failure (more than 80% of the failure within the adhesive or at the

dentin-adhesive interface), 2) cohesive failure within substrates (more than 80% of the failure in dentin or resin composite), and 3) mixed failure (the combination of adhesive and cohesive failure).

Statistical Analysis—Pretesting failures were considered left-censored data and were assigned a mean value between zero and the lowest μ TBS measured in the respective group. The mean μ TBSs calculated for each tooth were used as statistical units and analyzed using a two-way analysis of variance (ANOVA; factors material and storage condition). A multiple Student's *t*-test with Bonferroni correction was used for pairwise comparisons at a significance level of 0.05 (SPSS Statistics 23.0, IBM Corp, Armonk, NY, USA).

Weibull analysis was performed using the Weibull R package for R distribution (R Foundation for Statistical Computing, Vienna, Austria). Weibull scale (characteristic strength) and shape (modulus) parameters were calculated using a maximum likelihood estimation analysis, and 95% confidence intervals were calculated based on Monte Carlo simulations. The groups were compared using the strength at a 10% probability of failure (P10).

Degree of Conversion Analysis

Specimen Preparation—An additional five sound human third molars were sectioned using the Isomet saw (Isomet 1000, Buehler) under water cooling to obtain four to six thin coronal dentin specimens per tooth, approximately $1 \times 3 \times 4$ mm in size. Their surfaces were ground with a wet 600-grit SiC paper (DCCS, Sankyo Fuji Star) to produce a standardized smear layer.

ATR-FTIR Analysis—The absorption spectra of the adhesives were obtained from the region between 4000 and 400 cm^{-1} at a 2- cm^{-1} resolution using a Nicolet iS50 FT-IR spectrometer (Thermo Fisher Scientific, Waltham, MA, USA). Five dentin specimens were randomly selected for each adhesive. The adhesives were applied and air-blown according to their respective manufacturer's instructions (Table 1). The spectra were acquired first without light-curing, then, the adhesives were light-cured for 10 seconds (1000 mWcm^{-2} ; Valo, Ultradent Products Inc) and their spectra were immediately recorded. All the procedures were performed under standard room conditions, 23°C, and a constant relative humidity.

DC Calculation—DC is calculated using the equation $DC = 1 - (R_{\text{cured}}/R_{\text{uncured}})$, where *R* represents the ratio between the absorbance peak

of aliphatic C=C double bonds (around 1638 cm^{-1}) and a reference peak (internal standard) around 1608 cm^{-1} representing the aromatic double bonds.²⁶⁻²⁸ The DC data were statistically analyzed using a one-way ANOVA and Tukey's *post hoc* test ($\alpha=0.05$; SPSS Statistics 23.0, IBM Corp).

Correlation Between μ TBS and DC—The mean μ TBS values of each tooth (five per group) and the DC measurements (five per group) were used for the analysis. The Pearson correlation coefficient was calculated separately for each storage condition (24 hours, TC), $\alpha=0.05$ (SPSS Statistics 23.0, IBM Corp).

RESULTS

μ TBS

The results are summarized in Table 2. The two-way ANOVA revealed that both the material ($p<0.001$) and storage conditions ($p<0.001$) had a significant effect on the μ TBS and that their interaction also was significant ($p<0.019$). The highest μ TBS values were obtained with UBQ, followed by UBQ-A, PBU, GPB, and EB. It should be noted that the spread of the μ TBS values acquired with EB was huge both after 24 hours and TC, resulting in high SD and low Weibull modulus (shape parameter).

After 24 hours, the outcomes of the statistical analyses differed slightly. P10 of EB was significantly lower than that of all the other adhesives, which did not differ among each other. ANOVA did not identify any significant difference between the μ TBSs of EB and GPB ($p=1$), and it revealed that the μ TBS of GPB was significantly lower than that of UBQ ($p=0.022$).

After TC, the same results were obtained with ANOVA and Weibull analysis. The bond strength of EB was significantly lower than that of GPB ($p=0.002$), whose mean μ TBS and P10 were significantly lower compared with PB ($p=0.014$), UBQ-A ($p=0.001$), and UBQ ($p=0.001$). Compared with the 24-hour values, a significant decrease in bond strength was found for EB ($p<0.001$) and GPB ($p=0.024$) after TC.

SEM Analysis

The prevailing failure mode was adhesive (exemplary images are presented in Figure 1). In most groups, the fracture propagated partly through the adhesive resin and partly at the dentin-adhesive interface, as the adhesive layers were very thin. However, with EB, the failures occurred mainly at the dentin-adhesive interface. Round-shaped droplets were observed within the adhesive layer of GPB (Figure

Table 2: Results of Microtensile Bond Strength Test and Failure Mode Analysis						
Storage Condition	Adhesive	Mean ± SD (MPa) ^a	Characteristic Strength (95% CI)	Modulus (95% CI)	P10 (95% CI) ^b	Failure Mode, A/C/M (%) ^c
24-Hour water storage	PBU	66.3 ± 4.1 BCa	71.4 (68.4-74.4)	5.7 (4.7-6.7)	48.1 (43.4-52.3) cd	34/38/28
	EB	53.1 ± 13.2 Aa	59.9 (55.5-64.5)	3.0 (2.5-3.5)	28.2 (23.3-32.9) b	50/22/28
	GPB	60.0 ± 3.7 ABa	64.7 (62.1-67.3)	5.4 (4.6-6.2)	42.6 (39.0-46.0) c	76/16/8
	UBQ	74.5 ± 6.0 Ca	81.2 (77.3-85.1)	4.9 (4.1-5.8)	51.3 (45.7-56.3) cd	42/34/24
	UBQ-A	72.3 ± 4.4 BCa	78.4 (74.9-81.9)	5.2 (4.3-6.0)	50.6 (45.6-55.2) cd	33/51/15
Thermocycling	PBU	65.3 ± 4.9 Fa	71.4 (68.3-74.6)	5.5 (4.5-6.5)	47.3 (42.5-51.7) cd	25/36/39
	EB	31.2 ± 14.3 Db	36.2 (31.8-41.1)	1.7 (1.4-2.0)	9.6 (7.0-12.5) a	75/8/18
	GPB	49.9 ± 4.2 Eb	55.2 (52.6-57.8)	4.9 (4.1-5.7)	34.7 (31.0-38.1)b	78/18/4
	UBQ	70.2 ± 5.1 Fa	76.3 (73.1-79.5)	5.6 (4.7-6.7)	51.2 (46.2-55.6) d	37/35/28
	UBQ-A	69.6 ± 4.2 Fa	75.2 (72.2-78.3)	5.6 (4.7-6.6)	50.4 (45.6-54.7) cd	43/34/23
^a The mean microtensile bond strengths and SDs were analyzed using a two-way ANOVA and multiple Student t-tests with Bonferroni correction. Same uppercase letters indicate no statistically significant differences between adhesives under the same storage condition; same lowercase letters indicate no statistically significant differences between the storage conditions. ^b Same lowercase letters indicate no statistically significant differences between groups based on the results of Weibull analysis. ^c A, adhesive failure; C, cohesive failure within substrates (dentin or composite); M, mixed failure. Abbreviations: PBU: Prime&Bond universal, EB: Ecosite Bond, GPB: G-Premio Bond, UBQ: Clearfil Universal Bond Quick, UBQ-A: Clearfil Universal Bond Quick without amide monomer.						

1). Cohesive and mixed failures were more frequent with increasing bond strengths. In this study, only three pretesting failures occurred, all of them in the EB group after TC.

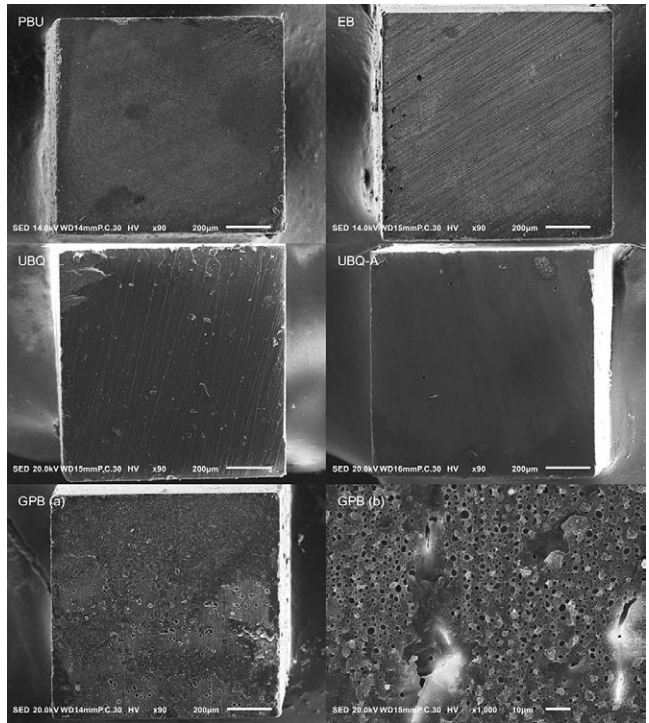


Figure 1. Exemplary SEM images of each adhesive. The images show representative adhesive failures for each material at a 90× magnification. It can be noted that round-shaped droplets (1000× magnified in GPB [b]) were present within the layer of GPB, presumably resulting from the phase separation of its components. Abbreviations: PBU: Prime&Bond universal, EB: Ecosite Bond, UBQ: Clearfil Universal Bond Quick, UBQ-A: Clearfil Universal Bond Quick without amide monomer, GPB: G-Premio Bond.

DC

DC in percentage is summarized in Table 3, and comparisons of spectra without and with light-curing for each material are presented in Figure 2. The highest DCs were obtained with PBU and UBQ, which were statistically similar ($p=0.842$). UBQ-A exhibited a DC significantly lower than PBU ($p=0.005$) and UBQ ($p=0.024$) but significantly higher than EB ($p<0.001$). GPB could not be evaluated because the peak at 1608 cm^{-1} was not detectable (Figure 2). Therefore, the correlations with μTBS were calculated only based on the results of PBU, EB, UBQ, and UBQ-A. A moderate positive correlation was revealed between DC and 24-hour μTBS ($r=0.716$), and a strong positive correlation was observed between DC and μTBS after TC ($r=0.856$). Both the correlations were statistically significant ($p<0.001$). The correlation graphs are presented in Figure 3.

DISCUSSION

New adhesives are frequently introduced to the market, often with spectacular claims regarding their properties. In this study, four recently commercialized universal adhesives and one experimental product were examined using a μTBS test and ATR-FTIR. The null hypothesis that there is no significant correlation between the μTBS and the DC of universal adhesives had to be rejected, because a significant correlation was revealed between DC and μTBS both after 24 hours and TC (Figure 3).

In this study, the DC of adhesives was evaluated after their application to dentin because it was

Table 3: Degree of Conversion (Mean $\pm$ SD)^a

Adhesive	Mean $\pm$ SD (%)
PBU	75.5 $\pm$ 2.8 c
EB	31.3 $\pm$ 9.1 a
GPB	—
UBQ	72.8 $\pm$ 7.8 c
UBQ-A	61.2 $\pm$ 5.2 b

^a Same lowercase letters indicate no statistically significant differences between the adhesives' degree of conversion in percentages. The degree of conversion of GPB could not be determined because the reference peak at 1608 cm⁻¹ was not detected.
Abbreviations: PBU: Prime&Bond universal, EB: Ecosite Bond, GPB: G-Premio Bond, UBQ: Clearfil Universal Bond Quick, UBQ-A: Clearfil Universal Bond Quick without amide monomer.

previously reported that the DC of experimental functional monomer-containing adhesives improved in the presence of hydroxyapatite.²⁹ Because the aim of this study was to examine the correlation between DC and μ TBS, we considered it essential to simulate the μ TBS experimental settings (and the clinical situation), where functional monomers can interact with the hydroxyapatite of hard dental tissues. Due to the low penetration depth of mid-range FTIR (about 1-3 μ m³⁰), the underlying dentin did not seem to interfere with the adhesives' spectra despite the thin adhesive layers of one-step adhesives.

On the other hand, the chemical properties of the adhesives may change after the application to dentin. This was probably the case of GPB, where the 1608 cm⁻¹ peak was successfully detected in previous studies when GPB was applied directly to the ATR crystal^{27,28} but not when it was applied to dentin²⁷ (Figure 2). Given that the 1608 cm⁻¹ peak represents aromatic carbon double bonds and that GPB does not contain bisphenol-A-glycidyl methacrylate (Bis-GMA), the only aromatic monomer that could be detected was 4-methacryloxyethyl trimellitic acid (4-MET). However, it is an acidic functional monomer and its conformation may change after the reaction of the carboxylic groups with hydroxyapatite, thus affecting the FTIR spectrum.

In some previous studies, the urethane (amide II) reference peak at 1538 cm⁻¹ was used for Bis-GMA-free adhesives,^{26,31} but it was not applicable in this study because the peak was not detected in all the adhesives. On the other hand, the carbonyl (C=O) peak at 1716 cm⁻¹ was detectable in all the materials tested, and it had been used as an internal standard for simplified adhesives in a previous study.²⁶ However, some FTIR studies reported that the vibrations of vinyl and carbonyl groups are not independent^{32,33} and that their conjugation is lost on polymerization.³³ Consequently, it was concluded

that the peak could not be used as an internal standard.³³ Therefore, only the peak at 1608 cm⁻¹ was used as the internal standard in this study, and the DC of GPB was not determined.

For the aforementioned reasons, the correlation between μ TBS and DC was examined only based on the results of PBU, EB, UBQ, and UBQ-A. A significant positive correlation was revealed both after 24 hours and TC, but it was stronger after TC, which indicates that DC may be an important factor in long-term bond strength stability. Besides that, the results showed that the DC of adhesives with reduced HEMA content (PBU, UBQ) was significantly higher compared with HEMA-rich materials (EB, UBQ-A). This may be due to the hydrophilicity of HEMA, because the water retained in the adhesive may lead to reduced polymerization.⁸ The presence of water in EB and UBQ-A was also confirmed by FTIR analysis, where the carbonyl peak at 1715 cm⁻¹ was not sharp and symmetric, presumably due to hydrogen bonding of water, which shifts the peak to $\sim$ 1700 cm⁻¹ (Figure 2).³²

UBQ exhibited the highest μ TBS both after 24 hours and TC. UBQ is a fluoride-releasing 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP)-based HEMA-containing adhesive. However, the content of HEMA was reduced by the incorporation of a new hydrophilic amide monomer, and it has been claimed that no waiting time is necessary prior to light-curing. Although the μ TBS obtained using the no-waiting approach was high, no significant difference from UBQ-A was found, which suggests that the incorporation of the amide monomer might not be the key feature. This is in contrast with a previous work where similar bond strengths of UBQ were obtained, but significantly lower μ TBS was acquired for UBQ-A.¹¹ We speculate that the different outcomes could be caused by differences in the application procedure, such as the presence/absence of scrubbing or slightly variable application time. Shorter application times were reported to compromise the bonding performance of universal adhesives,³⁴ especially on diamond-bur-cut dentin where a thicker and more compact smear layer is present.^{35,36}

Despite the addition of the new amide monomer, a previous study reported lower μ TBS of UBQ to bur-cut dentin and an improvement of its bond strength after phosphoric-acid etching, which suggests that the penetration in self-etch mode was insufficient due to the presence of a thicker smear layer.¹² It is also noteworthy that the μ TBS of UBQ was lower than that of Prime&Bond Active (Dentsply DeTrey

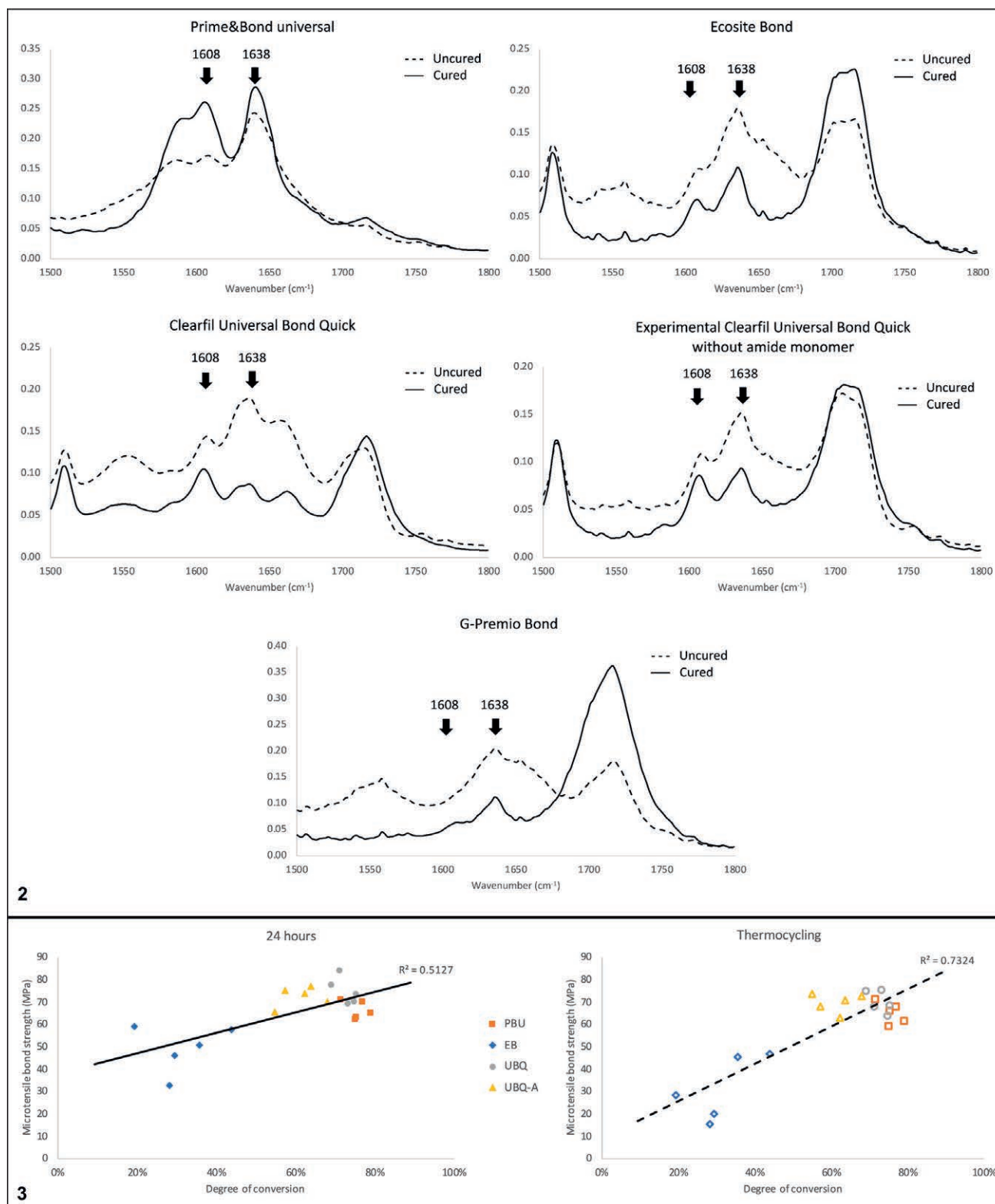


Figure 2. FTIR spectra of the adhesives ($1500\text{--}1800\text{ cm}^{-1}$). Comparison of the spectra before light-curing (dashed line) and immediately after light-curing (continuous line). The highlighted peaks at 1608 cm^{-1} (representing aromatic C=C double bonds) and at 1638 cm^{-1} (representing the aliphatic C=C double bonds) were used for the calculation of the degree of conversion. The peak at 1608 cm^{-1} is missing in the spectra of GPB, which is why its degree of conversion could not be calculated.

GmbH),¹² which is (based on the available information) probably another brand name for PBU used in this study. This may indicate that the prolonged application time (20 seconds with agitation for PBU) could be beneficial for UBQ. Although Kuno and others did not observe any significant benefits from increased application time on SiC paper-ground dentin, the authors suggested that the effect could be significant on bur-cut dentin.¹¹

Despite the arguable effect on application time, the presence of the amide monomer may bring more important advantages than saving a few seconds, such as improved DC and mechanical properties, higher durability, and lower water sorption. In this study, the DC of UBQ was significantly higher than that of UBQ-A, which could be due to its lower hydrophilicity compared with UBQ-A.^{37,38} The durability of UBQ was also favorable; TC did not significantly reduce the bond strengths of UBQ in this study and the study of Ahmed and others.¹² Furthermore, higher ultimate tensile strength after water storage and lower water sorption were reported for UBQ,¹¹ which can also be attributed to the reduced content of HEMA. Last, acrylamide monomers are more hydrolytically stable and were reported to improve shelf life.^{13,39}

PBU is another 10-MDP-based universal adhesive that features an acrylamide monomer, but unlike UBQ, HEMA is completely replaced. The DC of PBU was not significantly different from that of UBQ; both of them were significantly higher than the DC of HEMA-rich UBQ-A and EB. Slightly lower bond strengths were obtained with PBU compared with UBQ and UBQ-A; however, the difference was significant neither after 24 hours nor TC. No decrease in μ TBS was noted for PBU after TC in this study, similar to a previous report of no significant effect of 50,000 thermal cycles on the μ TBS of Prime&Bond Active.¹²

The durability of PBU may be attributed to lower hydrophilicity, resulting from the absence of HEMA, and to its DC. Given the longer application time⁴⁰ and the thin adhesive layer of PBU,⁴¹ solvent evaporation could possibly be improved, resulting in increased DC^{14,15} and lower adhesive permeability.^{18,19} The solvent contained in PBU, isopropanol, was previously reported to be ineffective in the

presence of HEMA⁴²; however, PBU is a HEMA-free adhesive. Therefore, given the similar vapor pressure of isopropanol and ethanol,²² they are likely to evaporate similarly. Last, the presence of PENTA (dipentaerythritol pentaacrylate monophosphate) could attribute to the DC, because its five double bonds make it an effective cross-linker.⁴³

HEMA-free adhesives are usually prone to phase separation⁴⁴; however, no droplets were observed within the adhesive layer of PBU using SEM (Figure 1). This is in accordance with the manufacturer's information, which claims that the bisacrylamide monomer is capable of keeping the hydrophobic monomers in solution, just like HEMA. In contrast to PBU, another HEMA-free universal adhesive, GPB, showed phase separation very clearly (Figure 1). Its bond strength was lower than those of PBU, UBQ-A, and UBQ, which is in agreement with a previous study, where GPB underperformed UBQ and Prime&Bond Active.¹² After 24 hours, the difference was significant compared with UBQ according to ANOVA, although not according to Weibull analysis. Both the analyses showed significant differences between GPB and all other adhesives after TC, which significantly reduced the μ TBS of GPB. We speculate that lower durability of GPB may be attributed to the presence of 4-MET, which forms less stable calcium salts than 10-MDP⁴⁵ and is more susceptible to aging by TC.⁴⁶ The purity of 10-MDP was also proved to affect the bond strengths and stability on aging.⁴⁷

Given the content of 4-MET, which is relatively hydrophobic due to its aromatic ring,⁴³ GPB is solvated by acetone. Acetone-based adhesives were reported to retain less water and solvents than ethanol-based ones,³⁸ which could favorably affect the DC of GPB. Moreover, GPB contains a diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide photoinitiator (known as TPO), which is very effective.²⁵ However, for the aforementioned reasons, the DC of GPB applied to dentin could not be evaluated in this study. In our previous study, where GPB was applied directly on the ATR crystal and the 1608 cm^{-1} peak was detected, its DC was $76.3 \pm 3.9\%$, which is comparable to the results of PBU and UBQ in this study. In contrast, another recent study reported that the DC of GPB ($67.2 \pm 3.8\%$) was the

Figure 3. Correlation graphs of micro-tensile bond strength and degree of conversion. Mean μ TBSs of each tooth associated with DC values of PBU, EB, UBQ, and UBQ-A are shown in the scatter plot. GPB is not included because its DC could not be determined. The continuous line visualizes the correlation of DC and μ TBS after 24 hours and the dashed line after TC. It can be noted that the correlation after TC is stronger than after 24 hours. Abbreviations: PBU: Prime&Bond universal, EB: Ecosite Bond, UBQ: Clearfil Universal Bond Quick, UBQ-A: Clearfil Universal Bond Quick without amide monomer, μ TBS: Micro tensile bond strength, DC: Degree of conversion.

lowest among six universal adhesives,²⁷ but it must be noted that a light-curing unit emitting light only in the blue region (440-480 nm) was used in that study²⁷ and was ineffective for the activation of TPO²³⁻²⁵ contained in GPB.

EB was included in the study because of its extremely low acidity (pH=4.5 according to the safety data sheet⁴⁸), which is much milder than the other tested adhesives (Table 1). However, the manufacturer did not provide details about the composition except for the fact that it is 10-MDP based, which hinders the discussion. After 24 hours, ANOVA did not show any significant difference between EB and GPB, whereas Weibull analysis found the bond strength of EB to be significantly lower. The differences to other adhesives after 24 hours were significant according to both the statistical analyses. After TC, which decreased the μ TBS of EB significantly, the bond strengths of EB were significantly lower compared with all other tested adhesives. The huge SD and low Weibull modulus of EB also imply its low reliability. Given the limited information, we assume that the pH was insufficient to interact with the smear layer and dentin, resulting in low bond strengths and failures at the dentin-adhesive interface. It must be noted that the insufficiency of EB's etching effect would be even more pronounced on bur-cut dentin.³⁵ The DC of EB was significantly lower than that of the other adhesives, which could also contribute to its low bond strengths.^{20,21} The considerable decrease in the μ TBS of EB after TC could be a consequence of increased water permeability due to low DC^{18,19} and HEMA content.⁴⁸

CONCLUSION

This study revealed that there is a significant positive correlation between μ TBS and DC. The correlation was more marked after TC, which suggests that DC may be an important factor for long-term bond stability.

Acknowledgements

This work was supported by the Ministry of Education, Culture, Sports, Science and Technology of Japan (Grants 18K09571 and 19K10106) and Charles University (Project PROGRES Q29/1LF).

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Human Research Ethics Committee of Tokyo Medical and Dental University. The approval code issued for this study is 2014-022.

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 27 November 2019)

REFERENCES

1. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, & Van Landuyt KL (2011) State of the art of self-etch adhesives *Dental Materials* **27**(1) 17-28.
2. Tay FR & Pashley DH (2003) Have dentin adhesives become too hydrophilic *Journal of the Canadian Dental Association* **69**(11) 726-731.
3. Pucci CR, Gu L-S, Zhang H-y, Song Q, Xia VW, Davis LB, de Souza Andrade D, Mazzoni A, Breschi L, Pashley DH, Tay FR, & Niu L-N (2018) Water-associated attributes in the contemporary dentin bonding milieu *Journal of Dentistry* **74** 79-89.
4. Tay FR, Pashley DH, Suh B, Carvalho R, & Miller M (2004) Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part I. Bond strength and morphologic evidence *American Journal of Dentistry* **17**(4) 271-278.
5. Tay FR & Pashley DH (2003) Water treeing: a potential mechanism for degradation of dentin adhesives *American Journal of Dentistry* **16**(1) 6-12.
6. Nishitani Y, Yoshiyama M, Hosaka K, Tagami J, Donnelly A, Carrilho M, Tay FR, & Pashley DH (2007) Use of Hoy's solubility parameters to predict water sorption/solubility of experimental primers and adhesives *European Journal of Oral Sciences* **115**(1) 81-86.
7. Ito S, Hashimoto M, Wadgaonkar B, Svizero N, Carvalho RM, Yiu C, Rueggeberg FA, Foulger S, Saito T, Nishitani Y, Yoshiyama M, Tay FR, & Pashley DH (2005) Effects of resin hydrophilicity on water sorption and changes in modulus of elasticity *Biomaterials* **26**(33) 6449-6459.
8. Van Landuyt KL, Snauwaert J, Peumans M, De Munck J, Lambrechts P, & Van Meerbeek B (2008) The role of HEMA in one-step self-etch adhesives *Dental Materials* **24**(10) 1412-1419.
9. Hosaka K, Nakajima M, Takahashi M, Itoh S, Ikeda M, Tagami J, & Pashley DH (2010) Relationship between mechanical properties of one-step self-etch adhesives and water sorption *Dental Materials* **26**(4) 360-367.
10. Yiu CKY, King NM, Pashley DH, Suh BI, Carvalho RM, Carrilho MRO, & Tay FR (2004) Effect of resin hydrophilicity and water storage on resin strength *Biomaterials* **25**(26) 5789-5796.
11. Kuno Y, Hosaka K, Nakajima M, Ikeda M, Klein Junior CA, Foxton RM, & Tagami J (2019) Incorporation of a hydrophilic amide monomer into a one-step self-etch adhesive to increase dentin bond strength: effect of application time *Dental Materials Journal* **38**(6) 892-899.
12. Ahmed MH, De Munck J, Van Landuyt K, Peumans M, Yoshihara K, & Van Meerbeek B (2019) Do universal adhesives benefit from an extra bonding layer *Journal of Adhesive Dentistry* **21**(2) 117-132.

13. Salz U, Zimmermann J, Zeuner F, & Moszner N (2005) Hydrolytic stability of self-etching adhesive systems *Journal of Adhesive Dentistry* **7**(2) 107-116.
14. Nunes TG, Ceballos L, Osorio R, & Toledano M (2005) Spatially resolved photopolymerization kinetics and oxygen inhibition in dental adhesives *Biomaterials* **26**(14) 1809-1817.
15. Cadenaro M, Breschi L, Antonioli F, Navarra CO, Mazzoni A, Tay FR, Di Lenarda R, & Pashley DH (2008) Degree of conversion of resin blends in relation to ethanol content and hydrophilicity *Dental Materials* **24**(9) 1194-1200.
16. Paul SJ, Leach M, Rueggeberg FA, & Pashley DH (1999) Effect of water content on the physical properties of model dentine primer and bonding resins *Journal of Dentistry* **27**(3) 209-214.
17. Ikeda T, De Munck J, Shirai K, Hikita K, Inoue S, Sano H, Lambrechts P, & Van Meerbeek B (2005) Effect of evaporation of primer components on ultimate tensile strengths of primer-adhesive mixture *Dental Materials* **21**(11) 1051-1058.
18. Cadenaro M, Antonioli F, Sauro S, Tay FR, Di Lenarda R, Prati C, Biasotto M, Contardo L, & Breschi L (2005) Degree of conversion and permeability of dental adhesives *European Journal of Oral Sciences* **113**(6) 525-530.
19. Breschi L, Cadenaro M, Antonioli F, Sauro S, Biasotto M, Prati C, Tay FR, & Di Lenarda R (2007) Polymerization kinetics of dental adhesives cured with LED: correlation between extent of conversion and permeability *Dental Materials* **23**(9) 1066-1072.
20. Sadr A, Shimada Y, & Tagami J (2007) Effects of solvent drying time on micro-shear bond strength and mechanical properties of two self-etching adhesive systems *Dental Materials* **23**(9) 1114-1119.
21. Sato K, Hosaka K, Takahashi M, Ikeda M, Tian F, Komada W, Nakajima M, Foxton R, Nishitani Y, Pashley DH, & Tagami J (2017) Dentin bonding durability of two-step self-etch adhesives with improved degree of conversion of adhesive resins *Journal of Adhesive Dentistry* **19**(1) 31-37.
22. Ekambaram M, Yiu CKY, & Matinlinna JP (2015) An overview of solvents in resin-dentin bonding *International Journal of Adhesion and Adhesives* **57** 22-33.
23. Ilie N & Hickel R (2008) Can CQ be completely replaced by alternative initiators in dental adhesives? *Dental Materials Journal* **27**(2) 221-228.
24. Cadenaro M, Antonioli F, Codan B, Agee K, Tay FR, Dorigo Ede S, Pashley DH, & Breschi L (2010) Influence of different initiators on the degree of conversion of experimental adhesive blends in relation to their hydrophilicity and solvent content *Dental Materials* **26**(4) 288-294.
25. Pongprueksa P, Miletic V, Janssens H, Van Landuyt KL, De Munck J, Godderis L, & Van Meerbeek B (2014) Degree of conversion and monomer elution of CQ/amine and TPO adhesives *Dental Materials* **30**(6) 695-701.
26. Borges B, Souza-Junior E, Brandt W, Loguercio A, Montes M, Puppini-Rontani R, & Sinhoreti M (2012) Degree of conversion of simplified contemporary adhesive systems as influenced by extended air-activated or passive solvent volatilization modes *Operative Dentistry* **37**(3) 246-252.
27. Papadogiannis D, Dimitriadi M, Zafiropoulou M, Gaintantzopoulou MD, & Eliades G (2019) Universal adhesives: setting characteristics and reactivity with dentin *Materials (Basel)* **12**(10) E1720.
28. Tichy A, Hosaka K, Bradna P, Ikeda M, Abdou A, Nakajima M, & Tagami J (2019) Subsequent application of bonding agents to a one-step self-etch adhesive: its effect with/without previous light-curing *Dental Materials* **35**(12) e299-e309.
29. Hanabusa M, Yoshihara K, Yoshida Y, Okihara T, Yamamoto T, Momoi Y, & Van Meerbeek B (2016) Interference of functional monomers with polymerization efficiency of adhesives *European Journal of Oral Sciences* **124**(2) 204-209.
30. Andrew Chan KL & Kazarian SG (2016) Attenuated total reflection Fourier-transform infrared (ATR-FTIR) imaging of tissues and live cells *Chemical Society Reviews* **45**(7) 1850-1864.
31. Stansbury JW & Dickens SH (2001) Determination of double bond conversion in dental resins by near infrared spectroscopy *Dental Materials* **17**(1) 71-79.
32. Gauthier MA, Stangel I, Ellis TH, & Zhu XX (2005) A new method for quantifying the intensity of the C=C band of dimethacrylate dental monomers in their FTIR and Raman spectra *Biomaterials* **26**(33) 6440-6448.
33. Sideridou I, Tserki V, & Papanastasiou G (2002) Effect of chemical structure on degree of conversion in light-cured dimethacrylate-based dental resins *Biomaterials* **23**(8) 1819-1829.
34. Huang XQ, Pucci CR, Luo T, Breschi L, Pashley DH, Niu LN, & Tay FR (2017) No-waiting dentine self-etch concept: merit or hype *Journal of Dentistry* **62** 54-63.
35. Saikaew P, Chowdhury AF, 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.
36. Saikaew P, Matsumoto M, Chowdhury A, 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.
37. Nihi FM, Fabre HS, Garcia G, Fernandes KB, Ferreira FB, & Wang L (2009) In vitro assessment of solvent evaporation from commercial adhesive systems compared to experimental systems *Brazilian Dental Journal* **20**(5) 396-402.
38. Yiu CK, Pashley EL, Hiraishi N, King NM, Goracci C, Ferrari M, Carvalho RM, Pashley DH, & Tay FR (2005) Solvent and water retention in dental adhesive blends after evaporation *Biomaterials* **26**(34) 6863-6872.
39. Nishiyama N, Suzuki K, Yoshida H, Teshima H, & Nemoto K (2004) Hydrolytic stability of methacrylamide in acidic aqueous solution *Biomaterials* **25**(6) 965-969.
40. Loguercio AD, Loeblein F, Cherobin T, Ogliari F, Piva E, & Reis A (2009) Effect of solvent removal on adhesive

- properties of simplified etch-and-rinse systems and on bond strengths to dry and wet dentin *Journal of Adhesive Dentistry* **11**(3) 213-219.
41. Zheng L, Pereira PN, Nakajima M, Sano H, & Tagami J (2001) Relationship between adhesive thickness and microtensile bond strength *Operative Dentistry* **26**(1) 97-104.
 42. Carvalho RM, Mendonca JS, Santiago SL, Silveira RR, Garcia FC, Tay FR, & Pashley DH (2003) Effects of HEMA/solvent combinations on bond strength to dentin *Journal of Dental Research* **82**(8) 597-601.
 43. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, & Van Meerbeek B (2007) Systematic review of the chemical composition of contemporary dental adhesives *Biomaterials* **28**(26) 3757-3785.
 44. 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.
 45. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, Inoue S, Tagawa Y, Suzuki K, De Munck J, & Van Meerbeek B (2004) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **83**(6) 454-458.
 46. Inoue S, Koshiro K, Yoshida Y, De Munck J, Nagakane K, Suzuki K, Sano H, & Van Meerbeek B (2005) Hydrolytic stability of self-etch adhesives bonded to dentin *Journal of Dental Research* **84**(12) 1160-1164.
 47. Yoshihara K, Nagaoka N, Okihara T, Kuroboshi M, Hayakawa S, Maruo Y, Nishigawa G, De Munck J, Yoshida Y, & Van Meerbeek B (2015) Functional monomer impurity affects adhesive performance *Dental Materials* **31**(12) 1493-1501.
 48. DMG (2018) Ecosite Bond SDS. Retrieved online February 12, 2019 from: https://www.dmg-america.com/fileadmin/DMG_America/SDS/Ecosite_Bulk_Fill_Set_SDS_04-2018.pdf

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 <https://meridian.allenpress.com/operative-dentistry> or enter the provided address into you address bar.

Post-gel and Total Shrinkage Stress of Conventional and Bulk-fill Resin Composites in Endodontically-treated Molars

RA da Silva Pereira • GF de Bragança • ABF Vilela • RA de Deus • RR Miranda • C Veríssimo • CJ Soares
Clinical Relevance: The clinician should consider the polymerization shrinkage stress when selecting a composite resin for posterior restorations. The use of post-gel shrinkage values should guide the selection of a composite resin for posterior teeth.

<https://doi.org/10.2341/19-187-L>

Tridimensional Evaluation of the Interfacial Gap in Deep Cervical Margin Restorations: A Micro-CT Study

N Scotti • A Baldi • EA Vergano • R Michelotto Tempesta • M Alovise • D Pasqualini • GC Carpegna • A Comba
Clinical Relevance: Using a material that optimizes marginal seal when using a margin elevation technique to manage deep class II cavities should enhance clinical outcomes.

<https://doi.org/10.2341/19-161-L>

Does the Type of Solvent in Dental Adhesives Influence the Clinical Performance of Composite Restorations Placed in Noncarious Cervical Lesions? A Systematic Review and Meta-analysis

RBE Lins • M Sebold • MB Magno • LC Maia • LRM Martins • M Giannini

Clinical Relevance: According to the clinical and scientific evidence presented in this systematic review and meta-analysis, dental adhesives containing either organic solvent (acetone or alcohol) can be used to achieve similar clinical performance and longevity of composite restorations.

<https://doi.org/10.2341/19-124-LIT>

A New Dual-cure Universal Simplified Adhesive: 18-month Randomized Multicenter Clinical Trial

E Gomes de Albuquerque • F Warol • F Signorelli Calazans • L Augusto Poubel • S Soares Marins • T Matos • JJ de Souza • A Reis • M de Oliveira Barceiro • AD Loguercio

Clinical Relevance: Non-carious cervical lesion restorations using a dual-cure universal adhesive in self-etch and etch-and-rinse mode showed satisfactory clinical performance after 18 months.

<https://doi.org/10.2341/19-144-C>

Influence of Silver Diamine Fluoride Treatment on the Microtensile Bond Strength of Glass Ionomer Cement to Sound and Carious Dentin

M Jiang • ML Mei • MCM Wong • CH Chu • ECM Lo

Clinical Relevance: This study provides valuable information about the influence of silver diamine fluoride (SDF) treatment on the microtensile bond strength of glass ionomer cement (GIC) to dentin.

<https://doi.org/10.2341/19-237-L>

Post-gel and Total Shrinkage Stress of Conventional and Bulk-fill Resin Composites in Endodontically-treated Molars

RA da Silva Pereira • GF de Bragança • ABF Vilela
RA de Deus • RR Miranda • C Veríssimo • CJ Soares

Clinical Relevance

The clinician should consider the polymerization shrinkage stress when selecting a composite resin for posterior restorations. The use of post-gel shrinkage values should guide the selection of a composite resin for posterior teeth.

SUMMARY

Objectives: The objective of this study was to evaluate the effect of the method used for calculation of polymerization shrinkage, total or post-gel, on the shrinkage stress of conventional and bulk-fill composite resins for restoring endodontically treated teeth using finite element analysis.

Renata Afonso da Silva Pereira, DDS, MSc, PhD, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil

Gabriel Felipe de Bragança, DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil

Andomar Bruno Fernandes Vilela, DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil

Raíssa Albuquerque de Deus, DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil

Methods and Materials: Four composite resins were tested for post-gel shrinkage (P-Shr) by the strain-gauge test and total shrinkage (T-Shr) using an optical method (n=10). Two conventional composite resins, Filtek Z350 XT (3M-ESPE; Z350) and TPH3 Spectrum (Dentsply; TPH3) and two bulk-fill composite resins, Filtek Bulk-Fill Posterior (3M-ESPE; POST) SureFil SDR flow (Dentsply; SDR) were tested. Elastic modulus (E), diametral tensile strength

Raíssa Ramos Miranda, DDS, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil

Crisnicaw Veríssimo, DDS, MSc, PhD, professor, Department of Operative Dentistry, School of Dentistry, Federal University of Goiás, Goiás, Brazil

*Carlos José Soares, DDS, MSc, PhD, professor and chair, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil

*Corresponding author: Avenida Pará, 1720, Bloco 4LA, sala 42, Campos Umuarama, Uberlândia, Minas Gerais 38400-902, Brazil; e-mail: carlosjsoares@ufu.br

<https://doi.org/10.2341/19-187-L>

(DTS), and compressive strength (CS) were also determined ($n=10$). The residual shrinkage stress was evaluated by finite element analysis with four restorative techniques: incremental with Z350 and TPH3; SDR/TPH3 (two bulk increments of 4 mm and two occlusal increments); and two bulk increments of 5 mm for POST. Data for P-Shr, T-Shr, E, DTS, and CS were analyzed by analysis of variance and Tukey's test ($\alpha=0.05$), and residual shrinkage was analyzed quantitatively and qualitatively by the modified von Mises criteria.

Results: SDR had the lowest CS values, POST and TPH3 had similar and intermediate values, and Z350 had the highest CS. TPH3 and Z350 had similar DTS values and values higher than SDR. Z350 and POST had higher P-Shr, and SDR had lower T-Shr. T-Shr resulted in higher shrinkage stress than P-Shr values. SDR/TPH3 resulted in higher shrinkage stress when using T-Shr and lower values when using the P-Shr value.

Conclusion: T-Shr resulted in higher stress in the enamel and in root dentin close to the pulp chamber than P-Shr values. The selection of the T-Shr or P-Shr changed the ranking of the shrinkage stress of the tested composite resin.

INTRODUCTION

Composite resin materials are widely used in restorative dentistry for restoring posterior vital and endodontically-treated teeth.^{1,2} During the polymerization reaction, resin undergoes a transition from a predominantly viscous state to a basically solid state due to a conversion of monomer molecules in polymer chains.^{3,4} Volumetric shrinkage is an inherent characteristic of resin-based materials.^{5,6} Several factors, such as mechanical properties, chemical composition, and the remaining tooth structure, determine the shrinkage stress,^{4,7-9} restorative technique, and curing method.¹⁰ This residual stress may result in propagation of enamel cracks, cuspal deflection,^{1,11,12} marginal loss, secondary caries, and postoperative sensitivity.⁷

Various restorative protocols, such as the incremental technique,¹³ gradual polymerization,¹⁴ and larger increments,^{4,8} have been advocated to reduce the shrinkage stress. Recent trends are focused on modifications of the resin matrix, mainly for developing materials with reduced polymerization shrinkage and consequently the shrinkage stress.¹⁵ Bulk-fill composite resins allow application of increments

from 4- to 5-mm thickness,^{1,16,17} with a high degree of conversion in all increments, reduced shrinkage, and shrinkage stress. Also, they are commercially available in low or high viscosity.^{12,18} The viscosity and composition of the bulk-fill composite resin may interfere with the performance of the material during shrinkage measurements.^{1,19} Bulk-fill composite resins have shown better or similar performance to the conventional materials in clinical trials and laboratory studies in terms of volumetric shrinkage, polymerization stress, cuspal deflection, and marginal quality.²⁰

When the material is light activated, the polymerization process develops, and the volumetric reduction causes shrinkage of the material.⁶ However, the relationship between shrinkage and stress is not that simple because not all shrinkage causes stress.^{6,19,21} Residual stresses will only be generated when the composite material can no longer relax in a timely manner; not all polymerization shrinkage causes shrinkage stresses.²¹ It is important to distinguish between total shrinkage and the shrinkage that truly causes stresses, called the "post-gel shrinkage."^{6,22} However, the manufacturers continue to inform the clinicians and propagate information regarding different materials using total shrinkage values. This information could be used for creating artificial benefit regarding specific composite resins that can present different rankings regarding total and post-gel shrinkage and total shrinkage reported values.

Shrinkage stresses cannot be measured directly; the use of finite element analysis (FEA) as a computational numerical analysis is considered the most comprehensive method to calculate the complex condition of the stress inside materials and structures.²³ Therefore, the aim of this study was to evaluate the effect of the method used for calculation of polymerization shrinkage on the shrinkage stress of conventional and bulk-fill composite resins for restoring endodontically-treated teeth. The null hypothesis was that there would be no difference in residual shrinkage stress when using the total shrinkage (T-Shr) or post-gel shrinkage (P-Shr) values.

METHODS AND MATERIALS

Study Design

Four commercial materials were used in this study, including two conventional composite resins, TPH3 Spectrum (Dentsply, Konstanz, Germany; TPH3) and Filtek Z350XT (3M ESPE, St Paul, MN, USA;

Table 1: Composition of Resin Composites (Manufacturer Information)

Material	CODE	Shade	Composite Resin Type	Increment Size and Light Activation Time	Organic Matrix	Filler	Filler % wt/vol	Manufacturer
TPH3 Spectrum	TPH3	A2	Nanohybrid	2.0 mm, 20 s	BisGMA and BisEMA Dimethacrylate	Boro-silicate/ aluminum/barium glass and silica	75.5/57.1	Dentsply
Filtek Z350 XT	Z350	A2	Nanofilled	2.0 mm, 20 s	BisGMA, BisEMA, UDMA, TEGDMA	Silica and zirconia nanofillers, agglomerated zirconia-silica nanoclusters	82/60	3M-ESPE
Surefil SDR flow	SDR	A2	Bulk-fill flowable	4.0 mm, 40 s	UDMA, BISGMA, EBPADMA, Procrylat resin	Silane treated ceramic and YbF3	68/44	Dentsply
Filtek Bulk-fill posterior	POST	A1	Bulk-fill regular paste	5.0 mm, 10 s occlusal, 10 s buccal, and 10 s palatal	AUDMA, UDDMA, UDMA, EDMAB	Silica, zirconia and YbF3	76.5/58.4	3M-ESPE
Abbreviations: BisEMA, bisphenol A polyethylene glycol diether dimethacrylate; BisGMA, bisphenol A diglycidylmethacrylate; EBPADMA, ethoxylated bisphenol A dimethacrylate; EDMAB, ethyl-4-dimethylaminobenzoate; TEGDMA, triethyleneglycol dimethacrylate; UDDDMA, dodecanediol dimethacrylate; UDMA, urethane dimethacrylate; YbF3: ytterbium fluoride.								

Z350), and two bulk-fill composite resins, the flowable viscosity Bulk Fill Surefil SDR Flow (Dentsply; SDR) and the regular viscosity Filtek Bulk Fill Posterior (3M ESPE; POST). The composition was described according to manufacturer's information (Table 1). All resin composites were tested for P-Shr and T-Shr. Shrinkage stresses were analyzed using FEA.

P-Shr

P-Shr was determined for all tested composite resins (n=10) using the strain-gauge method.²² The materials were shaped as a dome with 2.8 ± 0.4 mm in diameter and 1.7 ± 0.2 mm in height on top of a biaxial strain gauge (CEA-06-032WT-120, Measurements Group, Raleigh, NC, USA) that measured shrinkage strains in two perpendicular directions (x and y axes). A strain conditioner (ADS2000, Lynx, Tecnologia Eletrônica, São Paulo, Brazil) converted electrical resistance changes in the strain gauge to voltage changes through a quarter-bridge circuit with an internal reference resistance. The strain values measured along the two axes were averaged because the material properties were homogeneous and isotropic on a macro scale. All materials were light-cured using a light-emitting diode (LED) unit Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein) with an intensity of 1600 mW/cm^2 checked by a using Marc Resin Calibrator (BlueLight, Halifax, Canada) with the light tip held at a 1-mm distance from the surface of the composite and monitored for 10 minutes. The mean shrinkage strain was used as

linear P-Shr shrinkage input for the FEA and could be converted to the volumetric percentage by multiplying by 3 and 100%.

T-Shr

T-Shr was calculated using the optical method.⁵ Each resin sample (n=10) was shaped as a dome with 2.8 ± 0.4 mm in diameter and 1.7 ± 0.2 mm in height on a platform made of addition silicone (Express XT-Light body, 3M ESPE) in a blue color, for a better contrast between resin and surface. Each sample was slightly rounded and placed on the silicone platform. The platform was placed under a microscope coupled to a camera (SZX16 & UC30, Olympus, Tokyo, Japan) so that the images could be captured in a standardized way. Once positioned, a photograph was taken before photoactivation. The light from the microscope was attached only to capture the images to avoid premature polymerization of the compound through the optical microscope. Immediately after the first photograph and before polymerization of the material, the sample was light-cured for 40 seconds using the Bluephase G2 (Ivoclar Vivadent). After polymerization, the samples were monitored, and photographs were taken after 10 and 60 minutes of the initial light activation. All procedures were performed at room temperature (22°C). ImageJ software (National Institutes of Health, Bethesda, MD, USA), available free of charge on the internet, was used to evaluate pre- and post-polymerization photos. The brightness of the image was adjusted to determine the maximum contrast between the resin sample and the silicone

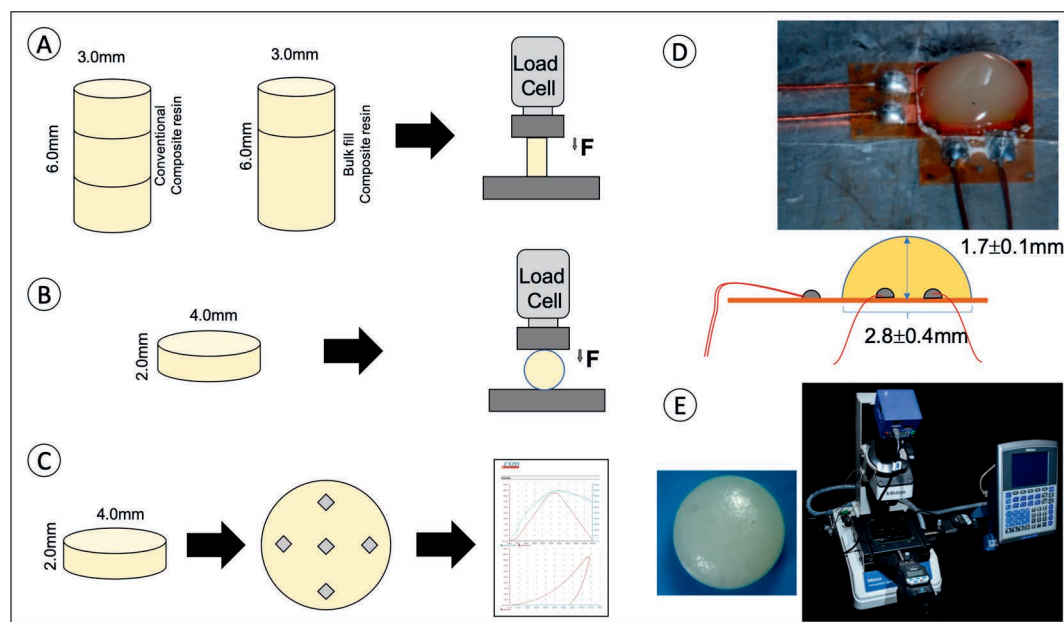


Figure 1. Schematic diagram of sample preparation and measurement methodology of mechanical properties recorded experimentally; (A) CS, (B) DTS, (C) E, (D) P-Shr, (E) T-Shr.

plate. Using the tool “wand tool,” the contour of the sample was defined to obtain the total area. Through the photograph of the total area of the samples before and after polymerization, it was possible to calculate the volume change before and after polymerization.

Compressive Strength and Diametral Tensile Strength

Compressive strength (CS) and diametral tensile strength (DTS; $n=10$) of the composite resins were calculated (Figure 1A and B, respectively). For characterization of the mechanical properties, the sample preparation for CS and DTS was performed using the maximum thickness recommended for each material and did not represent exactly the same protocol simulated in FEA. The composite resin was inserted into a cylindrical polytetrafluoroethylene mold for the CS test (6 mm height, 3 mm diameter) or the DTS test (2 mm height, 4 mm diameter). The specimens for the CS test made with bulk-fill composites were polymerized with 4.0 mm for the first increment and 2.0 mm for second increment. For conventional composites, the specimens were polymerized in three 2.0-mm increments using Bluephase G2 (Ivoclar Vivadent). The specimens were stored in distilled water for 24 hours at 37°C and afterward were submitted to CS and DTS testing in a universal testing machine (DL2000, EMIC) at a crosshead speed of 0.5 mm/min until

failure occurred. CS values were calculated by dividing the fracture load (F) by the cross-sectional area and converting it into MPa. DTS values were calculated using the following equation: $DTS = 2F / \pi dt$, where d is the specimen diameter, and t is the height of the specimen. DTS and CS values were converted into MPa.

Elastic Modulus

For elastic modulus (E) measurements, disc-shaped specimens similar to DTS samples (2 mm height, 4 mm diameter) were prepared.¹ Using a Vickers indenter (CSM Micro-Hardness Tester, CSM Instruments, Peseux, Switzerland), indentations were made at the surface of the finished and polished composite resin surface. The indentations were carried out with controlled force, whereby the test load was increased or decreased at a constant speed ranging between 0 and 500 mN in 20-second intervals. The maximum force of 500 mN was held for five seconds. The load and the penetration depth of the indenter were continuously measured during the load-unload cycle. The universal hardness was defined as the applied force divided by the apparent area of the indentation at the maximum force. The indentation modulus, comparable to the material's E, was calculated from the slope of the tangent of the indentation depth curve at the maximum force.

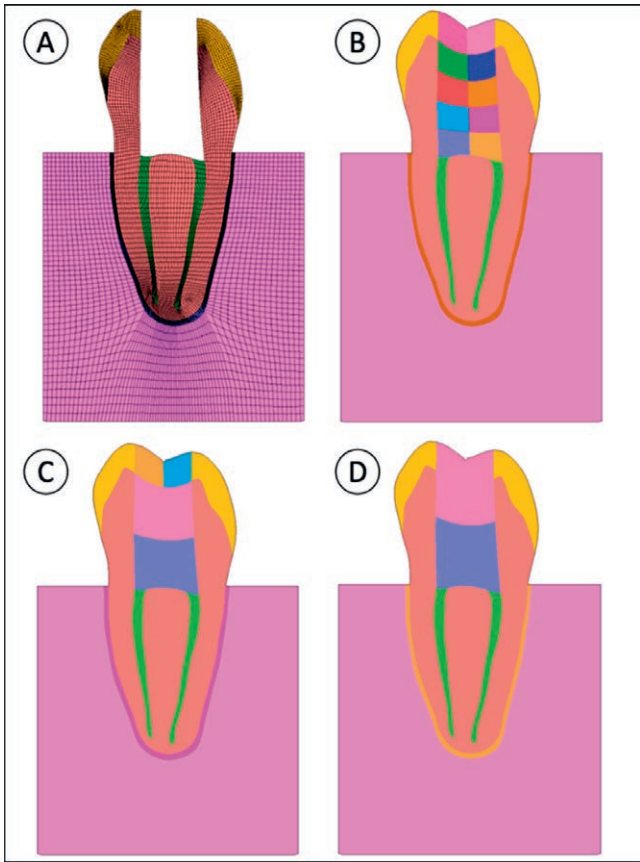


Figure 2. Two-dimensional FEA model generation. (A) Principal model: mesh of endodontically treated tooth with a MOD preparation. (B) Model simulating incremental filling technique using Z350 or TPH3, resulting in 10 increments. (C) Model simulating SDR flowable bulk fill used for restoring the dentin (two increments of 4.0 mm) and two occlusal increments of TPH3 replacing the enamel (2.0 mm), resulting in four increments. (D) Model simulating POST replacing the dentin and enamel (two increments of 5.0 mm), resulting in two increments.

Residual Stress Calculation: FEA

To calculate T-Shr and P-Shr residual shrinkage stresses, a two-dimensional (2D) finite element simulation was carried out simulating a mesial-occlusal-distal restoration of an endodontically-treated molar as described in a prior study.² The geometric model (Figure 2A) was based on a digitized buccolingual cross section of an endodontically-treated molar embedded in an acrylic resin cylinder

with a simulated periodontal ligament (PDL). The coordinates of points were obtained using public domain ImageJ software (ImageJ 1.48, National Institutes of Health). The mechanical properties used were as follows: enamel, $E = 84 \text{ GPa}$ and Poisson's ratio (η) = 0.30²⁴; dentin, $E = 18 \text{ GPa}$ and $\eta = 0.23$ ²³; polystyrene resin, $E = 13.5 \text{ GPa}$ and $\eta = 0.31$; and polyether, $E = 0.05$ and $\eta = 0.45$.²⁴ For the restorative composite resins, the E was experimentally determined and is shown in Table 2. The Poisson's ratio was chosen to be the same for all composites at 0.24.²³ It was assumed a plane stress condition for the composites and a plane strain condition for the tooth cross sections, PDL, and acrylic resin cylinder.

The FEA was performed using MSC.Mentat (preprocessor and postprocessor) and MSC.Marc (solver) software (MSC Software, Santa Ana, CA, USA). One FEA model was generated for each of the four restorative protocols of the experimental study (Figure 2A). The Z350 and TPH3 restorations were built and cured in six increments (Figure 2B and C, respectively). The bulk-fill flowable, SDR, was placed in two increments up to 4 mm and covered with TPH3 built with two increments of 2 mm thickness on the occlusal surface (Figure 2D). The bulk-fill regular paste, POST, was used in two increments of up to 5.0 mm (Figure 2D). Polymerization shrinkage was simulated by thermal analogy. The temperature was reduced by 1°C, and the linear shrinkage value P-Shr or T-Shr was entered as the coefficient of linear thermal expansion. Nodal displacements were the constraint in the x and y directions at the bottom and lateral surfaces of the support cylinder. Modified von Mises equivalent stress (mvm) was used to express the stress conditions, using the ratio of the CS and DTS strengths. The CS and DTS used for calculating the ratios for the restorative materials are shown in Table 2 and were calculated using the experimentally determined CS and DTS values. The CS and DTS strengths for enamel were 384.0 and 10.3 MPa and for dentin were 297.0 and 98.7 MPa, respectively.²³ The mean values of the 10% highest mvm stresses were determined for the enamel and

Table 2: Experimentally Determined Mean (and SD) Volumetric P-Shr, volumetric T-Shr, CS, DTS, and E. ^a					
Composite Resin	P-Shr (%)	T-Shr (%)	CS (MPa)	DTS (MPa)	E (GPa)
Filtek Z350 XT (Z350)	0.7 ± 0.1 c	2.6 ± 0.2 B	255.5 ± 30.4 A	47.5 ± 6.5 A	18.5 ± 2.0 A
TPH3 Spectrum (TPH3)	0.5 ± 0.0 B	2.5 ± 1.3 AB	164.4 ± 25.3 B	49.9 ± 7.2 A	15.6 ± 1.4 B
Filtek Bulk Fill Posterior (POST)	0.7 ± 0.1 c	2.1 ± 0.3 A	169.3 ± 15.8 B	42.4 ± 6.3 AB	12.8 ± 1.7 BC
Surefil SDR flow (SDR)	0.4 ± 0.1 A	3.9 ± 0.8 c	121.6 ± 13.4 c	39.4 ± 5.7 B	10.1 ± 0.9 c

^a Different letters mean significant difference between composite resins for each mechanical property provided by Tukey test ($p < 0.05$).

dentin structures when simulating the T-Shr and P-Shr. Fortran custom-made subroutines were generated for obtaining mvm stresses values from enamel-composite and dentin-composite interfaces.

Statistical Analysis

The P-Shr, T-Shr, E, DTS, and CS data were tested for normal distribution (Shapiro-Wilk) and equality of variances (Levene's test), followed by parametric statistical tests. One-way analysis of variance (ANOVA) was performed for each mechanical property. Multiple comparisons were made using Tukey's test. All tests used an $\alpha=0.05$ significance level, and all analyses were carried out with the statistical package Sigma Plot version 13.1 (Systat Software, Sao Jose, CA, USA). The modified von Mises stresses values were analyzed qualitatively.

RESULTS

P-Shr and T-Shr (%) of Resin Composites

The P-Shr and T-Shr mean values and standard deviations (SDs) of the four composites are shown in Table 2. ANOVA showed a significant difference between the resin composites ($p<0.05$). Tukey's test showed that POST and Z350 had the highest P-Shr values, and SDR and TPH3 had lower P-Shr values (Table 2). SDR showed higher T-Shr values than did other resin composites (Table 2). Z350 and TPH3 had intermediate values, and POST had the lowest T-Shr values.

DTS (MPa), CS (MPa), and E (GPa) of Resin Composites

The DTS, CS, and E mean values and SDs of the four composites are shown in Table 2. ANOVA showed a significant difference between the resin composites ($p<0.05$). Tukey's test showed that Z350 had the highest CS values, and SDR had lower Shr values (Table 2). TPH3 had the highest DTS values, but they were not significantly higher than Z350 or POST, and SDR had the lowest values. Regarding E results, SDR had significantly lower values than Z350 and TPH3; Z350 had higher value than all other resin composites.

Shrinkage Stress (MPa) FEA Results

Shrinkage stresses, expressed by modified von Mises values, generated by all tested restorative techniques are shown in Table 3 and Figures 3 and 4. TPH3 and Z350 groups showed the highest stress values at enamel and dentin structures at T-Shr and P-Shr (Table 3). The models generated with T-Shr

Table 3: The 10% Highest Mean (SD) Values of Modified von Mises Stress (MPa) Extracted From Enamel and Dentin Structures Calculated Using T-Shr and P-Shr of Conventional and Bulk-fill Composite Resins.

Composite Resins	T-Shr		P-Shr	
	Enamel	Dentin	Enamel	Dentin
Z350XT	209.3 (50.9)	53.9 (26.0)	55.3 (13.5)	14.3 (6.9)
TPH	187.2 (46.1)	49.3 (23.8)	38.4 (9.5)	10.1 (4.9)
SDR/TPH	156.1 (15.9)	36.9 (15.1)	23.1 (2.0)	4.3 (1.8)
POST	68.4 (10.8)	23.5 (8.6)	22.5 (3.5)	7.7 (2.8)

values (Figure 3) resulted in a substantial increase of shrinkage stress compared with models with P-Shr values, irrespective of the restorative technique.

T-Shr and P-Shr shrinkage-interface stress values for the different resin techniques are presented in Figure 4. SDR/TPH3 had the highest difference between the stress generated on the enamel-composite and dentin-composite interface when using T-Shr and P-Shr (Figure 4C). The T-Shr values generated elevated peak stress values at the limits of the increments and also at the transition of the tooth structures.

DISCUSSION

The selection of direct restorative material with high performance and durability has led to the development of new materials. The change from the use of the incremental technique to the bulk-filling method may be a result of the simplified restorative technique that reduces the possibility of errors, has fewer operative steps, and requires less clinical

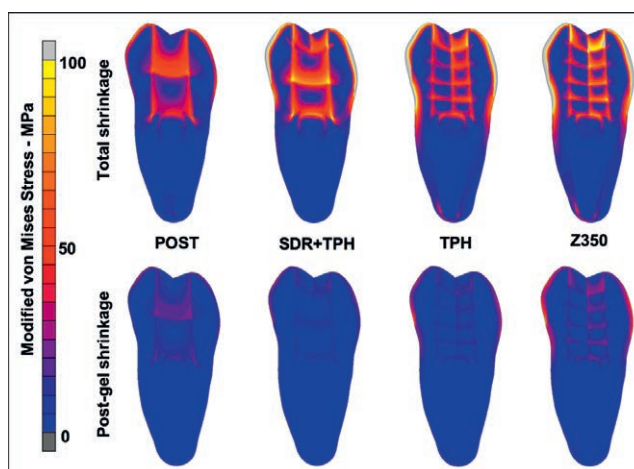


Figure 3. Modified von Mises shrinkage stress (MPa) distribution for the different restorative materials and techniques after polymerization using T-Shr values and P-Shr values.

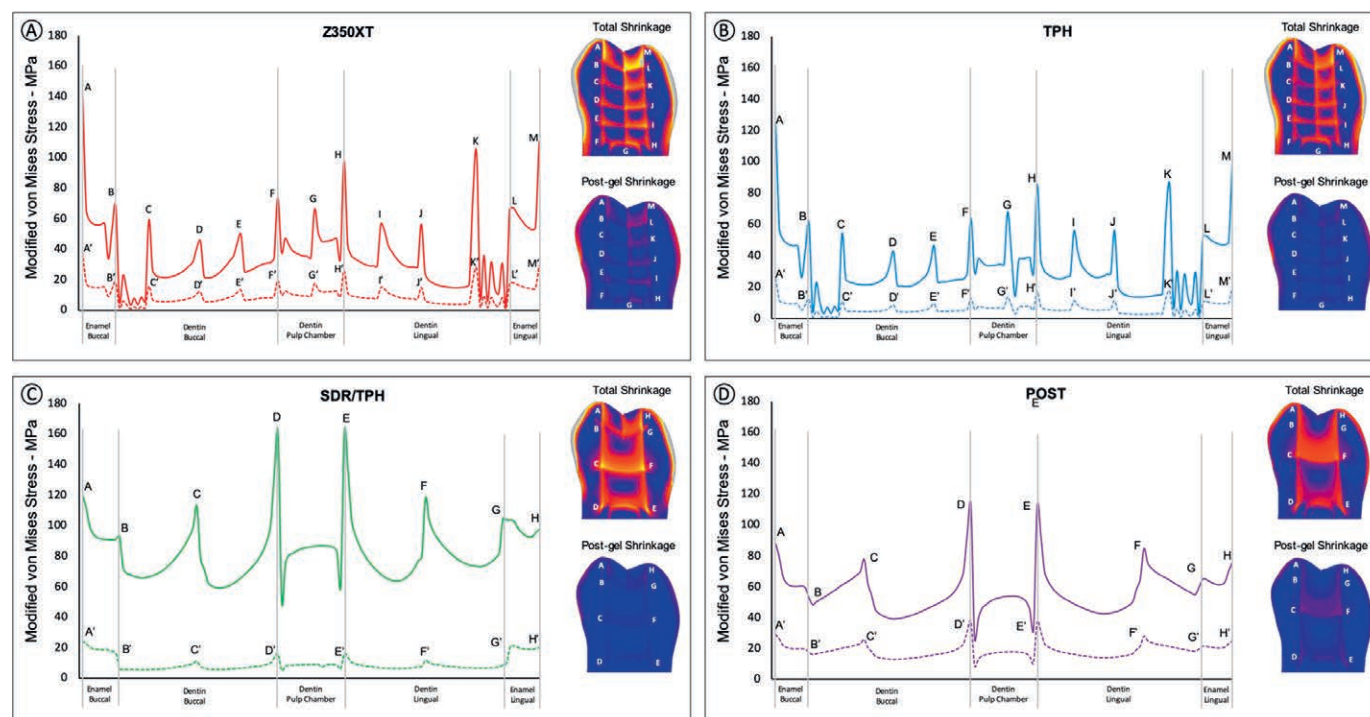


Figure 4. T-Shr and P-Shr shrinkage modified von Mises stresses from composite–enamel and dentin–composite interfaces values for the different resin techniques.

time.¹ The results of this study confirmed that P-Shr and T-Shr were dependent on material composition and that those values influenced residual shrinkage stress; therefore, the null hypothesis was rejected.

Volumetric shrinkage is a consequence of the polymerization of resin-based materials during formation of a polymer network, causing dimensional changes.⁵ It is known that not all polymerization shrinkage causes shrinkage stresses.^{19,21} The total shrinkage measures the entire amount of dimensional change of the composite during polymerization including the pre-gel stage, capable of flowing by releasing part of its tension, and P-Shr stage.^{21,26,27} This fact may explain why the percentage of volumetric total shrinkage of all four tested composites in this study presented values significantly higher than percentage results for P-Shr shrinkage. Residual stresses will only be generated when the composite material cannot timely relax anymore, which happens when a more rigid polymer network structure has developed, occurring only in the P-Shr phase.

The percentage of polymerization shrinkage is influenced by the amount of organic content of the composite²⁸ and the percentage of inorganic content.²⁹ The change of a resin composite to a solid material, characterized by the development of the

elastic modulus, during the polymerization results in rigid restorations and consequently residual shrinkage stresses by the effect of polymerization contraction.¹ The P-Shr of the Z350 composite resin was higher than the others due to the high content of filler in the resin matrix, which increases the modulus of elasticity and rigidity of the material⁸ and tends to increase the shrinkage stress. The higher P-Shr of the POST composite resin can probably be explained by the higher polymerization ratio obtained by the modification on the modulation of the polymerization reaction in deeper areas. The lower post-gel shrinkage value of SDR could be explained by the lower viscosity that results in more flexibility and also by the amount of inorganic filler content, an attribute of flowable composites.³⁰ Additionally, the SDR resin has a polymerization modulator that acts at propagation of the linear and branched chains of the polymers of the resin, reducing the formation velocity of the polymer network, keeping its viscosity longer and providing lower stress compared with traditional composites.³¹ Inversely, SDR presented the highest total shrinkage value, probably due to the smaller amount of filler content. The total volumetric contraction depends on factors such as the size of the charge particles and also the type of the organic matrix and concentration of monomers.³²

The Filtek Bulk Fill Posterior had intermediate E value and the highest P-Shr values; however, the POST showed the lowest modified von Misses shrinkage stress. The smaller number of increments, only two increments of 5 mm, and consequently, only two light activations can explain this finding. The SDR/TPH3 had four increments, and TPH3 and Z350XT had 10 increments. When restoring a tooth, increasing the number of increments results in higher stresses in the remaining tooth structure and at the tooth/restoration interface.^{4,8} The combination of higher elastic modulus of TPH3 and Z350XT and the significantly larger number of increments creates higher stress for both incremental filling techniques. Bulk-filling techniques reduce the increment number by using a larger volume of the composite, which results in lower residual shrinkage stress compared with the incremental filling techniques. The residual stress is known to concentrate at the cervical enamel.^{1,4,33} The lower shrinkage stress observed when flowable composite resin in combination with conventional composite resin may be explained by the lower P-Shr and E values that determine higher stress relief during the polymerization reaction.^{1,3} The stress presented at the root dentin close to the pulp chamber was already expected once the model simulated an endodontically-treated tooth with the pulp chamber totally filled with resin composite. Therefore, the bulk-fill resin composites show up as alternatives to restore endodontically-treated teeth with lower shrinkage stress than the incremental technique, due to the lower stress generated at severely compromised dental structures.³² The highest values of residual shrinkage stress were verified when the total shrinkage values were simulated compared with the post-gel values. The total shrinkage considers the total volumetric shrinkage of the increment, as presented by most manufacturers; however, in reality, the shrinkage is not correlated with the volume change involved in stress.¹⁹ The post-gel shrinkage concept has turned out to be a useful factor to develop an understanding of the development and calculation of shrinkage stresses. This study had limitations. Only a general 2D model was used. The use of a 3D model can demonstrate the stress concentration along the gingival margin. From a clinical perspective, this study highlighted that the use of a bulk-fill technique may contribute to less shrinkage stress generation on endodontically-treated molars, which are normally more susceptible to fracture and crack propagation.^{34,35} It is important that the clinician be capable of differentiating the type of

shrinkage when choosing a dental composite; total shrinkage does not reflect shrinkage stress behavior, which is much better characterized by post-gel shrinkage.

CONCLUSIONS

The use of total shrinkage values for finite element simulations resulted in higher stress in the enamel and in root dentin close to the pulp chamber than when post-gel shrinkage values were used. The selection of total or post-gel shrinkage changed the ranking of the shrinkage stress of the tested composite resins. The use of post-gel shrinkage values should guide the clinician in composite resin selection for posterior teeth.

Acknowledgements

This project was funded by grants from the CAPES, CNPq-National Council for Scientific and Technological Development, and FAPEMIG. This research was performed at CPbio-Biomechanics, Biomaterials, and Cell Biology Research Center, UFU.

Regulatory Statement

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

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 January 2020)

REFERENCES

1. Rosatto CM, Bicalho AA, Veríssimo C, Bragança 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.
2. Pereira R, Bicalho AA, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2016) Effect of restorative protocol on cuspal strain and residual stress in endodontically treated molars *Operative Dentistry* **41**(1) 23-33.
3. 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.
4. Bicalho AA, Pereira RD, Zanatta RF, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2014) Incremental filling technique and composite material—part I: cuspal deformation, bond strength, and physical properties *Operative Dentistry* **39**(2) E71-82.

5. Tantbirojn D, Pfeifer CS, Amini AN, & Versluis A (2015) Simple optical method for measuring free shrinkage *Dental Materials* **31**(11) 1271-1278.
6. Soares CJ, Faria-E-Silva AL, Rodrigues MP, Vilela ABF, Pfeifer CS, Tantbirojn D, & Versluis A (2017) Polymerization shrinkage stress of composite resins and resin cements: what do we need to know? *Brazilian Oral Research* **31**(Supplement 1) e62.
7. Tantbirojn D, Versluis A, Pintado MR, DeLong R, & Douglas WH (2004) Tooth deformation patterns in molars after composite restoration *Dental Materials* **20**(6) 535-542.
8. Bicalho AA, Valdivia AD, Barreto BC, Tantbirojn D, Versluis A, & Soares CJ (2014) Incremental filling technique and composite material—part II: shrinkage and shrinkage stresses *Operative Dentistry* **39**(2) E83-E92.
9. 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.
10. 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.
11. Ferracane JL (2013) Resin-based composite performance: are there some things we can't predict? *Dental Materials* **29**(1) 51-58.
12. 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.
13. Versluis A, Douglas WH, Cross M, & Sakaguchi RL (1996) Does an incremental filling technique reduce polymerization shrinkage stresses? *Journal of Dental Research* **75**(3) 871-878.
14. Zhu S & Platt J (2011) Curing efficiency of three different curing modes at different distances for four composites *Operative Dentistry* **36**(4) 362-371.
15. Stansbury JW (2012) Dimethacrylate network formation and polymer property evolution as determined by the selection of monomers and curing conditions *Dental Materials* **28**(1) 13-22.
16. Benetti AR, Havndrup-Pedersen C, Honoré D, Pedersen MK, & Pallesen U (2015) Bulk-fill resin composites: polymerization contraction, depth of cure, and gap formation *Operative Dentistry* **40**(2) 190-200.
17. Cerda-Rizo ER, de Paula Rodrigues M, Vilela A, Braga S, Oliveira L, Garcia-Silva TC, Soares CJ (2019) Bonding interaction and shrinkage stress of low-viscosity bulk fill resin composites with high-viscosity bulk fill or conventional resin composites *Operative Dentistry* **44**(6) 625-636.
18. Tarle Z, Attin T, Marovic D, Andermatt L, Ristic M, & Tauböck TT (2015) Influence of irradiation time on subsurface degree of conversion band microhardness of high-viscosity bulk-fill resin composites *Clinical Oral Investigation* **19**(4) 831-840.
19. Enochs T, Hill AE, Worley CE, Veríssimo C, Tantbirojn D, & Versluis A (2018) Cuspal flexure of composite-restored typodont teeth and correlation with polymerization shrinkage values *Dental Materials* **34**(1) 152-160.
20. Cidreira Boaro LC, Pereira Lopes D, de Souza ASC, Lie Nakano E, Ayala Perez MD, Pfeifer CS, Gonçalves F (2019) Clinical performance and chemical-physical properties of bulk fill composites resin: a systematic review and meta-analysis *Dental Materials* **35**(10) e249-e264.
21. Versluis A & Tantbirojn D (2009) Relationship between shrinkage and stress In: Daskalaki A (ed) *Dental Computing and Applications: Advanced Techniques for Clinical Dentistry* Information Science Reference, Hershey, PA 45-64
22. Sakaguchi RL, Versluis A, & Douglas WH (1997) Analysis of strain gage method for measurement of post-gel shrinkage in resin composites *Dental Materials* **13**(4) 233-239.
23. Versluis A, Tantbirojn D, Pintado MR, DeLong R, & Douglas WH (2004) Residual shrinkage stress distributions in molars after composite restoration *Dental Materials* **20**(6) 554-564.
24. Craig RG & Powers JM (2002) *Restorative Dental Materials* Mosby Elsevier, St Louis, MO.
25. Soares CJ, Soares PV, de Freitas Santos-Filho PC, Castro CG, Magalhaes D, & Versluis A (2008) The influence of cavity design and glass fiber posts on biomechanical behavior of endodontically treated premolars *Journal of Endodontics* **34**(8) 1015-1019
26. Bausch JR, de Lange K, Davidson CL, Peters A, & de Gee AJ (1982) Clinical significance of polymerization shrinkage of composite resins *Journal of Prosthetic Dentistry* **48**(1) 59-67.
27. Davidson CL & de Gee AJ (1984) Relaxation of polymerization contraction stresses by flow in dental composites *Journal of Dental Research* **63**(2) 146-148.
28. Gonçalves F, Azevedo CL, Ferracane JL, & Braga RR (2011) BisGMA/TEGDMA ratio and filler content effects on shrinkage stress *Dental Materials* **27**(6) 520-526.
29. Ferracane JL (2011) Resin composite: state of the art *Dental Materials* **27**(1) 29-38.
30. Fronza BM, Rueggeberg FA, Braga RR, Mogilevych B, Soares LE, 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.
31. Schattenberg A, Lichtenberg D, Stender E, Willershausen B, & Ernst CP (2008) Minimal exposure time of different LED-curing devices *Dental Materials* **24**(8) 1043-1049.
32. Ellakwa A, Cho N, & Lee IB (2007) The effect of resin matrix composition on the polymerization shrinkage and rheological properties of experimental dental composites *Dental Materials* **23**(10) 1229-1235.
33. Schliebe LRSO, Braga SSL, Da Silva Pereira RA, Bicalho AA, Veríssimo C, Novais VR, & Soares CJ (2016) The new generation of conventional and bulk-fill composites do not reduce the shrinkage stress in endodontically-treated molars *American Journal of Dentistry* **29**(6) 333-338.

34. 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.
35. Barcelos LM, Bicalho AA, Veríssimo C, Rodrigues MP, & Soares CJ (2017) Stress distribution, tooth remaining strain, and fracture resistance of endodontically treated molars restored without or with one or two fiberglass posts and direct composite resin *Operative Dentistry* **42**(6) 646-657.

Tridimensional Evaluation of the Interfacial Gap in Deep Cervical Margin Restorations: A Micro-CT Study

N Scotti • A Baldi • EA Vergano • R Michelotto Tempesta
M Alovisei • D Pasqualini • GC Carpegna • A Comba

Clinical Relevance

Using a material that optimizes marginal seal when using a margin elevation technique to manage deep class II cavities should enhance clinical outcomes.

SUMMARY

Objectives: The purpose of this laboratory study was to perform a tridimensional interfacial gap evaluation of class II cavities with

*Nicola Scotti, associate professor, DDS, PhD, Department of Surgical Sciences, University of Turin, Turin, Italy

Andrea Baldi, DDS, PhD student, Department of Surgical Sciences, University of Turin, Turin, Italy

Edoardo Alberto Vergano, DDS, Department of Surgical Sciences, University of Turin, Turin, Italy

Riccardo Michelotto Tempesta, DDS, PhD, Department of Surgical Sciences, University of Turin, Turin, Italy

Mario Alovisei, assistant professor, DDS, PhD, Department of Department of Surgical Sciences, Dental School, Endodontics, University of Turin, Turin, Italy

Damiano Pasqualini, associate professor, DDS, Department of Surgical Sciences, University of Turin, Turin, Italy

Giorgia Cristina Carpegna, PhD student, Department of Surgical Sciences, University of Turin, Turin, Italy

Allegra Comba, DDS, PhD, Department of Biomedical Sciences DIBINEM, University of Bologna, Bologna, Italy

*Corresponding author: Via Nizza 230, Torino 10126, Italy; e-mail: nicola.scotti@unito.it

<https://doi.org/10.2341/19-161-L>

enamel and dentin cervical margins, before and after cyclic fatigue, restored with different nanohybrid resin composites.

Methods and Materials: Standardized class II cavities were performed on 48 intact maxillary premolars, placing the mesial cervical margin 1 mm above the cement–enamel junction (CEJ) and the distal cervical margin 1 mm below the CEJ. Specimens were treated with two-step self-etch adhesive (Clearfil SE Bond2) and divided into six groups according to the restoration technique. Microcomputed Tomography imaging was executed before and after 1,000,000 cycles of chewing simulation at 50 N. Tridimensional interfacial gaps, expressed as cubic millimeters, were analyzed through a standardized software flowchart (Mimics). Data were analyzed with a two-way analysis of variance and Tukey *post hoc* tests ($\alpha=0.05$).

Results: Restoration technique ($p=0.001$) and chewing simulation ($p=0.00001$) significantly influenced interfacial gap on dentin but not on enamel. The *post hoc* test showed that, on dentin margins, flowable resins had a lower gap at baseline but a higher gap after chewing

simulation, especially when a 2-mm-thick layer was applied, compared with nanohybrid and bulk-fill composites.

Conclusions: Based on the obtained results, no differences in interfacial gap volume were found on enamel margins. On dentin margins, flowable resins showed better marginal seal at baseline, but they seem to be more prone to interfacial degradation during chewing simulation than traditional composites.

INTRODUCTION

Resin composites are the most widely used materials in direct posterior restorations, and consequently, interest in the longevity and reliability of these materials has grown over time.¹ Although widely used, composites still present various problems. During the polymerization phase, these materials are subject to shrinkage stress, which can lead to debonding, causing an interfacial gap.^{2,3} The resulting poor marginal seal is associated with postoperative sensitivity, secondary caries, periodontal problems, and infiltration of bacteria, liquids, and molecules, leading to marginal discoloration and failure of the restoration itself.^{4,5}

The clinical prognosis for adhesive restorations with margins made entirely of enamel is excellent,^{6,7} but the same cannot be stated for deep cavities; with cervical margins extending beyond the cemento-enamel junction (CEJ), it is difficult to obtain an effective and durable marginal seal.⁸⁻¹⁰ It has been reported that restorations with cervical margins in dentin and cementum are more susceptible to marginal staining, postoperative sensitivity, and the formation of secondary caries.^{4,11}

Different techniques and materials have been tested to improve the sealing of deep cervical margins, such as the open-sandwich technique,^{12,13} the use of ceramic inlays,¹⁴⁻¹⁶ and margin elevation with composites.¹⁶

Several studies have shown how the use of flowable composites, interposed between the cavity floor of the interproximal box and the restorative material, can reduce the interface stress related to volumetric contraction while curing.^{17,18} These materials have a low elasticity modulus, which allows increased elastic deformation and therefore greater absorption of contraction stress caused by polymerization; this minimizes the interfacial gap, especially in the cervical area.^{8,19} Another property of these materials is improved wetting, which facilitates adaptation, ensuring a more intimate contact with

the cavity walls.²⁰ However, flowable resins have inferior mechanical properties compared with conventional composites.²¹

Some studies have reported a better marginal fit with use of flowable composite layering with reduced thickness.^{22,23} However, Malmström and others showed that neither the thickness nor the presence of flowable composite as an initial cervical increment significantly influenced the marginal microleakage,²⁴ probably due to lower mechanical properties and a reduced resistance to deformation compared with conventional resin composites.²⁵

Bulk-fill flowable composites have been introduced to minimize internal polymerization stresses via a longer pre-gel phase. A study by Moorthy and others showed that minor contraction stress exerted by bulk-fill flowable composites translates into reduced cuspal deflection compared with traditional composites placed with an oblique layering technique.²⁶ However, a laboratory study by Furness and others showed that bulk-fill materials, both flowable and nonflowable, resulted in a similar gap-free marginal interface compared with conventional composites.²⁷

Interfacial gap formation has been evaluated in the literature by means of different destructive tests,²⁸⁻³² hindering a more detailed analysis of the interface before and after polymerization. Micro-computed Tomography (micro-CT) enables creation of a three-dimensional (3D) map of the tooth-restoration interface and detection of the deepest marginal leakage,^{33,34} allowing a detailed assessment not only of the entity but also of the topography of the interfacial gap.

To the best of our knowledge, no studies have performed a tridimensional interfacial gap analysis when the margin elevation technique with different resin composite materials and techniques for deep class II cavities is necessary. Thus, the aim of the present laboratory study was to evaluate the volumetric interfacial gap of composite restorations in class II cavities, with enamel and dentin cervical margins, before and after cyclic fatigue.

The null hypotheses were that the tridimensional interfacial gap during cervical margin elevation technique is not influenced by (1) the material used or (2) cyclic fatigue.

METHODS AND MATERIALS

Specimen Preparation

Forty-eight intact human maxillary premolars, extracted for periodontal reasons within the last

Table 1: Summary of Used Materials With Respective Classification and Main Components

Material	Classification	Main Components
Grandioso heavy flow (VOCO GmbH Cuxhaven)	Nanohybrid flowable composite resin	Bis-GMA, Bis-EMA, TEGDMA Fillers (83 wt% = 68 vol%): glass ceramic (average size: 1 μ m), functionalized SiO ₂ nanoparticles (20-40 nm)
Grandioso (VOCO GmbH Cuxhaven)	Nanohybrid composite resin	Bis-GMA, Bis-EMA, TEGDMA Fillers (89 wt% = 73 vol%): glass ceramic and silicone dioxide nanoparticles (20-40 nm, 0.5-3 μ m)
Admira Fusion flow (VOCO GmbH Cuxhaven)	Nanohybrid flowable ormocer	Matrix: resin ormocer Fillers (74 wt%): silicon dioxide (unspecified filler size)
Admira Fusion (VOCO GmbH Cuxhaven)	Nanohybrid ormocer	Matrix: resin ormocer Fillers (84 wt%): Silicon dioxide nanofillers (20-50 nm) and silicon oxide-based hybrid fillers (1 μ m)
Filtek Supreme XTE (3M Maplewood, MN, USA)	Nano-filled composite resin	Bis-GMA, Bis-EMA, PEGDMA, TEGDMA Fillers (65 wt% = 46 vol%): ytterbium trifluoride (0.1-5 μ m), nonagglomerated/nonaggregated surface modified 20-nm and 75-nm silica fillers, surface modified aggregated zirconia/silica fillers (cluster average size: 0.6-10 μ m)
Filtek Bulk-Fill Posterior (3M, Maplewood, MN, USA)	Bulk-fill nanofilled composite	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA Fillers (64.5 wt% = 42.5 vol%): ytterbium trifluoride (0.1-5 μ m), zirconia/silica (0.01-3.5 μ m)
Abbreviations: Bis-EMA, bisphenol A diglycidyl methacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; PEGDMA, Poly(ethylene glycol) dimethacrylate; UDMA, Urethane Dimethacrylate.		

three months, were selected and stored in distilled water after disinfection with an ultrasonic device. The selected teeth had no carious lesions, demineralization, cracks, or signs of wear. Two class II cavities, one mesial and one distal, of similar shape and size were created on each specimen by the same operator. The cavities were 4 mm in the buccal-lingual direction and 3 mm in the mesio-distal direction; the mesial cavity had an enamel cervical margin 1 mm above the cemento-enamel junction (CEJ), whereas the distal cavity had a dentin cervical margin 1 mm below the CEJ. A circumferential steel matrix was applied (Automatrix, Dentsply, Sirona, Germany) and tightened until a perfect fit with the cervical margin was achieved. Then, all specimens were subjected to the same adhesive procedure: selective enamel etching for 40 seconds with 35% phosphoric acid (K-etchant, Kuraray Noritake Dental, Mie, Japan), rinsing for 30 seconds, and air-drying. A two-step self-etch adhesive system (Clearfil SE Bond2, Kuraray Noritake Dental) was then applied following the manufacturer's instructions and lightly air-dried before light-curing for 40 seconds with a light-emitting diode (LED) lamp (Cefalux2, VOCO, Cuxhaven, Germany). Specimens were then divided into six groups (n=8 each) using the following restoration techniques.

- Group 1 (G1): A 1-mm-thick horizontal layer of flowable resin (Grandioso Heavy Flow, VOCO) was

applied over the cervical margin. The restoration was then finalized with 2-mm-thick oblique layers of nanofilled composite (Grandioso, VOCO).

- Group 2 (G2): A 1-mm-thick horizontal layer of ormocer flowable resin (Admira Fusion Flow, VOCO) was applied over the cervical margin. The restoration was then finalized with 2-mm-thick oblique layers of nanofilled ormocer (Admira Fusion, VOCO).
- Group 3 (G3): The same technique used in G1 was applied but with 2 mm of flowable composite (Grandioso Heavy Flow, VOCO).
- Group 4 (G4): The same technique used in G2 was applied but with 2 mm of flowable ormocer (Admira Fusion Flow, VOCO).
- Group 5 (G5): A nanohybrid composite (Filtek Supreme XTE, 3M ESPE, St Paul, MN, USA) was used, applying 2-mm-thick oblique layers.
- Group 6 (G6): A bulk restoration was performed using a bulk nanofilled composite (Filtek Bulk-Fill Posterior, 3M ESPE).

A summary concerning the used materials is given in Table 1.

In all specimens, each composite layer was light-cured with an LED lamp (Cefalux2, VOCO) at 1400 mW/cm² for 20 seconds. Finishing and polishing procedures were then performed with abrasive disks (SofLex, 3M ESPE) and silicon points (Enhance, Dentsply).

Micro-CT Analysis and Fatigue Treatment

The marginal adaptation of each restoration was evaluated using a micro-CT scanner (SkyScan 1172, Bruker, Billerica, MA, USA). High-resolution scans were performed on each specimen using the following parameters: voltage = 100 kV; current = 100 μ A; aluminum and copper (Al+Cu) filter; pixel size = 10 μ m; averaging = 5; rotation step = 0.1°; and total scan duration = five hours.

A CS-4.4 chewing simulator (SD Mechatronik, Feldkirchen-Westerham, Germany) was used for fatigue-cycling mechanical aging of the specimens. The resilience of the human periodontium was simulated by coating the roots of the teeth with a 1-mm polyether layer (Impregum, 3M ESPE). A 6-mm-diameter steatite sphere was used with the following settings: occlusal load = 50 N; frequency = 1 Hz; downward speed = 16 mm/s; and sliding movement = 2 mm over the buccal triangular crest. All restored specimens had a standardized anatomy and were similarly positioned to center the sphere exactly on the central fossa of the tooth. The test was performed for 1,000,000 cycles in distilled water.

Tridimensional Interfacial Gap Analysis

To reveal interfacial gap progression between the restorations and the tooth after cyclic fatigue, specimens were subjected to a second scan, with the same baseline parameters used to ensure consistency of the grayscale values. NRecon (Bruker, Kontich, Belgium) was used to reconstruct samples and obtain DICOM files (Digital Imaging and Communications in Medicine; .dcm) with the same Hounsfield unit (Hu) parameters and the following software corrections: beam hardening = 30%, smoothing = 3, smoothing kernel = 2 gaussian, and ring artifact correction = 7. A novel tridimensional method was used to analyze the interfacial gap. Using Mimics software (ver. 20.0, Materialise, Leuven, Belgium), thresholding of voids surrounding the restoration was performed automatically to include all voids surrounding the restoration in a 200- μ m range. The Hu values representative for gap voids (−1024/−990) were selected by an expert operator on the first sample and therefore applied to all samples. Using dynamic region growing and region growing functions, only internal and marginal gaps were included in the present analysis (Figures 1 and 2).

Volumetric calculation of the resulting mask was performed by the software, and volume data (expressed in cubic millimeters) were collected for both

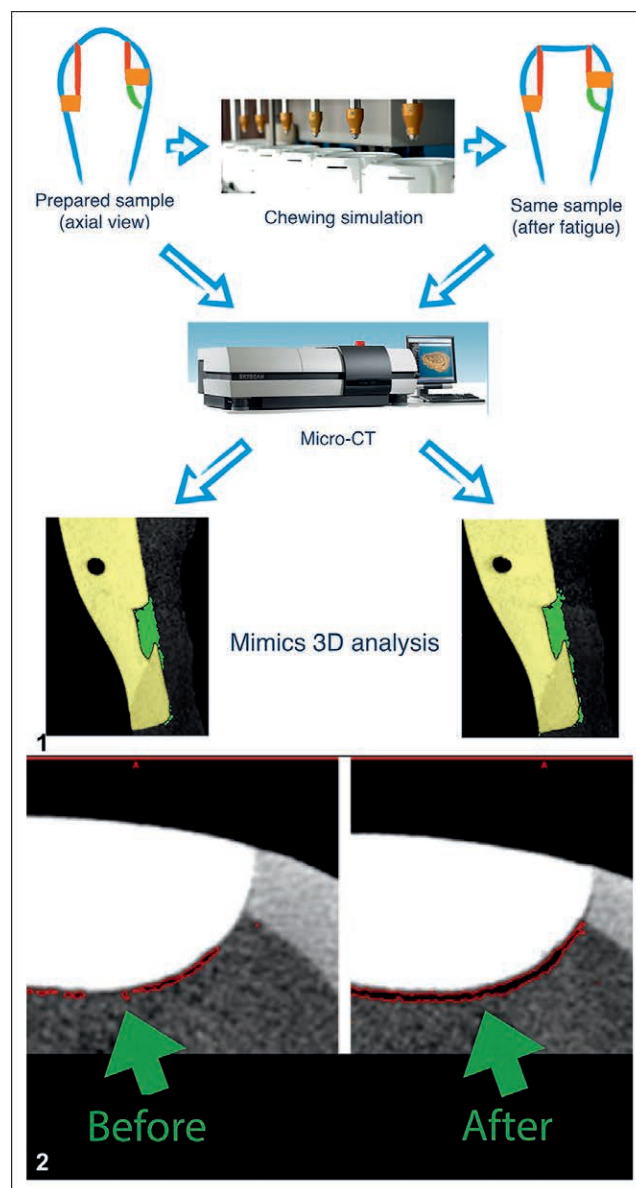


Figure 1. Representative scheme of the protocol.

Figure 2. Same coronal cuts of a random sample, before (left side) and after cyclic fatigue (right side). Void mask contour is highlighted with a red line. It is noticeable that the restoration interface degraded after cyclic fatigue, ultimately leading to a marginal gap opening internally.

the dentin (Figure 3) and enamel interfaces (Figure 4).

Statistical Analysis

To evaluate the effects of materials and techniques, a two-way analysis of variance (ANOVA) and *post hoc* Tukey tests were performed. The significance level was set to 95% ($\alpha=0.05$). All statistical analyses were

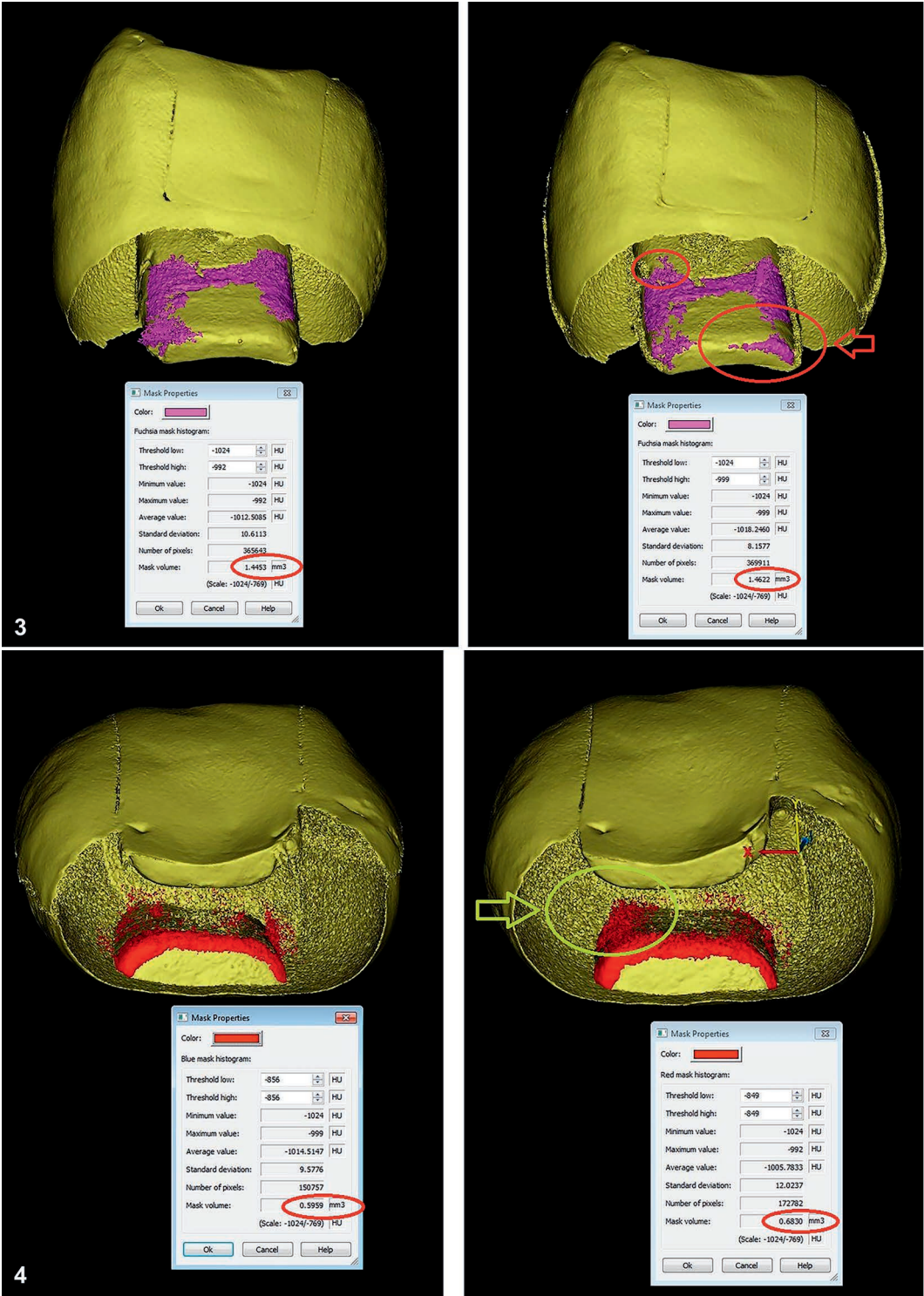


Figure 3. The same sample as Figure 2 is represented with 3D rendering before (left side) and after chewing simulation (right side). Yellow volume represents enamel and restorations. Violet volume represents interfacial voids of the deep restoration. It is noticeable that some areas showed a visible interfacial gap opening.

Figure 4. The same sample as Figure 3 before (left side) and after chewing simulation (right side). Yellow volume represents enamel and restorations. Red volume represents interfacial voids of the superficial restoration. It is noticeable that some areas showed a visible interfacial gap opening.

Table 2: Interfacial Volumetric Gaps of the Enamel Cervical Margin (mm³)^a

Material	Cyclic Fatigue	
	Before	After
Grandioso Heavy Flow, 1 mm	0.857 ± 0.254 Aa	1.162 ± 0.355 Aa
Admira Fusion Flow, 1 mm	0.727 ± 0.279) Aa	1.222 ± 0.246 Aa
Grandioso Heavy Flow, 2 mm	0.699 ± 0.388 Aa	1.457 ± 0.504 Ab
Admira Fusion Flow, 2 mm	0.879 ± 0.590) Aa	1.244 ± 0.446 Aa
Filtek Supreme XTE	1.322 ± 0.381 Aa	1.543 ± 0.159 Aa
Filtek Bulk-Fill Posterior	1.081 ± 0.485 Aa	1.665 ± 0.401 Aa

^a Same lowercase letters in rows indicate no difference between storage time ($p>0.05$); same uppercase letters in columns indicate no difference between luting procedures ($p>0.05$).

performed using the Stata software package (Stata-Corp, College Station, TX, USA).

RESULTS

Interfacial volumetric gaps ($\pm$ SD; expressed in cubic millimeters) are shown in Tables 2 (enamel cervical margins) and 3 (dentin cervical margins). The results of the ANOVA showed that restoration technique ($p=0.001$) and chewing simulation ($p=0.00001$) significantly influenced the interfacial gap on dentin but not on enamel. The *post hoc* test showed that, for deep dentin margins, flowable resins, either 1 or 2 mm, were better able to seal the interface before the chewing simulation but were more prone to interfacial degradation than nano-hybrid and bulk-fill composites. After cyclic fatigue, only the dentin margins closed with 2 mm of flowable composites showed greater interfacial gap than the other groups. On enamel margins, no differences were found between the restoration techniques tested.

DISCUSSION

Based on the results of the present study, the first null hypothesis was partially rejected because the use of flowable resins yielded a significantly better marginal seal on deep cervical margin elevation at baseline. The sealing ability of flowable resins also showed a significant reduction after artificial aging; therefore, the second null hypothesis was rejected.

Several laboratory studies have tested the performance of adhesive systems by evaluating marginal gap formation around restorations of extracted teeth.³⁵ This method assumes that if the forces generated by polymerization shrinkage or thermo-

Table 3: Interfacial Volumetric Gaps of the Dentin Cervical Margin (mm³)^a

Material	Cyclic Fatigue	
	Before	After
Grandioso Heavy Flow, 1 mm	0.931 ± 0.310 Aa	1.528 ± 0.363 Ab
Admira Fusion Flow, 1 mm	0.820 ± 0.276 Aa	1.592 ± 0.692 Ab
Grandioso Heavy Flow, 2 mm	0.719 ± 0.334 Aa	2.057 ± 0.506 Bb
Admira Fusion Flow, 2 mm	0.975 ± 0.206 Aa	2.298 ± 0.399 Bb
Filtek Supreme XTE	1.608 ± 0.209 Ba	1.922 ± 0.411 Aa
Filtek Bulk-Fill Posterior	1.422 ± 0.335 Ba	1.851 ± 0.178 Aa

^a Same lowercase letters in rows indicate no difference between storage time ($p>0.05$); same uppercase letters in columns indicate no difference between luting procedures ($p>0.05$).

mechanical strain exceed the bond strength, an observable gap will form at the margin of the restoration. Although there is no clear correlation between laboratory gap formation and interfacial failures observed clinically, it is reasonable to assume that this marginal gap formation is clinically relevant.³⁶ Many studies have found that all current adhesives appear incapable of completely sealing the restoration margins and thus preventing microleakage,^{37,38} especially for cavities with deep cervical margins.

Many techniques have been used to assess microleakage, and the results vary considerably.²⁸ Traditional laboratory methods to detect microleakage between a restoration and composite use organic dyes, such as basic fuchsin, methylene blue, and rhodamine, in conjunction with microscopy techniques²⁹ or transmission electron microscopy.³⁰ The disadvantage of these analyses include invasiveness, semiquantitative results, and limited ability to represent tridimensional geometry.³¹ Using scanning electron microscopy (SEM) to determine the presence of internal cracks or voids requires sample sectioning, which eliminates the possibility of evaluating the effects of artificial aging on samples after a baseline analysis. Epoxy replicas can also be used and evaluated with SEM, but this allows analysis only of the external margin. Moreover, such results can be affected by the accuracy of the impression, and a weak-to-moderate correlation with clinical findings has been reported.³² More recently, optical coherence tomography (OCT) has been used to evaluate interfacial adaptation and microleakage in composite restorations. OCT is a time-domain low-coherence interferometric technique that provides high-resolution cross-sectional (two-dimensional) or

volumetric (tridimensional) images without x-ray exposure by quantifying the reflection of infrared light from dental structures.³⁹ The resolution of OCT is approximately 5-15 μm , which is more than radiographic or current clinical CT images.⁴⁰ OCT initially showed some limits in detecting gaps in deep cavities due to the light transmission ability through dental tissues and materials, but new techniques and equipment are overcoming this problem.⁴¹ However, a recently published review by Sahyoun and others stated that image scaling, deformable registration, and fusion methods still must be implemented to superimpose OCT data onto another 3D surface.⁴² In the present study, micro-CT was used without any radio-opaque tracer to evaluate interfacial gaps. This approach has the advantages of being nondestructive, quantitative, and tridimensional. Specifically, it allows tridimensional visualization of the spatial distribution of the interfacial leakage along the cavity walls and floor, which cannot be obtained easily using traditional techniques that require sectioning of the specimen. These features make this method markedly more comprehensive. By contrast, traditional methods for microleakage studies can provide only limited, or even unrepresentative, information unless multiple sections of the sample are analyzed.³⁴ The volumetric evaluation of interfacial gaps between tooth hard tissues and restorations allows not only standardized, tridimensional measurement of the gap progression after cyclic fatigue, but also qualitative visualization of where the gap occurs. Stress propagation along adhesive restoration interfaces could be related to several factors and visualized by superimposition of baseline and after-chewing scans to determine the weakest point of the restoration-tooth interface.

When considering interface analysis between resin composite and tooth tissue, the adhesive system could represent a variable in marginal gap formation, particularly when margins are positioned apical to the CEJ. Bonding to dentin is different compared with enamel due to morphologic, histologic, and compositional differences; dentin contains a substantial proportion of water and organic materials, which impairs the bonding mechanism.⁴³ To improve the evaluation of restorative materials conducted in the present study, the same adhesive protocol was applied to all samples, as described in the previous section. Previous studies have shown an absence of resin tags in the cementum area,^{44,45} which reinforces the notion that cervical margins have the weakest bonding strength among all areas

of class II restorations. A two-step self-etch adhesive procedure with pre-etching of the peripheral enamel was performed on each sample in this study. As demonstrated in the literature, a mild etching effect causes a reduction in bond strength to enamel compared with that achieved using phosphoric acid-selective enamel etching.⁴⁶ This allows a considerable increase in the depth of resin penetration (longer resin tags), resulting in a better adhesion performance along enamel margins.⁴⁷ The results of the present study showed that the restoration techniques did not significantly influence the interfacial gap values of enamel margins at baseline or after chewing simulation. This can be explained by the greater adhesive reliability achievable on enamel substrate.⁴⁸

Consistent with previous studies, class II cervical margins in dentin, which usually yield to the deep margin elevation technique, showed a significantly greater marginal gap than those in enamel because dentin is a highly hydrophilic tissue that is only partially dehydratable and, therefore, more difficult to infiltrate by hydrophobic adhesives.²⁹ Water may persist within the adhesive layer on solvent evaporation, permeate the adhesive interface from the outer environment, or diffuse from the wet underlying dentin substrate. The amount of water uptake within the interface increases with time as bond strength decreases. In nonaqueous media, long-term preservation of dentin bond strength seems to be strongly linked to interface sealing.⁴⁹

The results of the present study also showed that marginal gaps along the dentin margins, treated with a deep margin elevation technique, were smaller when a flowable composite was used as the first horizontal layer, independent of its layer thickness and type. This finding is consistent with the results of two previous studies. Fabianelli and others reported that the open-sandwich technique was associated with significantly less dye penetration than the closed-sandwich technique,¹² whereas Korkmaz and others reported that the closed-sandwich technique required greater operator skill and achieved worse marginal adaptation.⁵⁰ Sadeghi and Lynch showed how better marginal adaptation could be achieved using an intermediate layer of flowable composites or compomers, especially when the cavity extended below the CEJ.⁵¹ Moreover, it has been widely reported that stresses generated during placement of a composite restoration can significantly influence the immediate marginal leakage, especially when dentin margins are present.⁵² With a low elastic modulus and better wettability,

flowable composites can create an intermediate flexible layer between the adhesive system and the composite resin, reducing contraction stress and improving the restoration seal.⁵³

Several authors reported significant effects of flow on the cavity floor, reducing microleakage in class II restorations. A previous study evaluated microleakage with and without flowable liners and concluded that flowable composites reduced, but did not eliminate, microleakage at the gingival cavosurface margins apical to the CEJ.⁵¹ The use of flow materials could reduce C-factor effects, leading to a reduction in polymerization stress and associated problems when applied in a 1-mm-thick layer. Lowering the C-factor may reduce the internal stresses within the composite restoration. However, the benefit of the gingival liner for reducing polymerization contraction stress is still somewhat controversial: some studies have reported that the use of flow did not reduce microleakage in class II restorations with margins below the CEJ,^{7,54} whereas other studies exhibited discordant results regarding microleakage.^{7,9,55,56} However, the methods used to evaluate marginal gaps were not precise or standardized, leading to greater variability in results due to differences in sample preparation, sectioning, and data collection procedures. Contrasting results were also presented by Kim and Park, who used micro-CT to evaluate the internal adaptation of composites.¹⁶ They showed that bulk-fill and layered resin composites had similar marginal sealing quality over dentin. However, differences in restorative materials, flowable liners, adhesive systems, and above all, testing procedures may explain variations in results.

The present laboratory study showed how flowable composites exhibited greater interfacial deterioration than nanohybrid composites, with a significantly increased gap volume after artificial aging procedures, especially when applied in thicknesses of 2 mm. Furthermore, the 3D analysis of the interfacial gap progression after cyclic fatigue allowed visualization of the microleakage increasing more at the level of the angle between the axial pulp wall and gingival floor. This could be related to the flowable resin's mechanical properties: Bayne and others evaluated the filler percent, wear, compressive strength, diametral tensile strength, indented biaxial flexure strength, and toughness of eight flowable and two hybrid composites.²⁵ Mechanical properties were approximately 60%-90% of conventional composites, resulting in a conclusion that flowable materials should be used with caution in

high stress-bearing areas. More recently, Baroudi and others found that the edge-fracture resistance of flowable composites was lower toward the margins than toward the center of a restoration,⁵⁷ explaining how, in the present study, the area between the axial pulp wall and gingival floor, near the cervical margin, showed the greatest increase in the interfacial gap. Even if the flowable composite's elastic modulus permits a stress-absorbing action, its higher amount of monomer could attenuate its mechanical resistance in long-term simulations, particularly in the area where internal stress consequent to functional loads concentrates. This would explain how flowable resins, if applied in 2-mm layers, showed greater interfacial deterioration. Pongprueksa and others also reported that conventional composites released significantly fewer monomers than flowable or bulk-fill composites, and a higher total monomer elution was recorded for both flowable composites, irrespective of the application method.⁵⁸ It is therefore assumable that higher quantities of unpolymerized monomers could lead to overlapping deformations. By consequence, fatigue microfailures would be more likely to appear in early stages of the simulation compared with a rigid material, with a more regular molecular structure that can dissipate forces.

CONCLUSIONS

All composite materials performed significantly better on enamel than on dentin. All flowable materials, regardless of the first horizontal layer thickness, were able to create a significantly better marginal seal than nanohybrid composites at baseline. However, nanohybrid and bulk-fill composites may be able to better maintain a marginal seal over time, because their use was not associated with any significant alteration of the marginal seal after mechanical treatment. Our results suggest that longitudinal clinical trials are necessary for precise clinical indications on the ideal approach to restoring cavities with deep cervical margins.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the CIR Dental School Lingotto. The approval code issued for this study is DS_2018_02.

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 23 December 2019)

REFERENCES

- Hickel R, Manhart J, & García-Godoy F (2000) Clinical results and new developments of direct posterior restorations *American Journal of Dentistry* **13**(Spec No) 41D-54D.
- Qvist V (1993) Resin restorations: leakage, bacteria, pulp *Endodontics & Dental Traumatology* **9**(4) 127-152.
- Aboushala A, Kugel G, & Hurley E (1996) Class II composite resin restorations using glass-ionomer liners: microleakage studies *Journal of Clinical Pediatric Dentistry* **21**(1) 67-70.
- Dennison JB & Sarrett DC (2012) Prediction and diagnosis of clinical outcomes affecting restoration margins *Journal of Oral Rehabilitation* **39**(4) 301-318.
- Jokstad A (2016) Secondary caries and microleakage *Dental Materials* **32**(1) 11-25.
- Soliman S, Preidl R, Karl S, Hofmann N, Krastl G, & Klaiber B (2016) Influence of cavity margin design and restorative material on marginal quality and seal of extended class II resin composite restorations in vitro *Journal of Adhesive Dentistry* **18**(1) 7-16.
- D'Alpino PH, Pereira JC, Rueggeberg FA, Svizero NR, Miyake K, & Pashley DH (2006) Efficacy of composite surface sealers in sealing cavosurface marginal gaps *Journal of Dentistry* **34**(3) 252-259.
- Chuang SF, Jin Y-T, Lin T-S, Chang C-H, & García-Godoy F (2003) Effects of lining materials on microleakage and internal voids of Class II resin-based composite restorations. *American Journal of Dentistry* **16**(2) 84-90.
- Tredwin CJ, Stokes A, & Moles DR (2005) Influence of flowable liner and margin location on microleakage of conventional and packable class II resin composites *Operative Dentistry* **30**(1) 32-38.
- Magne P & Spreafico RC (2012) Deep margin elevation: a paradigm shift *American Journal of Esthetic Dentistry* **2**(2) 11.
- Pereira PNR, Inokoshi S, & Tagami J (1998) In vitro secondary caries inhibition around fluoride releasing materials *Journal of Dentistry* **26**(5) 505-510.
- Fabianelli A, Sgarr A, Goracci C, Cantoro A, Pollington S, & Ferrari M (2010) Microleakage in class II restorations: open vs closed centripetal build-up technique *Operative Dentistry* **35**(3) 308-313.
- Kirsten GA, Rached RN, Mazur RF, Vieira S, & Souza EM (2013) Effect of open-sandwich vs. adhesive restorative techniques on enamel and dentine demineralization: an in situ study *Journal of Dentistry* **41**(10) 872-880.
- Zaruba M, Göhring TN, Wegehaupt FJ, & Attin T (2013) Influence of a proximal margin elevation technique on marginal adaptation of ceramic inlays *Acta Odontologica Scandinavica* **71**(2) 317-324.
- Hayashi M, Wilson NHF, Yeung CA, & Worthington HV (2003) Systematic review of ceramic inlays *Clinical Oral Investigations* **7**(1) 8-19.
- Kim HJ & Park SH (2014) Measurement of the internal adaptation of resin composites using micro-CT and its correlation with polymerization shrinkage *Operative Dentistry* **39**(2) E57-E70.
- Chuang S-F, Jin Y-T, Liu J-K, Chang C-H, & Shieh D-B (2004) Influence of flowable composite lining thickness on class II composite restorations *Operative Dentistry* **29**(3) 301-308.
- Leevailoj C, Cochran MA, Matis BA, Moore BK, & Platt JA (2001) Microleakage of posterior packable resin composites with and without flowable liners *Operative Dentistry* **26**(3) 302-307.
- Attar N, Tam LE, & McComb D (2003) Flow, strength, stiffness and radiopacity of flowable resin composites *Journal of the Canadian Dental Association* **69**(8) 516-521.
- Chuang SF, Liu JK, Chao CC, Liao FP, & Chen YH (2001) Effects of flowable composite lining and operator experience on microleakage and internal voids in class II composite restorations *Journal of Prosthetic Dentistry* **85**(2) 177-183.
- Ilie N, Rencz A, & Hickel R (2013) Investigations towards nano-hybrid resin-based composites *Clinical Oral Investigations* **17**(1) 185-193.
- Haak R, Wicht MJ, & Noack MJ (2003) Marginal and internal adaptation of extended class I restorations lined with flowable composites *Journal of Dentistry* **31**(4) 231-239.
- Lindberg A, van Dijken JWV, & Hörstedt P (2005) In vivo interfacial adaptation of class II resin composite restorations with and without a flowable resin composite liner *Clinical Oral Investigations* **9**(2) 77-83.
- Malmström HS, Schlueter M, Roach T, & Moss ME (2002) Effect of thickness of flowable resins on marginal leakage in class II composite restorations *Operative Dentistry* **27**(4) 373-380.
- Bayne SC, Thompson JY, Swift EJ, Stamatiades P, & Wilkerson M (1998) A characterization of first-generation flowable composites *Journal of the American Dental Association* **129**(5) 567-577.
- Moorthy A, Hogg CH, Dowling AH, Grufferty BF, Benetti AR, & Fleming GJP (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.
- 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.
- Hilton TJ (2002) Can modern restorative procedures and materials reliably seal cavities? In vitro investigations. Part 2 *American Journal of Dentistry* **15**(4) 279-289.
- Sano H, Shono T, Takatsu T, & Hosoda H (1994) Microporous dentin zone beneath resin-impregnated layer *Operative Dentistry* **19**(2) 59-64.
- Tay FR, Pashley DH, García-Godoy F, & Yiu CKY (2004) Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part II. Silver tracer penetration evidence *American Journal of Dentistry* **17**(5) 315-322.
- Bakhsh TA, Sadr A, Shimada Y, Tagami J, & Sumi Y (2011) Non-invasive quantification of resin-dentin inter-

- facial gaps using optical coherence tomography: validation against confocal microscopy *Dental Materials* **27**(9) 915-925.
32. Heintze SD (2013) Clinical relevance of tests on bond strength, microleakage and marginal adaptation *Dental Materials* **29**(1) 59-84.
 33. Rengo C, Goracci C, Ametrano G, Chieffi N, Spagnuolo G, Rengo S, & Ferrari M (2015) Marginal leakage of class V composite restorations assessed using microcomputed tomography and scanning electron microscope *Operative Dentistry* **40**(4) 440-448.
 34. Carrera CA, Lan C, Escobar-Sanabria D, Li Y, Rudney J, Aparicio C, & Fok A (2015) The use of micro-CT with image segmentation to quantify leakage in dental restorations *Dental Materials* **31**(4) 382-390.
 35. Roulet JF, Reich T, Blunck U, & Noack M (1989) Quantitative margin analysis in the scanning electron microscope *Scanning Microscopy* **3**(1) 147-158; discussion 158-159.
 36. Roulet JF (1994) Marginal integrity: clinical significance *Journal of Dentistry* **22**(Supplement 1) S9-12.
 37. 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.
 38. Bouillaguet S, Duroux B, Ciucchi B, & Sano H (2000) Ability of adhesive systems to seal dentin surfaces: an in vitro study *Journal of Adhesive Dentistry* **2**(3) 201-208.
 39. Otis LL, Everett MJ, Sathyam US, & Colston BW (2000) Optical coherence tomography: a new imaging technology for dentistry *Journal of the American Dental Association* **131**(4) 511-514.
 40. Fujimoto JG (2003) Optical coherence tomography for ultrahigh resolution in vivo imaging *Nature Biotechnology* **21**(11) 1361-1367.
 41. An L, Li P, Lan G, Malchow D, & Wang RK (2013) High-resolution 1050 nm spectral domain retinal optical coherence tomography at 120 kHz A-scan rate with 6.1 mm imaging depth *Biomedical Optics Express* **4**(2) 245-259.
 42. Sahyoun CC, Subhash HM, Peru D, Ellwood RP, & Pierce MC (2020) An experimental review of optical coherence tomography systems for noninvasive assessment of hard dental tissues *Caries Research* **54**(1) 43-54.
 43. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, & Van Meerbeek B (2005) A critical review of the durability of adhesion to tooth tissue: methods and results *Journal of Dental Research* **84**(2) 118-132.
 44. Gwinnett JA, Tay FR, Pang KM, & Wei SH (1995) Comparison of three methods of critical evaluation of microleakage along restorative interfaces *Journal of Prosthetic Dentistry* **74**(6) 575-585.
 45. Tay FR, Pang KM, Gwinnett AJ, & Wei SH (1995) A method for microleakage evaluation along the dentin/restorative interface *American Journal of Dentistry* **8**(2) 105-108.
 46. Haller B (2013) Which self-etch bonding systems are suitable for which clinical indications? *Quintessence International* **44**(9) 645-661.
 47. Devarasa GM, Subba Reddy VV, Chaitra NL, & Swarna YM (2012) Self-etching adhesive on intact enamel, with and without pre-etching *Microscopy Research and Technique* **75**(5) 650-654.
 48. Loguercio AD, Moura SK, Pellizzaro A, Dal-Bianco K, Patzlaff RT, Grande RHM, & Reis A (2008) Durability of enamel bonding using two-step self-etch systems on ground and unground enamel *Operative Dentistry* **33**(1) 79-88.
 49. Abdalla AI & Feilzer AJ (2008) Four-year water degradation of a total-etch and two self-etching adhesives bonded to dentin *Journal of Dentistry* **36**(8) 611-617.
 50. Korkmaz Y, Ozel E, & Attar N (2007) Effect of flowable composite lining on microleakage and internal voids in class II composite restorations *Journal of Adhesive Dentistry* **9**(2) 189-194.
 51. Sadeghi M & Lynch CD (2009) The effect of flowable materials on the microleakage of class II composite restorations that extend apical to the cemento-enamel junction *Operative Dentistry* **34**(3) 306-311.
 52. Choi KK, Condon JR, & Ferracane JL (2000) The effects of adhesive thickness on polymerization contraction stress of composite *Journal of Dental Research* **79**(3) 812-817.
 53. Labella R, Lambrechts P, Van Meerbeek B, & Vanherle G (1999) Polymerization shrinkage and elasticity of flowable composites and filled adhesives *Dental Materials* **15**(2) 128-137.
 54. Cavalcante LMA, Peris AR, Ambrosano GMB, Ritter AV, & Pimenta LAF (2007) Effect of photoactivation systems and resin composites on the microleakage of esthetic restorations *Journal of Contemporary Dental Practice* **8**(2) 70-79.
 55. Jain P & Belcher M (2000) Microleakage of class II resin-based composite restorations with flowable composite in the proximal box *American Journal of Dentistry* **13**(5) 235-238.
 56. Attar N, Turgut MD, & Güngör HC (2004) The effect of flowable resin composites as gingival increments on the microleakage of posterior resin composites *Operative Dentistry* **29**(2) 162-167.
 57. Baroudi K, Silikas N, & Watts DC (2008) Edge-strength of flowable resin-composites *Journal of Dentistry* **36**(1) 63-68.
 58. Pongprueksa P, De Munck J, Duca RC, Poels K, Covaci A, Hoet P, Godderis L, Van Meerbeek B, & Van Landuyt KL (2015) Monomer elution in relation to degree of conversion for different types of composite *Journal of Dentistry* **43**(12) 1448-1455.

Does the Type of Solvent in Dental Adhesives Influence the Clinical Performance of Composite Restorations Placed in Noncarious Cervical Lesions? A Systematic Review and Meta-analysis

RBE Lins • M Sebold • MB Magno • LC Maia • LRM Martins • M Giannini

Clinical Relevance

According to the clinical and scientific evidence presented in this systematic review and meta-analysis, dental adhesives containing either organic solvent (acetone or alcohol) can be used to achieve similar clinical performance and longevity of composite restorations.

*Rodrigo BE Lins, DDS, MSc, PhD, Department of Restorative Dentistry, Operative Dentistry Division, Piracicaba Dental School, University of Campinas, Piracicaba, Brazil

Maicon Sebold, DDS, MSc, PhD student, Department of Restorative Dentistry, Piracicaba Dental School, University of Campinas, Piracicaba, Brazil

Marcela B Magno, MSc, PhD, Post Doctoral student, Department of Pediatric Dentistry and Orthodontics, Federal University of Rio de Janeiro, Rio de Janeiro, Brazil

Lucianne C Maia, DDS, MSc, PhD, full professor, Department of Pediatric Dentistry and Orthodontics, Federal University of Rio de Janeiro, Rio de Janeiro, Brazil

Luís RM Martins, DDS, MSc, PhD, full professor, Department of Restorative Dentistry, University of Campinas, Piracicaba, Brazil

Marcelo Giannini, DDS, MSc, PhD, associate professor, Department of Restorative Dentistry, Piracicaba Dental School, University of Campinas, Piracicaba, Brazil

*Corresponding author: Avenida Limeira 901, Bairro Areião, Piracicaba, SP 13414-903, Brazil; e-mail: rodrigowlins@hotmail.com

<https://doi.org/10.2341/19-124-LIT>

SUMMARY

Objectives: This systematic review and meta-analysis compared the clinical performance and survival rates of composite restorations placed in noncarious cervical lesions (NCCLs) using dental adhesives containing acetone or alcohol-based solvents.

Methods and Materials: PubMed, Scopus, Web of Science, Virtual Health Library (VHL) LILACS, Cochrane Library, OpenGrey, Clinical Trials, and Rebec were searched. MeSH terms, supplementary concepts, synonyms, and free keywords were used in the search strategy. All references were crosschecked by two independent investigators following the PICOS strategy (population, NCCLs; intervention, acetone-based bonding agent; comparison, alcohol-based bonding agent; outcome, clinical evaluation parameters and survival rates; study design, randomized controlled clinical trials).

Cochrane Collaboration's tool was used to assess risk of bias, and two distinct meta-analyses were performed using the RevMan software. The prevalence of success and the total number of restorations for each group (acetone- or alcohol-based) were used to calculate the risk difference at a confidence interval of 95%. Random-effects models were applied, and heterogeneity was assessed using the I^2 index in the pooled and subgrouped meta-analyses. The certainty of evidence was evaluated through the Grading of Recommendations Assessment, Development, and Evaluation (GRADE) approach.

Results: A total of 7876 studies were retrieved, from which 27 studies were selected for the systematic review. Ten studies were classified as "low risk of bias" and included in the meta-analyses. Overall heterogeneity was not significant ($I^2 = 0.00\%$). The clinical performance of restorations placed with bonding agents based on both solvents for each of the available parameters presented no statistical significance for any of the meta-analyses ($p > 0.05$).

Conclusion: Scientific evidence suggests composite restorations placed with acetone or alcohol-based dental adhesives present similar clinical performance and survival rates in NCCLs.

INTRODUCTION

A solvent is the component of a solution that is present in the greatest amount,¹ and it is capable of dissolving or dispersing other substances.² Water, ethanol, and acetone are the most commonly added solvents in the composition of dental adhesives,^{3,4} in which they will dissolve monomers and consequently help reduce the viscosity of adhesive primers and/or resins, improving their diffusion through etched dentin.⁵ Therefore, solvents are included in the formulations of dental adhesives to facilitate the establishment of micro-retention with both enamel and dentin, regardless of adhesive mode of application, either etch-and-rinse or self-etch, or pH of adhesive formulation (mild/intermediately strong or strong, in the case of self-etch adhesives).^{3,5,6}

Alcohol and water are polar solvents that can create strong hydrogen bonds with collagen fibrils, maintaining the interfibrillar spaces, which improves monomer diffusion along etched dentin.⁴ Moreover, hydrogen bonds between ethanol and water increase evaporation rates, leading to more surface water

removal compared with pure water.⁵ Acetone-based solvents might be a great choice for bonding agents that contain hydrophilic and hydrophobic monomers in the same bottle, as acetone can dissolve both polar and nonpolar substances because of its high dipole moment and low dielectric constant.⁵

Conflicting information is available regarding the laboratory performance of composite restorations placed using acetone-based bonding agents: whereas some studies report higher nanoleakage and lower bond strengths,^{4,7-9} others describe similar results compared with ethanol-based adhesives.^{10,11} A randomized clinical trial (RCT)¹² reported better clinical performance for restorations placed with an ethanol/water-based bonding agent compared with an acetone-based one over 36 months of evaluation. Conversely, other clinical trials showed no differences between the clinical performance of restorations using bonding agents containing the aforementioned solvents for 24- or 36-month follow-up periods.¹³⁻¹⁵ Nonetheless, there are very few clinical trials designed to specifically compare acetone and alcohol-containing bonding agents.

Dental adhesives that use acetone might be expected to perform worse than water/alcohol-based systems, as they require a greater amount of water for optimal hybridization,⁴ and therefore are more sensitive to air-drying.¹⁶ This limitation occurs because, due to the absence of hydroxyl groups in the chemical structure of acetone, it cannot re-expand the collapsed collagen matrix,¹⁷ hindering adhesive resin diffusion. Additionally, acetone-based bonding agents are likely to form thinner adhesive layers that are more prone to polymerization inhibition by oxygen compared with ethanol and/or water.¹² However, acetone evaporates much more residual water from the tooth surface because of its higher vapor pressure and water-chasing effect,^{16,18} which has been suggested to cause less collagen degradation at the hybrid layer over time.¹⁸

In addition to the significant contributions of laboratory assessments about dental adhesives, randomized clinical trials of composite restorations in noncarious cervical lesions (NCCLs) are considered the best clinical condition to evaluate resin-based materials subjected to mechanical and chemical challenges.^{12,13,19,20} Thus, a systematic review and meta-analysis of the clinical evidence regarding the effect of the solvent type contained in dental adhesives on the performance of composite restorations in NCCLs might be important to guide future research and practice involving adhesive procedures. Therefore, this systematic review and meta-analysis aimed

to answer the following PICO (population, intervention, comparison, outcome) question: “does the type of solvent in dental adhesives influence the clinical performance of composite restorations placed in non-carious cervical lesions?” The null hypotheses were as follows: 1) there would be no difference in the clinical performance of composite restorations placed in NCCLs using acetone-based dental adhesives compared with alcohol-based dental adhesives; and 2) there would be no difference in the survival rates over several follow-up periods of composite restorations placed in NCCLs using acetone-based dental adhesives compared with alcohol-based dental adhesives.

METHODS AND MATERIALS

Study Protocol and Registration

The protocol of this study was registered in the International Prospective Register of Systematic Reviews (PROSPERO-CRD 42018106544) database, and its reporting followed the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA).²¹

Databases, Search Strategy, and Eligibility Criteria

An electronic search was carried out during the week of August 9, 2018 in the following databases: PubMed, Scopus, Web of Science, Virtual Health Library (VHL) LILACS, Cochrane Library, OpenGrey, Clinical Trials, and Rebec. The MeSH terms, supplementary concepts, synonyms, and free keywords used for the search strategy are presented in Table 1. A handmade search was also performed to find relevant articles that had not been retrieved in the electronic search of the selected databases. No restriction of date, language, or any other search filters were applied.

The selection criteria were defined based on the elements of the PICOS strategy²² described as follows:

Population (P): adult patients with NCCLs (class V);

Intervention (I): composite restorations performed with dental adhesives containing acetone-based solvents;

Comparison (C): composite restorations performed with dental adhesives containing alcohol-based solvents;

Outcome (O): clinical evaluation parameters of composite restorations (retention, marginal adaptation, marginal discoloration, surface texture,

color, postoperative sensitivity, secondary caries, anatomic form, pulp vitality), and overall survival rates, considering different follow-up periods (the “outcome” criterion was not used in the search strategy, because it would limit the number of retrieved studies);

Study design (S): randomized controlled clinical trials (the “study design” criterion was included in the search strategy to avoid a high number of laboratory studies).

RCTs with follow-ups shorter than 18 months, nonrandomized clinical trials, laboratory studies, and reviews were excluded. Additionally, studies performed on animals, primary teeth, carious or non-cervical cavities, or with restorative materials other than resin-based composites were also excluded.

Study Selection and Data Extraction Process

All references found on electronic databases were transferred to the EndNote X9 software (Clarivate Analytics, Philadelphia, PA, USA). Duplicates were excluded by the software considering title and year similarities and further by manual revision by two independent investigators (RBEL and MS). After title and abstract screening, all papers that could potentially be included in the systematic review were fully read to determine their eligibility. A third reviewer (MBM) was consulted in case the two main investigators could not reach an agreement.

Details of the studies (authors, year, country, and study design), participants (age—mean and range, number of NCCLs, and restored teeth), tested bonding agents (acetone or alcohol-based), methodologies (outcomes, follow-up in months, and overall survival rates), and results (success, failure rates, and statistical analyses) were extracted from the selected papers. Additionally, a classification regarding mode of action (etch and rinse [E&R] or self-etch [SE] with their respective number of application steps) and pH assessment (mild or intermediately strong, with a pH ≥ 1.5 , or strong, with a pH < 1.5) was also taken into consideration.⁶ If a certain study presented missing data, its corresponding or first author was contacted by email to obtain the necessary information so a decision could be made whether it should be included in the review. If no response was obtained after three attempts of contact by email, the manuscript was excluded.

Risk of Bias in Individual Studies

Quality and risk of bias of the eligible studies were assessed by two independent investigators (RBEL

Table 1: *Electronic Databases Searched and Strategies Used (up to August 9, 2018)*

Electronic Database	Strategy Used
PubMed (n=1615)	#1 Tooth erosion[MeSH Terms] OR Tooth abrasion[MeSH Terms] OR Tooth wear[MeSH Terms] OR Dental restoration, permanent[MeSH Terms] OR Erosion*, tooth[Title/Abstract] OR Tooth erosion*[Title/Abstract] OR Abrasion, dental[Title/Abstract] OR Abrasion, tooth[Title/Abstract] OR Teeth abrasions[Title/Abstract] OR Tooth abrasion*[Title/Abstract] OR Teeth abrasion[Title/Abstract] OR Wear*, dental[Title/Abstract] OR Dental wear[Title/Abstract] OR Wear*, tooth[Title/Abstract] OR Tooth wear*[Title/Abstract] OR Teeth abfraction*[Title/Abstract] OR Restoration, permanent dental[Title/Abstract] OR Dental restoration, permanent[Title/Abstract] OR Restorations, permanent dental[Title/Abstract] OR Dental restorations, permanent[Title/Abstract] OR Permanent Filling, Dental[Title/Abstract] OR Permanent Fillings, Dental[Title/Abstract] OR Dental Filling, Permanent[Title/Abstract] OR Dental Fillings, Permanent[Title/Abstract] OR Permanent dental fillings[Title/Abstract] OR Dental permanent filling*[Title/Abstract] OR Cervical lesion*[Title/Abstract] OR Class V[Title/Abstract] OR NCCL*[Title/Abstract] OR Cervical filling[Title/Abstract] OR Non-carious cervical lesion*[Title/Abstract] OR Noncarious cervical lesion*[Title/Abstract] OR Cervical restoration[Title/Abstract] OR Class V lesion*[Title/Abstract] OR Cervical fillings[Title/Abstract] OR (((((((Bonding, Dental[Title/Abstract] OR Dental Cements Curing[Title/Abstract] OR Dental Cement* Curing[Title/Abstract] OR Agent*, Dentin Bonding[Title/Abstract] OR Curing, Dental Cement*[Title/Abstract] OR Dental Cement Curing[Title/Abstract] OR Bonding Agents, Dentin[Title/Abstract] OR Bonding Agent*, Dentin[Title/Abstract] OR (((((((Bonding Agent, Dentin[Title/Abstract] OR Bis-GMA[Title/Abstract] OR Adhesive system*[Title/Abstract] OR Dentin bonding system*[Title/Abstract] OR HEMA[Title/Abstract] OR hydroxyethyl methacrylate[Title/Abstract] OR 2-hydroxyethyl methacrylate[Title/Abstract] OR glycol methacrylate[Title/Abstract] OR (((((((Bisphenol A-Glycidyl Methacrylate*[Title/Abstract] OR Solvent*[Title/Abstract] OR Methacrylate, Bisphenol A-Glycidyl[Title/Abstract] OR BisGMA[Title/Abstract] OR "Bisphenol A Glycidyl Methacrylate Polymer"[Title/Abstract] OR Dentin bonding*[Title/Abstract] OR (((((((Ethanol[Title/Abstract] OR Alcohol, Ethyl[Title/Abstract] OR Alcohol, Absolute[Title/Abstract] OR Alcohol, Grain[Title/Abstract] OR Absolute Alcohol[Title/Abstract] OR Acetone*[Title/Abstract] OR (((((((Cement*, Dental[Title/Abstract] OR Cement, Dental[Title/Abstract] OR Adhesive*, Dental[Title/Abstract] OR Self-etch adhesive*[Title/Abstract] OR Self-etching adhesive*[Title/Abstract] OR Etch-and-rinse adhesive[Title/Abstract] OR Adhesive material*[Title/Abstract] OR Universal adhesive*[Title/Abstract] OR Total-etch adhesive*[Title/Abstract] OR All-in-one adhesive*[Title/Abstract] OR One-bottle adhesive*[Title/Abstract] OR (((Etch-and-rinse bonding agents[Title/Abstract] OR Total-etch bonding agents[Title/Abstract] OR Self etching bonding agent*[Title/Abstract] OR Multimode adhesive*[Title/Abstract] OR Self-etch bonding agent*[Title/Abstract] OR Universal bonding agent*[Title/Abstract] OR One-bottle bonding agent*[Title/Abstract] OR "All-in-one bonding agent"[Title/Abstract] OR Total-etch bonding agent[Title/Abstract] OR "all in one bonding agents"[Title/Abstract] OR "all in one bonding agent"[Title/Abstract] #3 Clinical[Title/Abstract] OR Randomized[Title/Abstract] OR Intervention Study[Title/Abstract] OR Intervention Studies[Title/Abstract] OR Controlled Trial*[Title/Abstract] OR Prospective[Title/Abstract] OR Follow-up*[Title/Abstract] OR follow up[Title/Abstract] OR Trial*[Title/Abstract] OR Longitudinal[Title/Abstract] OR Quasi-Experimental[Title/Abstract] OR Non-Randomized[Title/Abstract] OR Nonrandomized[Title/Abstract]; #1 AND #2 AND #3

and MS) using the Cochrane Collaboration's tool for assessing risk of bias in randomized controlled clinical trials.²³ The evaluators compared and discussed the results of the selected papers, and a third investigator was consulted (LCM) when necessary. Assessment criteria were divided into seven domains: 1) random sequence generation; 2) allocation concealment; 3) blinding of participants and personnel; 4) blinding of outcomes assessment; 5) incomplete outcome data; 6) selective reporting; and 7) other bias. All domains were considered key domains, except for 3, and were classified as low, unclear, or high risk of bias for each study, following recommendations described in the Cochrane Handbook for Systematic Reviews of Interventions 5.1.0.²⁴ For a study to be considered low risk of bias, all of its key domains had to be classified as low. If a study presented any key domains registered as unclear, the corresponding or first author was contacted to

request additional information that could enable a precise risk of bias evaluation.

Meta-analyses

Clinical performance parameters and overall survival rates of composite restorations placed using acetone- or alcohol-based bonding agents presented in the low risk of bias studies were analyzed using the Revman 5.3 Software (Review Manager v. 5, The Cochrane Collaboration, Copenhagen, Denmark). Two separate meta-analyses were performed for 1) clinical evaluation parameters (retention, marginal adaptation, marginal discoloration, surface texture, color, postoperative sensitivity, secondary caries, anatomic form, and pulp vitality) and 2) survival rates (overall and at different evaluation periods: 6, 12, 18, 24, 36, 60, and 72 months). Clinical outcomes and overall survival rates were dichotomized as

Table 1: *Electronic Databases Searched and Strategies Used (up to August 9, 2018) (cont.)*

Electronic Database	Strategy Used
Cochrane Library (n=839)	#1MeSH descriptor: [Tooth Erosion] explode all trees #2(Erosion*, tooth OR Tooth erosion*):ti,ab,kw #3MeSH descriptor: [Tooth Abrasion] explode all trees #4("Abrasion, dental" OR "Abrasion, tooth" OR "Teeth abrasions" OR Tooth abrasion* OR "Teeth abrasion"):ti,ab,kw #5MeSH descriptor: [Tooth Wear] explode all trees #6(Wear*, dental OR Wear*, tooth OR "Dental wear" OR Tooth wear* OR Teeth abfraction*):ti,ab,kw #7MeSH descriptor: [Dental Restoration, Permanent] explode all trees #8("Restoration, permanent dental" OR "Restorations, permanent dental" OR "Dental restorations, permanent" OR "Permanent Filling, Dental" OR "Permanent Fillings, Dental" OR "Dental Filling, Permanent" OR "Dental Fillings, Permanent" OR "Permanent dental fillings" OR Dental permanent filling* OR "Dental restoration, permanent"):ti,ab,kw #9(Cervical lesion* OR "Class V" OR NCCL* OR "Cervical filling" OR Non-carious cervical lesion* OR Noncarious cervical lesion* OR "Cervical restoration" OR Class V lesion* OR "Cervical fillings"):ti,ab,kw #10#1 OR #2 OR #3 OR #4 OR #5 OR #6 OR #7 OR #8 OR #9 #11("Bis-GMA, BPDM, HEMA dental-bonding resin"):ti,ab,kw #12MeSH descriptor: [Dental Bonding] explode all trees #13("Bonding, Dental" OR "Dental Cements Curing" OR Dental Cement* Curing OR Curing, Dental Cement* OR "Dental Cement Curing"):ti,ab,kw #14MeSH descriptor: [Dentin-Bonding Agents] explode all trees #15("Bonding Agents, Dentin" OR Bonding Agent*, Dentin OR Agent*, Dentin Bonding OR "Bonding Agent, Dentin" OR Adhesive system* OR Dentin bonding system*):ti,ab,kw #16("hydroxyethyl methacrylate"):ti,ab,kw #17(HEMA):ti,ab,kw #18("2 hydroxyethyl methacrylate"):ti,ab,kw #19("glycol methacrylate"):ti,ab,kw #20MeSH descriptor: [Bisphenol A-Glycidyl Methacrylate] explode all trees #21("Bis-GMA" OR "Methacrylate, Bisphenol A-Glycidyl" OR "Bisphenol A Glycidyl Methacrylate Polymer" OR "Bisphenol A-Glycidyl Methacrylate" OR "Dentin bonding" OR BisGMA):ti,ab,kw #22MeSH descriptor: [Solvents] explode all trees #23(Solvent* OR Ethanol):ti,ab,kw #24MeSH descriptor: [Ethanol] explode all trees #25("Alcohol, Ethyl" OR "Alcohol, Absolute" OR "Alcohol, Grain" OR "Absolute Alcohol"):ti,ab,kw #26MeSH descriptor: [Acetone] explode all trees #27(Acetone*):ti,ab,kw #28MeSH descriptor: [Dental Cements] explode all trees #29(Cement*, Dental OR "Cement, Dental" OR Adhesive*, Dental):ti,ab,kw #30(Self-etch adhesive* OR Self-etching adhesive* OR "Etch-and-rinse adhesive" OR Adhesive material* OR Universal adhesive* OR Total-etch adhesive* OR All-in-one adhesive* OR One-bottle adhesive* OR "Etch-and-rinse bonding agents" OR "Total-etch bonding agents" OR Self-etching bonding agent* OR Multimode adhesive* OR Self-etch bonding agent* OR Universal bonding agent* OR One-bottle bonding agent* OR "All-in-one bonding agent" OR "All-in-one bonding agents" OR "All in one bonding agent" OR "Total-etch bonding agent"):ti,ab,kw #31#11 OR #12 OR #13 OR #14 OR #15 OR #16 OR #17 OR #18 OR #19 OR #20 OR #21 OR #22 OR #23 OR #24 OR #25 OR #26 OR #27 OR #28 OR #29 OR #30 #32("Intervention Study" OR "Intervention Studies" OR Clinical OR Follow-up* OR "Follow up" OR Trial* OR Randomized OR Controlled Trial* OR Prospective OR Longitudinal OR "Quasi-Experimental" OR "Non-Randomized" OR Nonrandomized):ti,ab,kw #33#10 AND #31 AND #32

success or failure according to the criteria used by each of the selected studies. The prevalence of success and the total number of restorations for each group (acetone- or alcohol-based) were used to calculate the risk difference at a confidence interval of 95%. Random-effects models were applied, and heterogeneity was tested using the I^2 index.

Certainty of Evidence Assessment

The quality of evidence (certainty in the estimates of effect) was determined for the outcomes using the

Grading of Recommendations Assessment, Development and Evaluation (GRADE) approach,²⁵ whereby randomized controlled clinical trials start as high evidence, and the quality or certainty of the body of evidence decreases to moderate, low, or very low evidence if serious or very serious issues related to risk of bias, inconsistency, indirectness, imprecision, and publication bias are present. Additionally, the quality of evidence of a study may be upgraded if the magnitude of effect is large or very large or if the effect of all plausible confounding factors would be to reduce or suggest a false effect. Therefore, the

Table 1: *Electronic Databases Searched and Strategies Used (up to August 9, 2018) (cont.)*

Electronic Database	Strategy Used
Lilacs VHL (n=268)	#1 (mh:(Tooth erosion)) OR (mh:(Tooth abrasion)) OR (mh:(Tooth wear)) OR (mh:(Dental restoration, permanent)) OR (tw:(Erosion\$, tooth)) OR (tw:(Tooth erosion\$)) OR (tw:(Abrasion, dental)) OR (tw:(Abrasion, tooth)) OR (tw:(Teeth abrasions)) OR (tw:(Tooth abrasion\$)) OR (tw:(Teeth abrasion)) OR (tw:(Wear\$, dental)) OR (tw:(Dental wear)) OR (tw:(Wear\$, tooth)) OR (tw:(Tooth wear\$)) OR (tw:(Teeth abfraction\$)) OR (tw:(Restoration, permanent dental)) OR (tw:(Dental restoration, permanent)) OR (tw:(Restorations, permanent dental)) OR (tw:(Dental restorations, permanent)) OR (tw:(Permanent Filling, Dental)) OR (tw:(Permanent Fillings, Dental)) OR (tw:(Dental Filling, Permanent)) OR (tw:(Dental Fillings, Permanent)) OR (tw:(Permanent dental fillings)) OR (tw:(Dental permanent filling\$)) OR (tw:(Cervical lesion\$)) OR (tw:(Class V)) OR (tw:(NCCL\$)) OR (tw:(Cervical filling)) OR (tw:(Non-cariou cervical lesion\$)) OR (tw:(Noncarious cervical lesion\$)) OR (tw:(Cervical restoration)) OR (tw:(Class V lesion\$)) OR (tw:(Cervical fillings)) #2 (mh:(Bis-GMA, BPD, HEMA dental-bonding resin)) OR (mh:(Dental bonding)) OR (mh:(Dentin-Bonding Agents)) OR (mh:(hydroxyethyl methacrylate)) OR (mh:(Bisphenol A-Glycidyl Methacrylate)) OR (mh:(Dental Cements)) OR (mh:(Solvents)) OR (mh:(Ethanol)) OR (mh:(Acetone)) OR (tw:(Bonding, Dental)) OR (tw:(Dental Cements Curing)) OR (tw:(Dental Cement\$ Curing)) OR (tw:(Agent\$, Dentin Bonding)) OR (tw:(Curing, Dental Cement\$)) OR (tw:(Dental Cement Curing)) OR (tw:(Bonding Agents, Dentin)) OR (tw:(Bonding Agent\$, Dentin)) OR (tw:(Bonding Agent, Dentin)) OR (tw:(Bis-GMA)) OR (tw:(Adhesive system\$)) OR (tw:(Dentin bonding system\$)) OR (tw:(HEMA)) OR (tw:(hydroxyethyl methacrylate)) OR (tw:(2-hydroxyethyl methacrylate)) OR (tw:(glycol methacrylate)) OR (tw:(“Bisphenol A-Glycidyl Methacrylate”)) OR (tw:(Solvent\$)) OR (tw:(Methacrylate, Bisphenol A-Glycidyl)) OR (tw:(BisGMA)) OR (tw:(“Bisphenol A Glycidyl Methacrylate Polymer”)) OR (tw:(Dentin bonding)) OR (tw:(Ethanol)) OR (tw:(Alcohol, Ethyl)) OR (tw:(Alcohol, Absolute)) OR (tw:(Alcohol, Grain)) OR (tw:(Absolute Alcohol)) OR (tw:(Acetone\$)) OR (tw:(Cement\$, Dental)) OR (tw:(Cement, Dental)) OR (tw:(Adhesive\$, Dental)) OR (tw:(Self-etch adhesive\$)) OR (tw:(Self-etching adhesive\$)) OR (tw:(Etch-and-rinse adhesive)) OR (tw:(Adhesive material\$)) OR (tw:(Universal adhesive\$)) OR (tw:(Total-etch adhesive\$)) OR (tw:(All-in-one adhesive\$)) OR (tw:(One-bottle adhesive\$)) OR (tw:(Etch-and-rinse bonding agents)) OR (tw:(Total-etch bonding agents)) OR (tw:(Self etching bonding agent\$)) OR (tw:(Multimode adhesive\$)) OR (tw:(Self-etch bonding agent\$)) OR (tw:(Universal bonding agent\$)) OR (tw:(One-bottle bonding agent\$)) OR (tw:(“All-in-one bonding agent”)) OR (tw:(Total-etch bonding agent)) OR (tw:(“all in one bonding agents”)) OR (tw:(“all in one bonding agent”)) #3 (tw:(Clinical)) OR (tw:(Randomized)) OR (tw:(Intervention Study)) OR (tw:(Intervention Studies)) OR (tw:(Controlled Trial\$)) OR (tw:(Prospective)) OR (tw:(Follow-up\$)) OR (tw:(follow up)) OR (tw:(Trial\$)) OR (tw:(Longitudinal)) OR (tw:(Quasi-Experimental)) OR (tw:(Non-Randomized)) OR (tw:(Nonrandomized)) #1 AND #2 AND #3

quality of evidence may vary from very low to high. Two GRADEs were performed: one for each clinical parameter and overall clinical performance and another for overall survival rates.²⁵

RESULTS

Study Selection

After an electronic search on all the aforementioned databases, 7876 studies were exported: 2826 duplicates were removed, and 4907 studies were excluded by title and abstract screening. One hundred forty-three studies were analyzed regarding eligibility. Four laboratory studies, 17 conference papers, 33 studies comparing two or more bonding agents with the same solvent composition, 52 studies that used only one bonding agent, and 10 studies that compared bonding agents containing alcohol/acetone/water with another water-based adhesive system were excluded. Thus, 27 remaining studies were included in the present systematic review, and 10 of these were used for the meta-analyses (Figure 1).^{14,26-34} Also, the handmade search returned no relevant papers.

Characteristics of the Selected Studies

Information about the 27 studies included in this systematic review is presented in Table 2 (supplemental material). All studies followed the randomized controlled clinical trial design, and they were developed in Australia, Belgium, Brazil, Egypt, Germany, Italy, Japan, Sweden, the United States, and Turkey. The selected papers were published between 2001 and 2019, comprising a total of 3959 dental restorations in 1087 patients, with follow-up periods ranging from 18 to 72 months. Twenty-two of the included studies used the modified United States Public Health Service (USPHS) criteria to evaluate dental restorations,^{12,14,19,26-31,34-46} whereas four studies used the FDI World Dental Federation criteria,^{15,20,32,33} and one study used its own customized criteria.¹³ All studies compared composite restorations performed using at least one bonding agent with acetone-based solvent against one bonding agent with alcohol-based solvent. Table 3 (supplemental material) presents the composition of the dental adhesives from the studies included in the meta-analyses, as well as adhesive classification: six 2E&R, one 3E&R, one 4E&R, seven 1SE, and two

Table 1: *Electronic Databases Searched and Strategies Used (up to August 9, 2018) (cont.)*

Electronic Database	Strategy Used
SCOPUS (n=3771)	#1 TITLE-ABS-KEY ("tooth erosion") OR TITLE-ABS-KEY ("tooth abrasion") OR TITLE-ABS-KEY ("tooth wear") OR TITLE-ABS-KEY ("dental restoration, permanent") OR TITLE-ABS-KEY (erosion*, AND tooth) OR TITLE-ABS-KEY (tooth AND erosion*) OR TITLE-ABS-KEY ("abrasion, dental") OR TITLE-ABS-KEY ("abrasion, tooth") OR TITLE-ABS-KEY ("teeth abrasions") OR TITLE-ABS-KEY (tooth AND abrasion*) OR TITLE-ABS-KEY ("teeth abrasion") OR TITLE-ABS-KEY (wear*, AND dental) OR TITLE-ABS-KEY ("dental wear") OR TITLE-ABS-KEY (wear*, AND tooth) OR TITLE-ABS-KEY (tooth AND wear*) OR TITLE-ABS-KEY (teeth AND abfraction*) OR TITLE-ABS-KEY ("restoration, permanent dental") OR TITLE-ABS-KEY ("dental restoration, permanent") OR TITLE-ABS-KEY ("restorations, permanent dental") OR TITLE-ABS-KEY ("dental restorations, permanent") OR TITLE-ABS-KEY ("permanent filling, dental") OR TITLE-ABS-KEY ("permanent fillings, dental") OR TITLE-ABS-KEY ("dental filling, permanent") OR TITLE-ABS-KEY ("dental fillings, permanent") OR TITLE-ABS-KEY ("permanent dental fillings") OR TITLE-ABS-KEY (dental AND permanent AND filling*) OR TITLE-ABS-KEY (cervical AND lesion*) OR TITLE-ABS-KEY ("class v") OR TITLE-ABS-KEY (nccl*) OR TITLE-ABS-KEY ("cervical filling") OR TITLE-ABS-KEY (non-carious AND cervical AND lesion*) OR TITLE-ABS-KEY (noncarious AND cervical AND lesion*) OR TITLE-ABS-KEY ("cervical restoration") OR TITLE-ABS-KEY (class AND v AND lesion*) OR TITLE-ABS-KEY ("cervical fillings") #2 TITLE-ABS-KEY("Bis-GMA, BPDm, HEMA dental-bonding resin") OR TITLE-ABS-KEY("Dental bonding") OR TITLE-ABS-KEY("Dentin-Bonding Agents") OR TITLE-ABS-KEY("hydroxyethyl methacrylate") OR TITLE-ABS-KEY("Bisphenol A-Glycidyl Methacrylate") OR TITLE-ABS-KEY("Dental Cements") OR TITLE-ABS-KEY("Solvents") OR TITLE-ABS-KEY("Ethanol") OR TITLE-ABS-KEY("Acetone") OR TITLE-ABS-KEY("Bonding, Dental") OR TITLE-ABS-KEY("Dental Cements Curing") OR TITLE-ABS-KEY("Dental Cement* Curing") OR TITLE-ABS-KEY("Agent*", Dentin Bonding) OR TITLE-ABS-KEY("Curing, Dental Cement") OR TITLE-ABS-KEY("Dental Cement Curing") OR TITLE-ABS-KEY("Bonding Agents, Dentin") OR TITLE-ABS-KEY("Bonding Agent*", Dentin) OR TITLE-ABS-KEY("Bonding Agent, Dentin") OR TITLE-ABS-KEY("Bis-GMA") OR TITLE-ABS-KEY("Adhesive system*") OR TITLE-ABS-KEY("Dentin bonding system*") OR TITLE-ABS-KEY("HEMA") OR TITLE-ABS-KEY("hydroxyethyl methacrylate") OR TITLE-ABS-KEY("2-hydroxyethyl methacrylate") OR TITLE-ABS-KEY("glycol methacrylate") OR TITLE-ABS-KEY("Bisphenol A-Glycidyl Methacrylate") OR TITLE-ABS-KEY("Solvent*") OR TITLE-ABS-KEY("Methacrylate, Bisphenol A-Glycidyl") OR TITLE-ABS-KEY("BisGMA") OR TITLE-ABS-KEY("Bisphenol A Glycidyl Methacrylate Polymer") OR TITLE-ABS-KEY("Dentin bonding") OR TITLE-ABS-KEY("Ethanol") OR TITLE-ABS-KEY("Alcohol, Ethyl") OR TITLE-ABS-KEY("Alcohol, Absolute") OR TITLE-ABS-KEY("Alcohol, Grain") OR TITLE-ABS-KEY("Absolute Alcohol") OR TITLE-ABS-KEY("Acetone") OR TITLE-ABS-KEY("Cement*", Dental) OR TITLE-ABS-KEY("Cement, Dental") OR TITLE-ABS-KEY("Adhesive*", Dental) OR TITLE-ABS-KEY("Self-etch adhesive*") OR TITLE-ABS-KEY("Self-etching adhesive*") OR TITLE-ABS-KEY("Etch-and-rinse adhesive") OR TITLE-ABS-KEY("Adhesive material*") OR TITLE-ABS-KEY("Universal adhesive*") OR TITLE-ABS-KEY("Total-etch adhesive*") OR TITLE-ABS-KEY("All-in-one adhesive*") OR TITLE-ABS-KEY("One-bottle adhesive*") OR TITLE-ABS-KEY("Etch-and-rinse bonding agents") OR TITLE-ABS-KEY("total-etch bonding agents") OR TITLE-ABS-KEY("Self etching bonding agent*") OR TITLE-ABS-KEY("Multimode adhesive*") OR TITLE-ABS-KEY("Self-etch bonding agent*") OR TITLE-ABS-KEY("Universal bonding agent*") OR TITLE-ABS-KEY("One-bottle bonding agent*") OR TITLE-ABS-KEY("All-in-one bonding agent") OR TITLE-ABS-KEY("Total-etch bonding agent") OR TITLE-ABS-KEY("all in one bonding agents") OR TITLE-ABS-KEY("all in one bonding agent") #3 TITLE-ABS-KEY("Clinical") OR TITLE-ABS-KEY("Randomized") OR TITLE-ABS-KEY("Intervention Study") OR TITLE-ABS-KEY("Intervention Studies") OR TITLE-ABS-KEY("Controlled Trial*") OR TITLE-ABS-KEY("Prospective") OR TITLE-ABS-KEY("Follow-up*") OR TITLE-ABS-KEY("follow up") OR TITLE-ABS-KEY("Trial*") OR TITLE-ABS-KEY("Longitudinal") OR TITLE-ABS-KEY("Quasi-Experimental") OR TITLE-ABS-KEY("Non-Randomized") OR TITLE-ABS-KEY("Nonrandomized") #1 AND #2 AND #3

"universal" (2E&R or 1SE). Also, pH assessment is described for self-etch systems (eight mild/intermediately strong and one strong).

Risk of Bias Assessment

Four of the 27 selected studies were rated as unclear for random sequence generation.^{13,35,36,45} Two studies were unclear,^{39,45} whereas one study¹³ did not perform the allocation concealment. Twenty-two papers were rated as unclear regarding the blinding of participants and personnel.^{13-15,19,20,30-46} However, this third domain was not regarded as a key domain in the risk of bias assessment. Eight studies

were classified as unclear,^{19,35,37-39,43-45} and one did not blind the evaluators.¹³ Ten studies presented high risk of bias for the incomplete outcome data domain for the following reasons: the clinical criteria used to assess the results were not described^{13,45}; only alpha scores were reported^{36-38,41-44}; or only bravo scores were reported.¹² Five studies showed high risk of bias for selective reporting,^{12,13,15,20,45} and six studies presented other bias.^{20,38-40,42,46}

Four papers were classified as low risk of bias.²⁶⁻²⁹ Nevertheless, six other papers were also included as low risk of bias,^{14,30-34} regardless of being checked as unclear for the blinding of participants and personnel, because this domain was not considered a key

Table 1: *Electronic Databases Searched and Strategies Used (up to August 9, 2018) (cont.)*

Electronic Database	Strategy Used
Web of Science (n=1371)	#1 TS=(Tooth erosion OR Tooth abrasion OR Tooth wear OR Dental restoration, permanent OR Erosion*, tooth OR Tooth erosion* OR Abrasion, dental OR Abrasion, tooth OR Teeth abrasions OR Tooth abrasion* OR Teeth abrasion OR Wear*, dental OR Dental wear OR Wear*, tooth OR Tooth wear* OR Teeth abfraction* OR Restoration, permanent dental OR Dental restoration, permanent OR Restorations, permanent dental OR Dental restorations, permanent OR Permanent Filling, Dental OR Permanent Fillings, Dental OR Dental Filling, Permanent OR Dental Fillings, Permanent OR Permanent dental fillings OR Dental permanent filling* OR Cervical lesion* OR Class V OR NCCL* OR Cervical filling OR Non-carious cervical lesion* OR Noncarious cervical lesion* OR Cervical restoration OR Class V lesion* OR Cervical fillings) #2 TS=(Bis-GMA, BPDM, HEMA dental-bonding resin OR Dental bonding OR Dentin-Bonding Agents OR hydroxyethyl methacrylate OR Bisphenol A-Glycidyl Methacrylate OR Dental Cements OR Solvents OR Ethanol OR Acetone OR Bonding, Dental OR Dental Cements Curing OR Dental Cement* Curing OR Agent*, Dentin Bonding OR Curing, Dental Cement* OR Dental Cement Curing OR Bonding Agents, Dentin OR Bonding Agent*, Dentin OR Bonding Agent, Dentin OR Bis-GMA OR Adhesive system* OR Dentin bonding system* OR HEMA OR hydroxyethyl methacrylate OR 2-hydroxyethyl methacrylate OR glycol methacrylate OR "Bisphenol A-Glycidyl Methacrylate" OR Solvent* OR Methacrylate, Bisphenol A-Glycidyl OR BisGMA OR "Bisphenol A Glycidyl Methacrylate Polymer" OR Dentin bonding OR Ethanol OR Alcohol, Ethyl OR Alcohol, Absolute OR Alcohol, Grain OR Absolute Alcohol OR Acetone* OR Cement*, Dental OR Cement, Dental OR Adhesive*, Dental OR Self-etch adhesive* OR Self-etching adhesive* OR Etch-and-rinse adhesive OR Adhesive material* OR Universal adhesive* OR Total-etch adhesive* OR All-in-one adhesive* OR One-bottle adhesive* OR Etch-and-rinse bonding agents OR Total-etch bonding agents OR Self etching bonding agent* OR Multimode adhesive* OR Self-etch bonding agent* OR Universal bonding agent* OR One-bottle bonding agent* OR "All-in-one bonding agent" OR Total-etch bonding agent OR "all in one bonding agents" OR "all in one bonding agent") #3 TS=(Clinical OR Randomized OR Intervention Study OR Intervention Studies OR Controlled Trial* OR Prospective OR Follow-up* OR follow up OR Trial* OR Longitudinal OR Quasi-Experimental OR Non-Randomized OR Nonrandomized) #1 AND #2 AND #3
REBEC (n=0)	Dental adhesives and non-carious cervical lesions
Gray literature (n=0)	Dental adhesives AND non-carious cervical lesions
Clinical trials (n=12)	Dental adhesives OR dentin bonding agents OR ethanol OR acetone non-carious cervical lesions OR noncarious cervical lesions

domain. The summary of risk of bias assessment is presented in Figure 2.

Meta-analyses

The meta-analysis included only papers with a low risk of bias, and the available data for each clinical parameter and overall survival rates were taken into account. Two meta-analyses were carried out for 10 of the selected studies^{14,26-34}: one for the clinical evaluation parameters and another one for the overall survival rates at different follow-up periods. Considering not all studies used the same clinical evaluation criteria, and the selected studies presented different follow-up periods, different numbers of studies were included in the meta-analyses for different clinical parameters. Although the study by Häfer and others²⁶ was classified as low risk of bias, it could not be used for the meta-analysis regarding clinical evaluation parameters, because this clinical trial used customized clinical evaluation criteria, which made it impossible to compare its results with the remaining studies. However, as survival rates could still be extracted from this paper, it was included in the second meta-analysis,

resulting in different numbers of studies for each one of the two meta-analyses.

In the first meta-analysis (Figure 3), which included nine studies, two studies did not provide data for marginal adaptation,^{32,33} seven studies did not provide data for surface texture and color,^{14,27,29,30,32-34} three studies did not provide data for postoperative sensitivity,^{14,27,31} one study did not provide data for secondary caries,²⁷ seven studies did not provide data for anatomic form,^{14,27,28,30,32-34} and one study supplied sufficient data only for the pulp vitality parameter.³⁰

In the second meta-analysis (Figure 4), which included 10 studies, two studies did not provide information for survival rates at six months,^{27,30} two studies did not provide data at 12 months,^{27,29} eight studies did not provide data at 18 months,^{14,26,27,30-34} three studies did not provide data at 24 months,²⁷⁻²⁹ six studies did not provide data at 36 months,^{14,27-29,31,34} and only two studies provided data at 60³³ and 72 months.²⁷

The overall heterogeneity between studies was not significant ($I^2 = 0.00\%$) for both meta-analyses. The heterogeneity values for each clinical evaluation

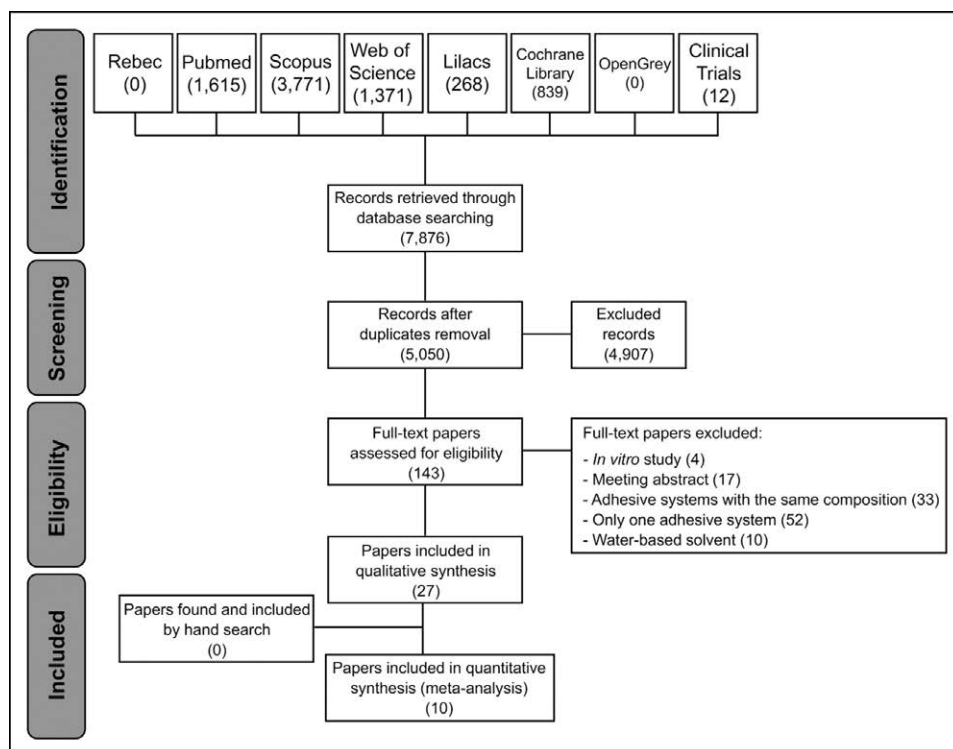


Figure 1. Flow diagram of the identified, eligible, and included studies of this systematic review and meta-analysis.

parameter were also not significant, ranging from 0% to 38% (38% for sensitivity, 34% for retention, and 0% for the remaining criteria). The heterogeneity for survival rates at each follow-up period was not significant either, except for the 12- and 18-month follow-ups, ranging from 0% to 88% (88% for 18 months, 62% for 12 months, 46% for 24 months, 0% for all the other follow-up periods).

The overall risk difference of each clinical evaluation parameter was 0.00 [−0.01, 0.00] ($p=0.57$), whereas it was −0.01 [−0.04, 0.02] ($p=0.46$) for retention, 0.00 [−0.02, 0.01] ($p=0.82$) for marginal adaptation, 0.00 [−0.01, 0.01] ($p=0.63$) for marginal discoloration, 0.00 [−0.04, 0.04] ($p=1.00$) for surface texture, 0.00 [−0.04, 0.04] ($p=1.00$) for color, −0.01 [−0.04, 0.02] ($p=0.65$) for sensitivity, 0.00 [−0.01, 0.01] ($p=1.00$) for secondary caries, 0.00 [−0.03, 0.03] ($p=1.00$) for anatomic form, and 0.00 [−0.04, 0.04] ($p=1.00$) for pulp vitality.

The overall risk of difference for survival rates was 0.00 [−0.01, 0.01] ($p=0.91$), whereas it was 0.00 [−0.01, 0.01] ($p=0.99$) for six months, 0.00 [−0.02, 0.02] ($p=0.88$) for 12 months, −0.14 [−0.39, 0.11] ($p=0.27$) for 18 months, 0.01 [−0.02, 0.04] ($p=0.52$) for 24 months, 0.00 [−0.04, 0.03] ($p=0.91$) for 36 months, −0.01 [−0.08, 0.06] ($p=0.82$) for 60 months, and −0.04 [−0.20, 0.12] ($p=0.65$) for 72 months (Figures 3 and 4).

High quality of evidence by the GRADE approach was evidenced for both meta-analyses (Tables 4 and 5), with very strong association of at least 919 events per 1000. Visual inspection of the funnel plot revealed a symmetric distribution, which suggests there were no publication biases for survival rates (Figure 5). The authors would also like to highlight that a meta-analysis including all studies, regardless of their risk of bias, was performed previously to the meta-analysis hereby presented, and the significance of their results, as well as the certainty of evidence, were similar to the statistical analysis included in the present study (that considered only studies with low risk of bias). Therefore, the authors opted to include only the last meta-analysis, without high risk of bias studies.

DISCUSSION

Several systematic reviews and meta-analyses regarding the differences in clinical performance between SE and E&R bonding agents can be found in the scientific literature.^{3,47-50} However, to the knowledge of the authors of this article, there is no review dealing with the potential influence of different solvents contained in bonding agents on the clinical outcomes of direct composite restorations in NCCLs. Therefore, the aim of this systematic review and meta-analysis was to compare the

	Random sequence generation (selection bias)	Allocation concealment (selection bias)	Blinding of participants and personnel (performance bias)	Blinding of outcome assessment (detection bias)	Incomplete outcome data (attrition bias)	Selective reporting (reporting bias)	Other bias
Abdalla & García-Godoy ²⁵	?	+	?	?	+	+	+
Boushell and others ²⁷	+	+	+	+	+	+	+
Burrow & Tyas ¹²	?	-	?	-	-	-	+
Celik and others ¹⁹	+	+	?	?	+	+	+
Häfer and others ²⁶	+	+	+	+	+	+	+
Kubo and others ¹⁹	+	+	?	+	+	+	+
Moretto and others ¹⁵	+	+	?	+	+	-	+
Oz and others ²⁸	+	+	+	+	+	+	+
Perdigão and others ²⁶	?	+	?	+	-	+	+
Peumans and others ²⁸	+	+	?	+	+	-	-
Reis & Loguercio ¹²	+	+	+	+	-	-	+
Reis and others ²⁹	+	+	+	+	+	+	+
Ritter and others ²⁷	+	+	?	?	-	+	+
Ritter and others ²⁸	+	+	?	?	-	+	-
Ruschel and others ²⁹	+	?	?	?	+	+	-
Saboia and others ⁴⁰	+	+	?	+	+	+	-
Sartori and others ⁴²	+	+	?	+	-	+	-
Sartori and others ⁴¹	+	+	?	+	-	+	+
Scotti and others ²⁸	+	+	?	+	+	+	+
Swift Jr and others ⁴²	+	+	?	?	-	+	+
Swift Jr and others ⁴⁴	+	+	?	?	-	+	+
Tuncer and others ³¹	+	+	?	+	+	+	+
van Dijken & Pallesen ⁴⁵	?	?	?	?	-	-	+
Van Dijken ⁴⁶	+	+	?	+	+	+	-
Van Landuyt and others ²⁸	+	+	?	+	+	+	+
Van Landuyt and others ²⁸	+	+	?	+	+	+	+
Zander-Grande and others ²⁴	+	+	?	+	+	+	+

Figure 2. Risk of bias assessment for the studies included in this systematic review and meta-analysis: (+) low; (?) unclear; or (-) high risk of bias.

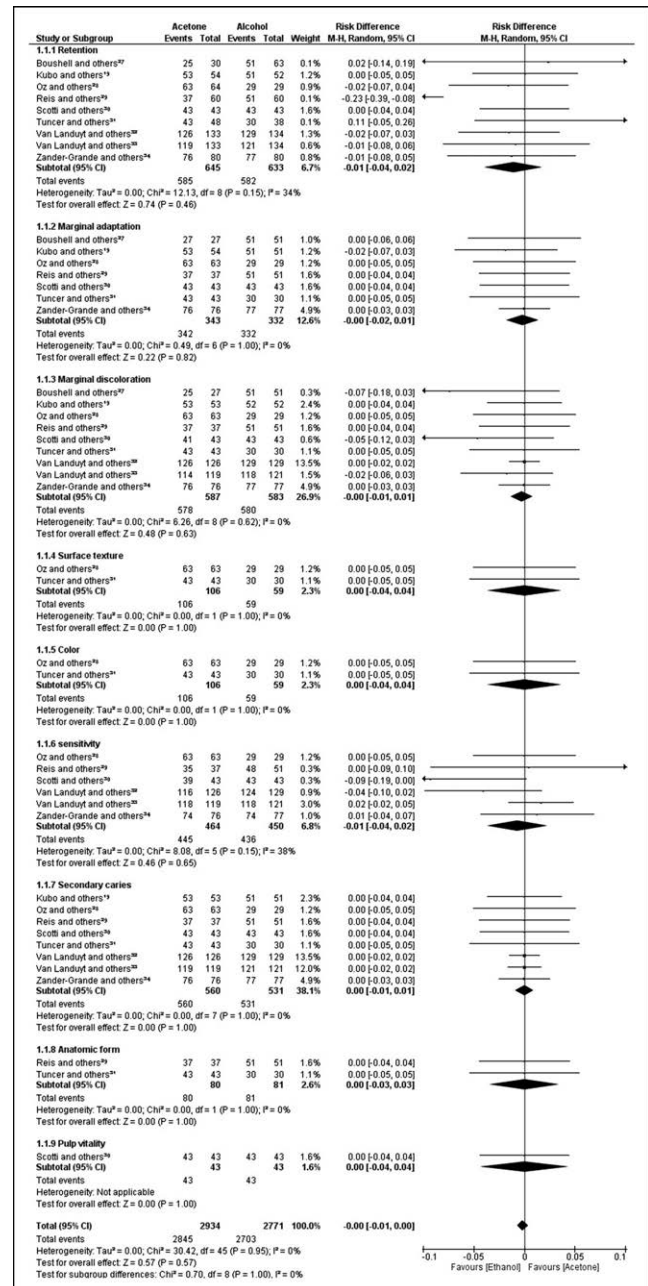


Figure 3. Forest plot of clinical evaluation parameters of acetone- vs alcohol-based bonding agents with all studies included in this systematic review and meta-analysis.

clinical evaluation parameters and survival rates of composite restorations placed in NCCLs using acetone- or alcohol-based dental adhesives that have been reported in randomized controlled clinical trials.

In the present paper, all risk of bias assessment criteria were considered key domains, except for the blinding of participants and personnel. Random

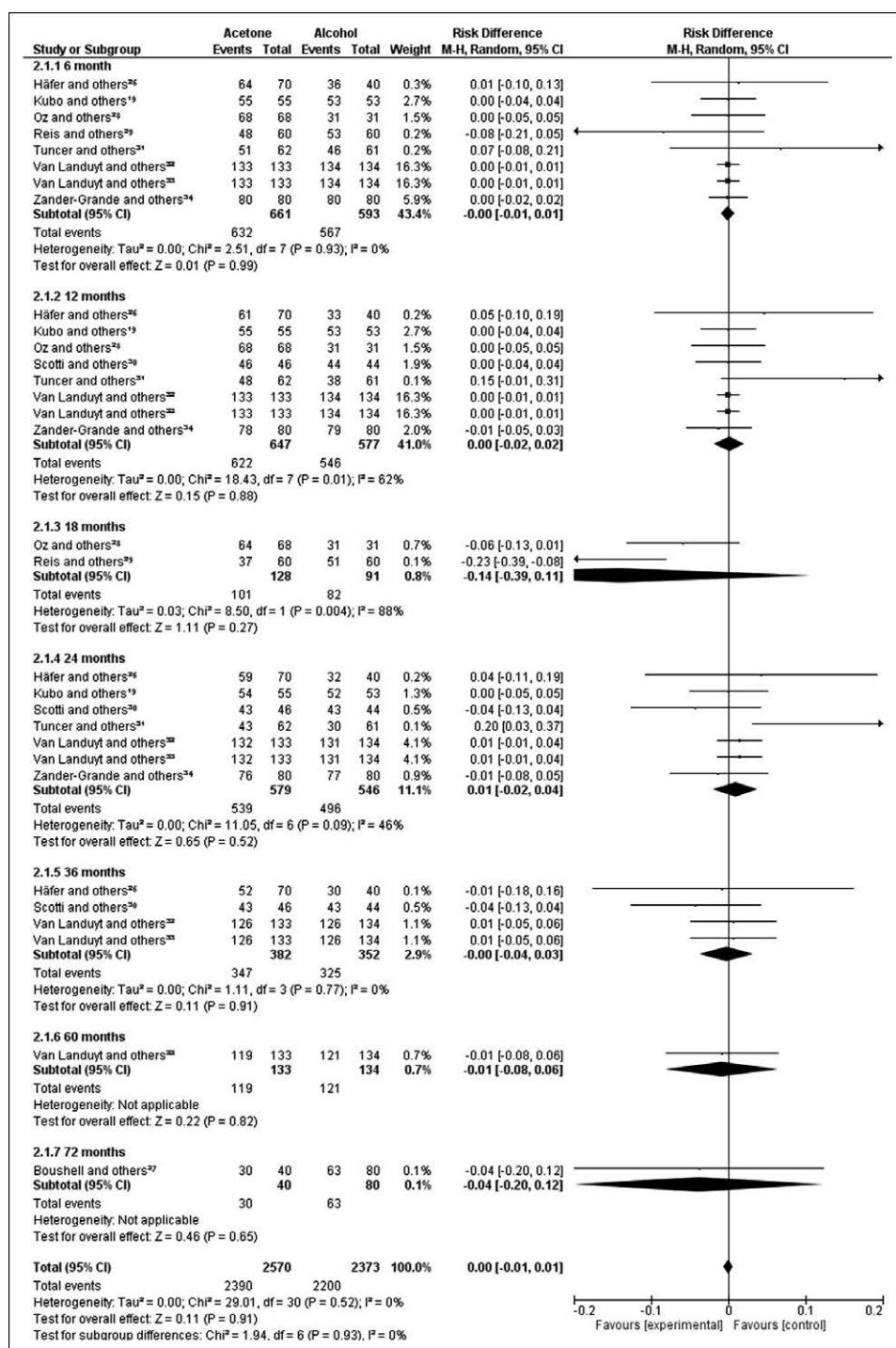


Figure 4. Forest plot of survival rates at different follow-up periods of acetone- × alcohol-based bonding agents with all studies included in this systematic review and meta-analysis.

sequence generation and allocation concealment were the first two criteria analyzed for each of the selected papers. Random sequence generation describes the method used for randomization, and allocation concealment ensures no intervention allocations could have been foreseen before or during patient enrollment.⁵¹ Randomization is essential in

clinical trials to guarantee that participants of both intervention and comparison groups present similar known and unknown prognostic factors.⁵¹ Another critical domain to avoid the selection of biased RCTs is the blinding of outcomes assessors, as it reduces the risk of detection bias (eg, observer, ascertainment, or assessment bias), and it also has a strong

< 1.5 had an inferior performance in comparison.⁶ Nonetheless, the mode of action of the adhesives evaluated in this systematic review and meta-analysis seemed to have played a minor role on their performance related to solvent content, which might have happened because both groups (alcohol- and acetone-based) presented even numbers of E&R and SE adhesive systems (four E&R, four SE, and one “universal” for alcohol-based; and four E&R, three SE, and one “universal” for the acetone-based).

Moreover, the role of solvent type might be more important for E&R adhesives, because they involve a prior dentin-etching step, and the collagen network might collapse during air drying,^{5,59} making these products more sensitive to operator mistakes. SE adhesives, on the other hand, need the addition of a solvent to their composition only to ensure ionization of functional monomers,⁵ but dentin demineralization and resin infiltration occur simultaneously, likely without a huge influence of solvent type. However, another more in-depth systematic review and meta-analysis would be necessary to correlate solvent type and mode of action of dental adhesive systems, as well as to analyze all subclasses of bonding agents, which is beyond the scope of the present paper.

Thus, the first null hypothesis was accepted. Different solvents have been suggested to affect the clinical performance of dental adhesives, because their type and concentration might impact the adhesive tolerance to dentin moisture.²⁸ Based on previous *in vitro* publications, one could expect the clinical performance of composite restorations placed using acetone-based bonding agents to be worse than alcohol-based bonding agents, considering acetone-containing products are believed to form thinner adhesive layers that are more prone to polymerization inhibition by oxygen and polymer degradation.¹² Also, increasing concentrations of acetone have been shown to negatively affect the microtensile bond strength of adhesive formulations.⁶⁰

Both high-vapor pressure solvents (acetone and alcohol) can wet the dentin surface and promote effective bonding, as evidenced by the results of the present systematic review and meta-analysis. The most likely influence of solvents on adhesive systems might be related to their application method^{16,59}: bonding agents with acetone are more sensitive to the air-drying of etched dentin, as this solvent cannot re-expand the shrunken collagen network.¹⁷ Acetone also tends to quickly dehydrate the etched dentin surface, because it has higher vapor pressure than ethanol and water. However, most of the trials included in the meta-analyses reported trained and/

or experienced operators performed the composite restorations in a controlled clinical situation,^{14,26,29,30,32-34} which might have led to similar results for both solvents, because the effects of operator mistakes were dramatically reduced due to operators strictly following the manufacturers' instructions.

Other aspects occasionally reported in RCTs involving dental adhesives are surface texture, color, and anatomic form of composite restorations. Although the meta-analysis revealed no differences between restorations performed with acetone- or alcohol-containing bonding agents, these parameters are much more related to the performance of dental composites than to bonding agents themselves, as they describe surface characteristics instead of dentin bonding parameters. Moreover, postoperative sensitivity is another important clinical evaluation parameter, because it is believed to be caused by a modification in the hydrodynamics of dentinal fluids due to incomplete adhesive penetration or inadequate hybridization.⁴⁷ The meta-analysis showed a similar performance for postoperative sensitivity between adhesive systems containing distinct solvents, and no secondary carious lesions were reported for any of the types of bonding agents. The results for these two parameters demonstrate that acetone- and alcohol-based adhesives present adequate bonding and comparable dentin sealing ability, providing long-lasting composite restorations.

A second meta-analysis was performed to compare survival rates of composite restorations placed using acetone- or alcohol-based adhesive systems over different follow-up periods (Figure 4). Survival rates can be described as the percentage of composite restorations that did not fail (loss or need of replacement/repair) at a certain evaluation time.^{56,61} Once again, there was no statistical difference between the two solvents, showing that composite restorations placed using both types of adhesives performed favorably in clinical trials with follow-ups ranging from 6 to 72 months. Hence, the second null hypothesis was also accepted. However, some of these results should be interpreted with caution. The follow-ups of 12 and 18 months presented high heterogeneity among studies (62% and 88%, respectively), which means the extracted data from the selected set of clinical trials varied from one to another,²² leading to more favorable survival rates (although not statistically significant) for restorations placed using alcohol-based bonding agents, especially at 18 months. Also, only one publication reported data for the 60-month follow-up,³³ and the

Table 4. Evidence Profile: Clinical Performance of NCCL Restorations With Acetone- and Alcohol-Based Dentin Bonding Agents

Certainty Assessment						
No. of Participants (Studies)	Risk of Bias	Inconsistency	Indirectness	Imprecision	Other Considerations	Overall Certainty of Evidence
Retention						
1278 (9 RCTs)	Not serious	Not serious	Not serious	Not serious	Very strong association	HIGH
Marginal adaptation						
675 (7 RCTs)	Not serious	Not serious	Not serious	Not serious	Very strong association	HIGH
Marginal discoloration						
1170 (9 RCTs)	Not serious	Not serious	Not serious	Not serious	Very strong association	HIGH
Surface texture						
165 (2 RCTs)	Not serious	Not serious	Not serious	Serious ^a	Very strong association	HIGH
Color						
165 (2 RCTs)	Not serious	Not serious	Not serious	Serious ^a	Very strong association	HIGH
Sensitivity						
914 (6 RCTs)	Not serious	Serious ^b	Not serious	Not serious	Very strong association	HIGH
Secondary caries						
1091 (8 RCTs)	Not serious	Not serious	Not serious	Not serious	Very strong association	HIGH
Anatomic form						
161 (2 RCTs)	Not serious	Not serious	Not serious	Serious ^a	Very strong association	HIGH
Pulp vitality						
86 (1 RCT)	Not serious	Not serious	Not serious	Serious ^a	Very strong association	HIGH
Clinical performance overall						
5705 (9 RCTs)	Not serious	Not serious	Not serious	Not serious	Very strong association	HIGH
Abbreviation: CI, confidence interval. ^a Number of events is lower than 300. ^b Little variation in the effect estimates across studies.						

72-month follow-up analysis also consisted of data extracted from a single study.²⁷ Thus, more long-term clinical trials are necessary to allow for a reliable prediction of the performance of composite

restorations placed using acetone- or alcohol-based bonding agents over time.

Methodologic variability is a limitation that must be considered in the present meta-analyses. Beveling

Table 5: Evidence Profile: Survival Rate of NCCL Restorations With Acetone- and Alcohol-Based Dentin Bonding Agents

Certainty Assessment						
No. of Participants (Studies)	Risk of Bias	Inconsistency	Indirectness	Imprecision	Other Considerations	Overall Certainty of Evidence
Survival Rate Overall						
4943 (10 RCTs)	Not serious	Serious ^a	Not serious	Not serious	Very strong association	HIGH
Abbreviation: CI, confidence interval. ^a Little variation in the effect estimates across studies.						

Table 4. Evidence Profile: Clinical Performance of NCCL Restorations With Acetone- and Alcohol-Based Dentin Bonding Agents (ext.)

Certainty Assessment		Summary of Findings			
No. of Participants (Studies)	Study Event Rates, No./Total (%)		Relative Effect (95% CI)	Anticipated Absolute Effects	
	With Alcohol	With Acetone		Risk With Alcohol	Risk Difference With Acetone
Retention					
1278 (9 RCTs)	582/633 (91.9)	585/645 (90.7)	Not estimable	919 per 1000	919 fewer per 1000 (919 fewer to 919 fewer)
Marginal adaptionation					
675 (7 RCTs)	332/332 (100.0)	342/343 (99.7)	Not estimable	1000 per 1000	1000 fewer per 1000 (1000 fewer to 1000 fewer)
Marginal discoloration					
1170 (9 RCTs)	580/583 (99.5)	578/587 (98.5)	Not estimable	995 per 1000	995 fewer per 1000 (995 fewer to 995 fewer)
Surface texture					
165 (2 RCTs)	59/59 (100.0)	106/106 (100.0)	Not estimable	1000 per 1000	1000 fewer per 1000 (1000 fewer to 1000 fewer)
Color					
165 (2 RCTs)	59/59 (100.0)	106/106 (100.0)	Not estimable	1000 per 1000	1000 fewer per 1000 (1000 fewer to 1000 fewer)
Sensitivity					
914 (6 RCTs)	436/450 (96.9)	445/464 (95.9)	Not estimable	969 per 1000	969 fewer per 1000 (969 fewer to 969 fewer)
Secondary caries					
1091 (8 RCTs)	531/531 (100.0)	560/560 (100.0)	Not estimable	1000 per 1000	1000 fewer per 1000 (1000 fewer to 1000 fewer)
Anatomic form					
161 (2 RCTs)	81/81 (100.0)	80/80 (100.0)	Not estimable	1000 per 1000	1000 fewer per 1000 (1000 fewer to 1000 fewer)
Pulp vitality					
86 (1 RCT)	43/43 (100.0)	43/43 (100.0)	Not estimable	1000 per 1000	1000 fewer per 1000 (1000 fewer to 1000 fewer)
Clinical performance overall					
5705 (9 RCTs)	2703/2771 (97.5)	2845/2934 (97.0)	Not estimable	975 per 1000	975 fewer per 1000 (975 fewer to 975 fewer)

of the enamel margins was carried out before restorative procedures in some of the selected studies,^{14,26,30,32,33} whereas other studies did not do it.^{27-29,31,34} Roughening of the superficial, hyper-mineralized dentin surface was performed in most of the selected clinical trials,^{14,26,27,30,32,33} but some of

them did not provide information regarding this aspect.^{28,29,31,34} Some papers included teeth with different levels of dentin sclerosis,^{27,29-34} and all studies had cavities of varied shapes and dimensions. All these clinical aspects can influence micro-retention, which might have affected the results of

Table 5: Evidence Profile: Survival Rate of NCCL Restorations With Acetone- and Alcohol-Based Dentin Bonding Agents (ext.)

Certainty Assessment		Summary of Findings			
No. of Participants (Studies)	Study Event Rates, No./Total (%)		Relative Effect (95% CI)	Anticipated Absolute Effects	
	With Ethanol	With Acetone		Risk With Ethanol	Risk Difference With Acetone
Survival Rate Overall					
4943 (10 RCTs)	2200/2373 (92.7)	2390/2570 (93.0)	Not estimable	927 per 1000	927 fewer per 1000 (927 fewer to 927 fewer)

the meta-analyses performed in this review. Furthermore, all the selected papers^{14,26-34} compared adhesives with not only different solvents, but also with distinct monomer compositions. Therefore, even if some adhesive systems contained a solvent type that could have affected the clinical performance of composite restorations unfavorably, their monomer chemistry might have compensated this disadvantage, improving their clinical results.

CONCLUSION

Based on the results of this systematic review and meta-analysis, there is no significant difference in the clinical performance of dental adhesives based on solvent type (alcohol- or acetone-based), regardless of adhesive mode of action or application.

Acknowledgements

This study was financed in part by the 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 7 February 2020)

REFERENCES

- Stenesh J (1989) *Dictionary of Biochemistry and Molecular Biology* John Wiley, New York.
- Morrison RT & Boyd RN (2002) *Organic Chemistry* Prentice-Hall of India Private Limited, New Dehli.
- Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, & Van Meerbeek B (2005) Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials *Dental Materials* **21**(9) 864-881.
- Manso AP, Marquezini L Jr, Silva SM, Pashley DH, Tay FR, & Carvalho RM (2008) Stability of wet versus dry bonding with different solvent-based adhesives *Dental Materials* **24**(4) 476-482.
- Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, & Van Meerbeek B (2007) Systematic review of the chemical composition of contemporary dental adhesives *Biomaterials* **28**(26) 3757-3785.
- Peumans M, De Munck J, Mine A, & Van Meerbeek B (2014) Clinical effectiveness of contemporary adhesives for the restoration of non-carious cervical lesions. A systematic review *Dental Materials* **30**(10) 1089-1103.
- Duarte S Jr, Perdigão J, & Lopes MM (2006) Effect of dentin conditioning time on nanoleakage *Operative Dentistry* **31**(4) 500-511.
- Zanchi CH, Lund RG, Perrone LR, Ribeiro GA, del Pino FA, Pinto MB, & Demarco FF (2010) Microtensile bond strength of two-step etch-and-rinse adhesive systems on sound and artificial caries-affected dentin *American Journal of Dentistry* **23**(3) 152-156.
- Cardoso PC, Lopes GC, Vieira LCC, & Baratieri LN (2005) Effect of solvent type on microtensile bond strength of a total-etch one-bottle adhesive system to moist or dry dentin *Operative Dentistry* **30**(3) 376-381.
- Loguercio AD, Loeblein F, Cherobin T, Ogliari F, Piva E, & Reis A (2009) Effect of solvent removal on adhesive properties of simplified etch-and-rinse systems and on bond strengths to dry and wet dentin *Journal of Adhesive Dentistry* **11**(3) 213-219.
- Ayar MK (2015) Effect of simplified ethanol-wet bonding on microtensile bond strengths of dentin adhesive agents with different solvents *Journal of Dental Sciences* **10**(3) 270-274.
- Reis A & Loguercio AD (2009) A 36-month clinical evaluation of ethanol/water and acetone-based etch-and-rinse adhesives in non-carious cervical lesions *Operative Dentistry* **34**(4) 384-391.
- Burrow MF & Tyas MJ (2012) Comparison of two all-in-one adhesives bonded to non-carious cervical lesions: results at 3 years *Clinical Oral Investigations* **16**(4) 1089-1094.
- Kubo S, Yokota H, Yokota H, & Hayashi Y (2009) Two-year clinical evaluation of one-step self-etch systems in non-carious cervical lesions *Journal of Dentistry* **37**(2) 149-155.
- Moretto SG, Russo EM, Carvalho RC, De Munck J, Van Landuyt K, Peumans M, Van Meerbeek B, & Cardoso MV (2013) 3-year clinical effectiveness of one-step adhesives in non-carious cervical lesions *Journal of Dentistry* **41**(8) 675-682.
- Irmak O, Baltacıoğlu IH, Ulusoy N, & Bagis YH (2016) Solvent type influences bond strength to air or blot-dried dentin *BMC Oral Health* **16**(1) 77.
- Aguilera FS, Osorio R, Osorio E, Moura P, & Toledano M (2012) Wetting ability of an acetone-based etch&rinse adhesive after NaOCl-treatment *Medicina Oral Patología Oral y Cirugía Bucal* **17**(4) e644-e648.
- Zhang ZY, Tian FC, Niu LN, Ochala K, Chen C, Fu BP, Wang XY, Pashley DH, & Tay FR (2016) Defying ageing: An expectation for dentine bonding with universal adhesives? *Journal of Dentistry* **45** 43-52.
- Celik C, Ozgunaltay G, & Attar N (2007) Clinical evaluation of flowable resins in non-carious cervical lesions: two-year results *Operative Dentistry* **32**(4) 313-321.
- Peumans M, Wouters L, De Munck J, Van Meerbeek B, & Van Landuyt K (2018) Nine-year clinical performance of a HEMA-free one-step self-etch adhesive in noncarious cervical lesions *Journal of Adhesive Dentistry* **20**(3) 195-203.
- Beller EM, Glasziou PP, Altman DG, Hopewell S, Bastian H, Chalmers I, Gotzsche PC, Lasserson T, Tovey D, & Group Pfa (2013) PRISMA for Abstracts: reporting

- systematic reviews in journal and conference abstracts *PLoS Medicine* **10**(4) e1001419.
22. Maia LC & Antonio AG (2012) Systematic reviews in dental research. A guideline *Journal of Clinical Pediatric Dentistry* **37**(2) 117-124.
 23. Higgins JPT, Altman DG, & Sterne JAC (2017) Chapter 8: assessing risk of bias in included studies In: Higgins JPT, Churchill R, Chandler J, Cumpston MS (eds) *Cochrane Handbook for Systematic Reviews of Interventions Version 5.2.0* The Cochrane Collaboration 1-8. Retrieved online September 13, 2018 from: <http://www.training.cochrane.org/handbook>.
 24. Higgins JPT & Green S (2011) *Cochrane Handbook for Systematic Reviews of Interventions Version 5.1.0* The Cochrane Collaboration. Retrieved online September 13, 2018 from: <http://www.handbook.cochrane.org>.
 25. Ryan R & Hill S (2016) How to GRADE the quality of the evidence *Cochrane Consumers and Communication Group Version 3.0* 1-25. Retrieved online November 5, 2018 from: <http://cccr.cochrane.org/author-resources>.
 26. Häfer M, Jentsch H, Haak R, & Schneider H (2015) A three-year clinical evaluation of a one-step self-etch and a two-step etch-and-rinse adhesive in non-carious cervical lesions *Journal of Dentistry* **43**(3) 350-361.
 27. Boushell LW, Heymann HO, Ritter AV, Sturdevant JR, Swift EJ Jr, Wilder AD Jr, Chung Y, Lambert CA, & Walter R (2016) Six-year clinical performance of etch-and-rinse and self-etch adhesives *Dental Materials* **32**(9) 1065-1072.
 28. Oz FD, Kutuk ZB, Ozturk C, Soleimani R, & Gurgan S (2019) An 18-month clinical evaluation of three different universal adhesives used with a universal flowable composite resin in the restoration of non-carious cervical lesions *Clinical Oral Investigations* **23**(3) 1443-1452.
 29. Reis A, Leite TM, Matte K, Michels R, Amaral RC, Geraldini S, & Loguercio AD (2009) Improving clinical retention of one-step self-etching adhesive systems with an additional hydrophobic adhesive layer *Journal of the American Dental Association* **140**(7) 877-885.
 30. Scotti N, Comba A, Gambino A, Manzon E, Breschi L, Paolino D, Pasqualini D, & Berutti E (2016) Influence of operator experience on non-carious cervical lesion restorations: clinical evaluation with different adhesive systems *American Journal of Dentistry* **29**(1) 33-38.
 31. Tuncer D, Yazici AR, Ozgunaltay G, & Dayangac B (2013) Clinical evaluation of different adhesives used in the restoration of non-carious cervical lesions: 24-month results *Australian Dental Journal* **58**(1) 94-100.
 32. Van Landuyt KL, Peumans M, De Munck J, Cardoso MV, Ermis B, & Van Meerbeek B (2011) Three-year clinical performance of a HEMA-free one-step self-etch adhesive in non-carious cervical lesions *European Journal of Oral Sciences* **119**(6) 511-516.
 33. Van Landuyt KL, De Munck J, Ermis RB, Peumans M, & Van Meerbeek B (2014) Five-year clinical performance of a HEMA-free one-step self-etch adhesive in noncarious cervical lesions *Clinical Oral Investigations* **18**(4) 1045-1052.
 34. Zander-Grande C, Ferreira SQ, da Costa TR, Loguercio AD, & Reis A (2011) Application of etch-and-rinse adhesives on dry and rewet dentin under rubbing action: a 24-month clinical evaluation *Journal of the American Dental Association* **142**(7) 828-835.
 35. Abdalla AI & Garcia-Godoy F (2006) Clinical evaluation of self-etch adhesives in Class V non-carious lesions *American Journal of Dentistry* **19**(5) 289-292.
 36. Perdigao J, Carmo AR, & Geraldini S (2005) Eighteen-month clinical evaluation of two dentin adhesives applied on dry vs moist dentin *Journal of Adhesive Dentistry* **7**(3) 253-258.
 37. Ritter AV, Heymann HO, Swift EJ, Jr., Sturdevant JR, & Wilder AD Jr (2008) Clinical evaluation of an all-in-one adhesive in non-carious cervical lesions with different degrees of dentin sclerosis *Operative Dentistry* **33**(4) 370-378.
 38. Ritter AV, Swift EJ, Jr., Heymann HO, Sturdevant JR, & Wilder AD Jr (2009) An eight-year clinical evaluation of filled and unfilled one-bottle dental adhesives *Journal of the American Dental Association* **140**(1) 28-37.
 39. Ruschel VC, Shibata S, Stolf SC, Chung Y, Baratieri LN, Heymann HO, & Walter R (2018) Eighteen-month clinical study of universal adhesives in noncarious cervical lesions *Operative Dentistry* **43**(3) 241-249.
 40. Saboia VPA, Almeida PC, Rittet AV, Swift EJ Jr, & Pimenta LA (2006) 2-year clinical evaluation of sodium hypochlorite treatment in the restoration of non-carious cervical lesions: a pilot study *Operative Dentistry* **31**(5) 530-535.
 41. Sartori N, Pacheco E, Souza PTdR, Lopes GC, & Peruchi LD (2011) Performance clínica de dois sistemas adesivos em lesões cervicais não cariosas: 30 meses de acompanhamento *Arquivos em Odontologia* **47**(3) 119-126.
 42. Sartori N, Lopes GC, & Vieira LC (2012) Clinical performance of cervical restorations with desensitizing agents: 18-month clinical trial *Journal of Adhesive Dentistry* **14**(2) 183-189.
 43. Swift EJ Jr, Perdigao J, Heymann HO, Wilder AD Jr, Bayne SC, May KN Jr, Sturdevant JR, & Roberson TM (2001) Eighteen-month clinical evaluation of a filled and unfilled dentin adhesive *Journal of Dentistry* **29**(1) 1-6.
 44. Swift EJ Jr, Perdigao J, Wilder AD Jr, Heymann HO, Sturdevant JR, & Bayne SC (2001) Clinical evaluation of two one-bottle dentin adhesives at three years *Journal of the American Dental Association* **132**(8) 1117-1123.
 45. van Dijken JW & Pallesen U (2008) Long-term dentin retention of etch-and-rinse and self-etch adhesives and a resin-modified glass ionomer cement in non-carious cervical lesions *Dental Materials* **24**(7) 915-922.
 46. van Dijken JW (2013) A randomized controlled 5-year prospective study of two HEMA-free adhesives, a 1-step self etching and a 3-step etch-and-rinse, in non-carious cervical lesions *Dental Materials* **29**(11) e271-e280.
 47. 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.

48. Reis A, Dourado Loguercio A, Schroeder M, Luque-Martinez I, Masterson D, & Cople Maia L (2015) Does the adhesive strategy influence the post-operative sensitivity in adult patients with posterior resin composite restorations?: a systematic review and meta-analysis *Dental Materials* **31**(9) 1052-1067.
49. Chee B, Rickman LJ, & Satterthwaite JD (2012) Adhesives for the restoration of non-carious cervical lesions: a systematic review *Journal of Dentistry* **40**(6) 443-452.
50. Masarwa N, Mohamed A, Abou-Rabii I, Abu Zaghlan R, & Steier L (2016) Longevity of self-etch dentin bonding adhesives compared to etch-and-rinse dentin bonding adhesives: a systematic review *Journal of Evidence-Based Dental Practice* **16**(2) 96-106.
51. Higgins JP, Altman DG, Gotzsche PC, Juni P, Moher D, Oxman AD, Savovic J, Schulz KF, Weeks L, Sterne JA, Cochrane Bias Methods G, & Cochrane Statistical Methods G (2011) The Cochrane Collaboration's tool for assessing risk of bias in randomised trials *BMJ* **343** d5928.
52. Boutron I, Guttet L, Estellat C, Moher D, Hrobjartsson A, & Ravaud P (2007) Reporting methods of blinding in randomized trials assessing nonpharmacological treatments *PLoS Medicine* **4**(2) e61.
53. Wood L, Egger M, Lotte Gluud L, Schulz K, Altman D, Juni P, & Sterne J (2006) *The Association of Allocation Concealment and Blinding with Estimated Treatment Effect Varies According to Type of Outcome: A Combined Analysis of Meta-Epidemiological Studies* Cochrane Colloquium, Dublin, UK.
54. Glasziou P, Altman DG, Bossuyt P, Boutron I, Clarke M, Julious S, Michie S, Moher D, & Wager E (2014) Reducing waste from incomplete or unusable reports of biomedical research *Lancet* **383**(9913) 267-276.
55. Dwan K, Altman DG, Arnaiz JA, Bloom J, Chan AW, Cronin E, Decullier E, Easterbrook PJ, Von Elm E, Gamble C, Gherzi D, Ioannidis JP, Simes J, & Williamson PR (2008) Systematic review of the empirical evidence of study publication bias and outcome reporting bias *PLoS One* **3**(8) e3081.
56. Hickel R, Roulet JF, Bayne S, Heintze SD, Mjor IA, Peters M, Rousson V, Randall R, Schmalz G, Tyas M, & Vanherle G (2007) Recommendations for conducting controlled clinical studies of dental restorative materials *Clinical Oral Investigations* **11**(1) 5-33.
57. Dennison JB & Sarrett DC (2012) Prediction and diagnosis of clinical outcomes affecting restoration margins *Journal of Oral Rehabilitation* **39**(4) 301-318.
58. Mahn E, Rousson V, & Heintze S (2015) Meta-analysis of the influence of bonding parameters on the clinical outcome of tooth-colored cervical restorations *Journal of Adhesive Dentistry* **17**(5) 391-403.
59. Sebold M, André CB, Carvalho RM, & Giannini M (2019) Dry-bonding to dentin using alternative conditioners based on iron-containing solutions or nitric acid *Journal of the Mechanical Behavior of Biomedical Materials* **94** 238-248.
60. Cho BH & Dickens SH (2004) Effects of the acetone content of single solution dentin bonding agents on the adhesive layer thickness and the microtensile bond strength *Dental Materials* **20**(2) 107-115.
61. Al-Khayatt AS, Ray-Chaudhuri A, Poyser NJ, Briggs PF, Porter RW, Kelleher MG, & Elias S (2013) Direct composite restorations for the worn mandibular anterior dentition: a 7-year follow-up of a prospective randomised controlled split-mouth clinical trial *Journal of Oral Rehabilitation* **40**(5) 389-401.

A New Dual-cure Universal Simplified Adhesive: 18-month Randomized Multicenter Clinical Trial

E Gomes de Albuquerque • F Warol • F Signorelli Calazans • L Augusto Poubel
S Soares Marins • T Matos • JJ de Souza • A Reis • M de Oliveira Barceleiro • AD Loguercio

Clinical Relevance

Non-carious cervical lesion restorations using a dual-cure universal adhesive in self-etch and etch-and-rinse mode showed satisfactory clinical performance after 18 months.

SUMMARY

Objectives: The objective of this multicenter, double-blind, split-mouth randomized clinical trial was to evaluate the clinical performance of a new dual-cure universal adhesive system (Futurabond U, Voco GmbH) when applied using different strategies over a period of 18 months.

Elisa Gomes de Albuquerque, DDS, MS, PhD student, professor, Department of Restorative Dentistry, School of Dentistry, Fluminense Federal University, Nova Friburgo, Brazil

Flavio Warol, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Fluminense Federal University, Nova Friburgo, Brazil

Fernanda Signorelli Calazan, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Fluminense Federal University, Nova Friburgo, Brazil

Luiz Augusto Poubel, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Fluminense Federal University, Nova Friburgo, Brazil

Stella Soares Marins, DDS, MSD student, Department of Restorative Dentistry, School of Dentistry, Fluminense Federal University, Nova Friburgo, Brazil

Thalita Matos, DDS, MS, PhD student, Department of Restorative Dentistry, School of Dentistry, Ponta Grossa State University, Ponta Grossa, Brazil

Methods and Materials: Fifty patients participated in this study. Two hundred non-carious cervical lesions were restored using the adhesive Futurabond U according to four adhesive strategies (n=50 per group): only self-etch (SEE), selective enamel etching + self-etch (SET), etch-and-rinse with dry dentin (ERDry), and etch-and-rinse with wet dentin (ERWet). After the adhesive application, cavities were restored using Admira Fusion composite resin. These restorations were evaluated according

Jullian J. de Souza, DDS, MS, PhD student, Department of Restorative Dentistry, School of Dentistry, Ponta Grossa State University, Ponta Grossa, Brazil

Alessandra Reis, DDS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Ponta Grossa State University, Ponta Grossa, Brazil

Marcos de Oliveira Barceleiro, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Fluminense Federal University, Nova Friburgo, Brazil

*Alessandro Dourado Loguercio, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Ponta Grossa State University, Ponta Grossa, Brazil

*Corresponding author: Avenida General Carlos Cavalcanti, 4748, Ponta Grossa, PR, Brazil. CEP 84010-330; e-mail: aloguercio@hotmail.com

<https://doi.org/10.2341/19-144-C>

to FDI World Dental Federation criteria for the following characteristics: retention/fracture, marginal adaptation, marginal staining, post-operative sensitivity, and caries recurrence.

Results: After 18 months, only four patients (12 months: one patient, $n=4$ restorations; and 18 months: three patients, $n=12$ restorations) were not evaluated. Fourteen restorations were lost after 18 months of clinical evaluation (four for SEE, three for SET, three for ERDry, and four for ERWet). The retention rates for 18 months (95% confidence interval) were 92% (81%-97%) for SEE, 94% (83%-97%) for SET, 94% (83%-97%) for ERDry, and 92% (81%-97%) for ERWet ($p>0.05$). Thirty-eight restorations were considered to have minor discrepancies in marginal adaptation at the 18-month recall (13 for SEE, 13 for SET, six for ERDry, and six for ERWet; $p>0.05$). Fourteen restorations were detected as a minor marginal discoloration at the 18-month recall (six for SEE, six for SET, one for ERDry, and one for ERWet; $p>0.05$). However, all were considered clinically acceptable. No restorations showed postoperative sensitivity or caries recurrence at the time.

Conclusion: The clinical performance of the Futurabond U did not depend on the bonding strategy used, and it was considered reliable after 18 months of clinical evaluation, although more marginal discrepancy was observed in the self-etch group.

INTRODUCTION

Non-carious cervical lesions (NCCLs) are commonly found specific tissue losses. They are reported in up to 60% of patients and are the most prevalent in the maxillary posterior teeth, mainly the premolars.^{1,2} Development of NCCLs is a pathological process characterized by the loss of dental hard tissues near the cemento-enamel junction³ not caused by a bacterial agent. NCCLs could be caused by toothbrush abrasion, erosion caused by acids, and abfraction due to occlusal problems.⁴ Recently, there have been worldwide increases in the prevalence and severity of NCCLs closely associated with people's lifestyles and the aging of the population.⁵ Because NCCLs have been associated with other conditions, such as dentin hypersensitivity and gingival recession, as well as the loss of the dental mineralization continuum,⁵ the restorative procedure is highly recommended.⁶

The current concepts of minimally invasive techniques associated with the need for simplification have prompted manufacturers to develop more user-friendly adhesive systems, by reducing the application steps and shortening the clinical application time.^{6,7} The newest adhesives within this philosophy are called "universal," "multipurpose," or "multi-mode" adhesives.^{8,9} This new class of adhesives could be used as a two-step etch-and-rinse or one-step self-etch, according to the dentist's preference and professional judgement regarding the selection of the adhesive strategy and number of steps.^{10,11}

Although there is no official definition for what qualifies an adhesive system as a universal adhesive, the literature describes it as a single-bottle adhesive that performs equally well with any adhesion strategy and bonds adequately to tooth structure as well as different direct and indirect restorative materials.¹¹

Universal adhesives are similar to the simplified one-step self-etch adhesives but contain specific functional monomers to provide better bonding to the hard dental substrates. The best known of these monomers is 10-methacryloyloxydecyl dihydrogen phosphate (MDP). Several studies showed that MDP ionically bonds to dentin, forming hydrolytically stable calcium salts on hydroxyapatite (nanolayering), which promotes a more effective and stable bonding in water than that provided by other functional monomers.¹²⁻¹⁴

The inclusion of MDP allowed the development of more hydrophobic (ie, less hydrophilic) adhesives¹⁵ as MDP is quite hydrophobic because of its long carbonyl chain.^{16,17} Due to the inclusion of MDP and an optimized amount of water content, the manufacturers claimed to have balanced the hydrophobic and hydrophilic properties to ensure bonding at varying moisture levels.^{18,19}

Although several universal adhesives have already been evaluated and shown reliable results through laboratory tests to establish the best application protocol for universal adhesives,^{10,20,21} it is known that only clinical evaluations are the ultimate proof of clinical efficacy.²²

Because of all these features, the clinical use of universal adhesives is increasing rapidly, and several clinical studies evaluating the universal adhesive systems were published with significant controversies.²³⁻³⁰ For instance, some studies^{26,30} showed that a better clinical performance was observed when the universal adhesives were applied in the etch-and-rinse approach. On the other hand, other

clinical studies showed that there were no differences for the universal adhesives applied in the self-etch or etch-and-rinse mode after 18-36 months of clinical service.^{23-25,28,29} The same controversial results could be observed in the conclusion of the authors of two systematic reviews of clinical studies evaluating universal adhesives,^{11,31} which indicates the need for more clinical studies to evaluate this new class of adhesive system.

Therefore, the objective of this multicenter, double-blind, randomized clinical trial was to evaluate the clinical behavior of a new dual-cure universal adhesive when applied using different application strategies during 18 months of clinical evaluation. The null hypothesis tested was that bonding to NCCLs using the self-etch strategy, compared to selective enamel etching or using the etch-and-rinse strategy with adhesive application on dry or moist dentin, would result in similar retention levels over 18 months of clinical service.

METHODS AND MATERIALS

Study Design

The description of the experimental design followed the Consolidated Standards of Reporting Trials (CONSORT) statement.³² Written informed consent was obtained from all participants prior to starting the treatment. This clinical trial was registered in clinicaltrials.gov clinical registry (#NCT03244124). All participants were informed about the nature and objectives of the study, but they were not aware of which tooth received the specific treatments under evaluation.

Trial Design, Settings, and Location of Data Collection

This was a multicenter, double-blind, superiority, split-mouth, randomized clinical trial. The study was carried out in the clinics of the School of Dentistry of the State University of Ponta Grossa (PR, Brazil) and the Federal University Fluminense (Polo de Nova Friburgo, RJ, Brazil) from October 2016 to November 2016.

Recruitment—Patients were recruited as they sought treatment in the dental clinics of both universities. No advertisement was used for participant recruitment. Patients were recruited in the order in which they reported for the screening session, thus forming a sample of convenience.

Eligibility Criteria—A total of 120 participants were examined by two calibrated dental residents to check if they met the inclusion and exclusion criteria

in each university (Figure 1). The evaluations were performed using a mouth mirror, an explorer, and a periodontal probe. Participants had to be in good general health, be at least 18 years old, have an acceptable oral hygiene level, and present at least 20 teeth under occlusion.

Participants were required to have at least four NCCLs to be restored in four different teeth. These lesions had to be nonretentive, be deeper than 1 mm, and involve both the enamel and dentin of vital teeth without mobility. The cavosurface margin could not involve more than 50% of enamel.³³ Patients who had extremely poor oral hygiene used orthodontic devices, had severe or chronic periodontitis, or had heavy bruxism habits were excluded from the study as they would need to receive other treatments before restorative intervention. Also, participants with known allergy to resin-based materials or any other material used in this study, pregnant or breastfeeding women, or participants under chronic use of anti-inflammatory, analgesic, and psychotropic drugs were not included in the study.

Sample-size Calculation

The annual retention rate for one-step self-etch adhesives in NCCLs was reported to be 4.4% in a recent systematic review.⁶ Considering that this decline follows a linear trend, the overall retention rate of one-step self-etch adhesives is approximately 78% after 5 years of clinical service. With an α of 0.05, a power of 80%, and a two-sided test, the minimal sample size was 50 restorations in each group in order to detect a difference of 25% among the test groups.

Random Sequence Generation and Allocation Concealment

The randomization was done on an intraindividual basis so that each subject ended up with four restorations, each one resulting from one of all possible combinations of adhesive strategy and roughening procedure. These randomization schemes were performed using software available at <http://www.sealedenvelope.com>.

A staff member not involved in the research protocol performed the randomization process with computer-generated tables. Details of the allocated groups were recorded on cards contained in sequentially numbered, opaque, sealed envelopes. Opening the envelope only on the day of the restorative procedure guaranteed concealment of the random sequence. In all cases, the tooth with the highest

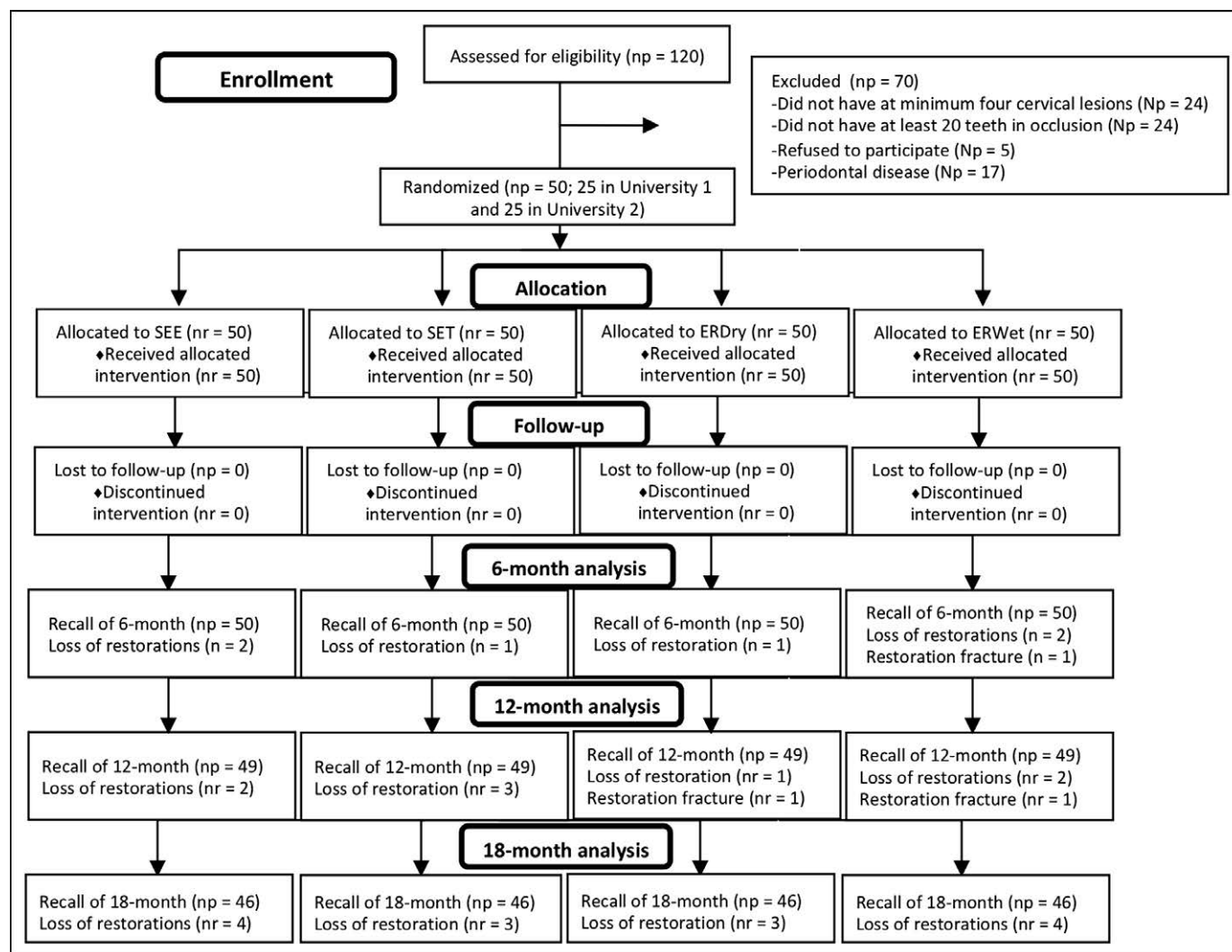


Figure 1. Flowchart of the experimental design.

tooth number received the treatment described first, whereas the tooth with the next number in sequence received the treatment mentioned second, with placement continuing in a similar manner until the fourth tooth (for the patients with four teeth needing treatment). All restorations in the same subject were always placed in different sextants.

Interventions: Restorative Procedure

All the patients selected for this study received dental prophylaxis with a suspension of pumice and water in a rubber cup and signed an informed consent form two weeks before the restorative procedures. The degree of sclerotic dentin from the NCCLs was measured according to the criteria described by Swift and others³⁴ (Table 1). The cavity dimensions in millimeters (height, width, and

depth), the geometry of the cavity (evaluated by profile photograph and labeled at <45°, 45° to 90°, 90° to <135°, and >135°),³⁵ the presence of an antagonist, and the presence of attrition facets were observed and recorded. Preoperative sensitivity was also evaluated by applying air for 10 seconds from a dental syringe placed 2 cm from the tooth surface and with an explorer. These features were recorded to allow comparison of the baseline features of the dentin cavities among experimental groups.

To calibrate the restorative procedure, the study director of each center (MOB and ADL) placed one restoration from each group to identify all steps involved in the application technique. Then, the four operators (EGA, FW, TM and JJS), who were resident dentists with more than five years of clinical experience in operative dentistry, two in each center,

Table 1: *Dentin Sclerosis Scale*^a

Category	Criteria
1	No sclerosis present; dentin is light yellowish or whitish, with little discoloration; dentin is opaque, with little translucency or transparency
2	More sclerosis than in category 1 but less than halfway between categories 1 and 4
3	Less sclerosis than in category 4 but more than halfway between categories 1 and 4
4	Significant sclerosis present; dentin is dark yellow or even discolored (brownish); glassy appearance, with significant translucency or transparency evident

^a Adapted from Swift and others.³⁴

placed four restorations, one of each group, under the supervision of the study director in a clinical setting. The restoration failures were shown to the operators before starting the study. At this point, the operators were considered calibrated to perform the restorative procedures. The operators restored all teeth. All participants received four restorations, one for each experimental group in different lesions previously selected according to the inclusion criteria.

Before restorative procedures, the operators anesthetized the teeth with a 3% mepivacaine solution (Mepisv, Nova DFL, Rio de Janeiro, Brazil) and cleaned all lesions with pumice and water in a rubber cup (No. 8040RA and No. 8045RA, KG Sorensen, Barueri, Brazil) followed by rinsing and drying. Then, shade selection was made using a shade guide. After a rubber dam was placed, the new universal adhesive system Futurabond U (Voco, GmbH, Cuxhaven, Germany) was applied as described later. The compositions, application modes, and batch numbers are described in Tables 2 and 3.

Self-etch Group (SEE)—One coat of adhesive was gently scrubbed on the entire enamel and dentin surface for approximately 20 seconds, according to the manufacturer's recommendations (Table 3). The adhesive was then evaporated by gentle air stream

for 5 seconds and light cured for 10 seconds at 1200 mW/cm² (Bluephase 20i, Ivoclar Vivadent, Schaan, Liechtenstein).

Self-etch Associated With Selective Enamel Etching Group (SET)—The 35% phosphoric acid (Vococid, Voco) was applied for 30 seconds only in enamel. Then, cavities were rinsed and air-dried, until dentin was kept visibly dry. The adhesive system was applied as described in the self-etch group (Table 3). Solvent evaporation and light curing procedures were also the same.

Etch-and-rinse Dry Dentin Group (ERDry)—The 35% phosphoric acid (Vococid) was applied for 30 seconds (enamel) and 15 seconds (dentin). Then, cavities were rinsed and air-dried, until dentin was kept visibly dry. The adhesive system was applied as described in the self-etch group (Table 3). Solvent evaporation and light curing procedures were also the same.

Etch-and-rinse Wet Dentin Group (ERWet)—The 35% phosphoric acid (Vococid) was applied for 30 seconds (enamel) and 15 seconds (dentin). Then, cavities were rinsed and slightly air-dried, keeping visible dentin moist. The adhesive system was applied as described in the self-etch group (Table 3). Solvent evaporation and light curing procedures were also the same.

After adhesive application, the resin composite Admira Fusion unidose (Voco) was used in up to three increments, inserted directly in the cavity with a Centrix device (Centrix, Shelton, CT, USA) and each one being light cured for 10 seconds at 1200 mW/cm². The restorations were finished immediately with fine and extra-fine No. 2200 diamond burs (KG Sorensen) and polished with Jiffy polisher (Ultradent) under constant water-cooling.

Calibration Procedures for Clinical Evaluation

For training purposes, two experienced and calibrated examiners in each center (FSC, LAP, AR and

Table 2: *Composition and Batch Number of Materials Used in the Restorative Procedures*

Material (Manufacturer)	Batch Number	Composition
Futurabond U (Voco GmbH, Cuxhaven, Germany)	1609415	35% phosphoric acid (Vococid): 35% phosphoric acid Adhesive: HEMA, Bis-GMA, HEDMA, acidic adhesive monomer, ^a urethane dimethacrylate, catalyst, silica nanoparticles, ethanol
Admira Fusion (Voco GmbH, Cuxhaven, Germany)	Shade A2: 1607524; Shade A3: 1606252; Shade A3.5: 1605482	Resin matrix: aromatic and aliphatic dimethacrylates, methacrylate-functionalized polysiloxane Inorganic filler: Ba-Al-glass, pyrogenic SiO ₂ , filler load: 78 mass %. Photoinitiator: camphorquinone. Synergist: NI

Abbreviations: Bis-GMA: Bisphenol-A-glycidyl dimethacrylate; HEDMA: 1,6-hexanediol dimethacrylate; HEMA: 2-hydroxyethyl methacrylate.

^a Acidic adhesive monomer in the composition of Futurabond U is 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate according to personal communication with Dr Martin Danebrock (January 30, 2018)

Table 3: Application Mode of the Adhesive System in the Groups ^a

Group	Application Mode			
	Etch		Adhesive	Resin Composite
Self-etch (SEE)	No	Keep dentin dry (do not over dry)	1. Activate single-dose adhesive package; 2. Apply adhesive to the cavity surface with Voco Single Tim Brush for 20 s with vigorous agitation; 3. Gently air dry for 5 s; 4. Light cure for 10 s at 1200 mW/cm ² .	Insert in the cavity at increases of up to 1 mm and light-cure each area of the surface of the restoration with a dental curing light appliance (wavelength of 470 nm, light power of 1200 mW/cm ²) for 30 s.
Self-etch associated to selective enamel etching (SET)	Apply etchant <i>only</i> in enamel (30 s), rinse for 30 s, air dry to remove excess water			
Etch-and-rinse, dentin dry (ERDry) ^b	Apply etchant in enamel (30 s) and dentin (15 s), rinse for 30 s, air dry to remove excess water	Keep dentin wet		
Etch-and-rinse, dentin wet (ERWet)				

^a According to the manufacturer's instructions.

^b Manufacturer does not indicate application in dry dentin.

ADL) observed 10 photographs that were representative of each score for each criterion. They evaluated 10 to 15 patients each on two consecutive days. These subjects had cervical restorations but were not part of this project. An intraexaminer and interexaminer agreement of at least 85% was necessary before beginning the evaluation.³⁶

Blinding The examiners were not involved with the restoration procedures and therefore blinded to the group assignment. Patients were also blinded to group assignment in a double-blind randomized clinical trial design.

Clinical Evaluation An individual, standardized, paper case report form was used for each evaluator at each recall time so that evaluators were kept blinded to earlier evaluations during the follow-up recalls. The restorations were evaluated by FDI World Dental Federation criteria^{37,38} and the classical US Public Health Service (USPHS) criteria (adapted by Bittencourt and others³⁹) at baseline and after 6, 12, and 18 months of clinical service. Only the clinically relevant measures for evaluation of adhesive performance were used and scored (Tables 4 and 5). The primary clinical endpoint was restoration retention/fracture, but the following secondary endpoints were also evaluated: marginal staining, marginal adaptation, postoperative sensitivity, and recurrence of caries. The evaluation of spontaneous postoperative sensitivity was performed one week after the restorative procedure by asking patients if they experienced any pain during the period.

These variables were ranked according to FDI criteria into clinically very good, clinically good, clinically sufficient/satisfactory, clinically unsatisfactory but repairable, and clinically poor (replacement required).^{37,38} In the case of marginal staining

and marginal adaptation, the semiquantitative criteria (SQUACE) proposed by Hickel and others^{37,38} was used. Each evaluator outlined the extent of the observed event on the sketch of each restoration using a pen according to defined criteria (marginal staining and marginal adaptation); after that, each margin was assessed quantitatively as a proportion of the total length of the margin. Also, all mentioned variables previously described were evaluated by USPHS criteria into Alpha, Bravo, and Charlie.³⁶

Both examiners evaluated all the restorations once and independently. When disagreements occurred during the evaluations, they had to reach a consensus before the participant was dismissed. The restoration retention rates were calculated according to the American Dental Association guidelines.⁴⁰ Cumulative failure percentage = $[(PF + NF) / (PF + RR)] \times 100\%$, where PF is the number of previous failures before the current recall, NF is the number of new failures during the current recall, and RR is the number of currently recalled restorations.

Statistical Analysis

The statistical analyses followed the intention-to-treat protocol according to CONSORT (Consolidated Standards of Reporting Trials) suggestion.³² Descriptive statistics were used to describe the distributions of the evaluated criteria. Statistical analysis for each individual item was performed for each evaluation criteria (FDI and USPHS criteria). The differences in the ratings of the four groups after 6, 12, and 18 months were tested with the Friedman repeated-measures analysis of variance by rank, and differences in the ratings of each group at baseline and after 6, 12, and 18 months were evaluated using the Wilcoxon test. Data from SQUACE after 18 months of clinical service was evaluated with the

Table 4: FDI Criteria Used for Clinical Evaluation^{37,38}

	Esthetic Properties	Functional Properties		Biological Properties	
	1. Staining Margin	2. Fractures and Retention	3. Marginal Adaptation	4. Postoperative (Hyper-)sensitivity	5. Recurrence of Caries
1. Clinically very good (A)	1.1 No marginal staining	2.1 Restoration retained, no fractures/cracks	3.1 Harmonious outline, no gaps, no discoloration	4.1 No hypersensitivity	5.1 No secondary or primary caries
2. Clinically good (B) (after correction very good)	1.2 Minor marginal staining, easily removable by polishing	2.2 Small hairline crack	3.2.1 Marginal gap (50 µm). 3.2.2 Small marginal fracture removable by polishing	4.2 Low hypersensitivity for a limited period of time	5.2 Very small and localized demineralization. No operative treatment required
3 Clinically sufficient/satisfactory (C) (minor shortcomings with no adverse effects but not adjustable without damage to the tooth)	1.3 Moderate marginal staining, not esthetically unacceptable	2.3 Two or more or larger hairline cracks and/or chipping (not affecting the marginal integrity)	3.3.1 Gap <150 µm not removable 3.3.2. Several small enamel or dentin fractures	4.3.1 Premature/ slightly more intense 4.3.2 Delayed/weak sensitivity; no subjective complaints, no treatment needed	5.3 Larger areas of demineralization, but only preventive measures are necessary (dentin not exposed)
4. Clinically unsatisfactory (D) (repair for prophylactic reasons)	1.4 Pronounced marginal staining; major intervention necessary for improvement	2.4 Chipping fractures, which damage marginal quality; bulk fractures with or without partial loss (less than half of the restoration)	3.4.1 Gap >250 µm or dentin/base exposed. 3.4.2. chip fracture damaging margins 3.4.3 Notable enamel or dentin wall fracture	4.4.1 Premature/ very intense 4.4.2 Extremely delayed/weak with subjective complaints 4.4.3 Negative sensitivity; intervention necessary but not replacement	5. 4 Caries with cavitation (localized and accessible and can be repaired)
5. Clinically poor (E) (replacement necessary)	1.5 Deep marginal staining not accessible for intervention	2.5 (Partial or complete) loss of restoration	3.5 Filling is loose but <i>in situ</i>	4.5 Very intense, acute pulpitis or nonvital; endodontic treatment is necessary and restoration has to be replaced	5.5 Deep secondary caries or exposed dentin that is not accessible for repair of restoration

Table 5: Modified USPHS Criteria According to Bittencourt and Others³⁹

	Marginal Staining	Retention	Fracture	Marginal Adaptation	Postoperative Sensitivity	Recurrence of caries
Alpha	No discoloration along the margin	Retained	None	Restoration is continuous with existing anatomic form	No postoperative sensitivity directly after the restorative process and during the study period	None evidence of caries contiguous with the margin
Bravo	Slight and superficial staining (removable, usually localized)	Partially retained	Small chip, but clinically acceptable	Detectable V-shaped defect in enamel only; catches explorer going both ways	-	-
Charlie	Deep staining cannot be polished away	Missing	Failure due to bulk restorative fracture	Detectable V-shaped defect to dentin-enamel junction	Sensitivity present at any time during the study period	Evidence of presence of caries



Figure 2. Examples of restorations after baseline and 18 months of clinical evaluation. All restorations were scored as clinically very good at baseline (A-D; FDI criteria). Teeth 23 (E; ErWet group), 12 (F; SET group), 28 (G; ErDry group), and 29 (G; SEE group) were scored as clinically very good (FDI criteria for marginal adaptation). Teeth 24 (E; SET group), 13 (F; SEE group), and 6 (H; ErWet group) were scored as clinically good (FDI criteria for marginal adaptation). Tooth 5 was scored as clinically poor (H; ErDry group; FDI criteria for marginal adaptation).

Kruskall-Wallis test. The Cohen kappa statistic was used to test interexaminer agreement. In all statistical tests, we preset the level of significance to 5%.

RESULTS

General Results

The restorative procedures were implemented exactly as planned, and no modification was performed. Of the 120 patients examined for eligibility, 70 were not enrolled in the study because they did not fulfill the inclusion criteria. Thus, a total of 50 subjects (23 men and 27 women) were selected. Two hundred restorations were placed, 50 for each group (Figure 1).

All baseline details relative to the research subjects and characteristics of the restored lesions are displayed in Table 6. The overall Cohen kappa statistic showed excellent agreement between the examiners at the 6-, 12-, and 18-month (0.82) follow-ups. All research subjects were evaluated at baseline and at the 6-month recall. One subject moved to a new city and was not included in the 12-month recall rate, and another three subjects did not attend the 18-month recall. Some examples of restorations after baseline and 18 months of clinical evaluation are depicted in Figure 2.

Retention/Fracture

Seven restorations were lost or fractured after 6 months of clinical evaluation (two for SEE, one for SET, one for ERDry, and three for ERWet). Accord-

ing to both evaluation criteria, the 6-month retention rates (95% confidence interval [CI]) were 96% (86%-98%) for SEE, 98% (89%-99%) for SET, 98% (89%-99%) for ERDry, and 94% (83%-97%) for ERWet with no statistical difference identified between any pair of groups ($p > 0.05$; Tables 7 and 8). When the data from the 6-month results from each group were compared with the baseline findings, no significant difference was found ($p > 0.05$; Tables 7 and 8).

Ten restorations were lost or fractured after 12 months of clinical evaluation (two for SEE, three for SET, two for ERDry, and three for ERWet). According to both evaluation criteria, the 12-month retention rates (95% CI) were 96% (86%-98%) for SEE, 94% (83%-97%) for SET, 96% (86%-98%) for ERDry, and 94% (83%-97%) for ERWet with no statistical difference identified between any pair of groups ($p > 0.05$; Tables 7 and 8). When the data from the 12-month results from each group were compared with the baseline findings, no significant difference was found ($p > 0.05$; Tables 7 and 8).

Fourteen restorations were lost after 18 months of clinical evaluation (four for SEE, three for SET, three for ERDry, and four for ERWet). According to both evaluation criteria, the 18-month retention rates (95% CI) were 92% (81% to 97%) for SEE, 94% (83% to 97%) for SET, 94% (83% to 97%) for ERDry, and 92% (81% to 97%) for ERWet, with no statistical difference identified between any pair of groups ($p > 0.05$; Tables 7 and 8). When the data from

Table 6: Characteristics of the Research Participants and the NCCLs by Experimental Group

Characteristics of Research Participants		No. of Participants			
Gender distribution					
Male		23			
Female		27			
Age distribution (y)					
20-29		3			
30-39		3			
39-49		20			
>49		24			
Characteristics of NCCLs	No. of Lesions				
	SEE	SET	ERDry	ERWet	
Shape (degree of angle)					
<45		0	0	0	0
45-90		8	10	13	6
90-135		17	13	15	17
>135		25	27	22	27
Cervico-incisal height (mm)					
<1.5		14	17	17	15
1.5-2.5		15	13	14	17
2.5-4.0		12	8	12	11
>4.0		9	12	7	7
Degree of sclerotic dentin					
1		29	28	28	31
2		13	14	11	11
3		6	7	9	6
4		2	1	2	2
Presence of antagonist					
Yes		41	49	42	44
No		9	1	8	6
Attrition facet					
Yes		37	38	40	43
No		13	12	10	7
Preoperative sensitivity (spontaneous)					
Yes		13	11	11	13
No		37	39	39	37
Preoperative sensitivity (air dry)					
Yes		17	13	13	15
No		33	37	37	35
Tooth distribution					
Anterior					
Incisor		9	12	10	11
Canines		8	5	10	9
Posterior					
Premolar		21	24	23	21
Molar		12	9	7	9
Arch distribution					
Maxillary		25	27	24	26
Mandibular		25	23	26	24
Abbreviations: ERDry, etch-and-rinse, dry dentin; ERWet, etch-and-rinse, wet dentin; NCCL, non-carious cervical lesion; SEE, self-etch without selective enamel etching; SET, self-etch with selective enamel etching.					

Abbreviations: EDRdy, etch-and-rinse, dry dentin; ERWet, etch-and-rinse, wet dentin; NCCL, non-carious cervical lesion; SEE, self-etch without selective enamel etching; SET, self-etch with selective enamel etching.

the 18-month results from each group were compared with the baseline findings, no significant difference was found ($p>0.05$; Tables 7 and 8).

Marginal Adaptation

Twenty-four restorations were considered to have minor discrepancies in marginal adaptation at the 6-month recall using the FDI criteria (eight for SEE, nine for SET, four for EDRdy, and three for ERWet; Table 7). No significant difference was detected between any pair of groups at the 6-month recall for either evaluation criteria ($p>0.05$; Table 7). When the USPHS criteria were used, three restorations were scored as Bravo for marginal adaptation (two for SEE and one for SET; $p>0.05$; Table 8). When the baseline and 6-month data from the FDI evaluation were compared, a significant difference was only observed between SEE and SET ($p<0.05$; Table 8). No significant difference was detected when the baseline and 6-month data were compared for EDRdy and ERWet ($p>0.05$; Table 8).

Thirty-one restorations were considered to have minor discrepancies in marginal adaptation at the 12-month recall using the FDI criteria (12 for SEE, 10 for SET, five for EDRdy, and four for ERWet; Table 7). No significant difference was detected between any pair of groups at the 12-month recall for either evaluation criteria ($p>0.05$; Table 7). When the USPHS criteria were used, nine restorations were scored as Bravo for marginal adaptation (four for SEE, three for SET, one for EDRdy, and one for ERWet; $p>0.05$; Table 8). When the baseline and 12-month data from the FDI evaluation were compared, a significant difference was observed only between SEE and SET ($p<0.05$; Table 8). No significant difference was detected when the baseline and 12-month data were compared for EDRdy and ERWet ($p>0.05$; Table 8).

Thirty-eight restorations were considered to have minor discrepancies in marginal adaptation at the 18-month recall using the FDI criteria (13 for SEE, 13 for SET, six for EDRdy, and six for ERWet; Table 7). When the baseline and 18-month data from the FDI evaluation were compared, a significant difference was observed only between SEE and SET ($p<0.05$; Table 7). No significant difference was detected when the baseline and 18-month data were compared for EDRdy and ERWet ($p>0.05$; Table 7). When the USPHS criteria were used, 16 restorations were scored as Bravo for marginal adaptation (six for SEE, six for SET, two for EDRdy, and two for ERWet; $p>0.05$; Table 8). No significant difference was detected when the baseline and 18-month data from

Table 7: Number of Evaluated Restorations for Each Experimental Group Classified According to FDI Criteria^{37,38}

FDI Criteria	Status ^a	6 Months				12 Months				18 Months			
		SEE	SET	ERDry	ERWet	SEE	SET	ERDry	ERWet	SEE	SET	ERDry	ERWet
Marginal staining	A	48	49	49	47	42	41	45	44	32	33	39	37
	B	-	-	-	-	03	04	01	01	06	06	01	01
	C	-	-	-	-	-	-	-	-	-	-	-	-
	D	-	-	-	-	-	-	-	-	-	-	-	-
	E	-	-	-	-	-	-	-	-	-	-	-	-
Fractures and retention	A	48	49	49	47	45	45	46	45	38	39	40	38
	B	-	-	-	-	-	-	-	-	-	-	-	-
	C	-	-	-	-	-	-	-	-	-	-	-	-
	D	-	-	-	01	-	-	01	01	-	-	-	-
	E	02	01	01	02	02	03	01	02	04	03	03	04
Marginal adaptation	A	40	40	45	44	33	35	41	41	25	26	34	32
	B	08	09	04	03	12	10	05	04	10	11	06	06
	C	-	-	-	-	-	-	-	-	03	02	-	-
	D	-	-	-	-	-	-	-	-	-	-	-	-
	E	-	-	-	-	-	-	-	-	-	-	-	-
Postoperative (hyper-) sensitivity	A	48	49	49	47	45	45	46	45	38	39	40	38
	B	-	-	-	-	-	-	-	-	-	-	-	-
	C	-	-	-	-	-	-	-	-	-	-	-	-
	D	-	-	-	-	-	-	-	-	-	-	-	-
	E	-	-	-	-	-	-	-	-	-	-	-	-
Recurrence of caries	A	48	49	49	47	45	45	46	45	38	39	40	38
	B	-	-	-	-	-	-	-	-	-	-	-	-
	C	-	-	-	-	-	-	-	-	-	-	-	-
	D	-	-	-	-	-	-	-	-	-	-	-	-
	E	-	-	-	-	-	-	-	-	-	-	-	-

Abbreviations: FDI, World Dental Federation; EREdry, etch-and-rinse, dry dentin; ERWet, etch-and-rinse, wet dentin; SEE, self-etch without selective enamel etching; SET, self-etch with selective enamel etching.
^a A, clinically very good; B, clinically good; C, clinically sufficient / satisfactory; D, clinically unsatisfactory; E, clinically poor.

the USPHS evaluation criteria were compared ($p>0.05$; Table 7). No significant difference was detected between any pair of groups at the 18-month recall for either evaluation criteria ($p>0.05$; Tables 7 and 8). At the 18-month evaluation, the results of SQUACE showed that, usually, the marginal discrepancy was observed in less than 10% or between 10% and 30% of the restorations' margins (Table 9). Only the SEE group showed a statistical difference at the 18-month recall, compared with other experimental groups ($p>0.05$; Table 9).

Marginal Discoloration

Marginal staining was not observed in any restoration at the 6-month recall using the FDI and USPHS criteria. However, nine restorations presented with minor marginal discoloration at the 12-month recall using the FDI criteria (three for SEE, four for SET, one for EREdry, and one for ERWet; Table 7). When the USPHS criteria were applied, only two restora-

tions (one for SEE and one for SET; Table 8) were considered to show marginal discoloration. When the baseline and 12-month data for both criteria were compared, no significant difference was detected ($p>0.05$; Tables 7 and 8).

After 18 months of clinical evaluation, 14 restorations were detected with a minor marginal discoloration using the FDI criteria (six for SEE, six for SET, one for EREdry, and one for ERWet; Table 7). When USPHS criteria were applied, only four restorations (two for SEE and two for SET; Table 8) were considered to show marginal discoloration. When the baseline and 18-month data for both criteria were compared, no significant difference was detected ($p>0.05$; Tables 7 and 8).

Other Parameters

No restorations had postoperative sensitivity to air at the 6-, 12-, and 18-month recalls using both

Table 8: Number of Evaluated Restorations for Each Experimental Group According to the Modified USPHS Criteria³⁹

USPHS Criteria	6 Months				12 Months				18 Months			
	SEE	SET	ERDry	ERWet	SEE	SET	ERDry	ERWet	SEE	SET	ERDry	ERWet
Marginal staining												
Alpha	48	49	49	47	44	44	46	45	36	37	40	38
Bravo	-	-	-	-	01	01	-	-	02	02	-	-
Charlie	-	-	-	-	-	-	-	-	-	-	-	-
Retention												
Alpha	48	49	49	47	45	45	46	45	38	39	40	38
Bravo	-	-	-	-	-	-	-	-	-	-	-	-
Charlie	02	01	01	02	02	03	01	02	04	03	03	04
Fractures												
Alpha	48	49	49	47	45	45	46	45	38	39	40	38
Bravo	-	-	-	-	-	-	-	-	-	-	-	-
Charlie	-	-	-	01	-	-	01	01	-	-	-	-
Marginal adaptation												
Alpha	46	48	49	47	41	42	45	44	32	33	38	36
Bravo	02	01	-	-	04	03	01	01	06	06	02	02
Charlie	-	-	-	-	-	-	-	-	-	-	-	-
Postoperative (hyper-) sensitivity												
Alpha	48	49	49	47	45	45	46	45	38	39	40	38
Bravo	-	-	-	-	-	-	-	-	-	-	-	-
Charlie	-	-	-	-	-	-	-	-	-	-	-	-
Recurrence of caries												
Alpha	48	49	49	47	45	45	46	45	38	39	40	38
Bravo	-	-	-	-	-	-	-	-	-	-	-	-
Charlie	-	-	-	-	-	-	-	-	-	-	-	-

Abbreviations: USPHS, US Public Health Service; EDRy, etch-and-rinse, dry dentin; ERWet, etch-and-rinse, wet dentin; SEE, self-etch without selective enamel etching; SET, self-etch with selective enamel etching.

criteria. No restoration showed a recurrence of caries after 6, 12, and 18 months for either criteria.

DISCUSSION

The comparison of bonding techniques and adhesive systems is usually performed with NCCLs, as these lesions lack macromechanical retention; therefore, restoration loss occurs due to ineffective bonding,

which is an objective and clinically important outcome for adhesive efficacy.^{6,41} This is considered a true endpoint because if restorations are lost, none of the other parameters can be evaluated. The results of the present study showed that after 18 months of clinical service, restorations placed with Futurabond U had similar retention rates regardless of the use of phosphoric acid or wetness in the surface of the dental substrate of NCCLs; this leads us to accept the null hypothesis.

In the present study, Futurabond U was applied after etching with phosphoric acid under dry or wet dentin (ERDry and ERWet) conditions. This took into account that the ideal universal adhesive would be one in which bonding to the tooth would not be influenced by clinically plausible variations in the surface moisture.¹⁹ Unfortunately, although the wet-bonding technique is very popular among clinicians, the manufacturers' instructions for the surface wetness of the substrates after phosphoric acid etching are not precisely specified, making variability in the clinic even more likely.^{42,43}

Table 9: Number of Evaluated Restorations for Each Experimental Group According to the Adhesive Classified by SQUACE criteria^{a,37,38}

SQUACE criteria	SEE	SET	ERDry	ERWet
Less than 10%	02	12	05	05
Between 10% and 30%	11	01	01	01
Between 31% and 50%	-	-	-	-
Statistical Analysis	B	A	A	A

Abbreviations: EDRy, etch-and-rinse, dry dentin; ERWet, etch-and-rinse, wet dentin; SEE, self-etch without selective enamel etching; SET, self-etch with selective enamel etching; SQUACE, semiquantitative criteria.

^a Different capital letters indicate statistically significant difference between groups ($p < 0.05$).

The rationale behind the wet-bonding technique is, after phosphoric acid etching (etch-and-rinse technique), it is important to keep the dentin moist to avoid the collapse of the exposed collagen matrix and, mainly, to guarantee the infiltration of the resin monomer into the demineralized dentin.⁴⁴ However, universal adhesives contain water in their composition to enable the self-etching potential, and several studies have already claimed that adhesives with a water concentration of 10% to 25% can re-expand the air-dried and collapsed collagen mesh to facilitate adhesive resin infiltration.^{19,45,46} Therefore, keeping the dentin wet or visibly dry after the phosphoric acid application would not make a difference in the bonding quality when using a universal adhesive with this water concentration in the composition. Unfortunately, the exact water content of the Futurabond U was not disclosed by the manufacturer.

It is worth mentioning that the active application of Futurabond U also helps to infiltrate the adhesive in the collapsed collagen network because the pressure of the collapsed collagen in demineralized dentin allows better monomer diffusion inward as well as solvent diffusion outward.^{47,48} The active application of the etch-and-rinse adhesives showed better clinical performance in adhesive restorations, even when the dentin was kept visibly dry, compared with passive application.^{33,49}

The results of the present study allowed us to conclude that Futurabond U is tolerant to dentin moisture variation because of the similar retention rate of Futurabond U when applied on dry dentin (ERDry; 94%) or moist dentin (ERWet; 92%) after 18 months of clinical evaluation. Compared with the literature, the clinical behavior of Futurabond U when applied in the EDRdry or ERWet condition could be considered very good and comparable to the universal adhesives evaluated in the same conditions.^{23,26}

As discussed in the Introduction, the presence of specific functional monomers, such as MDP, in the universal adhesives is responsible for the chemical interaction with the dental hard tissues. Unfortunately, the use of phosphoric acid removes the calcium from dentin and may preclude any potential chemical bonding (nanolayering) between the calcium and phosphate groups in the adhesive. Similar immediate dentin bond strength results are shown when etch-and-rinse and self-etch approaches are compared.⁵⁰ However, the bonding ability of Futurabond U in the etch-and-rinse mode was significantly lower after water storage compared with Futurabond U applied in the self-etch mode.⁵⁰ Although

extrapolation to clinical situations is not often recommended, the available research data suggest that etching dentin may not be the first choice for MDP-containing universal adhesives.²⁷

In the safety data sheet of Futurabond U the manufacturer describes its functional monomer only as a phosphate mono-methacrylate, and this caused some confusion in previously published laboratory studies⁵¹. However, it has already been confirmed that MDP is present in the Futurabond U composition,⁵² and this could be responsible for the higher retention rate of Futurabond U when applied in the self-etch mode (SEE and SET) after 18 months of clinical service, which is similar to the other universal adhesives available on the market.^{23-25,29}

A frequent concern regarding universal adhesives when used in the self-etch mode is related to the lower potential of etching enamel,^{53,54} resulting in a higher number of defective margins of the restorations and, consequently, the marginal staining at long-term follow-ups. In the present study, a closer view regarding the marginal adaptation data, mainly from the SEE group, showed that Futurabond U presented a lower percentage of marginal failures (30%) than found in previous studies when the SEE group was evaluated (47% to 57%).^{23,24} Although Futurabond U could be considered a mild pH, self-etch adhesive (pH range 2-2.5), the adhesives evaluated in previous studies^{23,24} are considered ultra-mild self-etch (pH>2.5). Therefore, it would be expected that Futurabond U should more adequately etch the enamel margins. However, no differences were observed in the enamel etching pattern when the mild and ultra-mild universal self-etch adhesives were compared.^{19,53,55} Actually, Futurabond U showed similar bonding properties to the enamel (bond strength and *in situ* degree of conversion) compared with the ultra-mild universal adhesives applied in the self-etch mode.^{24,53}

Several factors may be involved in the good marginal adaptation of a resin composite to the cavity. It was shown that the marginal cavity adaptation of resin restorations depends not only on material-related properties, such as the adhesive used, polymerization shrinkage, viscoelastic properties, and stiffness of the restorative material, but also on the individual treatment conditions, such as cavity size and geometry, restorative placement, and curing techniques.⁵⁶ Another factor is the method of resin composite application in the cavity. In this specific case, the resin composite was used in capsules, and applied directly to the cavity and not in syringes as in previous studies.²³⁻³⁰ The use of

capsules decreases the presence of voids and porosities in the final restorations.^{57,58} To the best of our knowledge, the effects of inserting resin composites in capsules or syringes on the clinical performance of the composite resin restorations are not known. Future clinical studies need to be done to evaluate the effect of inserting the resin composite in syringe versus capsule in the clinical performance of NCCL restorations.

It is worth mentioning that the percentage of marginal defects observed in several clinical studies that evaluated universal adhesives in NCCLs was still lower than in the present study.^{30,59} This could be explained by the criteria used to evaluate the restorations. Although in the present study, FDI criteria were applied, the restorations were evaluated using a modified USPHS criteria in the other studies.^{30,59} In a recent literature review,⁶⁰ it was shown that the use of FDI criteria for the clinical evaluation of direct restorations was more sensitive and precise in detecting minor failure compared with modified USPHS criteria.^{23,24,26,27,49} A few restorations were observed with marginal adaptation problems when evaluated by USPHS criteria in the present study as well as in results of previous studies evaluating NCCL adhesive restorations with the same criteria.^{23,24,30,59}

The use of selective etching of the enamel margins has been recommended prior to the application of self-etch adhesives to overcome this limitation.^{61,62} However, note that the results of the present study showed no significant difference in the marginal adaptation when the SEE and SET groups were compared using USPHS and FDI criteria. This could be attributed to the low number of restorations with marginal defects, as previously described.

On the other hand, when SQUACE was applied,³⁷ some differences were observed between SEE and SET. Although SQUACE, in addition to the FDI criteria, has been proposed to improve the marginal quality evaluation,³⁷ only a few clinical trials have used this auxiliary method.^{23,24,26} Perdigão and others²³ showed that, when SQUACE was used, SEE resulted in a significantly greater number of restorations, between 10% and 30% of the total length of the interface, showing a marginal discrepancy compared with the SET group or even compared with the ERWet and ERDry groups.

In the present study, when SQUACE was used, this same pattern was observed, as the SEE group showed a statistical difference at the 18-month recall rate compared with the other experimental

groups. However, it is worth mentioning that all marginal defects observed in the present study were considered clinically acceptable and easily corrected by the clinician through a repolish of the restorations.⁶³ Future long-term clinical follow-up studies are still necessary to prove the results obtained in this study.

Finally, this clinical study had some limitations. The study was conducted in a university setting, with all restorations placed in an ideal scenario by four well-calibrated and supervised operators. In this setting, only motivated patients with a low caries risk were included. Therefore, future clinical studies need to evaluate the universal adhesives in a practice-based study, preferably in patients with high caries risk. Also, 18 months of clinical evaluation could be considered a medium-term follow-up. Therefore, future long-term follow-up studies need to be done. However, the fact that Futurabond U belongs to a very versatile and moisture tolerant universal adhesive category warranted this medium-term evaluation.

CONCLUSION

After 18 months in NCCLs, the clinical performance of Futurabond U was very good when used in the etch-and-rinse technique and maintaining dentin moisture or slightly drying the dentin. The clinical performance was also very good when Futurabond U was used in the self-etch mode associated with selective enamel etching, although more marginal discrepancy was observed in the self-etch mode.

Acknowledgements

This project was supported by the National Council for Scientific and Technological Development (CNPq) under grants 305588/2014-1 and 303332/2017-4 and in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University Fluminense, Campus Nova Friburgo and State University of Ponta Grossa. The approval codes issued for this study are 165.357/2016 and 1.618.895/2016.

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 23 October 2019)

REFERENCES

- Aw TC, Lepe X, Johnson GH, & Mancl L (2002) Characteristics of noncarious cervical lesions: A clinical investigation *American Journal of Dentistry* **133**(6) 725-733.
- Smith WA, Marchan S, & Rafeek RN (2008) The prevalence and severity of non-carious cervical lesions in a group of patients attending a university hospital in Trinidad *Journal of Oral Rehabilitation* **35**(2) 128-134, <https://doi.org/10.1111/j.1365-2842.2007.01763.x>.
- Grippio JO, Simring M, & Coleman TA (2012) Abfraction, abrasion, biocorrosion, and the enigma of noncarious cervical lesions: A 20-year perspective *Journal of Esthetic and Restorative Dentistry* **24**(1) 10-23, <https://doi.org/10.1111/j.1708-8240.2011.00487.x>.
- 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, <https://doi.org/10.1177/154405910608500405>.
- Teixeira DNR, Zeola LF, Machado AC, Gomes RR, Souza PG, Mendes DC, & Soares PV (2018) Relationship between noncarious cervical lesions, cervical dentin hypersensitivity, gingival recession, and associated risk factors: A cross-sectional study *Journal of Dentistry* **76** 93-97, <https://doi.org/10.1016/j.jdent.2018.06.017>.
- Peumans M, De Munck J, Mine A, & Van Meerbeek B (2014) Clinical effectiveness of contemporary adhesives for the restoration of non-carious cervical lesions: A systematic review *Dental Materials* **30**(10) 1089-1103, <https://doi.org/10.1016/j.dental.2014.07.007>.
- Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, & Van Landuyt KL (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>.
- Alex G (2015) Universal adhesives: the next evolution in adhesive dentistry? *Compendium of Continuing Education in Dentistry* **36**(1) 15-26.
- Perdigão J & Loguercio AD (2014) Universal or multi-mode adhesives: Why and how? *Journal of Adhesive Dentistry* **16**(2) 193-194, <https://doi.org/10.3290/j.jad.a31871>.
- Muñoz MA, Luque I, Hass V, Reis A, Loguercio AD, & Bombarda NH (2013) Immediate bonding properties of universal adhesives to dentine *Journal of Dentistry* **41**(5) 404-411, <https://doi.org/10.1016/j.jdent.2013.03.001>.
- Nagarkar S, Theis-Mahon N, & Perdigão J (2019) Universal dental adhesive: Current status, laboratory testing and clinical performance *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **107**(6) 2121-2131, <https://doi.org/10.1002/jbm.b.34305>.
- Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, Inoue S, Tagawa Y, Suzuki K, De Munck J, & Van Meerbeek B (2004) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **83**(6) 454-458, <https://doi.org/10.1177/154405910408300604>.
- Yoshida Y, Yoshihara K, Nagaoka N, Hayakawa S, Torii T, Ogawa T, Osaka A, & Van B Meerbeek (2012) Self-assembled nanolayering at the adhesive interface *Journal of Dental Research* **91**(4) 376-381, <https://doi.org/10.1177/0022034512437375>.
- Yoshihara K, Yoshida Y, Nagaoka N, Hayakawa S, Okihara T, De Munck J, Maruo Y, Nishigawa G, Minagi S, Osaka A, & Van Meerbeek B (2013) Adhesive interfacial interaction affected by different carbon-chain monomers *Dental Materials* **29**(8) 888-897, <https://doi.org/10.1016/j.dental.2013.05.006>.
- Chen L & Suh BI (2013) Effect of hydrophilicity on the compatibility between a dual-curing resin cement and one-bottle simplified adhesives *Journal of Adhesive Dentistry* **15**(4) 325-331, <https://doi.org/10.3290/j.jad.a28604>.
- Mosznier N, Salz U, & Zimmermann J (2005) Chemical aspects of self-etching enamel-dentin adhesives: A systematic review *Dental Materials* **21**(10) 895-910, <https://doi.org/10.1016/j.dental.2005.05.001>.
- Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, & Van Meerbeek B (2007) systematic review of the chemical composition of contemporary dental adhesives *Biomaterials* **28**(26) 3757-3785, <https://doi.org/10.1016/j.biomaterials.2007.04.044>.
- Sugimura R, Tsujimoto A, Hosoya Y, Fischer NG, Barkmeier WW, Takamizawa T, Latta MA, & Miyazaki M (2019) Surface moisture influence on etch-and-rinse universal adhesive bonding *American Journal of Dentistry* **32**(1) 33-38.
- Tsujimoto A, Shimatani Y, Nojiri K, Barkmeier WW, Markham MD, Takamizawa T, Latta MA, & Miyazaki M (2019) Influence of surface wetness on bonding effectiveness of universal adhesives in etch-and-rinse mode *European Journal of Oral Sciences* **127**(2) 162-169, <https://doi.org/10.1111/eos.12596>.
- Vermelho PM, Reis AF, Ambrosano GMB, & Giannini M (2017) Adhesion of multimode adhesives to enamel and dentin after one year of water storage *Clinical Oral Investigations* **21**(5) 1707-1715, <https://doi.org/10.1007/s00784-016-1966-1>.
- Cuevas-Suárez CE, da Rosa WLO, Lund RG, da Silva AF, & Piva E (2019) Bonding performance of universal adhesives: An updated systematic review and meta-analysis *Journal of Adhesive Dentistry* **21**(1) 7-26, <https://doi.org/10.3290/j.jad.a41975>.
- Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, & De Munck J (2010) Relationship between bond-strength tests and clinical outcomes *Dental Materials* **26**(2) 100-121, <https://doi.org/10.1016/j.dental.2009.11.148>.
- Perdigão J, Kose C, Mena-Serrano AP, De Paula EA, Tay LY, Reis A, & Loguercio AD (2014) A new universal simplified adhesive: 18-month clinical evaluation *Operative Dentistry* **39**(2) 113-127, <https://doi.org/10.2341/13-045-C>.
- Loguercio AD, de Paula EA, Hass V, Luque-Martinez I, Reis A, & Perdigão J (2015) A new universal simplified adhesive: 36-month randomized double-blind clinical trial *Journal of Dentistry* **43**(9) 1083-1092, <https://doi.org/10.1016/j.jdent.2015.07.005>.

25. Lawson NC, Robles A, Fu CC, Lin CP, Sawlani K, & Burgess JO (2015) Two-year clinical trial of a universal adhesive in total-etch and self-etch mode in non-carious cervical lesions *Journal of Dentistry* **43**(10) 1229-1234, <https://doi.org/10.1016/j.jdent.2015.07.009>.
26. Lopes LS, Calazans FS, Hidalgo R, Buitrago LL, Gutierrez FF, Reis A, Loguercio AD, & Barceleiro MO (2016) Six-month follow-up of cervical composite restorations placed with a new universal adhesive system: A randomized clinical trial *Operative Dentistry* **40**(1) 465-480, <https://doi.org/10.2341/15-309-C>.
27. Loguercio AD, Luque-Martinez IV, Fuentes S, Reis A, & Muñoz MA (2018) Effect of dentin roughness on the adhesive performance in non-carious cervical lesions: A double-blind randomized clinical trial *Journal of Dentistry* **69** 60-69, <https://doi.org/10.1016/j.jdent.2017.09.011>.
28. Ruschel VC, Shibata S, Stolf SC, Chung Y, Baratieri LN, Heymann HO, & Walter R (2018) Eighteen-month clinical study of universal adhesives in noncarious cervical lesions *Operative Dentistry* **43**(3) 241-249, <https://doi.org/10.2341/16-320-C>.
29. Zanatta RF, Silva TM, Esper M, Bresciani E, Gonçalves SEP, & Caneppele T (2019) Bonding performance of simplified adhesive systems in noncarious cervical lesions at 2-year follow-up: A double-blind randomized clinical trial *Operative Dentistry* **44**(5) 476-487, <https://doi.org/10.2341/18-049-C>.
30. Oz FD, Ergin E, & Canatan S (2019) Twenty-four-month clinical performance of different universal adhesives in etch-and-rinse, selective etching and self-etch application modes in NCCL A randomized controlled clinical trial *Journal of Applied Oral Science* **27** e20180358, <https://doi.org/10.1590/1678-7757-2018-0358>.
31. Arbildo H, Lamas-Lara C, Cruzado-Oliva F, Rosas-Prado C, Gómez-Fuertes A, & Vásquez-Rodrigo H (2018) Comparison of the clinical effect of the adhesive strategies of universal adhesives in the treatment of non-carious cervical lesions: Systematic review and meta-analysis *Journal of Oral Research* **7**(5) 210-222.
32. Schulz KF, Altman DG, Moher D, & CONSORT Group (2011) CONSORT 2010 statement: Updated guidelines for reporting parallel group randomized trials *International Journal of Surgery* **9**(8) 672-677, <https://doi.org/10.1016/j.ijsu.2011.09.004>.
33. Zander-Grande C, Ferreira SQ, da Costa TR, Loguercio AD, & Reis A (2011) Application of etch-and-rinse adhesives on dry and rewet dentin under rubbing action: A 24-month clinical evaluation *Journal of the American Dental Association* **142**(7) 828-835.
34. Swift EJ Jr, Perdigão J, Heymann HO, Wilder Jr AD, Bayne SC, May Jr KN, Sturdevant JR, & Roberson TM (2001) Eighteen month clinical evaluation of a filled and unfilled dentin adhesive *Journal of Dentistry* **29**(1) 1-6.
35. Costa TRF, Loguercio AD, & Reis A (2013) Effect of enamel bevel on the clinical performance of resin composite restorations placed in non-carious cervical lesions *Journal of Esthetic and Restorative Dentistry* **25**(5) 346-356, <https://doi.org/10.1111/jerd.12042>.
36. Cvar JF & Ryge G (2005) Reprint of criteria for the clinical evaluation of dental restorative materials *Clinical Oral Investigation* **9**(4) 215-232, <https://doi.org/10.1007/s00784-005-0018-z>.
37. Hickel R, Roulet JF, Bayne S, Heintze SD, Mjör IA, Peters M, Rousson V, Randall R, Schmalz G, Tyas M, & Vanherle G (2007) Recommendations for conducting controlled clinical studies of dental restorative materials. Science committee project 2/98—FDI World Dental Federation study design (Part I) and criteria for evaluation (Part II) of direct and indirect restorations including onlays and partial crowns *Journal of Adhesive Dentistry* (Supplement 9) 121-147.
38. Hickel R, Peschke A, Tyas M, Mjör I, Bayne S, Peters M, Hiller KA, Randall R, Vanherle G, & Heintze SD (2010) FDI World Dental Federation: clinical criteria for the evaluation of direct and indirect restorations— Update and clinical examples *Clinical Oral Investigation* **12**(4) 259-272, <https://doi.org/10.1007/s00784-010-0432-8>.
39. Bittencourt DD, Ezecelevski IG, Reis A, Van Dijken JW, & Loguercio AD (2005) An 18-months' evaluation of self-etch and etch & rinse adhesive in non-carious cervical lesions *Acta Odontologica Scandinavica* **63**(3) 173-178, <https://doi.org/10.1080/00016350510019874>.
40. American Dental Association Council on Scientific Affairs (2001) *Acceptance Program Guidelines: Dentin and Enamel Adhesive Materials* American Dental Association, Chicago IL.
41. Reis A, 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, <https://doi.org/10.2341/17-060-L>.
42. Tay FR, Gwinnett AJ, & Wei SH (1996) The overwet phenomenon: An optical, micromorphological study of surface moisture in the acid-conditioned, resin-dentin interface *American Journal of Dentistry* **9**(1) 43-48.
43. Reis A, Loguercio AD, Azevedo CL, Carvalho RM, Julio Singer M, & Grande RH (2003) Moisture spectrum of demineralized dentin for adhesive systems with different solvent bases *Journal of Adhesive Dentistry* **5**(3) 183-192.
44. Pashley DH, Tay FR, Carvalho RM, Rueggeberg FA, Agee KA, Carrilho M, Donnelly A, & García-Godoy F (2007) From dry bonding to water-wet bonding to ethanol-wet bonding. A review of the interactions between dentin matrix and solvated resins using a macromodel of the hybrid layer *American Journal of Dentistry* **20**(1) 7-20.
45. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Ende A, Van Meerbeek B, & De Munck J (2012) Bonding effectiveness of a new 'multi-mode' adhesive to enamel and dentine *Journal of Dentistry* **40**(6) 475-484, <https://doi.org/10.1016/j.jdent.2012.02.012>.
46. Choi AN, Lee JH, Son SA, Jung KH, Kwon YH, & Park JK (2017) Effect of dentin wetness on the bond strength of universal adhesives *Materials (Basel)* **10**(11) E1224, <https://doi.org/10.3390/ma10111224>.
47. Dal-Bianco K, Pellizzaro A, Patzlaft R, Oliveira Bauer JR, & Loguercio AD (2006) Effects of moisture degree and rubbing action on the immediate resin-dentin bond strength *Dental Materials* **22**(12) 1150-1156, <https://doi.org/10.1016/j.dental.2005.10.010>.

48. Reis A, Pellizzaro A, Dal-Bianco K, Gomes OM, Patzlaff R, & Loguercio AD (2007) Impact of adhesive application to wet and dry dentin on long-term resin–dentin bond strengths *Operative Dentistry* **32**(4) 380-387, <https://doi.org/10.2341/06-107>.
49. Loguercio AD, Raffo J, Bassani F, Balestrini H, Santo D, do Amaral RC, & Reis A (2011) 24-month clinical evaluation in non-carious cervical lesions of a two-step etch-and-rinse adhesive applied using a rubbing motion *Clinical Oral Investigation* **15**(4) 589-596, <https://doi.org/10.1007/s00784-010-0408-8>.
50. Zhang ZY, Tian FC, Niu LN, Ochala K, Chen C, Fu BP, Wang XW, Pashley DH, & FR Tay (2016) Defying ageing: An expectation for dentine bonding with universal adhesives? *Journal of Dentistry* **45** 43-52, <https://doi.org/10.1016/j.jdent.2015.11.008>.
51. Siqueira FSF, Cardenas AM, Ocampo JB, Hass V, Bandeca MC, Gomes JC, Reis A, & Loguercio AD (2018) Bonding performance of universal adhesives to eroded dentin *Journal of Adhesive Dentistry* **20**(2) 121-132, <https://doi.org/10.3290/j.jad.a40300>.
52. Albuquerque EG, Warol F, Calazans FS, Poubel LA, Marins SS, Matos T, Hanzen T, Barceiro MO & Loguercio AD (2017) A new universal simplified adhesive: 6-month randomized multi-center clinical trial *Brazilian Journal of Dentistry* **74**(4) 251-260, <https://doi.org/10.18363/rbo.v74n4.p.251>.
53. Cardenas AM, Siqueira F, Rocha J, Szesz AL, Anwar M, El-Askary F, Reis A, & Loguercio AD (2016) Influence of conditioning time of universal adhesives on adhesive properties and enamel etching pattern *Operative Dentistry* **41**(5) 481-490, <https://doi.org/10.2341/15-213-L>.
54. Sato T, Takagaki T, Matsui N, Hamba H, Sadr A, Nikaido T, & Tagami J (2016) Morphological evaluation of the adhesive/enamel interfaces of two-step self-etching adhesives and multimode one-bottle self-etching adhesives *Journal of Adhesive Dentistry* **18**(3) 223-229, <https://doi.org/10.3290/j.jad.a36135>.
55. Loguercio AD, Munoz MA, Luque-Martinez I, Hass V, Reis A, & Perdigão J (2015) Does active application of universal adhesives to enamel in self-etch mode improve their performance? *Journal of Dentistry* **43**(9) 1060-1070, <https://doi.org/10.1016/j.jdent.2015.04.005>.
56. Takahashi H, Finger WJ, Wegner K, Utterodt A, Komatsu M, Wöstmann B, & Balkenhol M (2010) Factors influencing marginal cavity adaptation of nanofiller containing resin composite restorations *Dental Materials* **26**(12) 1166-1175, <https://doi.org/10.1016/j.dental.2010.08.189>.
57. Opdam NJ, Roeters JJ, Joosten M, & Veeke OV (2002) Porosities and voids in class I restorations placed by six operators using a packable or syringable composite *Dental Materials* **18**(1) 58-63.
58. Opdam NJ, Roeters JJ, de Boer T, Pesschier D, & Bronkhorst E (2003) Voids and porosities in class I micropreparations filled with various resin composite *Operative Dentistry* **28**(1) 9-14.
59. Canali GD, Ignácio SA, Rached RN, & Souza EM (2019) One-year clinical evaluation of bulk-fill flowable vs. regular nanofilled composite in non-carious cervical lesions *Clinical Oral Investigation* **23**(2) 889-897, <https://doi.org/10.1007/s00784-018-2509-8>.
60. Marquillier T, Doméjean S, Le Clerc J, Chemla F, Gritsch K, Maurin JC, Millet P, Pérard M, Grosogeat B, & Dursun E (2018) The use of FDI criteria in clinical trials on direct dental restorations: A scoping review *Journal of Dentistry* **68** 1-9, <https://doi.org/10.1016/j.jdent.2017.10.007>.
61. Frankenberger R, Lohbauer U, Roggendorf MJ, Naumann M, & Taschner M (2008) Selective enamel etching reconsidered: better than etch-and-rinse and self-etch? *Journal of Adhesive Dentistry* **10**(5) 339-344.
62. Erickson RL, Barkmeier WW, & Latta MA (2009) The role of etching in bonding to enamel: A comparison of self-etching and etch-and-rinse adhesive systems *Dental Materials* **25**(11) 1459-1467, <https://doi.org/10.1016/j.dental.2009.07.002>.
63. Hickel R, Brühaver 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>.

Influence of Silver Diamine Fluoride Treatment on the Microtensile Bond Strength of Glass Ionomer Cement to Sound and Carious Dentin

M Jiang • ML Mei • MCM Wong • CH Chu • ECM Lo

Clinical Relevance

This study provides valuable information about the influence of silver diamine fluoride (SDF) treatment on the microtensile bond strength of glass ionomer cement (GIC) to dentin.

SUMMARY

Objectives: To investigate the influence of silver diamine fluoride (SDF) treatment on the microtensile bond strength (mTBS) of glass ionomer cement (GIC) to sound and artificial carious dentin.

Methods: Thirty dentin blocks prepared from 30 noncarious human molars were randomly allocated into either the sound (Gp1) or arti-

cial carious dentin (Gp2) groups. A microbiological method was adopted to create artificial dentin caries lesions in Gp2 specimens. Each dentin block was sectioned into two halves perpendicularly, and each pair of block halves was randomly assigned to two subgroups to receive topical application of SDF (Gp1-SDF, Gp2-SDF) or water as control (Gp1-water, Gp2-water). An encapsulated GIC was bonded to the exposed dentin surfaces 14 days after the SDF/water application. After immersion for 7 days in artificial saliva, the GIC-dentin specimens were sectioned into beams for mTBS testing. Failure mode was examined after the mTBS test.

Results: There was no significant difference in the mean mTBS values between the SDF and control subgroups (Gp1-SDF vs Gp1-water, 10.57 ± 1.6 MPa vs 10.20 ± 1.8 MPa; Gp2-SDF vs Gp2-water, 6.14 ± 2.2 MPa vs 5.97 ± 2.3 MPa; paired *t*-test, $p > 0.05$). However, the mean mTBS value of the sound dentin group was significantly higher than that of the carious dentin group, irrespective of whether SDF was applied prior to GIC bonding (independent *t*-

Meng Jiang, BDS, MDS, Faculty of Dentistry, The University of Hong Kong, Sai Ying Pun, Hong Kong, China

May Lei Mei, BDS, MDS, PhD, Faculty of Dentistry, The University of Otago, Dunedin, New Zealand

May Chun Mei Wong, PhD, Faculty of Dentistry, The University of Hong Kong, Sai Ying Pun, Hong Kong, China

Chun Hung Chu, BDS, MDS, PhD, Faculty of Dentistry, The University of Hong Kong, Sai Ying Pun, Hong Kong, China

*Edward Chin Man Lo, BDS, MDS, PhD, Faculty of Dentistry, The University of Hong Kong, Sai Ying Pun, Hong Kong, China

*Corresponding author: 3F, Dental Public Health, The Prince Philip Dental Hospital, 34 Hospital Road, Sai Ying Pun, Hong Kong, China; e-mail: edward-lo@hku.hk

<https://doi.org/10.2341/19-237-L>

test, $p < 0.001$). Proportionally more cohesive failures occurred in the sound dentin groups (Gp1-SDF, 48.4%; Gp1-water, 42.9%) compared with the carious dentin groups (Gp2-SDF, 15.6%; Gp2-water, 9.8%; $p < 0.05$).

Conclusions: SDF treatment had no significant influence on the mTBS of GIC to dentin. Compared with sound dentin, dentin with caries had lower mTBS to GIC.

INTRODUCTION

Dental caries is a multifactorial disease involving interactions between the tooth structure, the microbial biofilm formed on tooth surface, and sugar intake.¹ Pathological factors can cause an imbalance between demineralization and remineralization, leading to a net loss of minerals from dental hard tissues and ultimately progressing into a carious lesion.¹ Complete removal of all carious dental tissues followed by placement of a restoration in the cavity has been considered the standard procedure for dental caries treatment. However, this approach has been challenged, as modern understanding of dental caries is that caries is not an infectious disease to be cured by removing all bacteria through extended cavity preparation. It is recommended to preserve not only sound dental tissues but also tissues with potential to remineralize, thus maximizing the healing potential of the tooth.^{2,3} Indeed, nonrestorative treatments, even with no removal of carious tissue, are options for the management of dental caries.^{4,5}

As a nonrestorative caries management method, topical application of silver diamine fluoride (SDF) solution has been shown to be effective in arresting dentin caries in young children⁶⁻⁹ as well as in older adults.¹⁰ SDF solution is a colorless solution containing diamine-silver ions and fluoride ions. The diamine-silver ion is a complex with two ammonia molecules attached to a silver ion, which makes it more stable and less oxidizing than the silver ion.¹¹ Silver can inhibit bacterial growth and dentin collagen degradation. The fluoride ion is known to promote remineralization of dental tissues by forming a mixture of fluorapatite and fluorohydroxyapatite on the tooth surface. The combination of silver and fluoride in an alkaline solution has a synergistic effect in arresting active dentin caries, which makes SDF different from other topical fluoride agents.¹² Hence, SDF solution not only acts as an anticariogenic agent to reduce the growth of cariogenic biofilm¹³ but also helps carious dentin to remineral-

ize and results in reduced solubility by transforming some hydroxyapatite into fluorohydroxyapatite.^{14,15}

Although application of SDF can arrest active caries effectively, there are limitations when only SDF treatment is used. Since SDF application is a nonrestorative approach, the cavities in teeth are still present after the application. This might cause problems in oral hygiene effectiveness because food debris and dental plaque can accumulate in the unrestored cavities. Furthermore, the chewing function of broken-down teeth might be compromised. In addition, a drawback of SDF application is the black stain on the arrested caries lesions,¹⁶ which may give rise to esthetic concerns. It was reported that placement of restorations in the SDF-treated caries lesions to cover the black stain increased parental satisfaction with their child's dental appearance.¹⁷ Hence, restoration placement after SDF application can aid plaque control, restore chewing function, and improve dental appearance.

Atraumatic restorative treatment (ART) developed in the 1980s is a minimally invasive operative approach for managing cavitated dentin carious lesions. The ART procedures require removal of soft carious dental tissues using hand instruments without administration of local anesthesia. The cavity is then cleaned and restored with adhesive dental materials, normally high-viscosity glass ionomer cement (GIC), and the adjacent pits and fissures are sealed concurrently.¹⁸ Single-surface ART restorations in both primary and permanent teeth have a high retention/survival rate and are recommended for use in clinical practice.¹⁹ Thus, the ART approach can be adopted to restore SDF-treated dentin caries lesions to reshape the tooth contour and to cover the black stain so as to improve both tooth function and esthetics. It is important to determine whether SDF treatment of the dentin surface influences the bond strength of dentin to GIC.

Previous studies achieved varying results regarding the effect of SDF application on the bond strength of dentin to GIC.²⁰⁻²⁴ Nonstandard protocols to prepare specimens can lead to contradictory results.²⁵ Some of these laboratory studies adopted methods of SDF application that were not recommended or practical in a clinical situation, such as rinsing the SDF away immediately after application²⁰ or leaving it to air-dry on the dentin surface for a very long period of time.²² In addition, the test dental substrates varied between studies, including sound, demineralized, and/or natural carious dentin.

It was reported that the mean microtensile bond strength (mTBS) value of GIC to demineralized dentin was significantly lower than that to sound dentin.²⁶ However, this was not supported by other studies,^{22,27,28} in which similar bond strengths were found irrespective of whether the substrate was carious or sound dentin.

In this study, specimens were prepared in ways that simulated clinical situations. The aim was to investigate the influence of SDF treatment on the bond strength of GIC to both sound and artificial carious dentin. Two null hypotheses were tested: 1) SDF treatment has no effect on the bond strength of GIC to sound and carious dentin and 2) there is no difference in the bond strength to GIC between sound and carious dentin.

METHODS AND MATERIALS

Specimen Preparation

The specimen preparation process is shown in Figure 1. Thirty extracted noncarious human molars stored in 0.5% thymol solution before use were selected from the tooth collection of a dental teaching hospital. A low-speed saw (ISOMET 1000, Buehler, LakeBluff, IL, USA) with a diamond blade was used to remove the occlusal, mesial, distal, buccal, and lingual enamel as well as the roots of all 30 teeth under running deionized water to create dentin blocks of approximately 6 mm × 4 mm × 6 mm each. The blocks were examined visually with magnification to ensure that there was no enamel on the surface. The dentin blocks were randomly allocated into two groups: the sound dentin group (Gp1) and the artificial carious dentin group (Gp2). With the occlusal surface facing up, each dentin block was sectioned into two halves perpendicularly using the same saw mentioned above. Each pair of tooth halves in both Gp1 and Gp2 was randomly assigned to two subgroups, SDF (Gp1-SDF, Gp2-SDF) and control (Gp1-water, Gp2-water) subgroups, with 15 dentin blocks in each subgroup. The exposed pulp chamber of each dentin block was cleaned with an excavator and then filled with resin composite (DUO-LINK, Bisco, Schaumburg, IL, USA) to act as support. The nonocclusal dentin surfaces were coated with a layer of acid-resistant nail varnish (Clarins, Paris, France) to prevent demineralization in the following artificial caries development procedure. The occlusal dentin surface was polished with a 2000-grit silicon carbide paper under running deionized water to create a fresh and uniform dentin surface.

In this study, 5 common species of cariogenic bacteria—*Streptococcus mutans* ATCC (American Type Culture Collection) 35668, *Streptococcus sobrinus* ATCC 33478, *Lactobacillus acidophilus* ATCC 9224, *Lactobacillus rhamnosus* ATCC 10863, and *Actinomyces naeslundii* ATCC 12014—were chosen to create artificial caries lesions in the dentin blocks. Five equal aliquots of 10⁷ CFU/mL of each bacterium in brain-heart infusion broth were mixed and inoculated on the exposed surface of the 30 dentin blocks in Gp2. Afterward, each dentin block was put into 1 mL of 5% sucrose solution in one well of a 24-well plate and incubated anaerobically at 37°C for three days.²⁹

After rinsing with water, the exposed surface of the dentin block was dried with cotton pellets. Then, 38% SDF solution (Saforide, Toyo Seiyaku Kasei Co Ltd, Osaka, Japan) was applied onto the exposed dentin surface of the dentin blocks in the SDF subgroups (Gp1-SDF and Gp2-SDF) with a microbrush (microapplicator, regular, Premium Plus International Ltd, Hong Kong) for 10 seconds and remained undisturbed for about 15 seconds. Dentin blocks in the control subgroups (Gp1-water and Gp2-water) received deionized water following the same procedure described above. After the application of SDF/water, the dentin blocks were immersed into artificial saliva for 14 days before bonding to GIC. The artificial saliva was refreshed every 24 hours.

Silicone impression material (DMG, Chemisch-Pharmazeutische Fabrik GmbH, Hamburg, Germany) was used to create a mold to help bond the GIC to the exposed dentin surface. Before bonding, the dentin surface was cleaned by deionized water and then dried with cotton pellets. The surface was then conditioned by the polyacrylic acid of a GIC product (Ketac-Molar, 3M ESPE Dental Products, St Paul, MN, USA) for 10 seconds. The surface was cleaned for one to two seconds with cotton pellets dipped in water and then blot dried. An encapsulated GIC material (Ketac-Molar Aplicap, 3M ESPE Dental Products) was bonded to the exposed dentin surface following the manufacturer's instructions, that is, mixed in a high-frequency electric-driven mixing device for 10 seconds and then placed onto the dentin surface with an applicator. After setting for two minutes, the GIC-dentin specimen was removed from the mold. The GIC-dentin specimen was then coated with a layer of petroleum jelly (Vaseline, Unilever, Englewood Cliffs, NJ, USA) to mimic the recommended clinical practice in ART

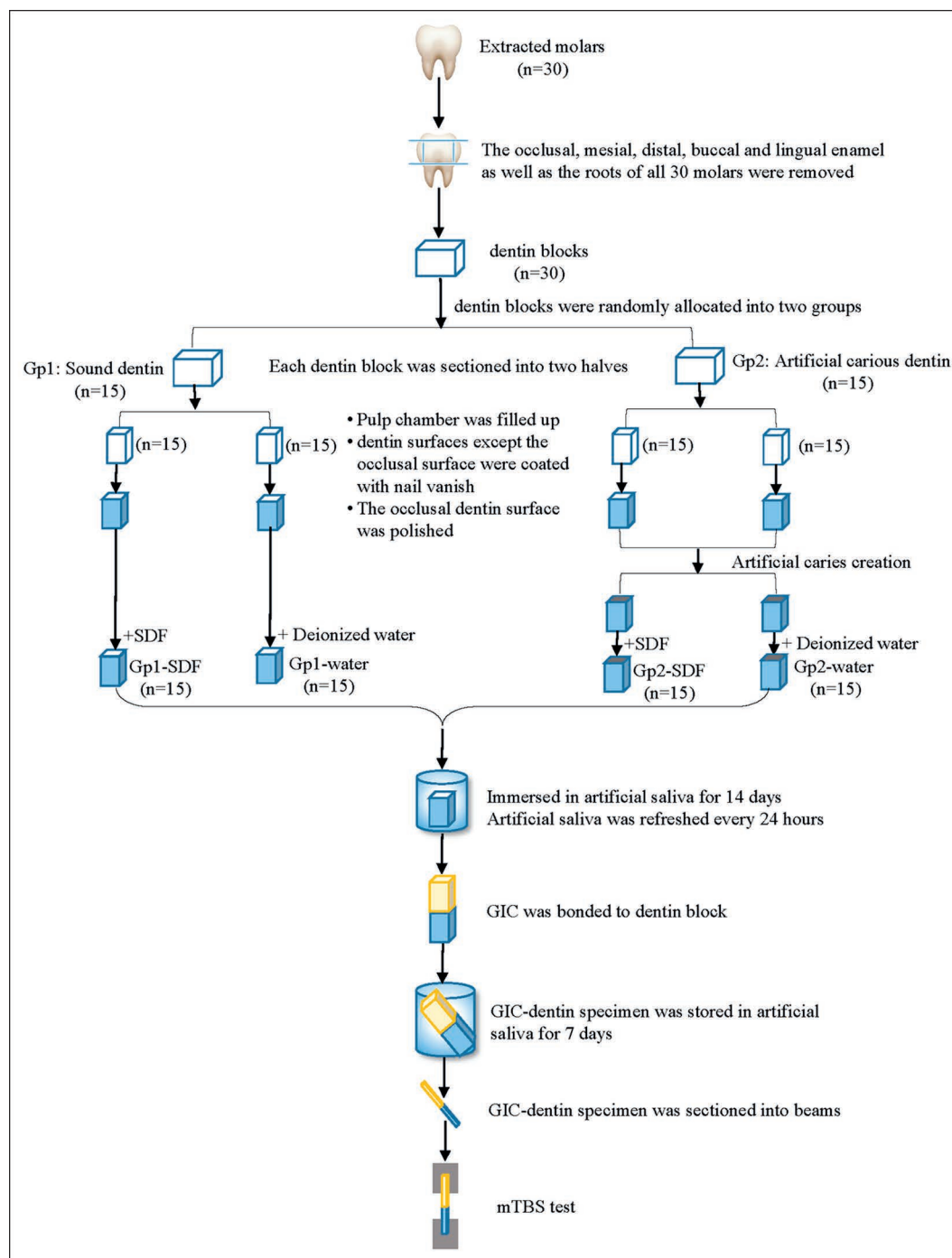


Figure 1. Schematic of sample preparation.

approach and stored in artificial saliva for seven days before mTBS testing.

mTBS Test

The GIC-dentin specimens were sectioned into beams perpendicular to the interface surface that were approximately 1 mm × 1 mm in cross section.

For each GIC-dentin specimen, three to five beams were successfully obtained. The length and width of the bonded surface area were measured by a digital caliper (Mitutoyo, Kawasaki, Japan). The mTBS of each beam was tested by a universal testing machine (model 4444, Instron, Norwood, MA, USA) using a cyanoacrylate glue (Zapit, Dental Ventures of Amer-

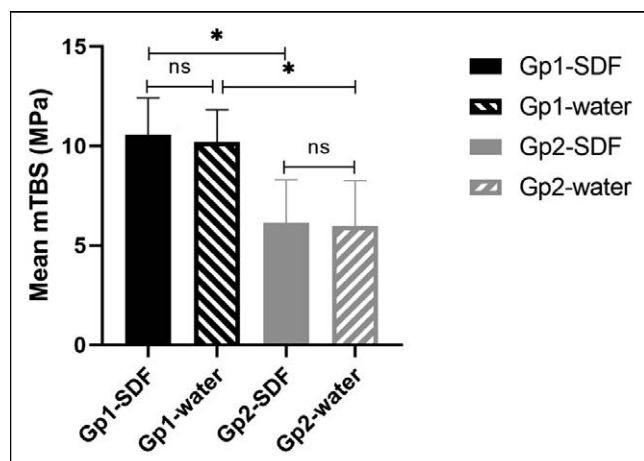


Figure 2. Mean mTBS values of the four study subgroups; ns, no significant difference (paired *t*-test); *, $p < 0.001$ (independent sample *t*-test).

ica, Corona, CA, USA) with a 100-N load cell at a crosshead speed of 1 mm/min. The mTBS value (MPa) of each beam was calculated by dividing the breaking peak value (N) by the bonded surface area (mm^2).

Failure Mode Analysis

Failure mode of the bonded surface was examined with a scanning electron microscope (SEM; SUI5IO, Hitachi, Japan). The specimens were coated with 80% Pt and 20% Pd for 2.5 minutes before scanning. Failure modes were classified into three types: 1) adhesive failure at the dentin and GIC material interface, 2) cohesive failure within the GIC material or dentin, and 3) mixed failure with a combination of both types of failures.

Data Analysis

Since the beams from the same GIC-dentin specimen cannot be considered as independent samples, the mean mTBS value of all successfully sectioned beams from the same GIC-dentin specimen was used as the mTBS value of that specimen.

All statistical analyses were conducted using statistical software (SPSS, Versin25, IBM Corp, Armonk, NY, USA). Paired *t*-tests were used to assess the mTBS values of paired specimens in the SDF and control subgroups. Independent sample *t*-tests were conducted to compare the mTBS values of the specimens in the sound and carious dentin groups. In addition, the distribution of failure modes in the four study subgroups was compared by chi-square testing with multiple comparisons using the Bonferroni method. The statistical significance of all tests was set at 0.05.

RESULTS

The mean area of the bonded surface was $1.087 \pm 0.159 \text{ mm}^2$, with no significant differences among the four study subgroups ($p > 0.05$). The mean mTBS values of Gp1-SDF ($10.57 \pm 1.6 \text{ MPa}$) and Gp1-water ($10.20 \pm 1.8 \text{ MPa}$) were not significantly different ($p > 0.05$; Figure 2). Similarly, there was no significant difference between the mean mTBS values of Gp2-SDF ($6.14 \pm 2.2 \text{ MPa}$) and Gp2-water ($5.97 \pm 2.3 \text{ MPa}$; $p > 0.05$). However, the mean mTBS values of the sound dentin groups were significantly higher than those of the carious dentin groups, irrespective of whether SDF was applied prior to GIC bonding ($p < 0.001$).

The distribution of failure modes of the four study subgroups is shown in Table 1. Three types of failure modes, adhesive (47.9%), cohesive (30.2%), and mixed (21.9%) failure, were observed. The representative SEM images of the GIC-dentin interfaces are displayed in Figure 3. All cohesive failures occurred in the GIC materials with no dentin surface exposed. Proportionally more cohesive failures were observed in the sound dentin groups (Gp1-SDF, 48.4%; Gp1-water, 42.9%) compared with the carious dentin groups (Gp2-SDF, 15.6%; Gp2-water, 9.8%; $p < 0.05$). Mixed failure mode was more often observed in the Gp2-SDF (34.4%) group compared with the Gp1-SDF group (10.9%; $p < 0.05$). There was no significant

Table 1: Distribution of Failure Modes in the Four Study Subgroups^a

Failure Mode	Group, n (%)				Total
	Gp1-SDF*	Gp1-Water*	Gp2-SDF*	Gp2-Water*	
Adhesive	26 (40.6)	26 (41.3)	32 (50.0)	32 (58.8)	116 (47.9)
Cohesive	31 (48.4) A	27 (42.9) A	10 (15.6) B	5 (9.8) B	73 (30.2)
Mixed	7 (10.9) A	10 (15.9) AB	22 (34.4) B	14 (27.5) AB	53 (21.9)
Total	64 (100.0)	63 (100.0)	64 (100.0)	51 (100.0)	242 (100.0)

^a Multiple comparison among the four subgroups was tested by Bonferroni method. Different letters denote that groups in the same failure mode (within the same row) differ significantly at the 0.05 level.

* $p < 0.001$; the *p*-value was derived from the chi-square test.

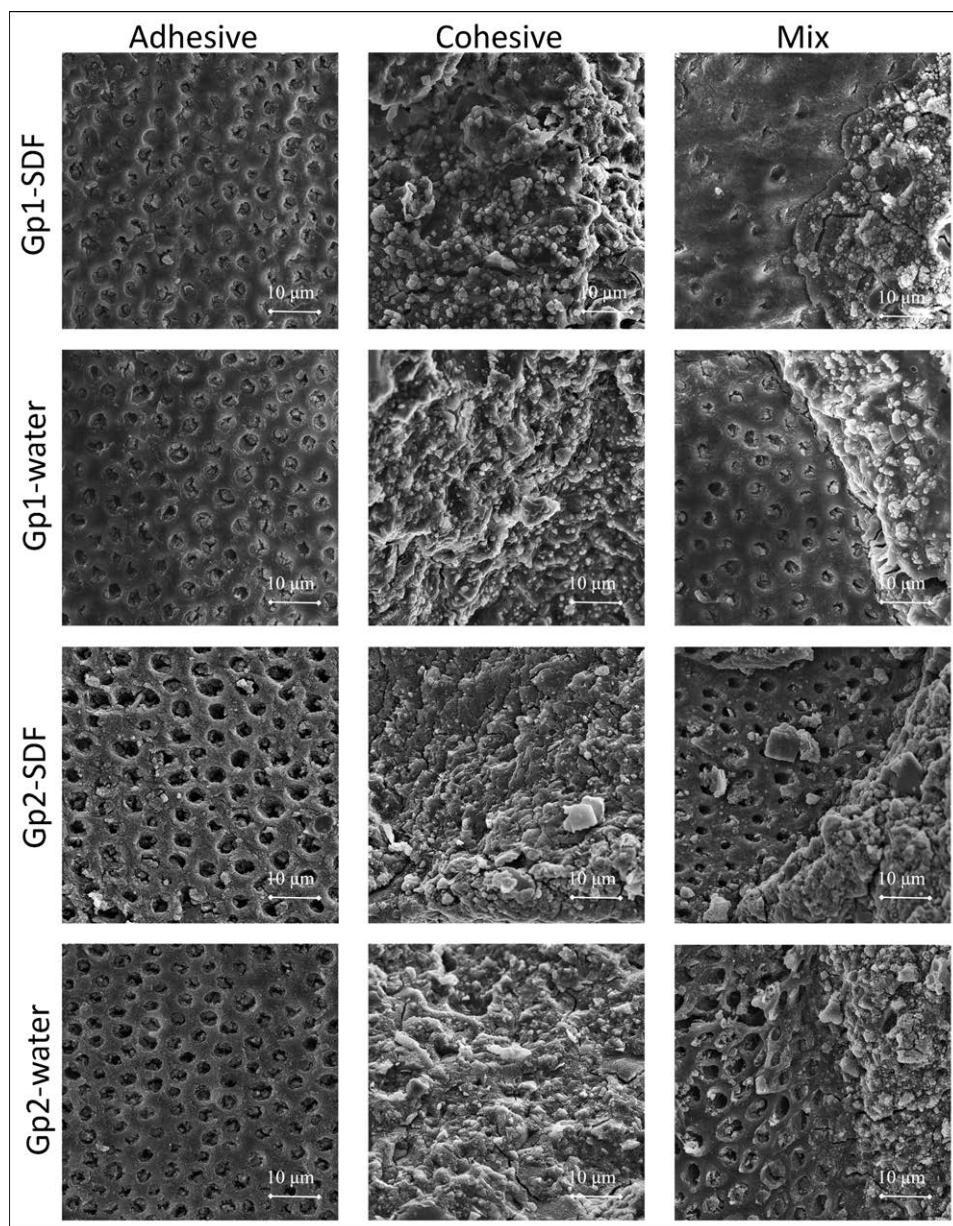


Figure 3. Representative SEM images of failure mode at 1500 $\times$ magnification.

difference in failure mode distribution between the SDF and control subgroups (Gp1-SDF vs Gp1-water, Gp2-SDF vs Gp2-water; $p>0.05$).

DISCUSSION

In the present study, because the differences in mTBS values between the SDF and control subgroups were not statistically significant, the first null hypothesis could not be rejected. On the contrary, the mTBS of carious dentin to GIC was significantly lower than that of sound dentin to GIC in both the SDF and control subgroups. Therefore, the second null hypothesis was rejected.

A microbiological method using five common species of cariogenic bacteria was adopted to create the artificial dentin caries lesions in the present study. In natural dentin caries lesions, the collagen in the outer carious dentin degrades quantitatively and qualitatively.³⁰ Compared with those induced by chemical methods, dentin caries lesions created by the microbiological method are morphologically closer to natural caries lesions.³¹ Subsequently, the SDF-treated dentin blocks were placed in artificial saliva for 14 days to provide an environment high in calcium and phosphate ions for remineralization. However, it was still different from the complex *in*

vivo situation. Therefore, caution should be exercised when interpreting the study results.

There was no significant difference in the bond strength between the SDF and control subgroups in the current study. Previous studies reported inconsistent results regarding the effect of SDF application on the bond strength of restorative materials to dentin. Some studies reported that SDF had no significant influence,^{20,21,32,33} while others concluded that SDF solution application reduced the bond strength.^{23,24,34,35} It is noted that the different studies used different protocols to prepare specimens for testing, and this might be a reason for the inconsistent findings.²⁵ In clinical practice, SDF is likely to be applied on dentin with a microbrush without immediate rinsing with water or drying with compressed air after the application. Patients are usually instructed not to eat or drink for at least half an hour after SDF application.⁸ It seems impossible to have the SDF naturally air dried on the dentin surface in the oral cavity since the mouth is always moist with saliva being present. Hence, in the present study, instead of rinsing with water or air-drying immediately after SDF application, the treated dentin blocks were stored in artificial saliva after SDF application to simulate the clinical situation.

Furthermore, different from previous studies in which the SDF-treated dentin surface was polished with a 600-grit silicon carbide paper,^{21,34} the SDF-treated dentin surface in the present study was simply cleaned with water without any polishing before GIC bonding. This was done to simulate the clinical practice of the ART procedure in which the SDF-arrested hard dentin surface would not be removed before restoring the cavity with GIC. *Ex vivo* studies found that there was a significant increase in microhardness with an elevated level of calcium and phosphorus in the outmost surface layer of SDF-arrested dentin caries lesions.^{14,36} Meanwhile, fluorohydroxyapatite with reduced solubility formed by the reaction of SDF with calcium and phosphate is considered a main factor favoring remineralization and caries arrest.¹⁵ In clinical practice, as recommended by the minimally invasive approach to maximize the preservation of tooth tissue, no action should be taken to remove the SDF-treated dentin before placement of GIC restorations. Therefore, it is important to keep the SDF-treated dentin surface without any removal and polishing when preparing specimens to assess the bond strength of GIC to SDF-treated dentin.

In the present study, it was found that the bond strength of carious dentin to GIC was significantly lower than that of sound dentin. This finding is in agreement with that of a previous study.²⁶ The chemical bonding between GIC and dental substrate involves the formation of ionic bonds between the carboxylate functional groups in the polyalkenoic acid molecules and calcium ions in the hydroxyapatite.³⁷ As caries progresses, demineralization will lead to a loss of calcium ions, and there will be less opportunity for bonding between calcium ions and the carboxyl groups, resulting in reduced bond strength. Therefore, it is expected that the bond strength of GIC to carious dentin is lower than that of GIC to sound dentin.

Three failure modes were observed under SEM in the present study, and the distribution of failure modes was not significantly different between the SDF and control subgroups. In contrast, compared with the artificial caries groups, the sound dentin groups had higher mean mTBS values and more cohesive failures within GIC. This finding is consistent with that of a previous study.²⁶ The difference in the prominent failure modes between the sound and carious dentin groups is probably related to the considerably lower bond strength of GIC to carious dentin. Nevertheless, cohesive failure is considered as a measure of the tensile strength of the GIC material, rather than the true adhesive bond strength.³⁷

In the present study, an encapsulated chemically cured GIC was used to test bond strength. Because the powder/liquid ratio influences the physical properties of GIC,³⁸ an encapsulated GIC instead of a hand-mixed one was used to standardize the preparation of specimen. Despite this, a limitation of this study is that only one commercial brand of conventional chemical-cured GIC was used. Resin-modified GIC (RMGIC) containing a polymerizable monomer, 2-hydroxyethyl methacrylate (HEMA), as an additional component is considered to have a better bonding performance to dental substrates compared with conventional GIC.^{26,37,39} The presence of HEMA enhances the micromechanical interlocking between the dentin surface and cement, which leads to a higher bond strength.⁴⁰ Therefore, further study is needed to determine whether application of SDF on the dentin surface affects HEMA polymerization, which may lead to a change of bond strength of RMGIC to dentin.

The mTBS test was adopted in the present study to investigate the bond strength of GIC to dentin. The mTBS test has several advantages over the

traditional TBS test, such as a greater percentage of adhesive failures and higher possibility of measuring a high bond strength value.⁴¹ In addition, the mTBS test is thought to have greater discriminative power than the traditional shear bond strength (SBS) test.²⁵ The SBS test is considered to have very little value in the prediction of clinical performance, whereas the mTBS was reported to be associated with the retention rate of Class V restorations in clinical studies.⁴² However, a study reported that there was no correlation between bond test results and the retention rate of restorations and only a moderate correlation between mTBS test results and marginal discoloration of restorations.⁴³ The uncertain correlation between bond strength test results and clinical parameters indicates that one should not solely rely on laboratory bond strength tests. Instead, well-designed clinical trials are needed to determine the performance of restorations in a clinical situation.

CONCLUSION

Within the limitations of the present study, it was concluded that SDF treatment had no significant influence on the mTBS of GIC to dentin. However, compared with sound dentin, carious dentin had a lower bond strength to GIC.

Acknowledgements

The authors would like to thank Ms Joyce Yau and Mrs Geoffrey Ng, Ying Yip Chui, and Paul Lee for their assistance in specimen preparation and SEM scanning in this study. This work was supported by the Tam Wah Ching endowed professorship in dental science of the University of Hong Kong.

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 Hong Kong. The approval code issued for this study is UW 19-253.

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 14 January 2020)

REFERENCES

1. Pitts NB, Zero DT, Marsh PD, Ekstrand K, Weintraub JA, Ramos-Gomez F, Tagami J, Twetman S, Tsakos G, & Ismail A (2017) Dental caries *Nature Reviews Disease Primers* **3**(4) 17030. <http://dx.doi.org/10.1038/nrdp.2017.30>
2. Featherstone JD & Domejean S (2012) Minimal intervention dentistry: part 1. From 'compulsive' restorative dentistry to rational therapeutic strategies *British Dental Journal* **213**(9) 441-445.
3. Frencken JE, Peters MC, Manton DJ, Leal SC, Gordan VV, & Eden E (2012) Minimal intervention dentistry for managing dental caries—a review: report of a FDI task group *International Dental Journal* **62**(5) 223-243.
4. Ricketts D, Lamont T, Innes NP, Kidd E, & Clarkson JE (2013) Operative caries management in adults and children *Cochrane Database Systematic Review* (3) CD003808. <http://dx.doi.org/10.1002/14651858.CD003808.pub3>
5. Urquhart OA-Ohoo, Tampi MP, Pilcher L, Slayton RL, Araujo MWB, Fontana M, Guzman-Armstrong S, Nascimento MM, Novy BB, Tinanoff N, Weyant RJ, Wolff MS, Young DA, Zero DT, Brignardello-Petersen R, Banfield L, Parikh A, Joshi G, & Carrasco-Labra A (2019) Nonrestorative treatments for caries: systematic review and network meta-analysis *Journal of Dental Research* **98**(1) 14-26.
6. Yee R, Holmgren C, Mulder J, Lama D, Walker D, & Helderman WV (2009) Efficacy of silver diamine fluoride for arresting caries treatment *Journal of Dental Research* **88**(7) 644-647.
7. Zhi QH, Lo ECM, & Lin HC (2012) Randomized clinical trial on effectiveness of silver diamine fluoride and glass ionomer in arresting dentine caries in preschool children *Journal of Dentistry* **40**(11) 962-967.
8. Duangthip D, Wong MCM, Chu CH, & Lo ECM (2018) Caries arrest by topical fluorides in preschool children: 30-month results *Journal of Dentistry* **70** 74-79.
9. Fung MHT, Duangthip D, Wong MCM, Lo ECM, & Chu CH (2018) Randomized clinical trial of 12% and 38% silver diamine fluoride treatment *Journal of Dental Research* **97**(2) 171-178.
10. Li R, Lo ECM, Liu BY, Wong MCM, & Chu CH (2016) Randomized clinical trial on arresting dental root caries through silver diamine fluoride applications in community-dwelling elders *Journal of Dentistry* **51** 15-20.
11. Chu CH & Lo EC (2008) Promoting caries arrest in children with silver diamine fluoride: a review *Oral Health & Preventive Dentistry* **6**(4) 315-321.
12. Mei ML, Lo ECM, & Chu CH (2018) Arresting dentine caries with silver diamine fluoride: what's behind it? *Journal of Dental Research* **97**(7) 751-758.
13. Chu CH, Mei L, Seneviratne CJ, & Lo EC (2012) Effects of silver diamine fluoride on dentine carious lesions induced by *Streptococcus mutans* and *Actinomyces naeslundii* biofilms *International Journal of Paediatric Dentistry* **22**(1) 2-10.
14. Mei ML, Ito L, Cao Y, Lo EC, Li QL, & Chu CH (2014) An *ex vivo* study of arrested primary teeth caries with silver diamine fluoride therapy *Journal of Dentistry* **42**(4) 395-402.
15. Mei ML, Nudelman F, Marzec B, Walker JM, Lo ECM, Walls AW, & Chu CH (2017) Formation of fluorohydroxyapatite with silver diamine fluoride *Journal of Dental Research* **96**(10) 1122-1128.
16. Duangthip D, Fung MHT, Wong MCM, Chu CH, & Lo ECM (2018) Adverse effects of silver diamine fluoride

- treatment among preschool children *Journal of Dental Research* **97**(4) 395-401.
17. Jiang M, Wong MCM, Chu CH, Dai L, & Lo ECM (2019) Effects of restoring SDF-treated and untreated dentine caries lesions on parental satisfaction and oral health related quality of life of preschool children *Journal of Dentistry* **88** 103171. <http://dx.doi.org/10.1016/j.jdent.2019.07.009>
 18. Holmgren CJ, Roux D, & Domejean S (2013) Minimal intervention dentistry: part 5. Atraumatic restorative treatment (ART)—a minimum intervention and minimally invasive approach for the management of dental caries *British Dental Journal* **214**(1) 11-18.
 19. de Amorim RG, Frencken JE, Raggio DP, Chen X, Hu X, & Leal SC (2018) Survival percentages of atraumatic restorative treatment (ART) restorations and sealants in posterior teeth: an updated systematic review and meta-analysis *Clinical Oral Investigations* **22**(8) 2703-2725.
 20. Zhao IS, Chu S, Yu OY, Mei ML, Chu CH, & Lo ECM (2019) Effect of silver diamine fluoride and potassium iodide on shear bond strength of glass ionomer cements to caries-affected dentine *International Dental Journal* **69**(5) 341-347.
 21. Puwanawiroj A, Trairatvorakul C, Dasanayake AP, & Auychai P (2018) Microtensile bond strength between glass ionomer cement and silver diamine fluoride-treated carious primary dentin *Pediatric Dentistry* **40**(4) 291-295.
 22. Wang AS, Botelho MG, Tsoi JKH, & Matinlinna JP (2016) Effects of silver diamine fluoride on microtensile bond strength of GIC to dentine *International Journal of Adhesion and Adhesives* **70** 196-203.
 23. Koizumi H, Hamama HH, & Burrow MF (2016) Effect of a silver diamine fluoride and potassium iodide-based desensitizing and cavity cleaning agent on bond strength to dentine *International Journal of Adhesion and Adhesives* **68** 54-61.
 24. Knight GM, McIntyre JM, & Mulyani (2006) The effect of silver fluoride and potassium iodide on the bond strength of auto cure glass ionomer cement to dentine *Australian Dental Journal* **51**(1) 42-45.
 25. De Munck J, Mine A, Poitevin A, Van Ende A, Cardoso MV, Van Landuyt KL, Peumans M, & Van Meerbeek B (2012) Meta-analytical review of parameters involved in dentin bonding *Journal of Dental Research* **91**(4) 351-357.
 26. Choi K, Oshida Y, Platt J, Cochran MA, Matis BA, & Yi K (2006) Microtensile bond strength of glass ionomer cements to artificially created carious dentin *Operative Dentistry* **31**(5) 590-597.
 27. Alves F, Hesse D, Lenzi T, Guglielmi CDB, Reis A, Loguercio AD, Carvalho T, & Raggio D (2013) The bonding of glass ionomer cements to caries-affected primary tooth dentin *Pediatric Dentistry* **35**(4) 320-324.
 28. Koizumi H, Hamama HH, & Burrow MF (2016) Evaluation of adhesion of a CPP-ACP modified GIC to enamel, sound dentine, and caries-affected dentine *International Journal of Adhesion and Adhesives* **66** 176-181.
 29. Mei ML, Ito L, Chu CH, Lo ECM, & Zhang CF (2014) Prevention of dentine caries using silver diamine fluoride application followed by Er:YAG laser irradiation: an *in vitro* study *Lasers in Medical Science* **29**(6) 1785-1791.
 30. Nakornchai S, Atsawasuwan P, Kitamura E, Surarit R, & Yamauchi M (2004) Partial biochemical characterisation of collagen in carious dentin of human primary teeth *Archives of Oral Biology* **49**(4) 267-273.
 31. Marquezan M, Correa FN, Sanabe ME, Rodrigues Filho LE, Hebling J, Guedes-Pinto AC, & Mendes FM (2009) Artificial methods of dentine caries induction: a hardness and morphological comparative study *Archives of Oral Biology* **54**(12) 1111-1117.
 32. Wu DI, Velamakanni S, Denisson J, Yaman P, Boynton JR, & Papagerakis P (2016) Effect of silver diamine fluoride (sdf) application on microtensile bonding strength of dentin in primary teeth *Pediatric Dentistry* **38**(2) 148-153.
 33. Quock RL, Barros JA, Yang SW, & Patel SA (2012) Effect of silver diamine fluoride on microtensile bond strength to dentin *Operative Dentistry* **37**(6) 610-616.
 34. Lutgen P, Chan D, & Sadr A (2018) Effects of silver diamine fluoride on bond strength of adhesives to sound dentin *Dental Material Journal* **37**(6) 1003-1009.
 35. Kucukyilmaz E, Savas S, Akcay M, & Bolukbasi B (2016) Effect of silver diamine fluoride and ammonium hexa-fluorosilicate applications with and without Er:YAG laser irradiation on the microtensile bond strength in sound and caries-affected dentin *Lasers in Surgery and Medicine* **48**(1) 62-69.
 36. Chu CH & Lo EC (2008) Microhardness of dentine in primary teeth after topical fluoride applications *Journal of Dentistry* **36**(6) 387-391.
 37. Nicholson JW (2016) Adhesion of glass-ionomer cements to teeth: a review *International Journal of Adhesion and Adhesives* **69**(September) 33-38.
 38. Crisp S, Lewis BG, & Wilson AD (1976) Characterization of glass-ionomer cements. 2. Effect of the powder: liquid ratio on the physical properties *Journal of Dentistry* **4**(6) 287-290.
 39. Palma-Dibb RG, de Castro CG, Ramos RP, Chimello DT, & Chinelatti MA (2003) Bond strength of glass-ionomer cements to caries-affected dentin *Journal of Adhesive Dentistry* **5**(1) 57-62.
 40. Lin A, McIntyre NS, & Davidson RD (1992) Studies on the adhesion of glass-ionomer cements to dentin *Journal of Dental Research* **71**(11) 1836-1841.
 41. Armstrong S, Geraldini S, Maia R, Araujo Raposo LH, Soares CJ, & Yamagawa J (2010) Adhesion to tooth structure: a critical review of "micro" bond strength test methods *Dental Materials* **26**(2) E50-E62.
 42. Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, & De Munck J (2010) Relationship between bond-strength tests and clinical outcomes *Dental Materials* **26**(2) e100-e121. <https://doi.org/10.1016/j.dental.2009.11.148>
 43. Heintze SD, Rousson V, & Mahn E (2015) Bond strength tests of dental adhesive systems and their correlation with clinical results—a meta-analysis *Dental Materials* **31**(4) 423-434. <https://doi.org/10.1016/j.dental.2015.01.011>

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 5
September/October 2020

www.jopdent.org
457-568

CLINICAL TECHNIQUE/CASE REPORT

- 457 Adapted Three-step Restorative Technique: Recovering Dental Substrate Compromised by Complex Erosive Wear in a Young Patient
CA Villavicencio-Espinoza • MC Giacomini • MH Narimatsu • AC Magalhães • MT Atta • L Wang
- 467 Occlusal and Esthetic Enhancement: A Noninvasive Approach to an Old Dilemma
GS Lima • LGB Albino • CS dos Santos • MS Cenci • T Pereira-Cenci

CLINICAL RESEARCH

- 473 Catechin-Based Dentin Pretreatment on the Clinical Performance of a Universal Adhesive: A Two-Year Randomized Clinical Trial
CAGA Costa • NLG Albuquerque • JS Mendonça • AD Loguercio • VPA Saboia • SL Santiago

LITERATURE REVIEW

- 484 Is Conventional Resin Cement Adhesive Performance to Dentin Better Than Self-adhesive? A Systematic Review and Meta-Analysis of Laboratory Studies
LL Miotti • AC Follak • AF Montagner • RT Pozzobon • BL da Silveira • AH Susin

LABORATORY RESEARCH

- 496 Effect of Shortened Light-Curing Modes on Bulk-Fill Resin Composites
CS Sampaio • PG Pizarro • PJ Atria • R Hirata • M Giannini • E Mahn
- 506 Fracture Resistance and Failure Mode of Custom-made Post-and-cores of Polyetheretherketone and Nano-ceramic Composite
KN Teixeira • TM Duque • HP Maia • TMSV Gonçalves
- 516 Effect of Methacryloyloxydecyl Dihydrogen Phosphate-Containing Silane and Adhesive Used Alone or in Combination on the Bond Strength and Chemical Interaction With Zirconia Ceramics Under Thermal Aging
MF Gutierrez • J Perdigão • P Malaquias • AM Cardenas • F Siqueira • V Hass • A Reis • AD Loguercio
- 528 Influence of Material and Surface Roughness of Resin Composite Cements on Fibroblast Behavior
N Rohr • N Bertschinger • J Fischer • A Filippi • NU Zitzmann
- 537 Simulated Cuspal Deflection and Flexural Properties of Bulk-Fill and Conventional Flowable Resin Composites
Y Shimatani • A Tsujimoto • WW Barkmeier • NG Fischer • Y Nagura • T Takamizawa • MA Latta • M Miyazaki
- 547 Effect of the Photo-initiator System Contained in Universal Adhesives on Radicular Dentin Bonding
RF Carvalho • AFM Cardenas • CN Carvalho • JJ de Souza • JRO Bauer • FSF Siqueira • A Armas-Vega • AD Loguercio • V Hass
- 556 Degree of Conversion Contributes to Dentin Bonding Durability of Contemporary Universal Adhesives
A Tichy • K Hosaka • A Abdou • M Nakajima • J Tagami

DEPARTMENTS

- 566 Online Only Articles

ONLINE ONLY ARTICLES

- E217 Post-gel and Total Shrinkage Stress of Conventional and Bulk-fill Resin Composites in Endodontically-treated Molars
RA da Silva Pereira • GF de Bragança • ABF Vilela • RA de Deus • RR Miranda • C Verissimo • CJ Soares
- E227 Tridimensional Evaluation of the Interfacial Gap in Deep Cervical Margin Restorations: A Micro-CT Study
N Scotti • A Baldi • EA Vergano • R Michelotto Tempesta • M Alovise • D Pasqualini • GC Carpegna • A Comba
- E237 Does the Type of Solvent in Dental Adhesives Influence the Clinical Performance of Composite Restorations Placed in Noncarious Cervical Lesions? A Systematic Review and Meta-analysis
RBE Lins • M Sebold • MB Magno • LC Maia • LRM Martins • M Giannini
- E255 A New Dual-cure Universal Simplified Adhesive: 18-month Randomized Multicenter Clinical Trial
E Gomes de Albuquerque • F Warol • F Signorelli Calazans • L Augusto Poubel • S Soares Marins • T Matos • JJ de Souza • A Reis • M de Oliveira Barceiro • AD Loguercio
- E271 Influence of Silver Diamine Fluoride Treatment on the Microtensile Bond Strength of Glass Ionomer Cement to Sound and Carious Dentin
M Jiang • ML Mei • MCM Wong • CH Chu • ECM Lo