OPERATIVE DENTISTRY

Nov/Dec 2016
Volume 41
Number 6
567–682

Academy of Operative Dentistry • American Academy of Gold Foil Operators • Academy of R.V. Tucker Study Clubs

OPERATIVE DENTISTRY

Volume 41 / Number 6
Nov/Dec 2016

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://www.jopdent.com/journal/policies.pdf

Permissions

For permission to reproduce material from Operative Dentistry please apply to Operative Dentistry at the Editorial Office address or via email at editor@jopdent.org.

Online Access

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

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

Editorial Board

Reviewer names available at: www.jopdent.com/journal/editorial_board.html

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

On The Cover

"Sunrise Through Mesa Arch" Canyonlands National Park, Utah, USA. Photo provided by Akimasa Tsujimoto of Tokyo, Japan. Photo taken with a Canon EOS 7D Mark II, 12mm f/5.6 1/30 sec. ISO-125 © Operative Dentistry, Inc.

We welcome the submission of pictures for consideration for use on the cover of Operative Dentistry! All photographs should be submitted via the forms at:

https://www.jopdent.com/journal/journal.html

Editorial Office

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

Operative Dentistry
Indiana University School of Dentistry, Room S411
1121 West Michigan Street, Indianapolis, IN 46202-5186
Phone 317-278-4800, Fax: 317-278-4900
http://www.jopdent.org

Editorial Staff

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 41 / Number 6
Nov/Dec 2016

www.jopdent.org
567–682

Clinical Technique / Case Report

567 Management of Amelogenesis Imperfecta: A 15-Year Case History of Two Siblings
E Dursun, E Savard, C Vargas, L Loison-Robert, H Cherifi, F Bdeoui, M-M Landru

Clinical Research

578 Clinical Evaluation of Genotoxicity of In-office Bleaching
M Rezende, JL De Geus, AD Loguercio, A Reis, D Kossatz

587 A Prospective Six-Year Clinical Study Evaluating Reinforced Glass Ionomer Cements with Resin Coating on Posterior Teeth: Quo Vadis?
LS Türkün, Ö Kanik

599 Clinical Evaluation of Silorane and Nano-hybrid Resin Composite Restorations in Class II Cavities up to 3 Years
F Öztürk-Bozkurt, T Toz, A Kara-Tuncer, H Gözükara-Bağ, M Özcan

Laboratory Research

607 Effect of Preparation Depth on the Marginal and Internal Adaptation of Computer-aided Design/Computer-assisted Manufacture Endocrowns
MD Gaintantzopoulou, HM El-Damanhoury

617 Surface Properties and Color Stability of Resin-Infiltrated Enamel Lesions
X Zhao, Y-F Ren

627 An In Situ Study of the Influence of Staining Beverages on Color Alteration of Bleached Teeth
AL Briso, TC Fagundes, MO Gallinari, J Moreira, LCAG de Almeida, V Rahal, RS Gonçalves, PH dos Santos

634 Influence of Finishing and Polishing Techniques and Abrasion on Transmittance and Roughness of Composite Resins
PMA Carneiro, TM Ramos, CS de Azevedo, E de Lima, SHJ de Souza, ML Turbino, PF Cesar, AB Matos

642 Bacterial Colonization in the Marginal Region of Ceramic Restorations: Effects of Different Cement Removal Methods and Polishing
SMB Pereira, LC Anami, CA Pereira, ROA Souza, KZ Kantorski, MA Bottino, AOC Jorge, LF Valandro

655 Filler Content, Surface Microhardness, and Rheological Properties of Various Flowable Resin Composites
S Jager, R Balthazard, A Dahoun, E Mortier

666 Premolar Axial Wall Height Effect on CAD/CAM Crown Retention
C Gillette, R Buck, N DuVall, S Cushen, M Wajdowicz, H Roberts

Departments

672 Letter to the Editor

675 Faculty Posting

677 Reviewer Recognition

679 Online Only Articles

Online Only Articles

E159 Effect of Intracanal Irrigants on Bond Strength of Fiber Posts Cemented With a Self-adhesive Resin Cement
MS Barreto, RA Rosa, VG Seballos, E Machado, LF Valandro, OB Kaizer, MVR Só, CAS Bier

E168 Multidisciplinary Treatment of Complicated Crown-Root Fractures: A Case Study
IL Stojanac, BV Bajkin, MT Premovic, BD Ramic, LM Petrovic

E174 Relined Fiberglass Post: Effect of Luting Length, Resin Cement, and Cyclic Loading on the Bond to Weakened Root Dentin
NC de Souza, ML Marcondes, DFF da Silva, GA Borges, LH Burnett Júnior, AM Spohr

E183 Influence of the Compliance and Layering Method on the Wall Deflection of Simulated Cavities in Bulk-fill Composite Restoration
Y-J Kim, R Kim, JL Ferracane, I-B Lee

E195 Degradation Potential of Bulk Versus Incrementally Applied and Indirect Composites: Color, Microhardness, and Surface Deterioration
M El Gezawi, D Kaisarly, H Al-Saleh, A ArRejaie, F Al-Harbi, KH Kunzelmann

©Operative Dentistry, 2016, **41-6,** 567-577

Management of Amelogenesis Imperfecta: A 15-Year Case History of Two Siblings

E Dursun • E Savard • C Vargas • L Loison-Robert
H Cherifi • F Bdeoui • M-M Landru

Clinical Relevance

Restoring amelogenesis imperfecta–affected teeth with composite is a viable medium-term treatment in young adults and a conservative and esthetic approach that allows for repairs. Stainless steel crowns may be used to restore deciduous and adult molars. Adjusting crown molds using a thermoforming procedure and buildup of composite shells is an interesting technique for restoring affected premolars and canines.

SUMMARY

Objective: Amelogenesis imperfecta (AI) is a heterogenous genetic disorder that interferes with normal enamel formation in the absence of systemic disorders. The patients' main concerns are caries susceptibility, poor esthetics, and generalized sensitivity. There is a broad clinical spectrum, from discolorations to consequent enamel alterations. This case report describes the 15-year case study and the full-mouth rehabilitation of two siblings affected by a hypocalcified AI.

Clinical Considerations: In these two patients, conservative care with stainless steel crowns and direct composite restorations was undertaken to restore function and esthetics and to reduce sensitivities in primary and mixed dentitions. The difficulties in monitoring resulted in severe infectious complications (dental abscess with cutaneous fistula), important dental defects, and loss of spaces with subsequent malocclusion. In the young adult dentition, they were treated by extractions, root canal therapies, and new restorations: stainless steel crowns for permanent molars, direct composite restorations (with strip crowns) for

*Elisabeth Dursun, DDS, PhD, associate professor, Dental Materials Research Unit, Innovations and Interfaces (URB2i) and Pediatric Dentistry Department, Faculty of Dental Surgery, Paris Descartes University, Montrouge, France; Pediatric Dentistry Department, Albert Chenevier Hospital, Créteil, France

Elodie Savard, DDS, Faculty of Dental Surgery, Paris Descartes University, Montrouge, France; Restorative Dentistry and Pediatric Dentistry Departments, Albert Chenevier Hospital, Créteil, France

Carole Vargas, resident, Orthodontic Department, Rangueil Hospital, Toulouse, France

Ludwig Loison-Robert, DDS, assistant professor, Biology Department, Faculty of Dental Surgery, Paris Descartes University, Montrouge, France; Albert Chenevier Hospital, Prosthodontic Department, Créteil, France

Hafida Cherifi, DDS, assistant professor, Public Health Department, Faculty of Dental Surgery, Paris Descartes University, Montrouge, France; Oral Surgery Department, Albert Chenevier Hospital, Créteil, France

Fadi Bdeoui, DDS, hospital practitioner, Pediatric Dentistry Department, Albert Chenevier Hospital, Créteil, France

Marguerite-Marie Landru, DDS, PhD, associate professor, Pediatric Department Faculty of Dental Surgery, Paris Descartes University, Montrouge, France; Pediatric Dentistry Department, Albert Chenevier Hospital, Créteil, France

*Corresponding author: 1 rue Maurice Arnoux, Montrouge, 92120 France; e-mail: elisabethdursun@gmail.com

DOI: 10.2341/15-372-T

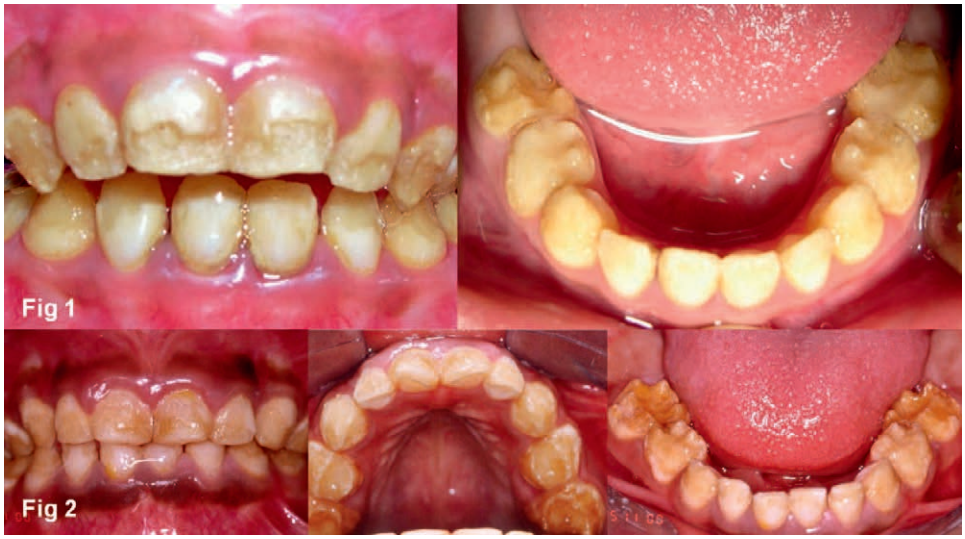


Figure 1. *Intraoral views of the initial situation for the sister in primary dentition (3 years old) affected with a hypocalcified form of AI.*
Figure 2. *Intraoral views of the initial situation for the brother in primary dentition (6 years old) affected with a hypocalcified form of AI.*

incisors and maxillary canines (to improve the crown morphology as well as to mask the discolorations and the malpositions), and adjusted composite crown molds using a thermoforming procedure for premolars and the mandibular canines. The main difficulties were rapid tooth surface loss, bonding to atypical enamel, developing dentition, long-term follow-up.

Conclusion: Restoring function and esthetics in AI-affected patients is a challenge from primary to adult dentition. Early corrections are essential to avoid dental damage and for psychological benefits. This clinical report highlights the adhesive rehabilitation for anterior and premolar areas and the difficulty of patient follow-up.

INTRODUCTION

Amelogenesis imperfecta (AI) is an inherited and clinically heterogeneous disorder of tooth development that could affect all (or nearly all) primary and permanent teeth. Its prevalence ranges from 1:700 to 1:14,000, according to the studied population.[1] Three types have been described: hypocalcified AI (with soft and friable enamel), hypoplastic AI (with normally calcified enamel but deficient in quantity), and hypomaturation AI (characterized by mottled enamel, with opaque white to red-brown discoloration).[2] Furthermore, several other dental anomalies have been associated with AI, such as taurodontism, multiple impacted teeth, congenitally missing teeth, and malocclusions (open bite or class III).[3-5]

Rehabilitation is complex and requires a multidisciplinary approach. The treatment objectives are to reduce sensitivity and caries susceptibility, protect the tooth structure from wear, establish good oral hygiene habits, and restore esthetics and function to improve the patient's quality of life. The multiple treatment phases last several years, and thus the patient's compliance is essential. Different treatment options have been proposed for the management of AI-affected teeth: microabrasion, laminate veneers, composite resin restorations, composite or ceramic onlays, stainless steel crowns, and metal-ceramic/all-ceramic crowns.[6-10]

Nowadays, the treatment approach is rather based on dental tissue preservation. Recent studies have successfully used composite restorations,[7] and some authors even showed that these offered good long-term performance in permanent hypomineralized molars.[11] The aim of this article is to report the 15-year case study of two siblings affected by the hypocalcified form of hereditary AI.

CASES PRESENTATION

During Primary and Mixed Dentition

Two siblings were referred to the Albert Chenevier hospital of Créteil in 2000 for the treatment of discolored and sensitive teeth. Both were diagnosed with hypocalcified form of hereditary AI (Figures 1 and 2). Neither the parents nor the two eldest children seemed to be affected.

The 6-year-old brother was treated first with stainless steel crowns on the primary molars and composite restorations on the primary maxillary anteriors using strip crowns (technique described later in the article; Figure 3). When he was 7 years old and then 9 years old, he received stainless steel

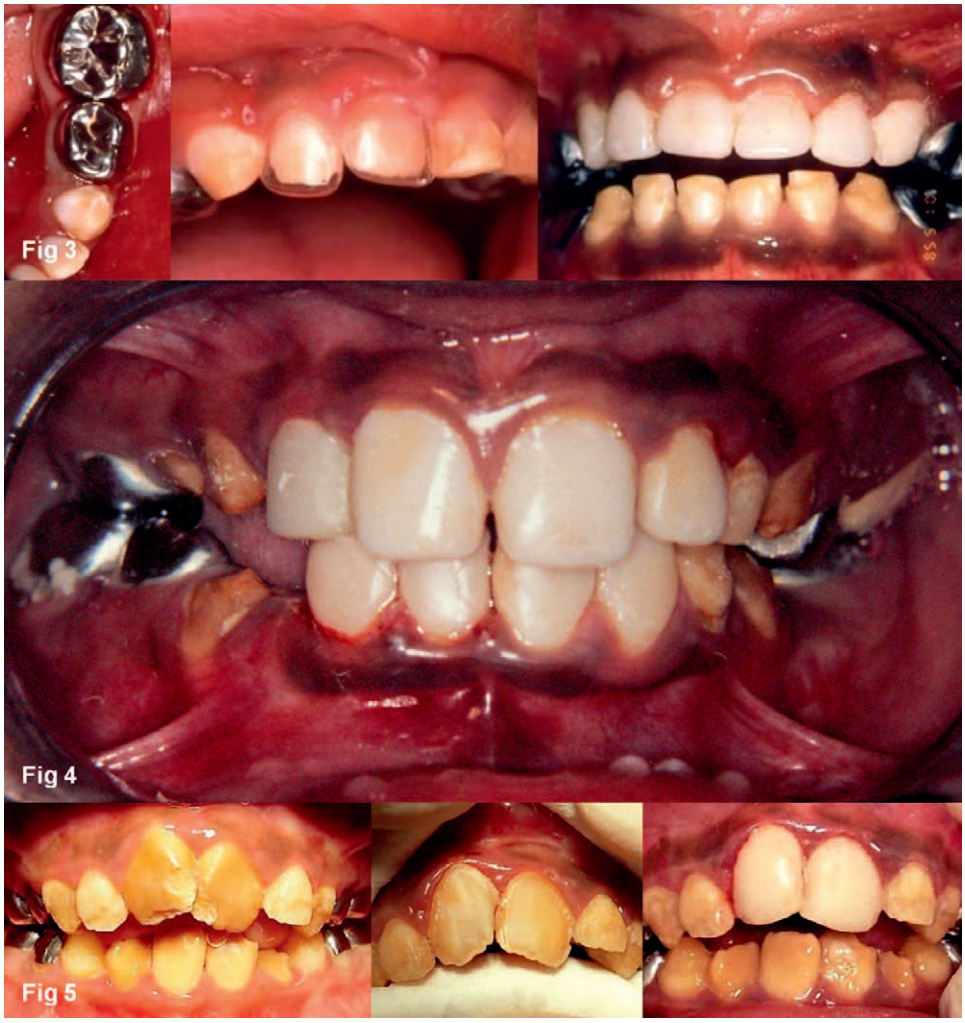


Figure 3. *Placement of stainless steel crowns on the boy's primary molars and composite restorations of the maxillary anterior primary teeth using strip crowns (6 years old).*
Figure 4. *Placement of stainless steel crowns on the boy's first permanent molars and labial composite restorations on the permanent incisors (between 7 and 9 years old).*
Figure 5. *Labial composite restorations of the sister's permanent maxillary central incisors (5 years old).*

crowns on the first permanent molars and direct composite restorations on the permanent centrals (Figure 4). At the age of 11, his maxillary premolars were covered with composite resin. As for the nearly 3-year-old sister, she showed no cooperation and thus treatment was not possible until she reached the age of 5, at which time the primary molars were restored with stainless steel crowns. When she was 9, a thin layer of composite resin was applied on the labial surfaces of the permanent maxillary central incisors (Figure 5). These patients did not show up for regular follow-up but rather for symptomatic treatments.

During Permanent Dentition at Adolescence

Case 1: The Sister—In 2013, the 14-year-old girl presented to the dental clinic, concerned about a chin fistula. She also complained of persistent tooth sensitivity to air, water, and brushing, as well as bad esthetics due to tooth discolorations.

Clinical Examination—Extraoral examination showed a slight facial asymmetry (nose deviation to the left) and a reduction in the dimension of the lower facial third. The cutaneous fistula was centered in the submental area. The 5-mm-wide lesion had a yellowish crust aspect, surrounded by an erythematous area (Figure 6). Intraoral examination revealed a red and swollen gingiva, heavy plaque deposits, as well as decayed, worn out, and discolored dentition (Figure 7). The old composite resins were no longer adapted to the gingival margins due to facial growth. An interincisal midline shift toward the left was noticed as well as a narrowed maxillary arch that caused an end-to-end molar relationship.

Radiographic Examination—The periapical radiographs revealed a large periapical radiolucency related to the mandibular right central incisor, which turned out to be the origin of the chin fistula. The bitewings showed several superficial radiolucencies related to the premolars and molars proximal

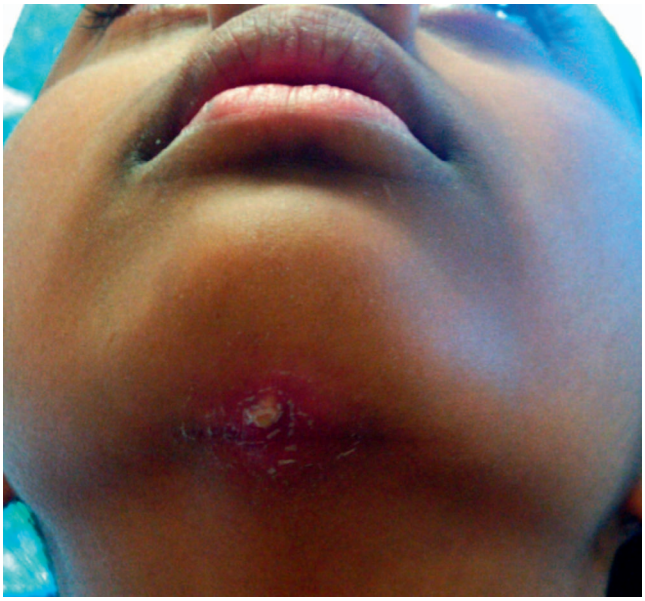


Figure 6. *Cutaneous fistula on the girl's chin (14 years old).*

areas, except for the second maxillary left molar, where the cavity was extending deep toward the pulp. The panoramic radiograph showed the impaction of the two maxillary permanent canines as well as the developing third molars. Cone beam computed tomography (CBCT) clarified the extent of the periapical radiolucency (which was approximately 10 mm wide) and the perforation of the external cortical bone next to the mandibular right central incisor. The impacted canines were positioned oblique and very close to the roots of the lateral incisors and first premolars (Figure 8).

Treatment Procedures—A treatment plan was elaborated to eliminate dental infections, reduce sensitivities, and improve esthetics and masticatory functions. The first steps were enhancement of oral hygiene and dental scaling. Endodontic treatment of the mandibular right central incisor and extraction of the severely decayed second maxillary left molar were carried out.

The preparation of the molars was as minimal as possible, especially on the occlusal surface, in order to compensate for the reduced vertical dimension. Stainless steel crowns were sealed using glass ionomer cement (Ketac Cem Radiopaque, 3M ESPE, Cergy Pontoise, France; Figure 9). The anterior teeth (incisors and retained deciduous canines) were restored in composite resin with strip crowns (transparent celluloid crown forms, Pella-ODUS). A slight circumferential preparation was performed to provide enough space for the strip crowns and the composite material, as well as to

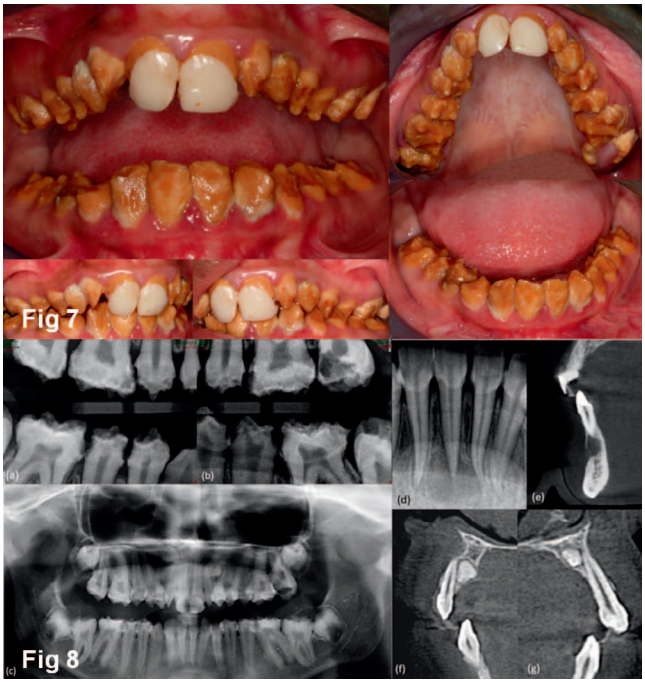


Figure 7. *Intraoral views of the sister's permanent dentition (14 years old).*
Figure 8. Radiographic images. (a, b) The bitewings reveal several superficial caries related to mandibular molars and premolars and deep cavity in the second maxillary left molar. (c) The panoramic radiograph shows impacted maxillary canines. (d) Large periapical radiolucent area next to the mandibular right central incisor. (e) Perforation of the external cortical bone (f). (g) CBCT examinations showing the impacted maxillary canines, close to the adjacent teeth.

remove the brownish-orange discolored surface layer, improving esthetic integration. The suitable size of strip crown was selected and trimmed with fine scissors to fit the tooth in length and cervical adaptation. The entire tooth surface was etched with a 37% phosphoric acid gel (Scotchbond Universal Etchant, 3M ESPE) for 30 seconds. The etchant was thoroughly rinsed, and the teeth were dried and subsequently treated with 5% sodium hypochlorite (NaOCl) for 1 minute to enhance the bond strength of the composite. Thin layers of primer and bonding agent (OptiBond, Kerr, Créteil, France) were then applied and light cured. The strip crown was filled with microhybrid composite resin (Herculite XRV, Kerr), carefully positioned on the tooth and the excess removed. After light curing for 80 seconds on each surface, the strip crown was peeled off with a probe or cut on the lingual side with a polishing disc if needed. Particular care was taken in the finishing and polishing sequence (using fine diamond burs, polishing discs, silicone cups), especially in the cervical region to avoid gingival inflammation. This procedure was repeated at a rate of two contralat-

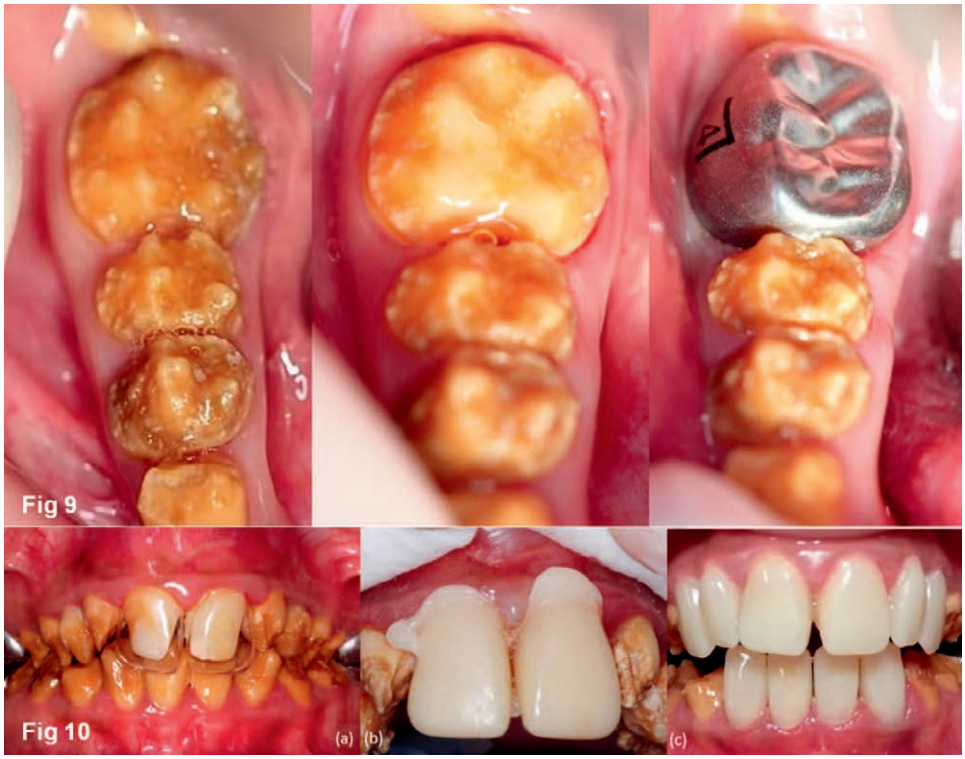


Figure 9. *Minimal molar preparation and placement of stainless steel crown.*
Figure 10. *(a) Strip crowns adjusted on the prepared incisors. (b) Strip crowns filled with composite resin positioned on the prepared teeth. (c) Anterior teeth restored with composite crowns using the same technique.*

eral teeth per treatment session, starting from the central incisors (Figure 10). Therapeutic abstention was decided regarding the maxillary impacted canines. Thus, the two deciduous canines were restored using strip crowns and subsequently bonded to a wire splint fixed to the palatal surface of the adjacent teeth. The impacted permanent teeth were to be followed up regularly.

Given that no suitably sized strip crowns were available for the premolars and the mandibular canines, adjusted crown molds were produced using a thermoforming procedure. The maxillary and mandibular teeth on the same side were prepared and full arch impressions were taken. A thick layer of self-etch adhesive (Adper EasyBond, 3M ESPE) was applied on the reduced teeth to prevent sensitivity and protect the dental tissues until the subsequent appointment, scheduled no more than two days later. The plaster casts were mounted on a semiadjustable articulator (Quick Master) and waxed up. Partial alginate impressions of the wax-ups were made and poured in plaster for the subsequent thermoforming procedure. The casts were trimmed as close as possible to the tooth crowns. Transparent plastic sheets were heated and pressed onto the casts using a vacuum thermoforming machine (Original Essix 110V). After allowing the thermopressed sheets to cool down for a few minutes, they were removed and trimmed so as to obtain the crown molds. On the initial model, the built-up wax was removed and light-cured individual composite shells were created for each tooth (Figure 11). In the following appointment, the patient's teeth were polished to remove the adhesive layer, and the composite shells were tried in for proper fitting and contouring and then bonded with flowable composite (Tetric EvoFlow, Ivoclar Vivadent, Saint-Jorioz, France) of the same shade. Polishing was performed as described above, and the occlusion contacts were verified.

The patient as well as her family were very satisfied with the treatment outcome (Figure 12). The dental sensitivity was relieved, and masticatory function, esthetics, and social life were improved. The patient and her parents were informed that these treatment options were a good compromise as a transitional restorative alternative until completion of bone growth, but once the latter is achieved, readjustment or replacement of several restorations will be needed. The patient was monitored at a 3-month interval for 18 months. The restorations remained intact with a satisfactory surface quality and no recurrent caries (Figure 13). The permanent mandibular right second molar began to erupt. As for the root canal therapy of the mandibular right central incisor, the two-dimensional and three-

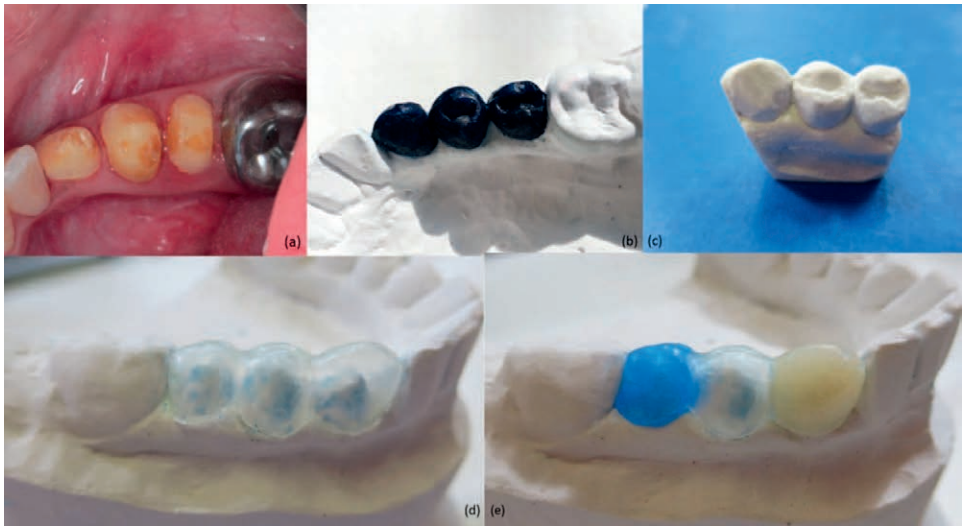


Figure 11. *(a) Mandibular canines and premolar preparations. (b) Wax-ups on plaster cast. (c) Reduction of the cast. (d) Crown template obtained using the thermoforming procedure. (e) Second individual composite shell elaboration thanks to wax isolation.*

dimensional radiographs showed a nearly complete regression of the radiolucency denoting bone regeneration. The chin fistula had completely disappeared (Figure 14).

Case 2: The Brother

While treating the young girl in 2013, her 18-year-old brother was encouraged to make an appointment for a checkup. He reported the persistence of mild tooth sensitivity since the last treatments in 2006.

Clinical Examination—Extraoral examination showed equal facial thirds, but an asymmetry was attributed to chin deviation to the left. Intraoral examination revealed heavy calculus deposits associated with moderate gingivitis. The second molars (except for the mandibular right one) were badly broken down. The stainless steel crown of the first mandibular left molar and all the composite restorations were no longer adapted to the gingival margins (Figure 15). The deciduous maxillary right canine showed high mobility. The absence of the contralateral canine was compensated for by the migration of adjacent teeth. Cross-bite on the left side, lateral infraocclusion on the right side, and edge-to-edge anterior bite were observable.

Radiographic Examination—The bitewings confirmed the maladaptation of the stainless steel crown on the first mandibular left molar. The periapical radiographs revealed a well-defined periapical radiolucency related to the maxillary left central incisor. The panoramic radiograph (Figure 16) showed the impaction of the two maxillary permanent canines and the mandibular third molars. According to CBCT sections, the impacted canines appeared close to the roots of adjacent teeth and generated internal and external cortical bone fenestrations. The third molars were in horizontal submerged position, in contact with the inferior alveolar nerve.

Treatment Procedures—Oral hygiene was enhanced and dental scaling performed. The maxillary left central incisor was endodontically treated. The broken-down second molars were extracted, as well as the impacted mandibular third molars, which led to perioperative pain and important postoperative complications for the mandibular left one (hemorrhage, trismus, bruise, and significant edema). The loose deciduous canine exfoliated by itself.

Regarding the mandibular left molars, the maladjusted crown was removed, the teeth were minimally prepared, and two new stainless steel crowns were simultaneously cemented.

The anterior teeth were restored with composite resin using strip crowns as described above. The designed wax-ups highlighted the impossibility of replacing the exfoliated deciduous canine because of insufficient space. Thus, it was decided to keep a narrow gap between the maxillary right lateral and first premolar. The premolars and the mandibular canines were restored following the same protocol described for the sister. Finding compromises for the buildup of composite restorations despite the malocclusion was challenging as the patient refused any orthodontic pretreatment.

At the end of the treatment, the patient was satisfied with the esthetic outcome and felt comfortable with his new occlusion. Follow-up was completed every 3 months for 12 months, during which oral hygiene motivation was always necessary. Except for a composite fragment that chipped off the buccal

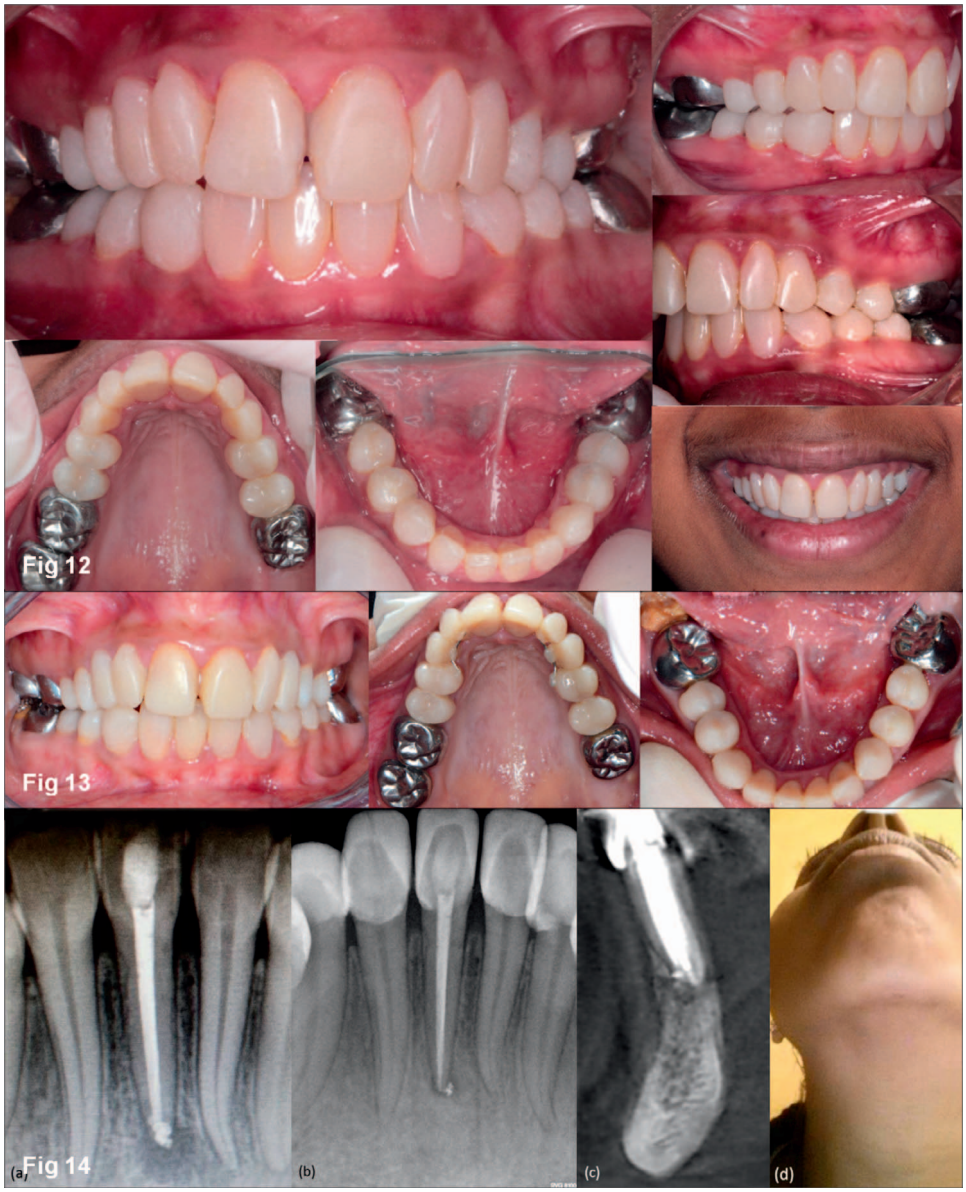


Figure 12. *Final intraoral views and smile photographs of the sister at the end of the treatment.*
Figure 13. *Intraoral views 18 months postoperative.*
Figure 14. *Radiographs showing the reduction of periapical radiolucency next to the mandibular right central incisor (a) 6 months after the endodontic treatment, (b, c) 18 months after the endodontic treatment, (d) skin healing of the fistula 6 months after the endodontic treatment.*

surface of the second left maxillary premolar at 7 months, the restorations remained satisfactory. After full eruption, the left maxillary third molar received a stainless steel crown (Figure 17).

DISCUSSION

Treatment Choice

Restoring esthetics and function in AI-affected patients is a challenging and multidisciplinary work and often requires compromises. Several therapeutic options were suggested: onlays, preformed stainless steel crowns, metallic or ceramic crowns for posterior teeth, and direct/indirect composite restorations, veneers, and ceramic crowns for anterior teeth. The treatment plan depends on numerous factors, including the type and severity of the enamel defects, the dentition stage, the hard and soft tissue development, and the cooperation and socioeconomic status of the patient.[12]

In primary and mixed dentitions, stainless steel crowns for primary molars and strip crowns or direct composites for anterior teeth remain efficient restorations and were chosen for the siblings' case. For permanent dentition, ceramic crowns and/or veneers are esthetically the more reliable solutions but are invasive, expensive, and require mature soft tissue. The treatment plan for these cases was based on the periodontal immaturity (which excluded fixed prosthodontics), the need of an acceptable esthetic

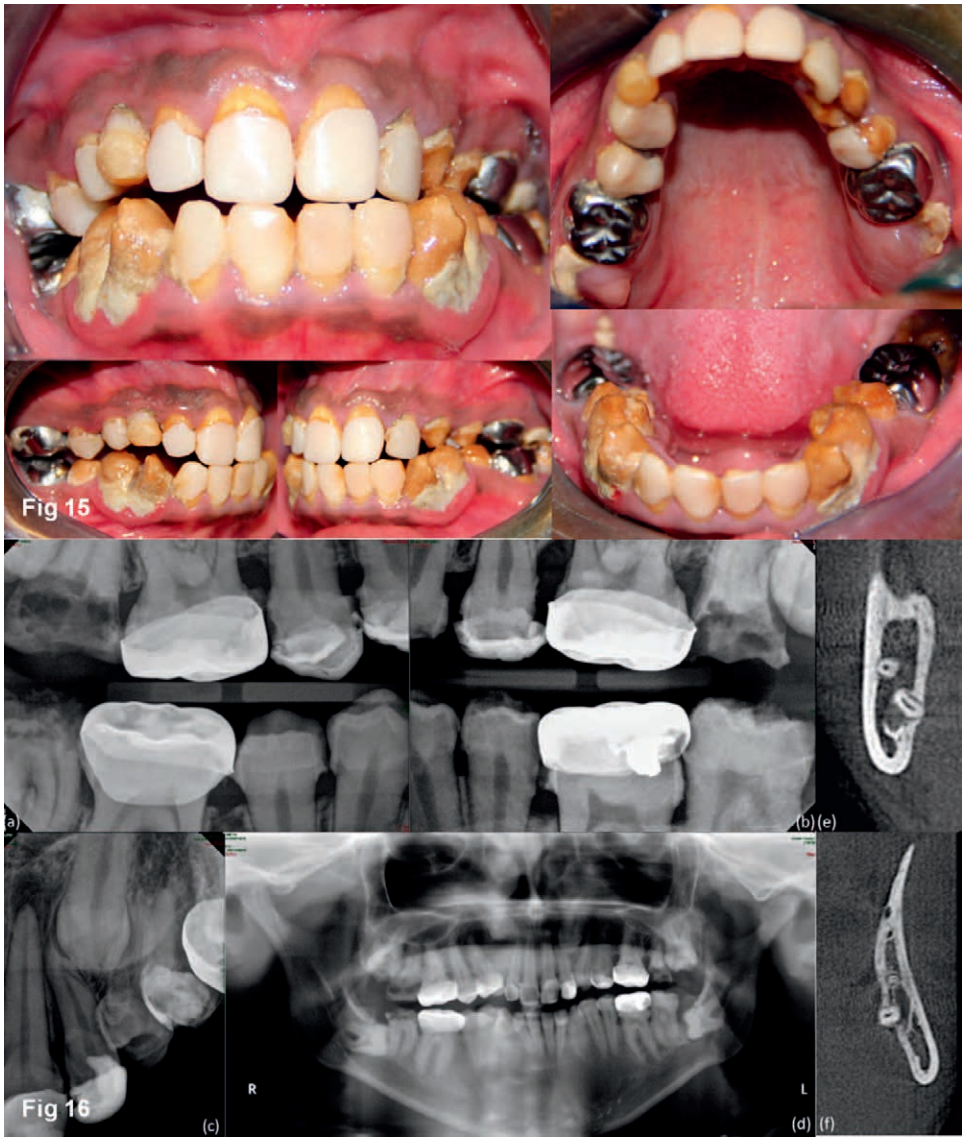


Figure 15. *Intraoral views of the brother's permanent dentition (18 years old).*
Figure 16. *Radiographic images. (a, b) The bitewings show the maladjustment of the stainless steel crown on the mandibular first left molar. (c) Periapical radiolucency next to the maxillary left central incisor. (d) Panoramic radiography showing impacted maxillary canines and mandibular third molars. (d, e) CBCT examinations of the impacted third molars showing their contact with the inferior alveolar nerve.*

appearance (requiring esthetic material), the relief of tooth sensitivity (indicating coverage of the entire crown surface), and the lack of financial resources of the patient's parents. These considerations led to the exclusion of ceramic crowns and veneer solutions. Composite restorations seemed to be a good compromise—esthetic and minimally invasive—as transitional restorations until bone and soft-tissue maturation. The crown molds for premolars and mandibular canines were also conservative, esthetic, functional, easily reparable, and affordable options. This transitional step allowed confirming the definitive form and shade for future prosthetic treatment.

Many studies reported a higher prevalence of impactions in AI patients.[10,13] In these siblings, the orthodontic traction of the impacted canines was not recommended as it might have led to root resorption of the adjacent teeth. Thus, these teeth will be subject to regular follow-ups. Their extraction will eventually be needed.

Bonding Composite Resin to AI Enamel

The conservative adhesive approach in young patients is more advantageous than the invasive prosthetic management.[14] In these cases, the bonding steps were difficult to perform because the siblings showed excessive salivation, and the placement of strip crowns or adjusted crown molds hindered the use of rubber dam or floss ligatures. Therefore, they needed to be performed by two practitioners and a dental assistant. Bonding to AI-affected enamel is more difficult compared with

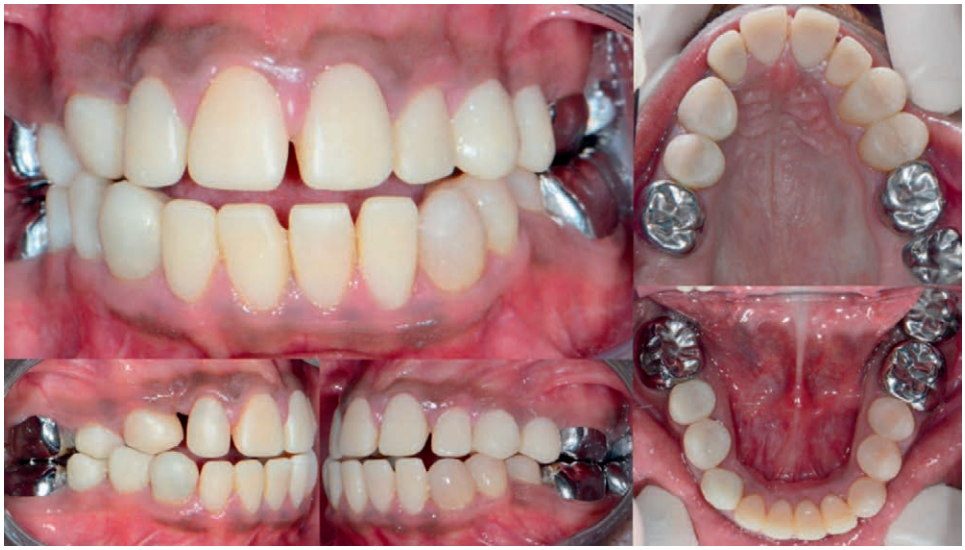


Figure 17. *Intraoral views of the brother at 11 months postoperative.*

sound enamel, and adhesive restorations display higher failure rates.[15-17] The hypocalcified form of AI shows higher porosity due to widened interprismatic spaces and irregular and disorganized prism architecture.[18-20] It also contains a lower mineral content than sound enamel due to increased retention of amorphous organic material (enamel proteins) between the enamel rods.[21,22] The protein content would be about five times higher than in sound enamel,[23] which would result in significantly lower bond strength.[24]

To overcome this difficulty, it has been suggested to apply 5% NaOCl on the enamel surface prior to bonding, in order to remove excess enamel proteins and enhance bond strength.[24,25] However, other studies indicated that this deproteinization procedure had no significant effect.[17,26,27] As AI teeth frequently exhibit a thin and/or locally disrupted enamel layer, dentin bonding is generally involved as well. In the hypocalcified form, the underlying dentin is hypermineralized and displays a reduced number of tubules with narrow and partly obliterated lumen, surrounded by thickened peritubular dentin.[20,28] This morphological pattern corresponds to sclerotic dentin, on which bonding is reported to be less efficient.[29,30] According to some studies, dentin pretreatment with NaOCl would not negatively influence bonding agent effectiveness.[24,31] Thus, it was decided in these cases to apply 5% NaOCl on the tooth surface, after phosphoric acid etching.

Psychological Impact and Follow-up

The management of AI-affected patients requires the patients' and parents' motivation throughout the growth period, with successive and long treatment phases at each dentition stage. The treatment in permanent dentition lasted seven months. The regular follow-up was not easy to obtain, especially for the treatments in primary and mixed dentition, during which the patients missed several appointments and many treatment steps were left out. Finally, in these patients, as described in most cases,[32] the positive psychosocial impact was noticeable. After the rehabilitation, the two siblings had become more self-confident and smiled more.

CONCLUSION

This case report described the 15-year case study and in particular the recent treatment in permanent dentition of two teenage siblings affected by the hypocalcified form of AI. They underlined the challenges encountered from the primary to adult dentition. The rehabilitation was implemented with minimal tooth preparation: the permanent molars were restored with stainless steel crowns and all other teeth with composite resins. Eighteen months after the end of the last treatment in permanent dentition, all of the restorations were intact (without discoloration or infiltration around the margins) and the patients were very pleased with the results. The use of composite as a conservative treatment option is a viable alternative in the medium-term treatment of AI-affected dentition, to restore function and esthetics. Psychological motivation and patient cooperation play major roles in the advancement and outcome of the treatment procedures.

Acknowledgment

The authors thank Laamia Sherif for proofreading this article in depth.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Hôpital Albert Chenevier in Créteil, France.

Conflict of Interest

The authors 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 4 April 2016)

REFERENCES

1. Bäckman B, & Holm AK (1986) Amelogenesis imperfecta: prevalence and incidence in a northern Swedish county *Community Dentistry and Oral Epidemiology* **14(1)** 43-47.
2. Witkop CJ Jr (1988) Amelogenesis imperfecta, dentinogenesis imperfecta and dentin dysplasia revisited: problems in classification *Journal of Oral Pathology* **17(9-10)** 547-553.
3. Collins MA, Mauriello SM, Tyndall DA, & Wright JT (1999) Dental anomalies associated with amelogenesis imperfecta: a radiographic assessment *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics* **88(3)** 358-364.
4. Aren G, Ozdemir D, Firatli S, Uygur C, Sepet E, & Firatli E (2003) Evaluation of oral and systemic manifestations in an amelogenesis imperfecta population *Journal of Dentistry* **31(8)** 585-591.
5. Gupta SK, Saxena P, Jain S, & Jain D (2011) Prevalence and distribution of selected developmental dental anomalies in an Indian population *Journal of Oral Science* **53(2)** 231-238.
6. de Souza JF, Fragelli CM, Paschoal MA, Campos EA, Cunha LF, Losso EM, & Cordeiro R de C (2014) Noninvasive and multidisciplinary approach to the functional and esthetic rehabilitation of amelogenesis imperfecta: a pediatric case report *Case Reports in Dentistry* **2014** 127175.
7. Alonso V, & Caserio M (2012) A clinical study of direct composite full-coverage crowns: long-term results *Operative Dentistry* **37(4)** 432-441.
8. Gerdolle D, Mortier E, Richard A, & Vailati F (2015) Full-mouth adhesive rehabilitation in a case of amelogenesis imperfecta: a 5-year follow-up case report *International Journal of Esthetic Dentistry* **10(1)** 12-31.
9. Yilmaz B, Oz U, & Yilmaz HG (2014) Interdisciplinary approach to oral rehabilitation of patient with amelogenesis imperfecta *New York State Dental Journal* **80(2)** 31-35.
10. Ayers KM, Drummond BK, Harding WJ, Salis SG, & Liston PN (2004) Amelogenesis imperfecta—multidisciplinary management from eruption to adulthood: review and case report *New Zealand Dental Journal* **100(4)** 101-104.
11. Feierabend S, Halbleib K, Klaiber B, & Hellwig E (2012) Laboratory-made composite resin restorations in children and adolescents with hypoplasia or hypomineralization of teeth *Quintessence International* **43(4)** 305-311.
12. Ozturk N, Sari Z, & Ozturk B (2004) An interdisciplinary approach for restoring function and esthetics in a patient with amelogenesis imperfecta and malocclusion: a clinical report *Journal of Prosthetic Dentistry* **92(2)** 112-115.
13. Seow WK (1995) Dental development in amelogenesis imperfecta: a controlled study *Pediatric Dentistry* **17(1)** 26-30.
14. Cogulu D, Becerik S, Emingil G, Hart PS, & Hart TC (2009) Oral rehabilitation of a patient with amelogenesis imperfecta *Pediatric Dentistry* **31(7)** 523-527.
15. Yip HK, & Smales RJ (2003) Oral rehabilitation of young adults with amelogenesis imperfecta *International Journal of Prosthodontics* **16(4)** 345-349.
16. Chen CF, Hu JC, Estrella MR, & Bresciani E (2013) Assessment of restorative treatment of patients with amelogenesis imperfecta *Pediatric Dentistry* **35(4)** 337-342.
17. Faria-e-Silva AL, De Moraes RR, Menezes M de S, Capanema RR, De Moura AS, & Martelli H Jr (2011) Hardness and microshear bond strength to enamel and dentin of permanent teeth with hypocalcified amelogenesis imperfecta *International Journal of Paediatric Dentistry* **21(4)** 314-320.
18. Gjørup H, Haubek D, Hintze H, Haukali G, Løvschall H, Hertz JM, & Poulsen S (2009) Hypocalcified type of amelogenesis imperfecta in a large family: clinical, radiographic, and histological findings, associated dentofacial anomalies, and resulting treatment load *Acta Odontologica Scandinavica* **67(4)** 240-247.
19. Poulter JA, Brookes SJ, Shore RC, Smith CE, Abi Farraj L, Kirkham J, Inglehearn CF, & Mighell AJ (2014) A missense mutation in ITGB6 causes pitted hypomineralized amelogenesis imperfecta *Human Molecular Genetics* **23(8)** 2189-2197.
20. Zhang C, Song Y, & Bian Z (2015) Ultrastructural analysis of the teeth affected by amelogenesis imperfecta resulting from FAM83H mutations and review of the literature *Oral Surgery, Oral Medicine, Oral Pathology and Oral Radiology* **119(2)** 69-76.
21. Wright JT, Duggal MS, Robinson C, Kirkham J, & Shore R (1993) The mineral composition and enamel ultrastructure of hypocalcified amelogenesis imperfecta *Journal of Craniofacial Genetics and Developmental Biology* **13(2)** 117-126.
22. El-Sayed W, Shore RC, Parry DA, Inglehearn CF, & Mighell AJ (2011) Hypomaturation amelogenesis imperfecta due to WDR72 mutations: a novel mutation and ultrastructural analyses of deciduous teeth *Cells, Tissues, Organs* **194(1)** 60-66.
23. Wright JT, Frazier-Bowers S, Simmons D, Alexander K, Crawford P, Han ST, Hart PS, & Hart TC (2009)

Phenotypic variation in FAM83H-associated amelogenesis imperfecta *Journal of Dental Research* **88(4)** 356-360.

24. Saroğlu I, Aras S, & Oztaş D (2006) Effect of deproteinization on composite bond strength in hypocalcified amelogenesis imperfecta *Oral Diseases* **12(3)** 305-308.
25. Venezie RD, Vadiakas G, Christensen JR, & Wright JT (1994) Enamel pretreatment with sodium hypochlorite to enhance bonding in hypocalcified amelogenesis imperfecta: case report and SEM analysis *Pediatric Dentistry* **16(6)** 433-436.
26. Sönmez IS, Aras S, Tunç ES, Küçükeşmen C (2009) Clinical success of deproteinization in hypocalcified amelogenesis imperfecta *Quintessence International* **40(2)** 113-118.
27. Pugach MK, Ozer F, Mulmadgi R, Li Y, Suggs C, Wright JT, Bartlett JD, Gibson CW, & Lindemeyer RG (2014) Shear bond strength of dentin and deproteinized enamel of amelogenesis imperfecta mouse incisors *Pediatric Dentistry* **36(5)** 130-136.
28. Sánchez-Quevedo MC, Ceballos G, García JM, Luna JD, Rodríguez IA, & Campos A (2004) Dentine structure and mineralization in hypocalcified amelogenesis imperfecta: a quantitative X-ray histochemical study *Oral Diseases* **10(2)** 94-98.
29. Perdigão J (2010) Dentin bonding: variables related to the clinical situation and the substrate treatment *Dental Materials* **26(2)** e24-37.
30. Hiraishi N, Yiu CK, & King NM (2008) Effect of acid etching time on bond strength of an etch-and-rinse adhesive to primary tooth dentine affected by amelogenesis imperfecta *International Journal of Paediatric Dentistry* **18(3)** 224-230.
31. Montagner AF, Skupien JA, Borges MF, Krejci I, Bortolotto T, & Susin AH (2015) Effect of sodium hypochlorite as dentinal pretreatment on bonding strength of adhesive systems *Indian Journal of Dental Research* **26(4)** 416-420.
32. Coffield KD, Phillips C, Brady M, Roberts MW, Strauss RP, & Wright JT (2005) The psychosocial impact of developmental dental defects in people with hereditary amelogenesis imperfecta *Journal of the American Dental Association* **136(5)** 620-630.

©Operative Dentistry, 2016, **41-6,** 578-586

Clinical Evaluation of Genotoxicity of In-office Bleaching

M Rezende • JL De Geus • AD Loguercio • A Reis • D Kossatz

Clinical Relevance

In-office bleaching with 35% hydrogen peroxide gel was not genotoxic for gingival and lip tissues during a one month evaluation period.

SUMMARY

Objective: The aim of this study was to evaluate the genotoxicity of in-office bleaching with 35% hydrogen peroxide in epithelial cells from the gingival and lip tissues.

Methods and Materials: Thirty volunteers with central incisors shade A1 or darker were selected for this study. The gingival tissue of the teeth to be bleached was isolated with a light-polymerized resin dam, and the 35% hydrogen peroxide gel was administered during three 15-minute applications over the course of the 45-minute application period. Two bleaching sessions with a one-week interval in between were performed. Exfoliated oral mucosa gingival epithelial cells and upper lip lining were collected at baseline and one month after the in-office dental bleaching. The scraped cells were placed on clean glass slides and smears were prepared. After staining with Giemsa solution, two blinded examiners performed cell and micronuclei counts under a 100× optical microscope. Tooth sensitivity was evaluated using the Visual Analogue Scale (VAS). Shade evaluation was recorded before and one month after the bleaching treatment with the value-oriented shade guide Vita Bleachedguide 3D-MASTER and the spectrophotometer Vita Easyshade. Data from the shade guide units and the micronuclei (MN) frequency were subjected to a Mann-Whitney test (α=0.05). The overall difference between before and one month after the bleaching treatment (ΔE and ΔSGU), absolute risk, and intensity of tooth sensitivity (TS) were calculated, as was the 95% confidence interval (CI).

Results: The frequency of MN was not increased after bleaching with 35% hydrogen peroxide in both study groups ($p>0.05$). The absolute risk of TS of the participants was 93% (95% CI, 79%-98%), with a mean VAS intensity of 5.7 ± 2.9 (95% CI, 4.6-6.8). Meaningful whitening was observed after bleaching. The change in shade guide units in the Bleached-

Márcia Rezende, DDS, MS, postdoctoral student, Department of Restorative Dentistry, University Estadual de Ponta Grossa, Ponta Grossa, Brazil

Juliana Larocca De Geus, DDS, MS, doctoral student, Department of Restorative Dentistry, University Estadual de Ponta Grossa, Ponta Grossa, Brazil

*Alessandro Dourado Loguercio, DDS, MS, PhD, professor, School of Dentistry, State University of Ponta Grossa, Ponta Grossa, Brazil

Alessandra Reis, DDS, PhD, professor, School of Dentistry, Department of Restorative Dentistry, University Estadual de Ponta Grossa, Ponta Grossa, Brazil

Stella Kossatz, DDS, MS, PhD, professor, School of Dentistry, Department of Restorative Dentistry, University Estadual de Ponta Grossa, Ponta Grossa, Brazil

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

DOI: 10.2341/15-207-C

guide 3D-MASTER was 2.3 ± 1.4. In terms of ΔE, the change in color was 7.7 ± 3.5.

Conclusions: The in-office bleaching did not induce DNA damage to the gingival and lip tissue during the bleaching period. Although effective whitening was observed, most of the participants experienced TS.

INTRODUCTION

In a survey[1] from a population of 407 adults, about one-third of participants were not satisfied with their dental appearance, and tooth color was the primary reason for this dissatisfaction. In a more recent study,[2] the same research group reported that more than 80% of the group of participants reported that they wished to have their teeth bleached. This desire has been responsible for the increase in the demand for bleaching procedures in dental offices.

Despite the effective results in whitening produced by bleaching procedures, the profession and the public have been aware of certain risks related to dental bleaching, such as tooth sensitivity (TS) and gingival irritation.[3] While the release of free radicals is capable of converting the complex pigment molecules from the dental structure into smaller and less saturated chains[3,4] to attain effective whitening, these free radicals may also be able to react with the soft tissue and cause injuries, such as burns and ulceration.[4-8]

It is known that the DNA of cells exposed to chemical or physical agents may become damaged. In this situation, chromosomal fragments called micronuclei (MN) are observed as a result of atypical mitoses. Depending on the extent of cellular damage, the consequences may include impairment of the cell cycle, cell death, and even the formation of a neoplasm.[4,9,10] An increased frequency of chromosome breaks has been recently demonstrated to be an initial event in carcinogenesis, suggesting that these alterations may play a significant role in assessing oncogenic risk.[11,12] An increased frequency of MN in exfoliated cells from oral mucosa has traditionally served as an index for evaluating the genotoxicity of exposure to various carcinogens, mainly because this technique is simple, painless, and cheap and has been used as an adjunct in molecular epidemiology.[13]

Although there are several studies in the literature that have evaluated the genotoxicity of bleaching agents, most of these studies are experimental in animals, and only a few of them have been conducted on humans.[10,14-17] Klaric and others[16] analyzed the genotoxic effect of two hydrogen peroxide (HP)–containing bleaching products, HP 28% and 35%, on oral mucosal cells. The authors concluded that both preparations demonstrated potential genotoxic effects. However, the study of De Geus and others[17] found that at-home bleaching with 10% carbamide peroxide did not induce DNA damage to the gingival tissue. The results of the study of Almeida and others[10] supported the findings of De Geus and others[17] because after the application of two concentrations of carbamide peroxide (10% and 16%), no difference between the two groups of carbamide peroxide gels was observed in terms of mutagenic stress on gingival epithelial cells. This controversy among studies highlights the need for further investigations into this issue. Therefore, the aim of this study was to evaluate an in-office bleaching process with 35% hydrogen peroxide in terms of its efficacy, TS, and genotoxicity in epithelial cells from the gingival and lip tissues.

The null hypothesis tested was that in-office dental bleaching with 35% hydrogen peroxide did not induce DNA damage to the gingival and lip tissue during the bleaching period.

METHODS AND MATERIALS

This clinical investigation was approved (protocol 172.988) by the Scientific Review Committee and by the committee for the protection of human subjects of the local university. This report follows the protocol established by the CONSORT statement.[18] Based on pre-established criteria, 30 volunteers who searched for dental bleaching were selected for this study. This study was performed between November 2013 and March 2014. Two weeks before the bleaching procedures, all of the volunteers received dental prophylaxis with pumice and water in a rubber cup and signed an informed consent form.

Eligibility Criteria

The participants who were included in this clinical trial were between 18 and 33 years of age and had good general and oral health. The participants were required to have six caries-free maxillary anterior teeth and no periodontal disease. The central incisors were shade A1 or darker, as judged by comparison with a value-oriented shade guide (Vita Classical, Vita Zahnfabrik, Bad Säckingen, Germany). Although patients with baseline tooth color A1 or darker are not often included in clinical trials, as such baseline color does not leave much ability to measure color differences using shade guide units, there is great demand from these patients, who are requesting bleaching treatments. As the primary outcome of this

study was not centered on color change, we included such patients (A1 or darker) in this clinical trial.

Participants with anterior restorations or dental prosthesis, orthodontics apparatus, or severe internal tooth discoloration (tetracycline stains, fluorosis, and pulpless teeth) were not included in the study. Additionally, pregnant/lactating women, participants with any other pathology that could cause sensitivity (such as recession, dentin exposure, or the presence of visible cracks in teeth), smokers, bruxers, or participants who had previously undergone tooth-whitening procedures were also excluded.

Sample Size Calculation

The sample size calculation was based on the frequency of MN per 1000 cells in adults. In the pilot study it was observed that the normal frequency of MN was about 1 ± 1.1.[17,19-22] In order for the bleaching procedure to be considered safe, it was expected that we would find a mean difference of not more than 1.0. Thus, we needed a minimum sample size of 30 participants with a power of 80% and an alpha of 5%.

Bleaching Procedure

A lip and cheek retractor (ArcFlex, FGM, Joinville, Santa Catarina, Brazil) was placed in the participant's mouth to avoid the contact of the bleaching gel with the cheek, lips, and tongue. Then the gingival tissue of the teeth to be bleached was isolated from the bleaching agent using a light-polymerized resin dam (Top Dam, FGM). In every two teeth, the light-cured gingival barrier was activated for 20 seconds using a LED light-curing unit (Gnatus, Ribeirão Preto, São Paulo, Brazil) set at 1200 mW/cm^2. The 35% hydrogen peroxide gel (Whiteness HP Maxx, FGM) was used during three 15-minute applications over the course of the 45-minute application period. Two bleaching sessions within a one-week interval were performed on each patient.

TS Evaluation

The TS was evaluated up to 24 hours using the Visual Analogue Scale (VAS).[6-8,17,23] The participants were asked to place a line perpendicular to a 10-mm-long line with zero at one end indicating "no TS" and a 10-mm end indicating "unbearable TS." Then the distance (in millimeters) from the zero end was measured with the aid of a millimeter ruler. The higher sensitivity score, as reported by the patient in the first 24 hours after the first and second tooth-whitening sessions, was used for estimation purposes.

Patients who had high levels of TS were instructed to get in touch with the researchers to be examined and treated with painkillers, anti-inflammatories, and/or desensitizing topicals.

Color Evaluation

Two calibrated evaluators with a previous agreement of at least 85%, as determined by weighted kappa statistics, recorded the shade of the maxillary right central incisor during different time assessments. Shade evaluation was recorded before the procedure and one month after the bleaching treatment. The color evaluation was performed with the value-oriented shade guide Vita Bleachedguide 3D-MASTER (Vita Zahnfabrik). The Vita Bleachedguide 3D-MASTER (Vita Zahnfabrik) contains 15 shade tabs with lighter shades and it is already organized from the highest (0M1) to the lowest (5M3) value.[24] Additionally, an objective color evaluation was performed with the spectrophotometer Vita Easyshade (Vita Zahnfabrik).

The measurement area of interest for shade matching was the middle one-third of the facial surface of the maxillary central incisor, according to the American Dental Association guidelines. Color changes were calculated from the beginning of the active phase through the individual recall times by calculating the change in the number of shade guide units (ΔSGU), which occurred toward the lighter end of the value-oriented list of shade tabs. In the event of disagreements between the examiners during the shade evaluation, a consensus was reached.

The spectrophotometer measurement with a silicone guide was determined using the parameters of the Easyshade device, which indicated the following values: L*, (a*), and (b*), in which L* represents the value from 0 (black) to 100 (white) and a* and b* represent the shade, where a* is the measurement along the red-green axis and b* is the measurement along the yellow-blue axis. The color comparison before and after treatment was given by differences between the two colors (ΔE), which were calculated using the formula $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.[25]

Sample Collection for MN

Epithelial cells that were exfoliated from the oral mucosa gingival and upper lip lining were collected at baseline and one month after the in-office dental bleaching. Before the cell collection, the participants

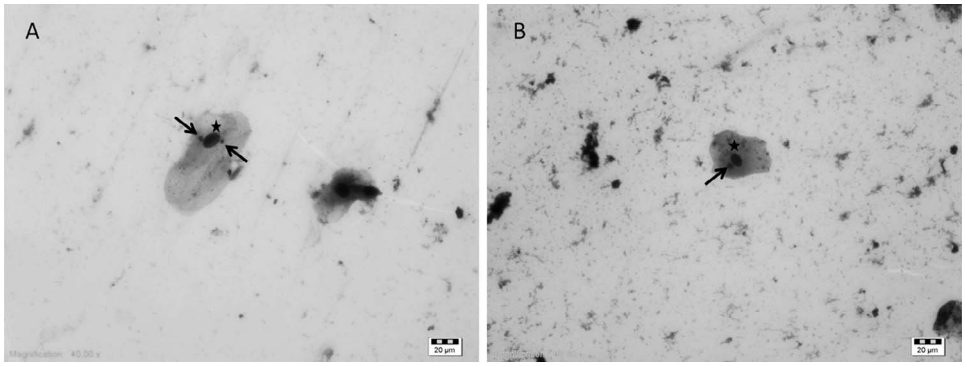


Figure 1. *Cells with micronuclei. In both (A) and (B) one can see the presence of micronucleus (indicated by an arrows) and the central nucleus of the cell (indicated with a star).*

rinsed their mouths with tap water for one minute. Subsequently, the cells were scraped with wooden spatulas from the marginal gingival and upper lip lining.[17,20,21,26,27] The scraped cells were placed on clean glass slides and smears were prepared. The smear was dried with a jet of air from a triple syringe for one minute at a distance of approximately 30 cm, thus avoiding excessive dehydration of the cells.[17,27]

Staining Procedures

The staining protocol was prepared immediately after the smear collection. Five to six drops of Giemsa stock solution (Cinética Química, Jandira, São Paulo, Brazil) were applied directly over the slide for two minutes and then the slides were washed in a container with tap water (container 1=three to four washes; container 2=two to three washes). The differentiation of the cells was performed in a third container (1200 mL of tap water and one drop of glacial acetic acid; Vetec Química Fina Ltda, Duque de Caxias, Rio de Janeiro, Brazil). After this process, the slide was dried for one minute in the same manner as described in the sample collection item. Then three drops of the adhesive Entellan (Merck KGaA, Darmstadt, Germany) were applied on the visibly dry slide for cover-glass positioning.[17,27]

Evaluation of the Slides

A single researcher performed the microscopic examination of the cells to avoid inter-examiner variation. An experienced oral pathologist trained this examiner. Calibration was made by reading five slides, randomly selected, from this study sample. An agreement of at least 80% was required (between the oral pathologist and the examiner) before beginning this phase of the study.

At least 1000 cells from each participant were evaluated during each period with the staining procedure. Cell counting was performed under an optical microscope with 100× magnification and, when MN were found, the magnification was increased to 400× (Nikon E800, Tokyo, Japan). The criteria for inclusion in the total cell count were the following: 1) the cytoplasm was intact and lying relatively flat; 2) there was little or no overlap with adjacent cells; 3) there was little or no debris; and 4) the nucleus was normal and intact, with the nuclear perimeter smooth and distinct.[28]

The parameters for identifying the micronucleus were as follows: 1) a rounded, smooth perimeter suggestive of a membrane; 2) less than one-third of the diameter of the associated nucleus but large enough to discern shape and color; 3) staining intensity similar to nucleus; 4) texture similar to nucleus; 5) same focal plane as nucleus; and 6) an absence of overlap with or bridge to nucleus (Figure 1).[28] Dead or degenerating cells (karyolysis, karyorrhexis, nuclear fragmentation) were excluded from evaluation. Nuclear blebbings (a micronucleus-like structure connected to the main nucleus by a bridge) were also not considered.

Statistical Analysis

The data were tabulated using the software SigmaPlot 5.0 for Windows (Systat Software Inc, San Jose, CA, USA). The Shapiro-Wilk test and the Bartlett test were used to test the normality and equal variance of the data. As at least one of these assumptions were not met, MN data were subjected to the Wilcoxon Signed Rank test with a level of 95% of confidence. The overall ΔE and ΔSGU, absolute risk, and intensity of TS were calculated, as was the 95% confidence interval (CI). Data from TS intensity between bleaching sessions were compared with the Wilcoxon Signed Rank test with a level of 95% of confidence.

RESULTS

A total of 104 participants were screened to select 30 participants who met the inclusion criteria. The distribution of patients according to the baseline

Table 1: *Distribution of the Participants According to the Baseline Tooth Color of the Vita Classical Scale*

	Baseline Tooth Color					
	A1	B2	A2	C1	C2	A3
No. of patients	8	7	5	3	1	6

tooth color can be seen in Table 1. The mean age of the participants was 23.5 ± 4.8 years, with a range of 18 to 33 years. Most of the participants were women (63%). All of the participants attended the recall visits during the bleaching protocol.

No significant difference was observed in the TS intensity between the first and second bleaching sessions ($p>0.05$) (Table 2). The overall absolute risk of TS of the participants was 93% (95% CI, 79%-98%), with a mean VAS intensity of 5.7 ± 2.9 (95% CI, 4.6-6.8). Five patients took an analgesic to alleviate the bleaching-induced TS (Tylenol, Janssen-Cilag Farmacêutica), and one patient self-administered an anti-inflammatory drug (Nimesulida, Medley, Campinas, Brazil).

Table 3 reports the means and standard deviations as well as medians and interquartile ranges of SGU, L*, a* and b* values. Meaningful changes towards whitening were observed after bleaching (Table 3; $p<0.05$). The change in the shade guide units within the Bleachedguide 3D-MASTER was 2.3 ± 1.4. In terms of ΔE, the change in color was 7.7 ± 3.5. The frequency of MN was not increased after bleaching with 35% hydrogen peroxide in both tissues (Table 4; $p>0.50$).

DISCUSSION

The authors of this study consider whitening at-home to be the first option for the treatment of discolored teeth.[29-31] The use of low concentrations of hydrogen peroxide gel causes less pulp irritation[32,33] and can minimize the risk and intensity of bleaching-induced TS, being a safer alternative for bleaching purposes. However, although at-home bleaching has some advantages over in-office bleaching, there are still some patients who do not adapt well to the at-home protocol, as it requires the daily use of a bleaching tray. Others desire to have a faster outcome and therefore ask for quicker ways to achieve the same whitening result. Under these circumstances, in-office bleaching is usually performed, and, thus, researchers should conduct further studies about the safety and efficacy of this protocol.

The present study showed an effective bleaching of approximately two SGU when evaluated with the Vita Bleachedguide. This is lower than the change detected by other authors[34-37] who also used 35% hydrogen peroxide. The small SGU changes detected in this study can be attributed to the fact that this study included patients with lighter teeth (shade A1 or darker), while the previous ones selected patients with darker teeth (shade C2 or darker). In a multivariable regression analysis, from pooled data of 11 clinical trials of dental bleaching performed by the same research group, Rezende and others[38] identified a significant relationship between baseline color in relation to color change estimates, meaning that the darker the baseline tooth color, the higher the degree of whitening. Using low-concentrated whitening strips, Gerlach and Zhou[39] observed the same. They reported that the lighter the baseline tooth color, the lower the degree of whitening. These studies corroborate our findings and suggest that patients with lighter teeth do not respond to the bleaching regimen as well as patients with darker teeth, perhaps because of the lower amount of available substrate for hydrogen peroxide oxidation.

Table 2: *Means and Standard Deviations of Tooth Sensitivity (TS) Intensity Experienced by Patients from the First and Second Session to Different Periods Using Visual Analog Scale (VAS) Pain Scales*[a]

Periods	First Session	Second Session
Immediately after	2.1 ± 2.5 A	2.4 ± 2.8 A
Up to 1 h	3.0 ± 2.7 B	2.9 ± 3.1 B
Up to 24 h	1.1 ± 2.0 C	1.9 ± 2.9 C

[a] *Means identified with the same letter are statistically similar.*

In the present study, we observed a high absolute risk of bleaching-induced TS that affected 93% of the patients who reported pain at least once during the onset of the treatment. This high risk is in accordance with the findings of previous studies.[8,40-42] Although the etiology of TS is not yet well understood, it is likely the result of the activation of nociceptors,[43,44] which is caused by the inflammatory reaction in the pulp tissue.[45,46] In the face of the high risk of bleaching TS after in-office bleaching, clinical alternatives to minimize this undesirable side effect were the focus of several investigations. The use of nonsteroidal anti-inflammatory drugs,[40,41,47] antioxidants,[48] and steroidal anti-inflammatory drugs[49] was not effective in minimizing this side effect. On the other hand, topical approaches, such as the application of potassium nitrate[34] or Gluma desensitizer PowerGel[8] (GLU; Heraeus Kulzer, Hanau, Germany), before in-office bleaching offer good alternatives to

Table 3: *Means, Standard Deviations (SDs), Medians, and Interquartile Ranges of the Shade Guide Units (SGUs) and L*, a*, and b* Parameters Before and After Bleaching*

Color Parameters	Mean ± SD		Medians and Interquartile Range	
	Baseline	1 Month	Baseline	1 Month
SGU (Bleachedguide)	6.5 ± 1.7	4.2 ± 1.1	6 (5/8)	4 (4/4)
L*	84.4 ± 3.9	86.8 ± 3.8	84.8 (80.9/86.7)	87.3 (85.6/88.5)
a*	−2.3 ± 1.2	−1.9 ± 1.2	−2.3 (−3.4/−1.6)	−1.9 (−2.7/−1.3)
b*	18.8 ± 4.3	13.6 ± 4.9	18.6 (16.3/21.6)	14.1(10.2/17.4)

significantly reduce the bleaching-induced TS produced by in-office bleaching.

With regard to the soft gingival tissues, clinicians can avoid the contact of in-office bleaching gels with the gingival tissue by applying a light-cured gingival barrier; however, the hydrogen peroxide often comes into contact with the oral tissue. This contact causes burns and ulcerations due to the oxidative stress induced by the hydrogen peroxide that may ultimately lead to genomic damage,[16] which highlights concerns about the safety of in-office bleaching protocols.

The frequency of MN in normal oral mucosa is between 0.5 and 2.0/1000 cells,[20-22] which is within the range we detected in the present study at baseline and one month post-bleaching. These findings suggest that the high-concentration hydrogen peroxide gel under controlled conditions did not seem to induce DNA damage to the gingival and lip tissues when applied in two bleaching sessions within a one-week interval. Although a higher concentration of hydrogen peroxide is used in the in-office bleaching protocol, clinicians can demonstrate good control of the product application. In general, the use of light-cured gingival barrier and the short exposure duration of the product may compensate for the increased hydrogen peroxide concentration used in in-office bleaching protocols.

Altogether, this explains why the results of this study are similar to those involving at-home bleaching, in which bleaching was not associated with an increased MN frequency.[17] On the other hand, the results of the present study are not in agreement with the findings of an in-office bleaching study.[16]

Table 4: *Means and Standard Deviations of the Micronuclei Frequency (MN) per 1000 Exfoliated Gingival and Lip Tissues*

	Before	After	*p*-Value[a]
MN gingival tissue	0.4 ± 0.6 A	0.5 ± 0.5 A	0.52
MN lip	0.3 ± 0.5 A	0.4 ± 0.6 A	0.50

[a] *Wilcoxon Signed Rank test, Capital letters indicate statistically similar groups.*

Klaric and others[16] speculated that the high number of MN after in-office bleaching could be attributed to the inadequate polymerization of the gingival light-curing barrier or even to the failure to promote an adequate protection to mitigate/eliminate the problem. Perhaps rubber dam isolation, with protection of the gingival tissue with petroleum jelly, might provide a better isolation of the operative field without the need to use a lip and cheek retractor.

An inadvertent contact of the highly concentrated bleaching material with the soft tissues of the oral cavity could induce increased levels of MN in the oral epithelial cells, as oxygen free radicals released from peroxides are important etiologic agents in the development of many pathological conditions.[50-52] This reinforces the need for caution during in-office bleaching,[50] as under controlled in-office conditions, such as those of the present study, this is not expected to occur.

Additionally, the study of Klaric and others[16] involved samples that were collected only 72 hours after bleaching. Considering that the oral epithelial cells turn over every 14 days,[53] it is theoretically not impossible to observe the genotoxic effects of acute exposure in shorter periods of time. In light of this, the present study collected samples one month after bleaching, as this period of time is within the regeneration cycle of the cells from the gingival tissue.[54] However, in light of these conflicting results, further clinical trials should focus on this topic and involve collection of samples at different time periods in order to investigate this controversy.

One should also mention the limitations of the current study. Most of the participants in this study were young adults with light baseline tooth color, which affects the generalizability of the findings of the present investigation to the overall population. Bonassi and others[13] compiled data from 5424 subjects with epithelial MN values obtained from 30 laboratories worldwide and concluded that several conditions may affect the MN frequency; in particular, age was shown to be highly significant. Further studies that include young and elderly

participants should be conducted in this field in order to prove this hypothesis.

CONCLUSIONS

In-office bleaching with 35% hydrogen peroxide did not induce DNA damage to the gingival and lip tissues during the bleaching period. Although effective whitening was observed, most of the participants experienced TS.

Acknowledgements

The authors would like to thank FGM Dental Products for the donation of the bleaching gel used in this investigation. The authors also declare that they have no conflict of interest.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Research Ethics Committee of the University Estadual de Ponta Grossa. The approval code for this study is 172.988.

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 9 May 2016)

REFERENCES

1. Samorodnitzki-Naveh GR, Geiger SB, & Levin L (2007) Patients' satisfaction with dental esthetics *Journal of the American Dental Association* **138(6)** 805-808.
2. Samorodnitzky-Naveh GR, Grossman Y, Bachner YG, & Levin L (2010) Patients' self-perception of tooth shade in relation to professionally objective evaluation *Quintessence International* **41(5)** e80-e83.
3. Carey CM (2014) Tooth whitening: What we now know *Journal of Evidence-based Dental Practice* **14(Supplement)** 70-76.
4. Goldberg M, Grootveld M, & Lynch E (2010) Undesirable and adverse effects of tooth-whitening products: A review *Clinical Oral Investigation* **14(1)** 1-10.
5. Dahl JE, & Pallesen U (2003) Tooth bleaching—A critical review of the biological aspects *Critical Reviews in Oral Biology and Medicine: An Official Publication of the American Association of Oral Biologists* **14(4)** 292-304.
6. Gurgan S, Cakir FY, & Yazici E (2010) Different light-activated in-office bleaching systems: A clinical evaluation *Lasers in Medical Science* **25(6)** 817-822.
7. He LB, Shao MY, Tan K, Xu X, & Li JY (2012) The effects of light on bleaching and tooth sensitivity during in-office vital bleaching: A systematic review and meta-analysis *Journal of Dentistry* **40(8)** 644-653.
8. Mehta D, Venkata S, Naganath M, LingaReddy U, Ishihata H, & Finger WJ (2013) Clinical trial of tooth desensitization prior to in-office bleaching *European Journal of Oral Science* **121(5)** 477-481.
9. Igarashi M, Nagata M, Itoh S, Yamoto T, & Tsuda S (2010) Relationship between DNA damage and micronucleus in mouse liver *Journal of Toxicological Science* **35(6)** 881-889.
10. Almeida AF, Torre Edo N, Selayaran M dos S, Leite FR, Demarco FF, Loguercio AD, & Etges A (2015) Genotoxic potential of 10% and 16% carbamide peroxide in dental bleaching *Brazilian Oral Research* **29(1)** 1-7.
11. Hagmar L, Bonassi S, Strömberg U, Brogger A, Knudsen LE, Norppa H, & Reuterwall C (1998) Chromosomal aberrations in lymphocytes predict human cancer: A report from the European Study Group on cytogenetic biomarkers and health (ESCH) *Cancer Research* **58(18)** 4117-4121.
12. Bonassi S, Hagmar L, Strömberg U, Montagud AH, Tinnerberg H, Forni A, Heikkilä P, Wanders S, Wilhardt P, Hansteen IL, Knudsen LE, & Norppa H (2000) Chromosomal aberrations in lymphocytes predict human cancer independently of exposure to carcinogens *Cancer Research* **60(6)** 1619-1625.
13. Bonassi S, Coskun E, Ceppi M, Lando C, Bolognesi C, Burgaz S, Holland N, Kirsh-Volders M, Knasmueller S, Zeiger E, Carnesoltas D, Cavallo D, da Silva J, de Andrade VM, Demircigil GC, Domínguez Odio A, Donmez-Altuntas H, Gattas G, Giri A, Giri S, Gómez-Meda B, Gómez-Arroyo S, Hadjidekova V, Haveric A, Kamboj M, Kurteshi K, Martino-Roth MG, Montero Montoya R, Nersesyan A, Pastor-Benito S, Favero Salvadori DM, Shaposhnikova A, Stopper H, Thomas P, Torres-Bugarín O, Yadav AS, Zúñiga González G, & Fenech M (2011) The Human Micro Nucleus project on exfoliated buccal cells (HUMN(XL)): The role of life-style, host factors, occupational exposures, health status, and assay protocol *Mutation Research* **728(3)** 88-97.
14. De Castro Albuquerque R, Gomez RS, Dutra RA, Vasconcellos WA, Gomez RS, & Gomez MV (2002) Effects of a 10% carbamide peroxide bleaching agent on rat oral epithelium proliferation *Brazilian Dental Journal* **13(3)** 162-165.
15. Da Costa Filho LC, da Costa CC, Sória ML, & Taga R (2002) Effect of home bleaching and smoking on marginal gingival epithelium proliferation: A histologic study in women *Journal of Oral Pathology and Medicine: Official Publication of the International Association of Oral Pathologists and the American Academy of Oral Pathology* **31(8)** 473-480.
16. Klaric E, Par M, Profeta I, Kopjar N, Rozgaj R, Kasuba V, Zeljezic D, & Tarle Z (2013) Genotoxic effect of two bleaching agents on oral mucosa *Cancer Genomics & Proteomics* **10(5)** 209-215.
17. De Geus JL, Rezende M, Margraf LS, Bortoluzzi MC, Fernández E, Loguercio AD, Reis A, & Kossatz S (2015) Evaluation of genotoxicity and efficacy of at-home bleaching in smokers: A single-blind controlled clinical trial *Operative Dentistry* **40(2)** E47-E55.
18. 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.

19. Holland N, Bolognesi C, Kirsch-Volders M, Bonassi S, Zeiger E, Knasmueller S, & Fenech M (2008) The micronucleus assay in human buccal cells as a tool for biomonitoring DNA damage: The HUMN project perspective on current status and knowledge gaps *Mutation Research* **659(1-2)** 93-108.
20. Angelieri F, Moleirinho TCG, Carlin V, Oshima CTF, & Ribeiro DA (2010) Biomonitoring of oral epithelial cells in smokers and nonsmokers submitted to panoramic Xray: Comparison between buccal mucosa and lateral border of the tongue *Clinical Oral Investigation* **14(6)** 669-674.
21. Nerseyan A, Muradyan R, Kundi M, & Schulte-Hermann R (2011) Impact of smoking on the frequencies of micronuclei and other nuclear abnormalities in exfoliated oral cells: A comparative study with different cigarette types *Mutagenesis* **26(2)** 295-301.
22. Naderi NJ, Farhadi S, & Sarshar S (2012) Micronucleus assay of buccal mucosa cells in smokers with the history of smoking less and more than 10 years *Indian Journal of Pathology and Microbiology* **55(4)** 433-438.
23. Rezende M, Loguercio AD, Reis A, & Kossatz S (2013) Clinical effects of exposure to coffee during at-home vital bleaching *Operative Dentistry* **38(6)** E229-E236.
24. Paravina RD, Johnston WM, & Powers JM (2010) New shade guide for evaluation of tooth whitening—Colorimetric study *Journal of Esthetic and Restorative Dentistry* **19(5)** 276-278.
25. Commission Internationale de l'Eclairage (1978) Recommendations on Uniform Color Spaces, Color Difference Equations, Psychometric Color Terms, 1978; Supplement 2 to CIE Publication 15 (E2-31.1) 1971 (TC-1.3) 1978 Paris *Paris, France: Bureau Central de la CIE*.
26. Bansal H, Sandhu VS, Bhandari R, & Sharma D (2012) Evaluation of micronuclei in tobacco users: A study in Punjabi population *Contemporary Clinical Dentistry* **3(2)** 184-187.
27. Bortoluzzi MC, Campagnoli EB, Milan JR, Reinheimer A, Masson M, & Capella DL (2014) Frequency of micronucleus in oral epithelial cells after exposure to mate-tea in healthy humans *Medicina Oral Patologia Oral y Cirugia Bucal* **19(4)** e345-e349.
28. Tolbert PE, Shy CM, & Allen JW (1992) Micronuclei and other nuclear anomalies in buccal smears: Methods development *Mutation Research* **271(1)** 69-77.
29. Zekonis R, Matis BA, Cochran MA, Al Shetri SE, Eckert GJ, & Carlson TJ (2003) Clinical evaluation of in-office and at-home bleaching treatments *Operative Dentistry* **28(2)** 114-121.
30. Haywood VB (2003) Frequently asked questions about bleaching *Compendium Continuing Education in Dentistry* **24(4A)** 324-338.
31. Auschill TM, Hellwig E, Schmidale S, Sculean A, & Arweiler NB (2005) Efficacy, side-effects and patients' acceptance of different bleaching techniques (OTC, in-office, at-home) *Operative Dentistry* **30(2)** 156-163.
32. Bernardon JK, Sartori N, Ballarin A, Perdigão J, Lopes GC, & Baratieri LN (2010) Clinical performance of vital bleaching techniques *Operative Dentistry* **35(1)** 3-10.
33. Mondelli RF, Azevedo JF, Francisconi AC, Almeida CM, & Ishikiriama SK (2012) Comparative clinical study of the effectiveness of different dental bleaching methods—Two year follow-up *Journal of Applied Oral Science* **20(4)** 435-443.
34. Tay LY, Kose C, Loguercio AD, & Reis A (2009) Assessing the effect of a desensitizing agent used before in office-tooth bleaching *Journal of the American Dental Association* **140(10)** 1245-1251.
35. Reis A, Dalanhol AP, Cunha TS, Kossatz S, & Loguercio AD (2011) Assessment of tooth sensitivity using a desensitizer before light-activated bleaching *Operative Dentistry* **36(1)** 12-17.
36. Papathanasiou A, Kastali S, Perry RD, & Kugel G (2002) Clinical evaluation of a 35% hydrogen peroxide in-office whitening system *Compendium of Continuing Education in Dentistry* **23(4)** 335-338.
37. Onwudiwe UV, Umesi DC, Orenuga OO, & Shaba OP (2013) Clinical evaluation of 16% and 35% carbamide peroxide as in-office vital tooth whitening agents *Nigerian Quarterly Journal of Hospital Medicine* **23(2)** 80-84.
38. Rezende M, Loguercio AD, Kossatz S, & Reis A (2016) Predictive factors on the efficacy and risk/intensity of tooth sensitivity of dental bleaching: A multi regression and logistic analysis *Journal of Dentistry* **45** 1-6.
39. Gerlach RW, & Zhou X (2001) Vital bleaching with whitening strips: Summary of clinical research on effectiveness and tolerability *Journal of Contemporary Dental Practice* **2(3)** 1-16.
40. Paula E, Kossatz S, Fernandes D, Loguercio A, & Reis A (2013) The effect of perioperative ibuprofen use on tooth sensitivity caused by in-office bleaching *Operative Dentistry* **38(6)** 601-608.
41. De Paula EA, Loguercio AD, Fernandes D, Kossatz S, & Reis A (2013) Perioperative use of an anti-inflammatory drug on tooth sensitivity caused by in-office bleaching: A randomized, triple-blind clinical trial *Clinical Oral Investigations* **17(9)** 2091-2097.
42. Bonafé E, Bacovis CL, Iensen S, Loguercio AD, Reis A, & Kossatz S (2013) Tooth sensitivity and efficacy of in-office bleaching in restored teeth *Journal of Dentistry* **41(4)** 363-369.
43. Fried K, Sessle BJ, & Devor M (2011) The paradox of pain from tooth pulp: Low-threshold "algoneurons"? *Pain* **152(12)** 2685-2689.
44. Gibbs JL, Melnyk JL, & Basbaum AI (2011) Differential TRPV1 and TRPV2 channel expression in dental pulp *Journal of Dental Research* **90(6)** 765-770.
45. Caviedes-Bucheli J, Ariza-García G, Restrepo-Méndez S, Ríos-Osorio N, Lombana N, & Muñoz HR (2008) The effect of tooth bleaching on substance P expression in human dental pulp *Journal of Endodontics* **34(12)** 1462-1465.
46. Costa CA, Riehl H, Kina JF, Sacono NT, & Hebling J (2010) Human pulp responses to in-office tooth bleaching

Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology and Endodontics **109(4)** e59-e64.

47. Charakorn P, Cabanilla LL, Wagner WC, Foong WC, Shaheen J, Pregitzer R, & Schneider D (2009) The effect of preoperative ibuprofen on tooth sensitivity caused by in-office bleaching *Operative Dentistry* **34(2)** 131-135.
48. De Paula EA, Kossatz S, Fernandes D, Loguercio AD, & Reis A (2014) Administration of ascorbic acid to prevent bleaching-induced tooth sensitivity: A randomized triple-blind clinical trial *Operative Dentistry* **39(2)** 128-135.
49. Rezende M, Bonafe E, Vochikovski L, Farago PV, Loguercio A, Reis A, & Kossatz S (2016) Pre- and postoperative dexamethasone does not reduce bleaching-induced tooth sensitivity: A randomized, triple-masked clinical trial *Journal of the American Dental Association* **147(1)** 41-49.
50. Floyd RA (1997) The effect of peroxides and free radicals on body tissues *Journal of the American Dental Association* **128(Supplement)** 37S-40S.
51. Fenech M, Crott J, Turner J, & Brown S (1999) Necrosis, apoptosis, cytostasis and DNA damage in human lymphocytes measured simultaneously within the cytokinesis-block micronucleus assay: Description of the method and results for hydrogen peroxide *Mutagenesis* **14(6)** 605-612.
52. Ribeiro DA, Marques ME, & Salvadori DM (2005) Assessment of genetic damage induced by dental bleaching agents on mouse lymphoma cells by single cell gel (comet) assay *Journal of Oral Rehabilitation* **32(10)** 766-771.
53. Thomson PJ, Potten CS, & Appleton DR (1999) Mapping dynamic epithelial cell proliferative activity within the oral cavity of man: A new insight into carcinogenesis? *British Journal of Oral & Maxillofacial Surgery* **37(5)** 377-383.
54. Newman MG, Takei HH, Klokkevold PR, & Carranza FA (2012) *Carranza's Clinical Periodontology* **11th edition** Elsevier, St Louis, MO.

©Operative Dentistry, 2016, **41-6,** 587-598

A Prospective Six-Year Clinical Study Evaluating Reinforced Glass Ionomer Cements with Resin Coating on Posterior Teeth: Quo Vadis?

LS Türkün • Ö Kanik

Clinical Relevance

Despite minor reparable defects, the overall clinical performance of EquiaFil was found to be excellent even in large posterior class II restorations after a period of six years compared to Riva SC.

SUMMARY

Objective: The aim of this study was to evaluate the long-term clinical performance of two encapsulated glass ionomer cements (GICs) (EquiaFil and Riva SC) covered with two different coatings (Equia Coat and Fuji Varnish) over six years using modified US Public Health Service (USPHS) criteria.

Methods: Fifty-four patients having class I and II restorations/caries were included in the study. A total of 256 restorations were made with EquiaFil and Riva SC. Equia Coat or Fuji Varnish was used randomly on the surface of the restorations. After cavity preparations, the teeth were randomly restored with one GIC and coated with Equia Coat or Fuji Varnish. The restorations were evaluated at baseline; six, 12, and 18 months; and six years after placement using modified USPHS criteria. Two evaluators checked color match, marginal discoloration, marginal adaptation, caries formation, anatomical form, postoperative sensitivity, and retention rate, and photographs were taken at each recall. The results were evaluated with Pearson chi-square and Mann-Whitney U-test ($p<0.05$).

Results: Thirty-seven patients were evaluated. There was a significant difference between EquiaFil and Riva SC regarding retention rate and color match after six years ($p=0.033$ and 0.046). When comparing baseline to six years, the overall success of EquiaFil was better than Riva SC, having significant problems regarding retention rate and anatomical form ($p=0.016$ and 0.031). Class II cavities were

*Lezize Sebnem Turkun, DDS, PhD, professor, Department of Restorative Dentistry Bornova, Ege University School of Dentistry, Izmir, Turkey

Özgur Kanik, DDS, PhD, assistant professor, Department of Restorative Dentistry, Kocatepe University School of Dentistry, Afyon, Turkey

*Corresponding author: Izmir 35100, Turkey; e-mail sebnemturkun@hotmail.com

DOI: 10.2341/15-331-C

significantly worse in marginal adaptation, anatomical form, and retention rate in the Riva SC groups (p=0.033, 0.015, and 0.007) but not in the EquiaFil groups. The combination of the coatings had no effect on the overall success of the materials.

Conclusions: The EquiaFil system was more successful than Riva SC regarding color match, marginal adaptation, anatomic form, and retention rate after a six-year clinical evaluation period.

INTRODUCTION

For a few decades, a broad range of restorative dental materials has been emerging onto the market. Some of them needed special cavity designs, whereas others were intended to be used with adhesive dentistry. More recently, another treatment approach, minimal invasive dentistry, has gained popularity, especially in the field of operative dentistry. Today, restoring a tooth is not the only objective; it is also important to protect the existing tooth structures for a long period of time from any invasive treatment procedures. For that purpose, besides being esthetically pleasing, the new restorative materials of choice need to have good physical and mechanical properties and at the same time induce the remineralization of the tooth. One of the most popular and effective dental materials belonging to this group is glass ionomer cement (GIC).[1-3]

GICs, introduced by Wilson and Kent[4] in 1972, are a special group of dental materials having some very unique properties. They adhere to slightly moist enamel and dentin without the need for any adhesive system, they release fluoride and thus have anticariogenic effects for an extended period of time, they can absorb and release fluoride from topical fluoride solutions, they have thermal expansion similar to enamel, and they are biocompatible with a low toxicity. This group of materials is also considered to be "bioactive" due to these unique features. However, traditional GICs have some drawbacks, such as low fracture toughness, higher occlusal wear, and the need to be protected from initial dehydration and moisture uptake at the early maturation stage compared to other restorative materials, such as amalgam and modern resin composite restorative materials.[1-3,5] To avoid problems in the first 24 hours, copal varnish, light-cured bonding resins, petroleum jelly, cocoa butter, and even nail varnish have been used over the years.[6,7] It is presumed that this layer will prevent the restoration from dehydrating and maintain the all-important water balance in the system.[8] The longer this protective material is in contact with the restoration, the smaller the chance that its mechanical properties will decrease.[9]

In the late 1990s, many improvements and modifications have been made in GICs to overcome their disadvantages and to develop a material that may replace amalgam and that can be used in field conditions with atraumatic restorative treatment (ART) techniques.[10] However, these GICs exhibited a wear rate five times higher than amalgam and three times higher than resin composite materials[11] and were not found very successful in early clinical studies.[12,13] Consequently, their reputation did not change much, and they were still considered a semipermanent restorative material for small to moderate-size class I and II fillings in permanent teeth.[14] A few years later, due to the need for a cheap, tooth-colored material with easy handling properties, the development of high-viscosity, encapsulated, and packable GICs occurred. These were introduced to the market claiming superior physical and mechanical properties and a more translucent appearance than their predecessors due to the incorporation of glass particles.[1]

Early reviews published in 2004 and 2005 on the clinical performance of highly viscous GICs in posterior teeth indicated that their annual failure rate was estimated to be around 8%.[15,16] Scholtanus and Huysmans[17] published a six-year study in 2007 performed by two dentists in private practice with 116 class II restorations. According to their results, until 18 months, there were no problems; at the 3.5-year recall, the survival was 93%; and at six years, the survival dropped to 60% for the GIC tested. On the other hand, clinical trials performed with ART techniques and highly viscous GICs were very promising. Frencken and others[18] performed an ART study with Fuji IX and Ketec Molar, reporting a cumulative survival rate of 66.1% for GIC and 57% for amalgam after 6.3 years. Ersin and others[19] conducted a two-year clinical study with Fuji IX GP and a packable resin composite on primary molars restored again with ART and found a survival rate of 96.7% in class I and 76.1% in class II GIC restorations with no significant difference with the resin composite tested. A few years later, in 2011, Burke and others[20] evaluated 169 Fuji IX restorations in class I and II cavities placed by three dentists and found a survival rate of 98% after two years of follow-up, the main reason for replacement being fracture.

Table 1: *Description of Materials Used in This Study*

Material	Type	Manufacturer	Composition
EquiaFil	Conventional glass ionomer cement	GC (Tokyo, Japan)	Powder: 95% strontium fluoro alumino-silicate glass, 5% polyacrylic acid Liquid: 40% aqueous polyacrylic acid
Riva SC	Conventional glass ionomer cement	SDI (Bayswater, VIC, Australia)	Powder: Strontium fluoro alumino-silicate glass, polyacrylic acid copolymer powders, pigments Liquid: polyacrylic acid copolymers
Equia Coat	Low-viscosity nanofilled surface coating resin	GC	40%-50% methyl methacrylate, 10%-15% colloidal silica, 0.09% camphorquinone, 30%-40% urethane methacrylate, 1%-5% phosphoric ester monomer
Fuji Varnish	Classical varnish	GC	Isopropyl acetate, acetone, vinyl acetate, vinyl chloride copolymer, glycerol, resin acid esters

In 2007, a new glass ionomer concept consisting of a highly viscous encapsulated GIC, Fuji IX GP Extra (GC, Tokyo, Japan), and a nanofilled light-cured coating material, G-Coat Plus (GC), named Equia, was developed.[6] The changes in the formulation over the traditional GICs included a reduction in the size of the glass particles in the matrix, offering improved physical properties and stiffer syringeable materials that allow some packability to the material. The G-Coat Plus, with its low viscosity, can be used as a glaze on the top of the GICs and resin composites and can adhere to enamel and dentin as well. Furthermore, the nanofillers protect the GIC against abrasive wear that may occur during the early stage of a few months until the material becomes fully mature and resistant to intraoral situations.[1,8,21,22] A few years later, in 2009, this system was renamed EquiaFil and the coating Equia Coat.

To date, few prospective clinical studies have compared the long-term clinical performance of this new restorative system to other dental materials, these usually being resin composites. The available published studies derive mainly from data obtained from retrospective studies, including many operators of private practices,[8,23] or are performed with ART techniques in field conditions on primary teeth. The authors of this study could not find any other long-term clinical study comparing the clinical performance of encapsulated GICs between themselves. Furthermore, there are also no clinical studies comparing the effect of light-cured coatings to classical varnishes on GIC restorations.

Therefore, the aim of this study was to evaluate the long-term clinical performance of two encapsulated GICs (EquiaFil and Riva SC) covered with two different coatings (Equia Coat and Fuji Varnish) over six years using modified US Public Health Service (USPHS) criteria. The null hypothesis of the study was that after six years, there would be no difference in the clinical performance of the two GICs tested for the assessed criteria.

METHODS AND MATERIALS

In this six-year randomized prospective controlled clinical trial, two encapsulated GIC restorative materials—EquiaFil and Riva SC (SDI, Bayswater, VIC, Australia) in combination with two different coatings (Equia Coat and Fuji Varnish)—were evaluated. The materials used are described in Table 1.

Patient Recruitment

Patients applying for routine dental care at Ege University, School of Dentistry, were screened by the Department of Restorative Dentistry. The inclusion criteria for the selection of the patients for the study were as follows: 1) the patient should have good oral hygiene, 2) the patient has a need for at least two or more posterior restorations in contact with the neighbouring tooth and in occlusion with the antagonist teeth, 3) teeth planned to be restored should be vital and symptomless, and 4) the cavity isthmus size should be more than one-third the intercuspal distance. The exclusion criteria were as follows: 1) absence of adjacent and antagonist teeth, 2) teeth with periodontal problems, 3) teeth with preoperative pain or pulpal inflammation, 4) teeth formerly subjected to direct pulp capping, and 5) patients having severe systemic diseases, allergies, or adverse medical histories.

A total of 54 patients satisfying the inclusion criteria were selected to participate in this clinical trial. The average age of the patients was 34.6 years (range 17-55 years). All included patients were voluntary and signed a written informed consent.

Table 2: *Distribution of the Restorative Material Groups Among Dental Arches and Cavity Classes at the Beginning of the Study*

Restorative Materials	Maxillar		Mandibular		Class I	Class II	Total
	Premolar	Molar	Premolar	Molar			
EquiaFil + Equia Coat	10	19	10	37	28	44	**72**
EquiaFil + Fuji Varnish	9	14	11	20	33	21	**54**
Riva SC + Equia Coat	15	22	7	21	28	40	**68**
Riva SC + Fuji Varnish	11	29	9	16	35	27	**62**
Total	45	84	37	94	124	132	**256**

Restorative Procedures

One experienced dentist placed 124 class I and 132 class II restorations (256 restorations in total). The distribution of the materials, cavity classes, and tooth groups are depicted in Table 2. The filling materials EquiaFil or Riva SC were randomized over these two cavity groups using a randomization list.

Before treatment, preoperative digital photographs and periapical radiographs were taken, and the vitality of the tooth was recorded. Old restorations were removed and the enamel was prepared using diamond round and fissure burs (Komet, Gebr. Brasseler GmbH & Co KG, Lemgo, Germany) at high speed with water cooling. To remove caries, hand instruments and slow-speed steel burs were used. Local anesthesia was used only in patients complaining about pain or sensitivity. The isolation of the cavities was performed with cotton rolls and high-speed evacuation. For initial caries excavations, a minimal invasive cavity design was used. The two surface class II cavities were mostly medium size to large, but none of them involved any cusps. No beveling was performed in any of the cavities. A $Ca(OH)_2$ cavity liner (Dycal, Dentsply DeTrey, Konstanz, Germany) was applied where needed as a limited base material. A sectional matrix system (Palodent, Dentsply) was used to restore the class II cavities.

The cavities were restored using a bulk fill method with one of the encapsulated GICs (EquiaFil or Riva SC) and covered with a light-cured coating (Equia Coat) or a classical varnish (Fuji Varnish) according to the manufacturer's instructions. The P/L ratios of the glass ionomers were similar, being 0.40/0.12 for EquiaFil and 0.45/0.14 for Riva SC.

EquiaFil and Riva SC Restorative Procedures

After the cavity preparations, the dentin and enamel were conditioned with 20% polyacrylic acid for 10 seconds (Cavity Conditioner, GC) to remove the smear layer, rinsed thoroughly, and briefly air-dried without desiccating the surface. Both EquiaFil and Riva SC capsules were mixed for 10 seconds in a capsule mixer (Silvermix90, GC) and packed into the cavities with their respective gun injectors. The major grooves and fissures were modeled and the restorations left undisturbed for two minutes for initial setting according to the manufacturer's recommendations. Then the occlusion was checked with two layers of articulation paper and adjusted with water-cooled high-speed fine diamond burs (Komet).

As a last step, the restorations were quickly dried and covered randomly with one of the coating materials to be tested. Equia Coat was applied in one coat with its microbrush and light cured for 20 seconds using a light-curing unit (Bluephase C5, Ivoclar Vivadent, Liechtenstein), while Fuji Varnish was applied in two coats and left undisturbed until setting.

Evaluations and Recall Periods

One week after the restorations were placed, the patients were recalled, and two independent blinded clinicians making the baseline evaluations scored the restorations according to modified USPHS criteria.[10] The criteria evaluated were color match, marginal discoloration, marginal adaptation, anatomic form, caries development, postoperative sensitivity, and retention loss. The clinicians used mirrors and probes and took bitewing radiographs and intraoral digital photographs at baseline, six, 12, 18, and 72 months. If disagreement occurred during the evaluation stages, the final score decision was made by consensus of both examiners.

We anticipated that some marginal ridges of class II restorations might show some chipping during the course of the study that might not require the replacement of the entire restoration, only its repair. Thus, in agreement with the study of Peumans and others,[24] we thought that these restorations should be scored differently because they were not proper failures and could be maintained and monitored throughout the trial. For that purpose, we divided

Table 3: *Modified US Public Health Service (Ryge) Direct Evaluation Criteria*

Criteria	Inspection Method	Rating Scale
Color match	Visual inspection with a mirror at 18 inches	Alpha: The restoration matches the adjacent tooth structure in color and translucency. Bravo: Light mismatch in color, shade, or translucency between the restoration and the adjacent tooth. Charlie: The mismatch in color and translucency is outside the acceptable range of tooth color and translucency.
Marginal discoloration	Visual inspection with a mirror at 18 inches	Alpha: No discoloration anywhere along the margin between the restoration and the adjacent tooth. Bravo: Slight discoloration along the margin between the restoration and the adjacent tooth. Charlie: The discoloration penetrated along the margin of the restorative material in a pulpal direction.
Marginal adaptation	Visual inspection with a mirror at 18 inches	Alpha 1: Harmonious outline. Alpha 2: Marginal gap (max 100 μ) with discoloration (removable). Bravo: Marginal gap (>100 μ) with discoloration (unremovable). Charlie: The restoration is fractured or missing.
Anatomical form	Visual inspection with a mirror at 18 inches	Alpha 1: Continuous with existing anatomical form. Alpha 2: Slightly discontinuous due to some chipping on the proximal ridge. Bravo: Discontinuous with existing anatomical form due to material loss, but proximal contact still present. Charlie: Proximal contact is lost with ridge fracture.
Caries formation	Visual inspection with a mirror at 18 inches	Alpha: No evidence of caries. Bravo: Evidence of caries along the margin of the restoration.
Postoperative sensitivity	Visual inspection with a mirror at 18 inches	Alpha: No evidence of postoperative sensitivity. Bravo: Sensitivity present.
Retention rate	Visual inspection with a mirror at 18 inches	Alpha 1: Clinically excellent. Alpha 2: Clinically good with slight deviations from ideal performance; correction possible without damage of tooth or restoration. Bravo: Clinically sufficient with few defects; corrections or repair of the restoration possible. Charlie: Restoration is partially missing. Delta: Restoration is totally missing.

score Alpha into two groups, A1 and A2, for marginal adaptation, anatomic form, and retention rate criteria, as shown in Table 3.

Statistical Analysis

Statistical analysis was performed with SPSS version 13.0 software. To test the performance of the restorative materials according to USPHS criteria over the study period and to compare the acceptable versus unacceptable scores, the Pearson chi-square test was used. The Mann-Whitney U-test evaluated the comparison of the different coatings on the performance of the GIC tested. The level of significance was set at $\alpha=0.05$ for all tests.

RESULTS

Through 18 months, 52 patients with 248 restorations were evaluated. However, after six years, 37 patients and 88 restorations per glass ionomer group (total of 176 restorations, recall rate 68.75%) were available for recall. The scores obtained during the recalls and the distribution of the materials/coating combinations over the study period are detailed in Table 4.

In the modified USPHS evaluation criteria, scores A and A1 meant the highest degree of clinical acceptability, while A2 meant a clinically acceptable restoration having minor reparable problems. Score B stood for a clinically acceptable restoration that did not need replacement but could survive with some repairs, and scores C/D stood for unacceptable restorations that needed to be replaced.

None of the patients at any time interval of the study experienced pain or postoperative sensitivity from the restored teeth, and no incidence of caries formation was observed.

Table 4: *US Public Health Service Scores Obtained During the Recall Periods*

Criteria	Baseline			Six Months			12 Months			18 Months			Six Years			
	A1/A2	B	C	A1/A2	B	C	A1/A2	B	C	A1/A2	B	C	A1/A2	B	C	D
Color match																
EG	19	36	7	19	36	7	19	36	7	19	36	19	43	2	0	—
EV	16	36	10	16	36	10	16	36	10	16	36	16	43	0	0	—
RG	0	8	54	0	8	54	0	8	54	0	8	0	30	5	12	—
RV	1	6	55	1	6	55	1	6	55	1	6	1	28	5	8	—
Marginal adaptation																
EG	62	0	0	59/1	2	0	55/4	2	1	53/4	1	53/4	35/0	4	1	—
EV	62	0	0	60/0	1	1	59/1	1	1	58/1	1	58/1	43/2	3	0	—
RG	62	0	0	62/0	0	0	59/3	0	0	56/4	1	56/4	39/4	3	3	—
RV	62	0	0	59/3	0	0	56/6	0	0	55/6	1	55/6	30/3	4	2	—
Anatomic form																
EG	62	0	0	59/1	2	0	56/3	2	1	53/3	1	53/3	40/1	4	1	—
EV	62	0	0	60/0	1	1	59/1	1	1	58/1	1	58/1	37/1	3	1	—
RG	62	0	0	62/0	0	0	60/2	0	0	57/3	1	57/3	43/2	4	4	—
RV	62	0	0	59/3	0	0	56/6	0	0	55/6	1	55/6	28/4	1	2	—
Retention rate																
EG	62	0	0	59	3	0	55	6	1	52	5	52	40	3	1	0
EV	62	0	0	60	1	1	59	2	1	58	2	58	41	3	0	0
RG	62	0	0	62	0	0	58	4	0	55	6	55	37	2	3	1
RV	62	0	0	59	3	0	58	4	0	57	4	57	40	2	2	1
Marginal discoloration																
EG	62	0	0	62	0	0	62	0	0	62	0	62	43	0	0	—
EV	62	0	0	61	1	0	61	1	0	61	1	61	43	2	0	—
RG	62	0	0	61	1	0	60	2	0	60	2	60	43	0	0	—
RV	62	0	0	62	0	0	62	0	0	62	0	62	43	2	0	—
Caries formation																
EG	62	0	—	62	0	—	62	0	—	62	0	62	44	0	—	—
EV	62	0	—	62	0	—	62	0	—	62	0	62	44	0	—	—
RG	62	0	—	62	0	—	62	0	—	62	0	62	44	0	—	—
RV	62	0	—	62	0	—	62	0	—	62	0	62	44	0	—	—
Postoperative sensitivity																
EG	62	0	—	62	0	—	62	0	—	62	0	62	44	0	—	—
EV	61	1	—	62	0	—	62	0	—	62	0	62	44	0	—	—
RG	61	1	—	62	0	—	62	0	—	62	0	62	44	0	—	—
RV	62	0	—	62	0	—	62	0	—	62	0	62	44	0	—	—

Abbreviations: EG, EquiaFil + Equia Coat; EV, EquiaFil + Fuji Varnish; RG, Riva SC + Equia Coat; RV, Riva SC + Fuji Varnish.

The resin coating and the varnish applied on all restorations were worn away in nearly all of the restorations after six months. However, this was not seen as a problem, and during the other study recalls, we could not find any significant difference or influence of the different coatings on the success rate of either glass ionomer ($p \geq 0.05$) (Figure 1).

Regarding the color-match criteria, there was a significant difference between EquiaFil and Riva SC in all of the recall periods ($p \leq 0.05$). After 18 months, there was a pronounced color mismatch (score C) in Riva SC coated with Equia Coat (87.1%) and Riva SC coated with Fuji Varnish (88.7%) compared to both EquiaFil groups. The color match of both EquiaFil groups was eight times better than all the Riva SC groups. The match of these groups was found to be better after six years, but the significant difference was maintained ($p \leq 0.05$).

Compared to baseline, the difference in the marginal discoloration scores of EquiaFil and Riva SC groups was not significantly different in any of the recalls during the six years ($p \geq 0.05$) (Figure 2).

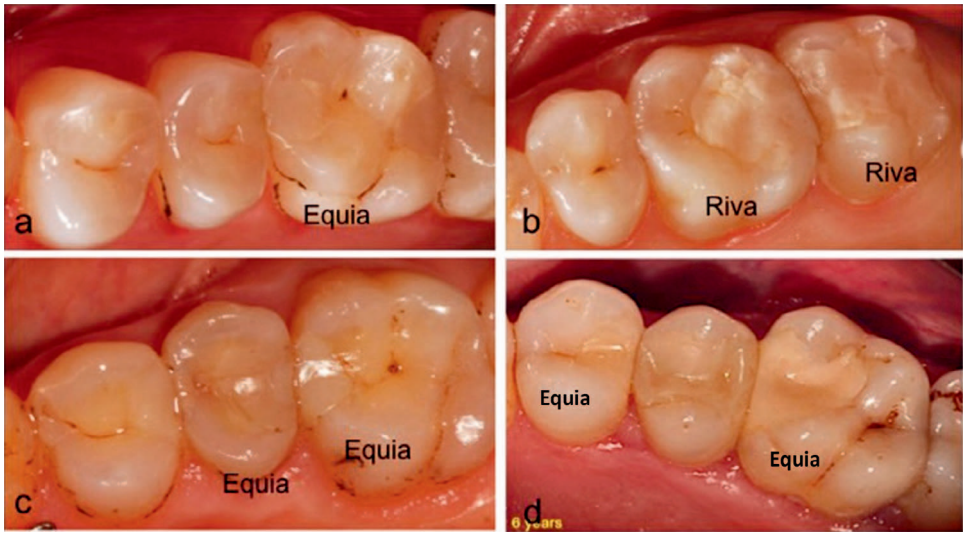


Figure 1. *(a-d): EquiaFil and Riva SC class II restorations scored as 'A' after six-year recall.*

Until the 18-month recall, for marginal adaptation, anatomic form, and retention rate, due to the low number of restorations that had problems in all the groups, Pearson chi-square could not be performed. All the class I restorations were found to be perfect, while a total of nine class II restorations had to be replaced: five cases of EquiaFil + G Coat (8.1%), two cases of EquiaFil + Fuji Varnish (3.2%), one case of Riva SC + G Coat (1.6%), and one case of Riva SC + Fuji Varnish (1.6%). However, at the six-year recall, the difference was significant for all three criteria mentioned above for class II restorations between the two main GIC groups regardless of the coating used ($p \leq 0.05$) (Figure 3). Regarding the marginal adaptation criteria, in the EquiaFil group, one restoration had to be replaced (1.1%), while in the Riva SC group, five cases (5.6%) were scored as unacceptable ($p=0.033$). For the anatomic form criteria, in the EquiaFil group, two restorations had to be replaced (2.27%), while in the Riva SC group, six cases (6.81%) were scored as unacceptable ($p=0.015$) (Figure 4).

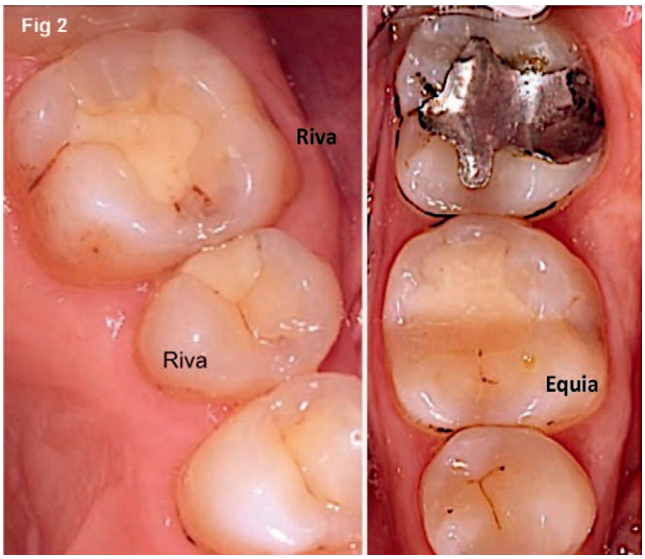


Figure 2. *(Left): Two Riva SC restorations having a color mismatch and some marginal discolorations after six years. (Right): One EquiaFil occlusal restoration having an 'A' score in all the criteria after six years.*

In the retention rate criteria, both partial loss (score C) and total loss of the restorations (score D) were considered as failure. In the EquiaFil group, one restoration had to be replaced (1.13%), while six had to be repaired. In the Riva SC group, five cases (5.68%) were scored as partial loss, while two restorations (2.27%) had to be replaced and were scored as unacceptable ($p=0.007$) (Figure 5). The total failure rate for Riva SC was found to be 7.95% after six years of clinical service.

DISCUSSION

Clinical evaluations of restorative materials are essential in order to obtain data on their clinical performance and *in vivo* longevity. Clinical trials with GICs as permanent restorative materials were conducted mostly on class I cavities,[10,25,26] and there are limited data showing their performance in class II cavities. Moreover, in the past, clinical studies on GICs were performed usually in primary molars and with ART techniques.[19,27,28] The results were mostly positive; however, the materials were powder/liquid, and the coatings were usually classical varnishes. In the past decade, long-term clinical studies on GICs have been performed on adult patients' posterior teeth having small to moderate-size cavities[18] with

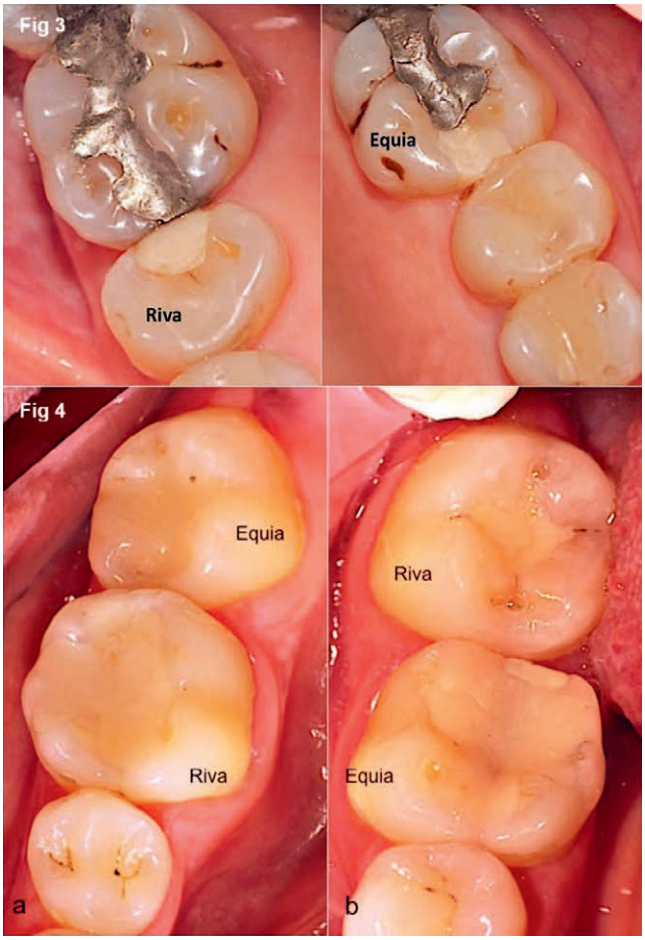


Figure 3. *(Left): One class II Riva SC restoration receiving an 'A2' score for marginal adaptation and anatomic form and a 'C' score for color match after six years. (Right): One class II EquiaFil restoration receiving an 'A' score for all the criteria after six years.*
Figure 4. *(a): Six-year recall of perfect EquiaFil restoration and a 'B' score for Riva SC restoration. (b): Six-year recall of an occlusal Riva SC restoration having a slight color mismatch and a large class II EquiaFil restoration being repaired at 18 months and still functioning at the six-year recall.*

better results with reinforced GICs and in small restorations.[14,17,23] The present study was performed on adult patients with an average age of 34.6 years and having moderate-size to large class I and II cavities. Many of the class II cavities had very large proximal boxes that were beyond the indicated application of both glass ionomers. This procedure was an especially great challenge for Riva SC, which was more clearly indicated for non–stress-bearing class II cavities.

According to the American Dental Association, a material intended to be used in posterior teeth needs to have a retention rate of at least 90% after 18 months of clinical service to become fully accepted as a definitive restorative material.[29] In our study, after 18 months, only a few changes were noted in the evaluated GICs, making both materials suitable for permanent posterior teeth restorations. However, at the six-year recall, the difference between the materials was more pronounced.

Color match was a great problem in conventional GICs due to the lack of translucency of the material. However, some reinforced and modern GICs exhibit a better match with adjacent tooth structures, partially due to the small glass particles and filled resin-based coating materials. In our study, both GICs were reinforced; however, EquiaFil's color match was found to be eight times better than Riva SC after 18 months. The difference was less visible after six years due to the improvement in translucency over time of the materials as the cements mature.[7] To overcome these shade problems, in 2014, the manufacturer of Riva SC launched more translucent and esthetic shades (T-A2, T-A3, and T-A3.5).

The applied coatings sealed the restorations in a thickness of 35-40 μ,[30] protected the margins, and created a regular and glossy surface. However, they disappeared in nearly all cases at the six-month recall, leaving a slightly rougher surface than the adjacent enamel. With time, these coatings were lost by oral mastication wear, but during this time, the cements are expected to become more resistant to variation in water balance and fully mature with maximum mechanical strength.[31] The same findings were obtained in a study by Miletic and others.[32] Lohbauer and others[25] conducted an *in vitro* study to compare the three-point fracture strength (FS) and three-body wear of G-Coat Plus–coated and –non-coated Fuji IX GP Extra samples. According to their findings, the GIC surface must be resin coated to improve its mechanical strength and wear resistance. Similarly, Bonifacio and others[21] measured the FS and wear of coated versus uncoated Fuji IX GP Extra specimens and concluded that the resin coating improved the FS and wear resistance of the GIC tested. In contrast to these studies, Bagheri and others[33] compared the fracture toughness (FT) by a four-point bending test of Fuji IX GP Extra and a resin-modified GIC and found that the FT of the GIC was not affected by the resin coating.

The absence of failures due to secondary caries after six years is most likely due to the anticariogenic effect and fluoride release of the GICs.

Marginal discoloration was moderate and was seen only in a few cases during the six-year period

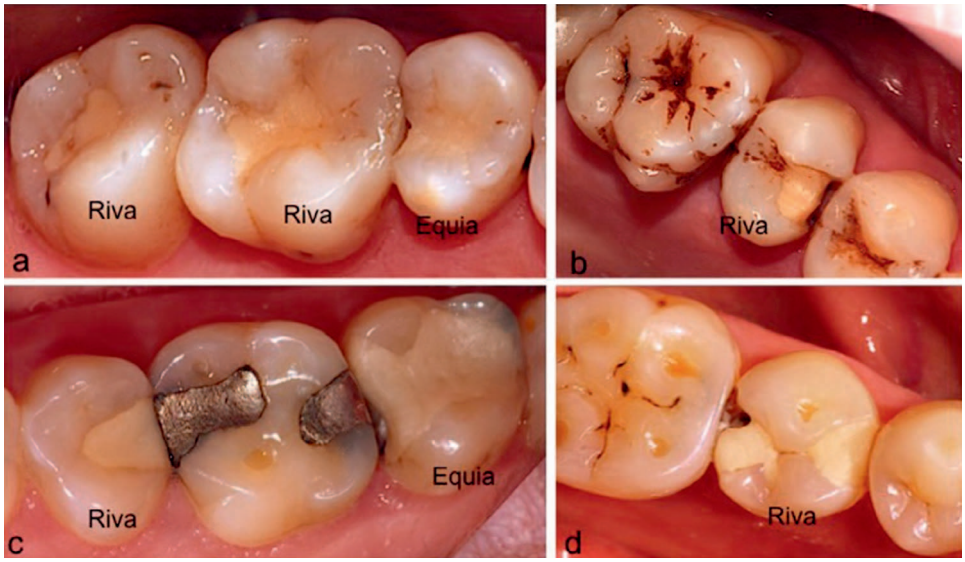


Figure 5. *(a): EquiaFil OD restoration receiving a 'B' score for anatomic form and retention rate after six years. (b): Riva SC restoration being scored as 'C' for marginal adaptation, anatomic form, and retention rate after six years. (c): Perfect Riva SC restoration and EquiaFil restoration scored as 'B' for anatomic form and 'A2' for retention rate after six years. (d): Riva restoration scored as 'C' for anatomic form and retention rate after six years.*

of evaluation. This is probably due to the self-adhesion of the glass ionomers to tooth structures without the need of adhesive systems.

Anatomic form, marginal adaptation, and retention loss are more or less related USPHS criteria for posterior restorative materials. The loss of anatomical form of restorations is an indication of wear and morphological changes, especially in class II restorations. In the present study, as in the study of Peumans and others,[24] all of the three criteria were modified for the Alpha score because of the nature of the GIC. In the original USPHS criteria, an Alpha score meant that the monitored restoration was in perfect condition, while a Bravo score meant that it had some minor problems that did not affect the performance of the material. In our study, an Alpha 2 score was added for marginal adaptation and meant that a marginal gap less than 100 μ was present with a slight discoloration that could be removed by polishing. Regarding anatomical form, an Alpha 2 score meant that the restoration was slightly discontinuous due to some chipping on the proximal ridge of the class II restorations. For the retention loss criteria, an Alpha 2 score was set mainly for class II restorations that deviated slightly from ideal performance and that had minimal material loss that may be corrected without inducing any damage to the adjacent tooth structures or the restoration itself.

In the present study, the evaluated clinical performance of GIC until 18 months was found to be excellent without any difference between the materials or coating applications.[34] However, between 18 months and six years, the clinical performance of Riva SC GIC in moderate-size to large class II restorations was significantly worse that EquiaFil for marginal adaptation, anatomic form, and retention loss. Similar to our results, Scholtanus and others[17] found no failure at 18 months in their long-term clinical study evaluating the previous version of EquiaFil: Fuji IX GP. However, at six years, the survival rate dropped to 60%. Our results might be due to the composition and amount of the strontium fluoro alumino-silicate glass present in the Riva SC material, probably being more close to conventional glass ionomers than to reinforced materials. In addition, Riva SC was developed for the restoration of only small and non–stress-bearing class II restorations, and this could have made it less successful in moderate-size to large restorations. It is our opinion that to be able to have long-term successful results with this material, the cavity size should be small to moderate, and the width of the proximal box should not exceed the half of the intercuspal distance.

In a review performed in 2005 by Hickel and others[15] on primary molar teeth and with conventional GICs, the main reason of failure in class II restorations was reported to be fracture. Again, in the study by Frankenberger and others,[14] Ketac Molar covered with Ketac Glaze in class I and II restorations was evaluated for two years, and they reported having lost the interproximal contacts in 40% of the class II restorations due to bulk

fracture, meaning an annual failure of 20%. However, the dropoff rate was 76%, and this may have influenced the low performance of the material. According to Basso and others,[30] the possibility of achieving durable class II restorations with GICs is related to the width of the mesial or distal box of the cavity. Much chipping and some failures of their 48-month clinical study performed with Equia were located in the marginal proximal crest of wider restorations. Similarly, in our study, marginal chipping and fractures were seen mainly in large proximal ridges of the class II restorations. Some of them could be repaired and maintained, while some had to be replaced. For repairing the chipping, we performed a retentive slot cavity in the proximal box of the class II restoration, placed a sectional matrix, and filled the new cavity with a fresh glass ionomer capsule to restore the proximal contact. When the fracture is large and the dentin is exposed or the proximal contact is totally lost, the replacement of the filling would be more appropriate than a repair.

Reinforced restorative GICs are a relatively new group of material; thus, published long-term clinical studies with these materials are few. To the authors' knowledge, there are no long-term clinical studies comparing the performance of two glass ionomers and two coating materials. Of the few published studies, many compared GICs to resin composites and were mainly retrospective and performed with more than one operator in general dental practices. However, carefully designed prospective studies performed in ideal conditions by a single operator and evaluated by two independent calibrated clinicians are superior to retrospective studies that provide data that were recorded for other reasons than research and evaluated by the operator him- or herself.

In a clinical study comparing EquiaFil and a resin composite, Gradia Direct Posterior, performed by Gürgan and others,[35] it was found that after four years, the success rates for class I and class II Gradia Direct Posterior restorations were 100%, whereas the failure rate was 7.7% for class II EquiaFil restorations. No significant change over time was found for the anatomical form, color match, secondary caries, postoperative sensitivity, surface texture, and retention for either restorative material. There was also no significant difference between the two restorative materials in terms of marginal discoloration at any recall period.

Diem and others[8] reported one of the few published studies comparing Fuji IX Extra (EquiaFil) with and without coating (Equia Coat) and also a microfine hybrid resin composite in the restoration of the first premolars of young children with the ART technique in field conditions. After three years, the color match of all the restorations improved, with no differences between the materials. Moderate marginal staining was depicted, and marginal adaptation loss was minimal for all restorations. They concluded that Fuji IX GP Extra with or without the coating showed acceptable clinical performance compared to the tested resin composite. Moreover, according to casts obtained from these restorations, the application of Equia Coat was found beneficial in reducing the wear of this GIC in class I restorations. Similar to our results, they found a rate of 3% of surface chipping or cracks in the GIC and of 2% in the resin composite. They concluded that notable marginal fractures did not become apparent until the third year in the Fuji IX Extra group.

Friedly and others[23] evaluated the performance of 151 Equia restorations in single- and multisurface posterior restorations for 24 months and found that large cavities had more volume loss, but all the restorations were scored as satisfactory at the end of the study. Khandelwal and others[36] evaluated the EquiaFil system for a period of two years. They reported 88.8% success in class I restorations and a perceptible roughness in 11.5% of the restorations with very few marginal disintegrations. Miletic and others[32] evaluated 45 Equia system restorations in a one-year pilot study and found that all the restorations were clinically acceptable.

In the present study, the EquiaFil system in both cavity types exhibited significantly better clinical outcomes over the observation period of six years than Riva SC. Therefore, the null hypothesis formulated at the beginning of the study was rejected.

Reinforced GICs may be considered as the material of the future in restorative dentistry and minimally invasive dentistry. Their long-term clinical success is making them promising as a permanent restorative material, even in moderate-size class II restorations. Further developments are needed to improve their mechanical properties and extend their indications.

CONCLUSION

The highly viscous reinforced GIC restorative system EquiaFil showed acceptable clinical performance according to modified USPHS criteria in

class I and moderate-size to large class II restorations over a period of six years.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subject's oversight committee guidelines and policies of Ege University. The approval code for this study is 08-10.1/6, 29.12.2007.

Acknowledgment

The authors of this manuscript would like to acknowledge the dental companies of GC Europe and SDI Australia for providing the materials free of charge for this clinical trial. A special thanks to Hayal Boyacıoglu for helping us with the statistical analysis.

Conflict of Interest

The authors 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 February 2016)

REFERENCES

1. Sidhu SK (2011) Glass-ionomer cement restorative materials: A sticky subject? *Australian Dental Journal* **56(Supplement 1)** 23-30.
2. Lohbauer U (2010) Dental glass ionomer cements as permanent filling materials? Properties, limitations and future trends *Materials* **3(1)** 76-96.
3. McLean J, & Wilson A (1977) The clinical development of the glass ionomer cement: II. Some clinical applications *Australian Dental Journal* **22(2)** 120-127.
4. Wilson AD, & Kent BE (1972) A new translucent cement for dentistry: The glass-ionomer cement *British Dental Journal* **132(4)** 133-135.
5. Ilie N, Hickel R, Valceaune AS, & Huth KC (2012) Fracture toughness of dental restorative materials *Clinical Oral Investigations* **16(2)** 489-498.
6. McLean JW, & Wilson AD (1977) The clinical development of the glass-ionomer cement II. Some clinical applications *Australian Dental Journal* **22(2)** 120-127.
7. Valera VC, Navarro MFL, Taga EM, & Pascotto RC (1997) Effect of nail varnishes and petroleum jelly combinations on glass ionomer dye uptake *American Journal of Dentistry* **10(5)** 251-253.
8. Diem VTK, Tyas MJ, Hien CN, Phuong LH, & Khanh ND (2014) The effect of a nano-filled resin coating on the 3-year clinical performance of a conventional high-viscosity glass-ionomer cement *Clinical Oral Investigations* **18(3)** 753-759.
9. Marquezan M, Osorio R, Ciamponi AL, & Toledano M (2010) Resistance to degradation of bonded restorations to simulate caries-affected primary dentin *American Journal of Dentistry* **23(1)** 47-52.
10. Tyas MJ (2006) Clinical evaluation of glass-ionomer cement restorations *Journal of Applied Oral Science* **14(Supplement)** 10-13.
11. Kunzelmann KH (1996) Glass-ionomer cements, cermet cements, hybrid-glass-ionomers and compomers—Laboratory-trials- wear resistance *Trans Academic Dental Materials* **9** 89-104.
12. Kilpatrick NM (1993) Durability of restorations in primary molars *Journal of Dentistry* **21(2)** 67-73.
13. Welbury RR, Walls AW, Murray JJ, & McCabe JF (1991) The 5-year results of a clinical trial comparing a glass polyalkenoate (ionomer) cement restoration with an amalgam restoration *British Dental Journal* **170(5)** 177-181.
14. Frankenberger R, Garcia-Godoy F, & Kramer N (2009) Clinical performance of viscous glass ionomer cement in posterior cavities over two years *International Journal of Dentistry* Article ID781462.1-7, doi:101155/2009/781462.
15. Hickel R, Kaaden C, Paschos E, Buerkle V, Garcia-Godoy F, & Manhart J (2005) Longevity of occlusually-stressed restorations in posterior primary teeth *American Journal of Dentistry* **18(3)** 198-211.
16. Manhart J, Chen HY, Hamm G, & Hickel R (2004) Buonocore memorial lecture review of the clinical survival of direct and indirect restorations in posterior teeth of the permanent dentition *Operative Dentistry* **29(5)** 481-508.
17. Scholtanus JD, & Huysmans MCDNJM (2007) Clinical failure of class II restorations of a highly viscous glass-ionomer over 6-year period: A retrospective study *Journal of Dentistry* **35(2)**156-162.
18. Frencken JE, Taifour D, & van't Hof MA (2006) Survival of ART and amalgam restorations in permanent teeth of children after 6.3 years *Journal of Dental Research* **85(7)** 622-626.
19. Ersin NK, Candan U, Aykut A, Önçag Ö, Eronat C, & Kose A (2006) A clinical evaluation of resin-based composite and glass ionomer cement restorations placed in primary teeth using the ART approach: Results at 24 months *Journal of the American Dental Association* **137(11)** 1529-1536.
20. Burke FJ, Siddons C, Cripps S, Bardha J, Crisp RJ, & Dopheide B (2007) Clinical performance of reinforced glass ionomer restorations placed in UK dental practices *British Dental Journal* **203(1) E2** (discussion 40-41).
21. Bonifacio CC, Werner A, & Kleverlaan CJ (2012) Coating glass-ionomer cements with a nanofilled resin *Acta Odontologica Scandinavica* **70(6)** 471-477.
22. Kato K, Yarimizu H, Nakaeseko H, & Sakuma T (2008) Influence of coating materials on conventional glass-ionomer cement *Journal of Dental Research* **87(Special Issue B)** Abstract #487.
23. Friedly K, Hiller KA, & Friedl KH (2011) Clinical performance of a new glass ionomer based restoration system: A retrospective cohort study *Dental Materials* **27(10)** 1031-1037.
24. Peumans M, De Munck J, Van Landuyt K, Poitevin A, Lambrechts P, & Van Meerbeek B (2010) Two-year clinical evaluation of a self-adhesive luting agent for ceramic inlays *Journal of Adhesive Dentistry* **12(2)** 151-61.

25. Lohbauer U, Kramer N, Siedschlag G, Schubert EW, Lauerer B, Muller FA, Petschelt A, & Ebert J (2011) Strength and wear resistance of a dental glass-ionomer cement with a novel nanofilled resin coating *American Journal of Dentistry* **24(2)** 124-128.

26. Aljamhan A (2012) Resin-coated glass ionomer cement abrasion and wear resistance. *Journal of Dental Research* **91(Special Issue A)** Abstract #1045.

27. Hubel S, & Mejare I (2003) Conventional versus resin-modified glass-ionomer cement for class II restorations in primary molars: A 3-year clinical study *International Journal of Paediatric Dentistry* **13(1)** 2-8.

28. Rutar J, McAllan L, & Tyas MJ (2000) Clinical evaluation of a glass ionomer cement in primary molars *Pediatric Dentistry* **22(6)** 486-488.

29. Loguercio AD, & Reis A (2008) Application of a dental adhesive using the self-etch and etch-and-rinse approaches: An 18-month clinical evaluation *Journal of the American Dental Association* **139(1)** 53-61.

30. Basso M, Brambilla E, Benites MG, Giovannardi M, & Ionescu AC (2015) Glass ionomer cement for permanent dental restorations: A 48-months, multi-center, prospective clinical trial *Stomatology Education Journal* **2(1)** 25-35.

31. Serra MC, Navarro MF, Freitas SF, Carvalho RM, Cury JA, & Retief DH (1994) Glass ionomer cement surface protection *American Journal of Dentistry* **7(4)** 203-206.

32. Miletic I, Baraba A, Juic IB, & Anic I (2013) Evaluation of a glass-ionomer based restoration system—A one year pilot study *Journal of Minimum Intervention in Dentistry* **6** 87-95.

33. Bagheri R (2012) Effect of G-Coat Plus on the properties of aesthetic restorations *Journal of Dental Research* **91(Special Issue B)** Abstract #753.

34. Turkun LS, & Kanic O (2010) Clinical evaluation of new glass ionomer coating combined systems for 18-months *Journal of Dental Research* **89(Special Issue B)** Abstract #402.

35. Gurgan S, Kutuk ZB, Ergin E, Oztas ZZ, & Cakir FY (2015) Four-year randomized clinical trial to evaluate the clinical performance of a glass ionomer restorative system *Operative Dentistry* **40(2)** 134-143.

36. Khandelwal P, Hiller K-A, Friedl K, & Friedl K-H (2010) Clinical performance of a glass ionomer based restorative system *Journal of Dental Research* **89 (Special Issue B)** Abstract #3240.

©Operative Dentistry, 2016, **41-6,** 599-606

Clinical Evaluation of Silorane and Nano-hybrid Resin Composite Restorations in Class II Cavities up to 3 Years

F Öztürk-Bozkurt • T Toz • A Kara-Tuncer • H Gözükara-Bağ • M Özcan

Clinical Relevance

Both nano-hybrid and silorane-based resin composites performed similarly in Class II restorations for up to three years except for marginal adaptation where silorane-based composite demonstrated significant marginal deterioration.

SUMMARY

In this study, the clinical performance of a silorane-based resin composite (SC) vs a nano-hybrid resin composite (NHC) was evaluated in Class II cavities. From January 2012 to February 2013, a total of 29 patients (eight men, 21 women; mean age, 24 ± 5 years) received 29 pairs of restorations using both SC (Filtek Silorane, 3M ESPE) and NHC (Filtek Z550, 3M ESPE) materials. Patients were followed until February 2015. One operator performed all restorations using the corresponding adhesive resins according to the manufacturers' instructions. Two calibrated independent examiners evaluated the restorations at one week, six months, and then annually using the modified United States Public Health Service (USPHS) criteria for anatomic form, marginal adaptation, color match, surface roughness, marginal discoloration, secondary caries, and postoperative sensitivity. Changes in the USPHS parameters were analyzed with the McNemar test (α=0.05). The mean observation period was 31.2 months. Marginal adaptation was the only parameter that showed a significant difference and was worse for SC than NHC (p=0.012). At the final recall, 17 restorations from the SC group and five from the NHC group received a score of 1 (explorer catches). These scores were significantly different be-

*Funda Öztürk-Bozkurt, DDS, PhD, assistant professor, Istanbul Medipol University, School of Dentistry, Department of Restorative Dentistry, Istanbul, Turkey

Tuğba Toz, DDS, PhD, assistant professor, Istanbul Medipol University, School of Dentistry, Department of Restorative Dentistry, Istanbul, Turkey

Aysun Kara-Tuncer, DDS, PhD, assistant professor, Bezmialem University, School of Dentistry, Department of Endodontics, Istanbul, Turkey

Harika Gözükara-Bağ, PhD, assistant professor, Inonu University, School of Medicine, Department of Biostatistics, Malatya, Turkey

Mutlu Özcan, DrMedDent, PhD, professor, University of Zurich, Center for Dental and Oral Medicine, Dental Materials Unit, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zurich, Switzerland

*Corresponding author: Ataturk Bulvarı 34083, Fatih, Unkapanı, Istanbul, Turkey; e-mail: fbozkurt@medipol.edu.tr

DOI: 10.2341/15-259-C

tween baseline and final recall for SC ($p<0.001$) but not for NHC ($p>0.05$). Both NHC and SC performed similarly in Class II restorations up to three years except for marginal adaptation, for which the latter demonstrated significant deterioration at the final recall compared with baseline.

INTRODUCTION

Since their introduction, dental composites have undergone many structural changes in order to achieve easier application and better clinical results. Their low cost, better optical properties, and less need for preparation made them the first choice of material for posterior direct restorations as opposed to other materials.[1] They also have shown acceptable prognoses according to some clinical studies.[2,3] However, the longevity of posterior resin restorations remains a matter of concern among clinicians, given that some other studies reported failures generally after 5 to 6 years.[1,4,5] According to these clinical studies, the most frequently reported defects and failures are fractures, secondary caries, and marginal leakage that is often attributed to polymerization shrinkage.[1,4,5]

Despite many improvements in dental adhesive technologies, polymerization shrinkage remains a problem because it is caused by an exchange of van der Waals forces in shorter covalent bond spaces during the conversion of monomers into a polymer network.[6] Polymerization shrinkage generates stresses between the tooth and the restoration and may increase the risk of early failure.[5] Current adhesive technologies aim for achieving "low-shrinkage" materials. With higher filler content and incorporation of prepolymerized resin fillers, the absence of low-molecular-weight diluents and the use of high-molecular-weight monomers, methacrylate-based resin composite materials present less polymerization shrinkage.[5]

Alternative efforts have been made by introducing an epoxide ring-opening polymerization type of chemistry.[7] Siloranes, a new class of ring-opening monomers, comprise two molecules: siloxane, which makes the material hydrophobic, and oxirane, with volumetric shrinkage of 0.99 vol%.[8-10] With this low polymerization shrinkage, the incidence of microleakage, secondary caries, postoperative sensitivity, and enamel fractures are claimed to decrease.[11] Siloranes have clinically acceptable physical, biological, and mechanical properties, and the performance of such resins is similar[12,13] or superior[14,15] to that of methacrylate-based resins.

Although clinical trials are costly and time-consuming, and prediction of the clinical results on the basis of a single or multiple *in vitro* studies may be desirable,[1] the overall clinical behavior of resin restorations is multifactorial and unlikely to be predicted by either of the methods alone. Because siloranes are recently introduced adhesive restorative technologies, they have not been evaluated widely *in vivo*.[16,17] Thus, the objective of this clinical evaluation was to investigate the longevity of silorane-based resin composite (SC) vs a methacrylate-based nano-hybrid resin composite (NHC) for posterior Class II restorations. The null hypothesis tested was that there would be no statistically significant differences between the two resin composite materials.

METHODS AND MATERIALS

The brands, manufacturers, chemical composition, and batch numbers of the materials used in this study are listed in Table 1.

Study Design

The Ethics Committee of Istanbul University approved this clinical study (2012/644-1047). Patients in need of at least two posterior restorations were recruited for the study. Inclusion criteria were as follows: Adults of at least 18 years of age, with good oral hygiene, having at least two primary approximal caries in the posterior teeth having an antagonist tooth in occlusion, being mentally in a good state to provide written consent to participate in the clinical study, and willing to attend the scheduled follow-up appointments. Exclusion criteria included no systemic diseases, presence of teeth with severe periodontal problems, extensive caries that need to be treated endodontically, and/or composite or amalgam replacements.

Placement of Restorations

From January 2012 to February 2013, one operator with experience in adhesive dentistry (more than 18 years since graduation) placed two pairs of restorations in 29 patients (eight men, 21 women; mean age, 24 ± 5 years). Following cavity preparation, the decision to apply the test or control material was randomized by tossing a coin; the distribution of restorations is shown in Table 2.

Isolation of the preparation was achieved with suction and cotton rolls. When the remaining dentin thickness was very thin, to perform indirect pulp capping, calcium hydroxide paste (Dycal, Dentsply

Table 1: *The Brand, Type, Manufacturer, and Chemical Composition of the Main Materials Used in This Study*

Brand	Type	Manufacturer	Chemical Composition
Silorane Self-etch Primer	Dentin primer	3M ESPE AG, St Paul, MN, USA	Phosphorylated methacrylates, bis-GMA, HEMA, water, ethanol, silane-treated silica filler, Vitrebond copolymer, initiators, stabilizers
Silorane Bond	Adhesive bond	3M ESPE AG	Hydrophobic methacrylates, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers
Filtek Silorane	Low-shrink resin composite	3M ESPE AG	Organic matrix: 3,4-epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-epoxycyclohexylethylphenylmethylsilane, yttrium fluoride, camphorquinone, iodonium salt, initiators, stabilizers
Ultra-Etch	Etching gel	Ultradent, South Jordan, USA	35% phosphoric acid
Adper Single Bond 2	Etch-&-rinse dental adhesive	3M ESPE AG	bis-GMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system, and a methacrylate functional copolymer of polyacrylic and polyitaconic acids
Filtek Z550	Resin composite	3M ESPE AG	bis-GMA, UDMA, TEGDMA, bis-EMA Filler: silica, zirconia Particle size: 0.6 to 10 μ 78.5 wt%, 63.3 vol%

Abbreviations: bis-EMA, Bisphenol A ethoxylated dimethacrylate; bis-GMA, Bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, Triethylene glycol dimethacrylate; UDMA, Urethane dimethacrylate.

Caulk, Milford, DE, USA) was placed in the deepest part and covered with a resin-modified glass ionomer liner (Glass-Liner, Willmann & Pein GmbH, Hamburg, Germany) and polymerized. Silorane Self-Etch Primer (3M ESPE Dental Products, St Paul, MN, USA) was scrubbed on the cavity walls of the preparation for 15 seconds, gently air-thinned, and photo-polymerized for 10 seconds with an LED device (Elipar Free Light, 3M ESPE AG, Germany; $\geq$400 mW/cm^2). Silorane Bond was applied with a brush to the cavity walls, air-thinned, and photo-polymerized for 10 seconds. SC (Filtek Silorane, 3M ESPE AG) was then placed incrementally and photo-polymerized separately for 40 seconds.

The adhesive for the control group was an etch-and-rinse system. Enamel margins and dentin were etched simultaneously with 35% phosphoric acid (Ultra-Etch, Ultradent, South Jordan, UT, USA) for 20 seconds. Following water rinsing for approximately 10 seconds, the cavity was dried for one to two seconds to prevent desiccation. Adper Single Bond 2 (3M ESPE Dental Products) was applied for 15 seconds using a microbrush, gently air-thinned, and photo-polymerized for 10 seconds. The NHC (Filtek Z550, 3M ESPE Dental Products) was then placed in 2-mm thickness incrementally and photo-polymerized for 40 seconds. All restorations were finished and polished with abrasive disks (Sof-Lex Finishing and Polishing Systems, 3M ESPE AG) and rubber cups (Jiffy Polishers, Ultradent, South Jordan, UT, USA).

Patients were given routine oral hygiene instructions and asked to contact the clinician if they perceived any problems with the restored teeth.

Evaluation

Two specialist dentists who were blinded to the study groups evaluated the restorations. In cases of differing scores, the observers reevaluated the restorations and reached a consensus. At baseline (one week following restoration placement for evaluation of postoperative sensitivity), six months, and for final recall, the restorations were evaluated using modified United States Public Health Service (USPHS) criteria[18] for the following parameters: anatomical form, marginal adaptation, color match, surface roughness, marginal staining, secondary caries, and postoperative sensitivity (Table 2).

Statistical Analysis

Statistical analysis was performed using SPSS 11.0 software for Windows (SPSS Inc, Chicago, IL, USA). The McNemar test was used to evaluate the difference between the two materials. A p-value of <0.05 was considered to indicate statistical significance.

Table 2: *Distribution of Restored Teeth and Restoration Types in the Maxilla and Mandible*

	Filtek Silorane		Filtek Z550	
	MO/DO	MOD	MO/DO	MOD
Maxilla				
Premolars (n)	6	0	6	1
Molars (n)	7	2	6	1
Mandible				
Premolars (n)	2	0	7	0
Molars (n)	9	3	7	1
Total (N)	24	5	26	3
	29		29	

Abbreviations: MO/DO, mesioocclusal/distoocclusal; MOD, mesioocclusodistal.

RESULTS

The distribution of restored teeth and restoration types in the maxilla and mandible are presented in Table 2. USPHS evaluation scores for the SC and NHC groups are provided in Table 3.

All patients (100%) attended the final recall visit. The mean observation period was 31.2 months. Indirect pulp capping was performed in nine restorations in the SC group and five in the NHC group.

At baseline, one restoration from each group (one with indirect pulp capping) was scored as 1 (sensitivity disappeared in one week) for postoperative sensitivity (Table 4). At baseline and the six months recall, no statistically significant difference was observed in any parameter ($p>0.05$). At the final recall, the only parameter that showed a significant

Table 3: *Modified United States Public Health Service (USPHS) Criteria*

Category	Score		Criteria
	Acceptable	Unacceptable	
Anatomical form	0		The restoration is continuous with tooth anatomy
	1		Slightly undercontoured or overcontoured restoration; marginal ridges slightly undercontoured, contact slightly open (may be self-correcting); occlusal height reduced locally
		2	Restoration is undercontoured, dentin or base exposed; contact is faulty, not self-correcting; occlusal height reduced, occlusion affected
		3	Restoration is missing partially or totally, fracture of tooth, shows traumatic occlusion; restoration causes pain in tooth or adjacent tissue
Marginal adaptation	0		Restoration is continuous with existing anatomic form; explorer does not catch
	1		Explorer catches; no crevice is visible into which explorer will penetrate
	2		Crevice at margin, enamel exposed
		3	Obvious crevice at margin; dentin or base exposed
		4	Restoration mobile, fractured, or missing
Color match	0		Very good color match
	1		Good color match
	2		Slight mismatch in color, shade, or translucency
		3	Obvious mismatch, outside the normal range
		4	Gross mismatch
Surface roughness	0		Smooth surface
	1		Slightly rough or pitted
	2		Rough, cannot be refinished
		3	Surface deeply pitted, irregular grooves
Marginal discoloration	0		No discoloration evident
	1		Slight staining, can be polished away
	2		Obvious staining cannot be polished away
		3	Gross staining
Caries	0		No evidence of caries contiguous with the margin of the restoration
		1	Caries is evident contiguous with the margin of the restoration
Postoperative sensitivity	0		No sensitivity
	1		Sensitivity lost in one week
		2	Continuous sensitivity

Table 4: *Summaries of USPHS Evaluations Expressed in Percentage at Baseline and up to Final Recall*

Criteria	Baseline		6 months		Final Recall	
	SC, n (%)	NHC, n (%)	SC, n (%)	NHC, n (%)	SC, n (%)	NHC, n (%)
Anatomic form						
0	29 (100)	29 (100)	29 (100)	28 (96.55)	27 (93.1)	26 (89.65)
1	0 (0)	0 (0)	0 (0)	1 (3.45)	2 (6.89)	3 (10.35)
2	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
3	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
Marginal adaptation						
0	29 (100)	29 (100)	28 (96.55)	27 (93.1)	12 (41.38)	24 (82.76)
1	0 (0)	0 (0)	1 (3.45)	2 (6.89)	17 (58.62)	5 (17.24)
2	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
3	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
4	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
Color match						
0	29 (100)	29 (100)	28 (96.55)	29 (100)	27 (93.1)	28 (96.55)
1	0 (0)	0 (0)	1 (3.45)	0 (0)	2 (6.89)	1 (3.45)
2	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
3	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
4	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
Surface roughness						
0	29 (100)	29 (100)	28 (96.55)	28 (96.55)	28 (96.55)	25 (86.21)
1	0 (0)	0 (0)	1 (3.45)	1 (3.45)	1 (3.45)	4 (13.79)
2	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
3	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
Marginal discoloration						
0	29 (100)	29 (100)	28 (96.55)	28 (96.55)	28 (96.55)	26 (89.65)
1	0 (0)	0 (0)	1 (3.45)	1 (3.45)	1 (3.45)	3 (10.35)
2	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
3	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
Secondary caries						
0	29 (100)	29 (100)	29 (100)	29 (100)	29 (100)	29 (100)
1	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
Postoperative sensitivity						
0	28 (96.55)	28 (96.55)	29 (100)	29 (100)	29 (100)	29 (100)
1	1 (3.45)	1 (3.45)	0 (0)	0 (0)	0 (0)	0 (0)
2	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)

difference was marginal adaptation being worse for SC than NHC ($p=0.012$). In total, 17 restorations from the SC and five from the NHC group received a score of 1 (explorer catches). The scores were significantly different between baseline and final recall for the SC group ($p<0.001$) but not for the NHC group ($p>0.05$).

DISCUSSION

Due to their optical and clinical properties, resin composites have become the favored restorative materials for direct posterior restorations, despite some shortcomings. Advances in material formulations, such as advanced filler morphologies and contributions in monomer technology are expected to improve the clinical success of resin composites.[19] In this regard, siloranes have been introduced to dentistry as alternatives to methacrylates due to their hydrophobicity and decreased polymerization shrinkage.[20,21] The goal in developing siloranes was to create a material with reduced polymerization shrinkage and less polymerization stress.[19] Limited clinical studies are available that involve silorane-based materials.[16,17,22]

In our study, we compared the clinical behavior of silorane and a conventional methacrylate-based NHC. Laboratory investigations of silorane have generally revealed that this material exhibits properties at least as good as those of methacrylate-based resin composites.[23-25] Therefore, the results of our clinical investigation may contribute to knowledge about the clinical success of siloranes. Both materials tested showed successful clinical outcomes after 37 months.

A similar clinical study with three years of follow-up indicated no statistically significant difference in any parameter between SC and methacrylate-based composite resins according to the modified USPHS criteria.[22] According to our clinical observations at the final recalls, only the marginal adaptation criteria showed a significant difference. Margin-related problems such as discoloration and chipping of material were usually observed in recalls up to 24 months, which is considered a medium time frame.[26] In this study, the scores were worse for SC (17 restorations) than NHC (five restorations). The worse scores for marginal adaptation could have originated from the degradation of the adhesive interface as a result of slow hydrolysis. Monomers in adhesive systems generally absorb water and chemicals from the oral environment, which eventually affects the adhesion and can result in deleterious effects on marginal adaptation over time.[22,27]

SC had its own adhesive system and was applied according to the manufacturer's instructions. The primer, including HEMA and bis-GMA, was applied directly on dentin tissues and photo-polymerized; the adhesive resin, including TEGDMA, was then applied and photo-polymerized again for 10 seconds. The high content of HEMA and water in the self-etching primer makes the adhesive systems more hydrophilic, increasing the susceptibility of the hybrid layer to water absorption and, consequently, reducing the durability of the adhesion.[28] It has already been shown that HEMA-containing adhesive systems are most prone to hydrolysis, generally resulting in a decline in mechanical properties.[29] However, the application of a hydrophobic bonding resin decreases this permeability and improves bonding stability.[10] In addition, it was found that the mildly acidic (pH=2.7) silorane self-etching primer caused demineralization of the superficial dentin, incorporated the smear layer, chemically bound with calcium in hydroxyapatite,[10] and bonded to tooth tissue more strongly. The worse marginal adaptation score may not be related only to the possible degradation of the adhesive system of the SC over time because the chemical bonding of the adhesive to tooth tissues improved the adhesion of the material.

In a five-year clinical observation, the clinical behavior of three restorative systems were evaluated, including a SC and a methacrylate-based composite combined either with an etch-and-rinse or a self-etch adhesive.[30] Deterioration of the marginal adaptation was observed with all restorative systems, whereas marginal staining was more frequently seen only around the restorations performed with self-etch adhesives for both SC and methacrylate-based composites. It is possible that using the etch-and-rinse technique could have resulted in more reliable clinical performance on the basis of the marginal quality of the restorations. However, Duarte and others[31] concluded in their *in vitro* study that a SC is compatible only with its dedicated adhesive. Thus, the use of well-established different adhesive protocols could not be used with SC. Similarly, in an *in vitro* study, silorane exhibited significantly lower shrinkage forces and better marginal adaptation than did a methacrylate-based composite.[32] The worse observations for SC in our study differed from their results. These findings also could be related to the polishability of SC because for that criterion, one restoration for SC and four for NHC were scored as 1, indicating that the polishability of NHC was not better than that of SC.

Adebayo and others[33] showed lower bond strength with siloranes than with methacrylate-based composites, which also supports our significant differences for the marginal adaptation criteria. Similar to our findings, one clinical trial that examined a methacrylate-based composite resin and silorane in Class II cavities revealed that the marginal adaptation of the silorane was inferior to that of the methacrylate-based resin composite, both occlusally and approximally.[16] On the other hand, one other study found no statistically significant difference in the clinical performance between the restorative materials (nano-hybrid, packable, and silorane) except for marginal adaptation.[34] Silorane showed worse marginal adaptation at the end of their three-year observations. Similar to the results of previous studies,[34,35] significantly worse scores were obtained for the marginal integrity of SC than a methacrylate-based resin composite.

It has also been reported that choosing products from the same manufacturer will favor chemical interaction between the resin composite and the corresponding adhesive system.[36] In this clinical study, NHC (Filtek Z550) was used in combination with the Adper Single Bond 2, products from the

same manufacturer (3M ESPE AG). Five restorations received a score of 1 for the marginal adaptation criteria for NHC, and this result was better than that for SC. In fact, SC was also applied with its own adhesive system from the same manufacturer. Apart from the polishability of the restorations, the type of adhesive system could be related to the success of the marginal adaptation criteria. SC was bonded with a two-step self-etching adhesive, whereas NHC was applied with a two-step etch-and-rinse adhesive. The difference in application procedures of these adhesive systems could affect the marginal durability of these restorations after three years of clinical service.

None of the available clinical studies emphasized the superiority of SC over methacrylate-based resin composites with minimum of six months and maximum of five years evaluation period, despite the excellent performance reported by the manufacturer.[16,17,22,30,34,35,37] We can conclude that both restorative materials tested were clinically acceptable after three years of service. The null hypothesis was rejected due to the statistically significant difference in the criterion of marginal adaptation. Long-term clinical observations are required to fully assess the performance of this material introduced to dentistry with promising expectations.

CONCLUSIONS

From this study, the following could be concluded:

1. Both SC and NHC showed acceptable clinical performance over an evaluation period of three years for posterior Class II restorations when used in conjunction with their corresponding adhesive systems.
2. Significant marginal deterioration was observed for SC restorations compared with NHC at final recall.

Acknowledgement

The authors would like to thank Miss Tugce Yucel for organizing the recalls of the patients attended in the present study.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethical Committee of Istanbul University. The approval code for this study is 2012/644-1047.

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 10 March 2016)

REFERENCES

1. Ferracane JL (2013) Resin-based composite performance: Are there some things we can't predict? *Dental Materials* **29(1)** 51-58.
2. Da Rosa Rodolpho PA, Donassollo TA, Cenci MS, Loguercio AD, Moraes RR, Bronkhorst EM, Opdam NJ, & Demarco FF (2011) 22-year clinical evaluation of the performance of two posterior composites with different filler characteristics *Dental Materials* **27(10)** 955-963.
3. Lempel E, Toth A, Fabian T, Krajczar K, & Szalma J (2014) Retrospective evaluation of posterior direct composite restorations: 10-year findings *Dental Materials* **31(2)** 115-122.
4. Tyas MJ (2005) Placement and replacement of restorations by selected practitioners *Australian Dental Journal* **50(2)** 81-89.
5. Boaro LC, Goncalves F, Guimaraes TC, Ferracane JL, Pfeifer CS, & Braga RR (2013) Sorption, solubility, shrinkage and mechanical properties of "low-shrinkage" commercial resin composites *Dental Materials* **29(4)** 398-404.
6. Arrais CA, Oliveira MT, Mettenburg D, Rueggeberg FA, & Giannini M (2013) Silorane- and high filled-based "low-shrinkage" resin composites: Shrinkage, flexural strength and modulus *Brazilian Oral Research* **27(2)** 97-102.
7. Weinmann W, Thalacker C, & Guggenberger R (2005) Siloranes in dental composites *Dental Materials* **21(1)** 68-74.
8. Eick JD, Smith RE, Pinzino CS, & Kostoryz EL (2006) Stability of silorane dental monomers in aqueous systems *Journal of Dentistry* **34(6)** 405-410.
9. Nanjundasetty JK, Nanda S, Panuganti V, & Marigowda JC (2013) Marginal sealing ability of silorane and methacrylate resin composites in Class II cavities: A scanning electron microscopic study *Journal of Conservative Dentistry* **16(6)** 503-508.
10. Ruschel VC, Baratieri LN, Monteiro Junior S, & Andrada MA (2014) Silorane adhesive system: A case report *Journal of Canadian Dental Association* **80** 4.
11. Ferracane JL (2008) Buonocore lecture. Placing dental composites—A stressful experience *Operative Dentistry* **33(3)** 247-257.
12. Gao BT, Lin H, Zheng G, Xu YX, & Yang JL (2012) Comparison between a silorane-based composite and methacrylate-based composites: Shrinkage characteristics, thermal properties, gel point and vitrification point *Dental Materials Journal* **31(1)** 76-85.
13. Ruiz-de-Castaneda E, Gaton-Hernandez P, Rodriguez EG, Silva RA, Nelson-Filho P, & Silva LA (2013) Pulpal and periapical response after restoration of deep cavities in dogs' teeth with Filtek Silorane and Filtek Supreme XT systems *Operative Dentistry* **38(1)** 73-81.

14. Agrawal VS, Parekh VV, & Shah NC (2012) Comparative evaluation of microleakage of silorane-based composite and nanohybrid composite with or without polyethylene fiber inserts in Class II restorations: An *in vitro* study *Operative Dentistry* **37(5)** 1-7.

15. Krifka S, Federlin M, Hiller KA, & Schmalz G (2012) Microleakage of silorane- and methacrylate-based Class V composite restorations *Clinical Oral Investigations* **16(4)** 1117-1124.

16. Schmidt M, Dige I Kirkevang LL, Vaeth M, & Horsted-Bindslev P (2015) Five-year evaluation of a low-shrinkage silorane resin composite material: A randomized clinical trial *Clinical Oral Investigations* **19(2)** 245-251.

17. Baracco B, Perdigao J, Cabrera E, Giraldez I, & Ceballos L (2012) Clinical evaluation of a low-shrinkage composite in posterior restorations: One-year results *Operative Dentistry* **37(2)** 117-129.

18. van Dijken JW, Kieri C, & Carlén M (1999) Longevity of extensive Class II open-sandwich restorations with a resin-modified glass-ionomer cement *Journal of Dental Research* **78(7)** 1319-1325.

19. Guggenberger R, & Weinmann W (2000) Exploring beyond methacrylates *American Journal of Dentistry* **13(Spec No)** 82D-84D.

20. Palin WM, Fleming GJ, Burke FJ, Marquis PM, & Randall RC (2005) The influence of short and medium-term water immersion on the hydrolytic stability of novel low-shrink dental composites *Dental Materials* **21(9)** 852-863.

21. Maia RR, Reis RS, Moro AF, Perez CR, Pessoa BM, & Dias KR (2015) Properties evaluation of silorane, low-shrinkage, non-flowable and flowable resin-based composites in dentistry *PeerJ* **3** e864 https://peerj.com/articles/864/

22. Mahmoud SH, Ali AK, & Hegazi HA (2014) A three-year prospective randomized study of silorane- and methacrylate-based composite restorative systems in Class II restorations *Journal of Adhesive Dentistry* **16(3)** 285-292.

23. Ernst CP, Meyer GR, Klocker K, & Willershausen B (2004) Determination of polymerization shrinkage stress by means of a photoelastic investigation *Dental Materials* **20(4)** 313-321.

24. Ilie N, & Hickel R (2006) Silorane-based dental composite: Behavior and abilities *Dental Materials Journal* **25(3)** 445-454.

25. Papadogiannis D, Kakaboura A, Palaghias G, & Eliades G (2009) Setting characteristics and cavity adaptation of low-shrinking resin composites *Dental Materials* **25(12)** 1509-1516.

26. 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.

27. Mahmoud SH, & Al-Wakeel Eel S (2011) Marginal adaptation of ormocer-, silorane-, and methacrylate-based composite restorative systems bonded to dentin cavities after water storage *Quintessence International* **42(10)** 131-139.

28. 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.

29. Papadogiannis D, Lakes RS, Papadogiannis Y, & Tolidis K (2013) Mechanical viscoelastic behavior of dental adhesives *Dental Materials* **29(6)** 693-701.

30. Baracco B, Fuentes MV, & Ceballos L (2015) Five-year clinical performance of a silorane- vs a methacrylate-based composite combined with two different adhesive approaches *Clinical Oral Investigations* online September 21, 2015.

31. Duarte S Jr, Phark JH, Varjão FM, & Sadan A (2009) Nanoleakage, ultramorphological characteristics, and microtensile bond strengths of a new low shrinkage composite to dentin after artificial aging *Dental Materials* **25(5)** 589-600.

32. Gregor L, Bortolotto T, Feilzer AJ, & Krejci I (2013) Shrinkage kinetics of a methacrylate- and a silorane-based resin composite: Effect on marginal integrity *Journal of Adhesive Dentistry* **15(3)** 245-250.

33. Adebayo OA, Burrow MF, Tyas MJ, & Palamara J (2012) Effect of tooth surface preparation on the bonding of self-etching primer adhesives *Operative Dentistry* **37(2)** 137-149.

34. Yazici AR, Ustunkol I, Ozgunaltay G, & Dayangac B (2014) Three-year clinical evaluation of different restorative resins in Class I restorations *Operative Dentistry* **39(3)** 248-255.

35. Goncalves FS, Leal CD, Bueno AC, Freitas AB, Moreira AN, & Magalhaes CS (2013) A double-blind randomized clinical trial of a silorane-based resin composite in Class 2 restorations: 18-month follow-up *American Journal of Dentistry* **26(2)** 93-98.

36. D'Alpino PH, de Farias NC, Silva MS, de Goes MF, Gonzalez AH, & Di Hipolito V (2013) Compatibility between silorane adhesive and simplified methacrylate-based adhesive systems *Dental Materials Journal* **32(2)** 263-273.

37. Santos MJ, Kunnilathu A, Steele S, & Santos GC Jr (2014) Clinical evaluation of silorane-based and dimethacrylate-based resin composites: 1-year follow-up *General Dentistry* **62(6)** 6-10.

©Operative Dentistry, 2016, **41-6,** 607-616

Effect of Preparation Depth on the Marginal and Internal Adaptation of Computer-aided Design/Computer-assisted Manufacture Endocrowns

MD Gaintantzopoulou • HM El-Damanhoury

Clinical Relevance

Computer-aided design/computer-assisted manufacture endocrown restorations have a clinically acceptable marginal accuracy; however, increasing the intraradicular extension of the crown may have a reverse effect on the marginal and internal fit of the restorations.

SUMMARY

The aim of the study was to evaluate the effect of preparation depth and intraradicular extension on the marginal and internal adaptation of computer-aided design/computer-assisted manufacture (CAD/CAM) endocrown restorations. Standardized preparations were made in resin endodontic tooth models (Nissin Dental), with an intracoronal preparation depth of 2 mm (group H2), with extra 1- (group H3) or 2-mm (group H4) intraradicular extensions in the root canals (n=12). Vita Enamic polymer-infiltrated ceramic-network material endocrowns were fabricated using the CEREC AC CAD/CAM system and were seated on the prepared teeth. Specimens were evaluated by microtomography. Horizontal and vertical tomographic sections were recorded and reconstructed by using the CTSkan software (TView v1.1, Skyscan).The surface/void volume (S/V) in the region of interest was calculated. Marginal gap (MG), absolute marginal discrepancy (MD), and internal marginal gap were measured at various measuring locations and calculated in microscale (μm). Marginal and internal discrepancy data (μm) were analyzed with nonparametric Kruskal-Wallis analysis of variance by ranks with Dunn's post hoc, whereas S/V data were analyzed by one-way analysis of variance and Bonferroni multiple comparisons (α=0.05). Significant differences were found in MG, MD, and internal gap width values between the groups, with H2 showing the lowest values from all groups. S/V calculations presented significant differences between H2 and the other two groups (H3 and H4) tested, with H2 again showing the lowest values. Increasing the intraradicular extension of endocrown

*Marianna D Gaintantzopoulou, DDS, MSD, PhD, assistant professor, Department of Preventive and Restorative Dentistry, College of Dental Medicine, University of Sharjah, Sharjah, United Arab Emirates

Hatem M El-Damanhoury, BDS, MDS, PhD, assistant professor, Department of Preventive and Restorative Dentistry, College of Dental Medicine, University of Sharjah, Sharjah, United Arab Emirates; and Department of Operative Dentistry, Faculty of Dentistry, Suez Canal University, Ismailia, Egypt

*Corresponding author: M28-135, PO Box 27272, Sharjah, UAE; e-mail: mdimitra@sharjah.ac.ae

DOI: 10.2341/15-146-L

restorations increased the marginal and internal gap of endocrown restorations.

INTRODUCTION

Endocrowns are considered alternative restorations for severely damaged endodontically treated posterior teeth.[1-3] Initially proposed by Pissis,[4] they were then described by Bindl and Mörmann[5] as adhesive restorations consisting of the entire core and crown as a single unit (ie, monobloc). Endocrowns strictly follow the rationale of minimally invasive preparations owing to a decay-oriented concept. They are anchored to the internal portion of the pulp chamber and on the cavity margins, thus obtaining macromechanical retention provided by the axial opposing pulpal walls and micro-retention/chemical bonding attained with the use of adhesive cementation.[6]

The precise dimensions of the central intrapulpal retention cavity are not clearly determined. Especially in cases of excessive loss of tooth structure, where only 1- to 2-mm of intact tooth structure above the dentinoenamel junction is left, the need for further intraradicular extension might be a prerequisite. The utilization of the available space inside the pulp chamber adds to the stability and retention of the restoration.[2] The deeper the pulp cavity and resulting intracoronal extension, the greater the surface area that can be utilized for adhesive retention and transmission of masticatory forces.[6]

Endocrown restoration is made available through computer-aided design/computer-aided manufacturing (CAD/CAM) technology, which provides the possibility for chairside design and fabrication. Despite the advantages of in-office CAD/CAM systems and software, there are limitations in the optical depth of field of the intraoral scanning camera.[7] This may lead to reduced impression capacity of the crown, pulp chamber, and part of the canal, with potential discrepancies in marginal fit and cavity adaptation of the restorations.

Marginal and internal adaptation of indirect restorations are both very important parameters that may affect the periodontal status and longevity of the restorations.[8] Increased marginal discrepancies are related to increased exposure of the luting material to the oral environment, leading to chemomechanical degradation of the cement and the adhesive interface between the tooth structure, luting agent, and esthetic indirect restoration.[9] Internal fit is another key factor related to the long-term stability of esthetic indirect restorations. The cement interface has been described as a crack initiation area.[10,11] Increased interfacial space and resin cement thickness may create increased polymerization shrinkage and interfacial stresses, resulting in decreased strength of the tooth-restoration interface.[10,12] A sufficient three-dimensional (3D) fit of the restoration has been considered mandatory to obtain maximum mechanical support of all-ceramic restorations from the tooth structure.[13] Intrapulpal extension of endocrowns may influence the retention and the adaptation of the restoration.[7] Presently, there are limited data available on the marginal internal adaptation[14] and no information about the effect of the intrapulpal extension on the fit of the endocrown restorations. Therefore, the intent of the present study was to evaluate the effect of cavity preparation depth and intraradicular extension on the marginal and internal fit and of resin-ceramic CAD/CAM endocrown restorations. The null hypothesis assumed that there is no statistically significant difference in marginal and internal fit between the different intraradicular preparation depths.

METHODS AND MATERIALS

Fabrication of the Master Models

Three acrylic resin first mandibular right molars (Endodontic Tooth Model) were used as the master die models for the three tested groups. All endodontic and restorative procedures were done on the same typodont model (model A12A-200, Nissin Dental, Kyoto, Japan), so teeth were positioned in the same position alternately. One experienced operator made all preparations, took the optical impressions, and designed the restorations.

Endodontic Procedures

An access cavity was prepared on the mounted tooth models with a diamond-coated stainless steel bur, and standardized canal enlargement was performed with an engine-driven rotary NiTi system (ProTaper, Dentsply Maillefer, Ballaigues, Switzerland) using a crown-down technique. Root canals were obturated with a thermo-plasticized gutta percha (Calamus Dual, Dentsply Maillefer, Woodinville, WA, USA). The superior aspect of the gutta-percha material was removed to 3 mm below the orifice of each canal, and then flowable resin composite (Filtek Z350XT flowable, 3M ESPE, St. Paul, MN, USA) was used to fill the canals up to the level of the pulp chamber.

Tooth Preparation for Endocrown

The occlusal portion of the three tooth master die models was trimmed using a model trimmer (3/4 HP

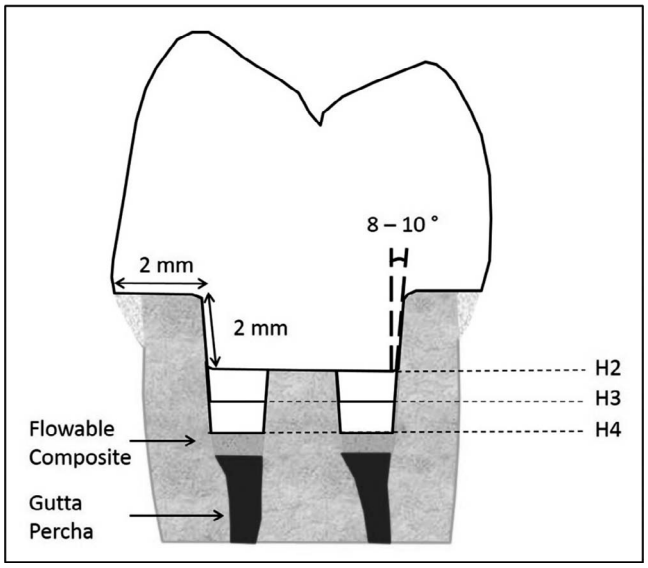


Figure 1. *Diagram of the three types of preparations. Intracoronal height of the preparation was 2.0 mm (group H2). Intacoronal height of 3 mm with an intraradicular extension of 1 mm (group H3) and a 2.0-mm intraradicular extension to reach a total intacoronal height of 4 mm (group H4).*

Wet Model Trimmer, Whip Mix Corporation, Louisville, KY, USA) to reach a standardized height of the internal axial walls of the pulp chamber of approximately 2.0 ± 0.2 mm as measured with a Digital Depth Micrometer (Mitutoyo, Aurora, IL, USA).

Cavity preparations were limited to removal of undercut areas of the pulp chamber and alignment of its internal axial walls with an internal taper of 8-10 degrees using a tapered diamond-coated stainless steel bur with a rounded end (G845KR, Edenta, Au St. Gallen, Switzerland) held perpendicular to the pulpal floor. All internal line angles were rounded and smoothed using the same type of bur. The axial walls were prepared from the pulpal side to provide for a standardized cavity wall thickness of 2.0 ± 0.2 mm measured with a digital caliper (Mitutoyo, Aurora, IL, USA).

In one master die model, the intracoronal height of the preparation was kept at 2.0 mm (group H2). A standardized intraradicular extension of 1 mm was performed in the second master die to reach a total intracoronal height of 3 mm (group H3) and a 2.0-mm intraradicular extension in the third master die to reach a total intracoronal height of 4 mm (group H4; Figure 1). The intraradicular extensions were done in the three canals of each master die using a tapered diamond-coated bur with a round edge (845KR, Edenta).

Endocrown Fabrication

Scanning and designing procedures were repeated for each restoration, resulting in the fabrication of 12 restorations for each group (n=12, according to pilot study results and power analysis). Teeth were air-dried for 10 seconds and evenly covered with antireflection powder (CEREC Optispray, Sirona, Bensheim, Germany). The scanning procedure was done in a standardized way using a digital scanner (CEREC Bluecam, Sirona) with the tip of the scanner always resting at the center of the occlusal surface of the distal adjacent tooth to maintain the same distance between the camera and the prepared tooth models in all the scans. Three scans of the prepared master die were taken for each restoration: one scan with the camera parallel to the occlusal surface and two scans at 30 degrees buccally and lingually to the long axis of the tooth, respectively.

After scanning, contrasting powder was removed using air-water spray for 20 seconds, and each master die sample was evaluated under a stereomicroscope attached to a digital camera (Zeiss, Oberkochen, Germany) for remnants of the contrasting powder, which had to be vigorously washed out by water and dried for complete removal. Endocrowns were designed using a software package (CEREC 3D, version 4.0, Sirona Dental Systems GmbH, Bensheim, Germany) to have similar occlusal anatomy, dimensions, and occluso-gingival height by using the biogeneric reference option in the software. The spacer thickness was set to 0 mm both at the margins and the internal fitting surface of the crowns. Endocrowns were fabricated from polymer-infiltrated ceramic network material (Vita Enamic, Vita Zahnfabrik, Bad Säckingen, Germany) using the CEREC AC system. Each crown was tried for fit on the master models.

Microtomography Testing and Statistics

Adaptation of the restorations was investigated by computerized x-ray microtomography (micro-XCT). A compact desktop system for nondestructive high-resolution x-ray microscopy and microtomography (1072 micro-CT, Skyscan, Kontich, Belgium) was used to obtain a 3D reconstruction of the inner microstucture of the object of interest (endocrown and master die) from 2D x-ray shadow projections. For all three different groups, the master die was positioned precisely within the x-ray beam into a special socket, and the restorations were seated individually, secured in place, checked for fit, and scanned. The scanner operated under the following conditions: W source, 100-KV accelerating voltage,

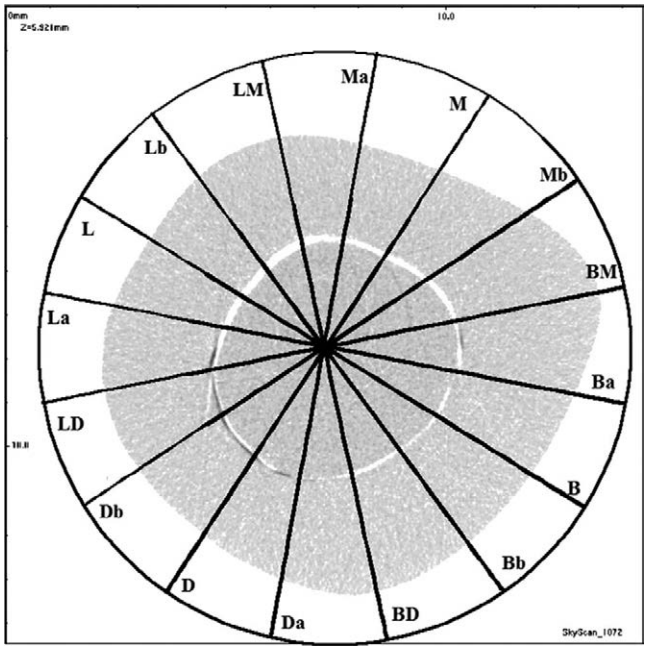


Figure 2. *Circle with eight diameters positioned equally onto a 2D sagittal image.*

98-μA beam current, 14.16-μm pixel size, 180° rotation at 0.45° step, 1.9-second exposure time per step, and 1-mm Al filter. Horizontal tomographic sections were recorded and reconstructed (2D and 3D) by using CT-analyzer software (CTan, Skyscan, Kontich, Belgium).

For the evaluation of the marginal fit, the marginal gap (MG) and the absolute marginal discrepancy (MD) were determined according to the criteria stated by Holmes and others.[15] Measurements of both MG and MD were made at 16 selected landmarks after computed reconstruction (TView version1.1, SkyScan) of eight vertical cuts (jpeg files) of the scanned specimens. For this reason, a circle was drawn with eight diameters equally distributed from each other, at the same image position of every scanned specimen. One diameter of the circle was directed from the middle of the buccal and the lingual surface, whereas another perpendicular to the latter was directed from the middle of the mesial and the distal surface. The center of the circle was placed over the midpoint of the mesio-distal and bucco-lingual diameter (Figure 2). Therefore, the number and the orientation of the slices were standardized for all specimens. Images produced were transferred to Photoshop CS2 digital image editing software (Adobe Systems Incorporated, San Jose, CA, USA) and measurements were performed under the same magnification using digital image editing software (Image J software, National Institute of Health, Bethesda, MD, USA), with a reduction ratio of the given size of the pixel of 14.16 μm (Figure 3a). For each specimen, 16 MG and 16 MD measurements were obtained. All values, either for overextended or underextended margins, were gathered as positive values (Figure 3b). For the analysis of the data, landmarks B, Ba, and Bb, L, La, and Lb, M, Ma, and Mb, and D, Da, and Db (Figure 2) were merged together as Bl, Ll, Ml, and Dl, respectively, due to their symmetric location.

Internal adaptation was calculated by measurements taken from nine internal landmarks on each obtained cross-section: two (C1 and C2) on the cervical seats (C), oriented in the middle of the cervical seat; four (A1, A2, A3, and A4), on the axial walls (A), 400 μm from the axio-cervical and the axio-

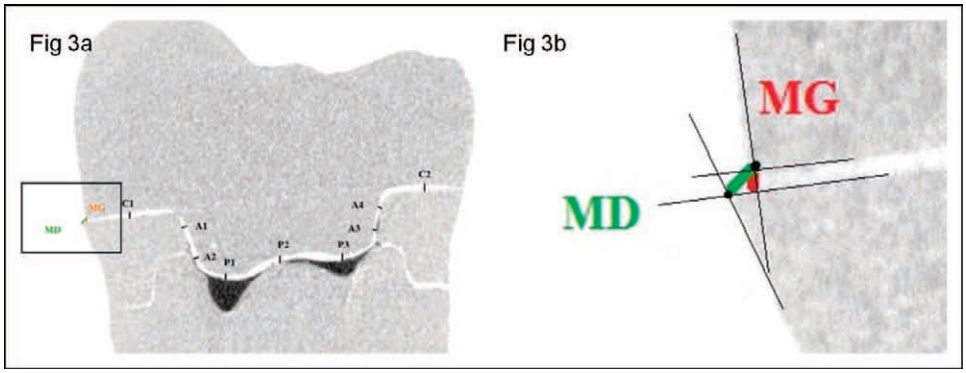


Figure 3. *(a) Vertical cut of a coping on the master die showing the measuring locations: MG; MD; cervical seat area C (C1, C2), at the midpoint of the cervical seat floor; axial wall A (A1, A4), 400 μm pulpal to the axio-cervical line angle; axial wall A (A2, A3), 400 μm occlusal to the axio-pulpal line angle; intrapulpal wall P (P1, P3), the deepest point above the orifice; intrapulpal wall P (P2), the top of the bulkiest intrapulpal floor area. (b) Closer view of the marginal area MD (the distance between contact point of the tangential lines between prepared and unprepared surface of the master die near to the margin and the contact point of the tangential lines between the inner and outer surface of the restoration near to the margin and MG (the distance of the vertical projection of the margin of the die to the inner surface of a hyperextended endocrown or the distance of the vertical projection of the margin of a hypoextended endocrown to the inner prepared surface of the die).*

Table 1: *Mean, SD, and Median Values of the Marginal and Internal Gaps (μm) at the Measured Points*

N	Measuring location	H2				H3				H4			
		Mean (SD)	Median	25%	75%	Mean (SD)	Median	25%	75%	Mean (SD)	Median	25%	75%
192	MG	40.6 (4.1)	40.5^{aA}	28.9	49.3	48.9 (13.8)	43.9^{bA}	29.8	63.4	59.4 (9.6)	56.4^{cA}	43.7	70.5
192	MD	65.9 (12.1)	46.9^{aB}	33.3	80.5	76.2 (12.0)	61.8^{bB}	38.4	97.7	77.6 (5.9)	65.8^{bA}	47.2	97.5
192	C (C1, C2)	150.3 (48.2)	141.5^{aC}	117.6	179.3	169.5 (77.56)	155.2^{aC}	119.0	196.5	161.8 (45.7)	155.1^{aB}	126.9	197.4
384	A (A1, A2, A3, A4)	93.2 (45.7)	89.5^{aD}	59.5	125.9	113.7 (57.9)	104.1^{bD}	70.5	148.3	121.8 (79.7)	103.7^{bC}	79.7	146.2
288	P (P1, P2, P3)	129.6 (55.5)	122.6^{aC}	85.1	169.2	135.5 (59.5)	129.9^{abC}	99.2	162.1	152.3 (65.5)	150.4^{bB}	105.7	182.9

Within a row, the same lowercase superscript letters show mean values with no statistically significant difference (p>0.05). Within a column, the same uppercase superscript letters show mean values with no statistically significant difference (p>0.05).

pulpal lines angles, respectively; and three (P1, P2, and P3) on the intrapulpal wall (P), P1 and P3 oriented on the deepest area of the orifices and P2 oriented in the middle of the bulkiest area of the intrapulpal wall (Figure 3a). In total, 72 measurements were made for each specimen.

The surface/void volume ratio (S/V) in the region of interest (ROI) was calculated using special software (TView, Skyscan). The ROI was set 100 μm from the margin inside the cavity.

Statistical Analysis

Analysis of the data was performed regarding the preparation depth and the measured location. All statistical computations were carried out with the SigmaPlot (ver. 12.3) software package (Systat Software, Inc., San Jose, CA, USA). Saphiro-Wilk's test and Levene's test were performed to verify departures from basic assumptions about variance and normality. All marginal and internal discrepancy data (μm) were analyzed with nonparametric Kruskal-Wallis analysis of variance by ranks with Dunn's post hoc, because normality tests failed. S/V data were analyzed by one-way analysis of variance (ANOVA) and Bonferroni multiple comparisons. A 95% (α=0.05) level of significance was used for all comparisons.

RESULTS

A summary of the results of the descriptive statistical analysis of the marginal and internal discrepancies on the measured areas (MG, MD, C, A, and P) for all the groups is listed in Table 1.

Overall median MG widths of 40.5, 43.9, and 56.4 μm were revealed for H2, H3, and H4 preparation depth groups, respectively, all being significantly different ($p<0.001$). No significant difference was identified within any of the three groups tested in respect to the different groups of landmarks measured (Table 2).

The MD evaluation showed significantly lower ($p<0.001$) median gap values for H2 compared with H3 and H4 (Table 1). No significant difference existed between H3 and H4. Statistically significant differences were revealed in the various landmarks tested ($p=0.007$; Table 3). Within each preparation depth group, Ll, LD, and LM values were lower than the ones measured at the rest of the landmarks, with differences not being significant in all cases.

Endocrowns fabricated for the H2 group presented overextension or underextension of the margin in 30% of the measurements, whereas over- or underextended margins were identified in 26.25% and 25% of the measurements for H3 and H4 groups,

Table 2: *Mean, SD, and Median Values of the MG Evaluated in Different Landmarks*

N	Landmarks (LM)	H2				H3				H4			
		Mean (SD)	Median	25%	75%	Mean (SD)	Median	25%	75%	Mean (SD)	Median	25%	75%
36	Bl (B, Ba, Bb)	42.6 (19.7)	35.2^{A}	25.4	57.5	55.1 (27.3)	49.1^{A}	31.7	84.6	60.3 (22.8)	56.4^{A}	49.3	66.5
36	Ll (L, La, Lb)	38.0 (18.3)	35.2^{A}	28.2	37.8	33.7 (8.7)	29.8^{A}	27.9	42.3	58.4 (43.3)	37.6^{A}	29.3	94.4
36	Ml (M, Ma, Mb)	42.9 (8.6)	38.7^{A}	40.3	49.3	44.8 (19.8)	43.3^{A}	25.4	64.7	80.52 (46.9)	81.9^{A}	35.2	126.1
36	Dl (D, Da, Db)	45.7 (8.3)	47.0^{A}	42.8	49.3	59.3 (21.4)	56.4^{A}	42.3	84.6	69.5 (40.5)	56.4^{A}	47.5	119.6
12	BD	53.2 (11.5)	49.8^{A}	44.6	63.6	62.3 (19.8)	56.6^{A}	45.8	81.7	75.3 (14.9)	75.2^{A}	63.8	86.9
12	LM	36.8 (12.9)	32.9^{A}	26.2	49.3	58.1 (36.7)	57.8^{A}	26.6	89.8	40.7 (9.1)	42.3^{A}	31.7	48.8
12	BM	29.2 (10.2)	23.3^{A}	21.2	40.2	31.1 (22.3)	35.2^{A}	10.4	50.0	59.4 (7.8)	57.2^{A}	53.1	66.9
12	LD	30.6 (7.8)	28.2^{A}	24.6	37.7	52.1 (11.8)	51.7^{A}	41.2	63.0	51.1 (7.7)	56.4^{A}	42.6	56.8

Within a column, the same uppercase superscript letters show mean values with no statistically significant difference (p>0.05).

Table 3: *Mean, SD, and Median Values of the Absolute MD Evaluated in Different Landmarks*

N	Landmark (LM)	H2				H3				H4			
		Mean (SD)	Median	25%	75%	Mean (SD)	Median	25%	75%	Mean (SD)	Median	25%	75%
36	BI (B, Ba, Bb)	42.2 (27.2)	35.2^{A}	25.4	57.5	113.5 (53.5)	99.6^{A}	58.1	159.0	71.9 (31.9)	65.8^{AB}	51.7	77.5
36	LI (L, La, Lb)	58.9 (43.3)	37.6^{A}	29.2	94.5	44.7 (41.9)	33.8^{B}	28.2	42.9	59.9 (39.3)	56.1^{A}	38.7	91.0
36	MI (M, Ma, Mb)	80.5 (46.9)	81.9^{B}	35.2	126.1	55.7 (36.5)	49.3^{AB}	29.9	65.0	91.6 (53.3)	79.7^{AB}	47.9	151.4
36	DI (D, Da, Db)	99.5 (80.5)	73.6^{B}	43.8	191.6	91.3 (49.54)	85.8^{AB}	43.3	146.6	71.9 (33.6)	47.5^{A}	47.0	85.5
12	BD	53.3 (11.5)	49.8^{AB}	44.6	63.6	106.2 (52.1)	92.7^{AB}	58.8	160.2	75.3 (14.9)	75.2^{AB}	63.8	86.9
12	LM	56.5 (41.3)	42.3^{AB}	28.2	91.8	66.6 (36.3)	63.4^{AB}	43.4	98.2	40.7 (9.0)	42.3^{A}	31.7	48.8
12	BM	63.2 (34.1)	73.0^{B}	29.4	92	75.9 (6.6)	75.9^{AB}	20.9	159.6	148.8 (35.7)	134.6^{B}	126.3	178.3
12	LD	30.6 (7.8)	28.2^{A}	24.6	37.7	52.1 (11.8)	51.7^{AB}	41.2	63.0	60.2 (16.7)	56.4^{AB}	49.6	72.7

Within a column, the same uppercase superscript letters show mean values with no statistically significant difference ($p > 0.05$*).*

respectively (Table 4). Comparison of MG and MD values in each group revealed a significant difference in groups H2 and H3, with MD values being higher in both cases ($p<0.001$).

In the evaluation of internal gaps (Figure 3a), for the cervical seat measuring points (C1 and C2), there was no significant difference between the three tested groups ($p=0.258$). At the axial measuring locations (A1, A2, A3, and A4), group H2 exhibited significantly lower median gap width values compared with the other two groups H3 and H4 ($p<0.001$). The intrapulpal gap width measured at three points (P1, P2, and P3) was significantly wider for H4 compared with H2 ($p=0.011$). No significant difference was identified among groups H2 and H3 and among H3 and H4, respectively. Significant differences existed between the various measured positions within all groups, with median values measured in axial walls (A) being significantly lower than the ones measured in cervical seat (C) and the intrapulpal floor (P), but significantly higher than the gap width values measured at the marginal area (MG and MD), in all preparation groups ($p<0.001$; Table 1).

S/V calculation (Table 5) showed significant differences among the three groups. H4 showed higher mean values (46.99 ± 2.01) than H2 (39.33 ± 3.83) but not significantly different than H3 (44.69 ± 2.89; $p=0.005$).

Table 4: *Percentages of Over- and Underextended Restorations at the Marginal Area in Different Preparation Depths Groups*

Preparation type	N	Overextension, %	Underextension, %	Equal, %
H2	160	12.5	17.5	70
H3	160	13.75	12.5	73.75
H4	160	6.25	18.75	75

DISCUSSION

The aim of this study was to evaluate the marginal and internal fit of endocrown restorations with different intraradicular extension using micro-XCT. The null hypothesis assuming that there would be no significant difference between the groups tested should be rejected.

In the present study, micro-XCT, a nondestructive evaluation method, was used to investigate the marginal and internal fit of the endocrowns. In the literature, a variety of qualitative and quantitative and destructive and nondestructive methods for evaluating the marginal and the internal fit have been applied. Micro-XCT is an alternative technique for 3D evaluation of precementation space due to the ability to acquire 3D relationships between structures of different coefficients of absorption without sample sectioning or chemical fixation. It allows 2D and 3D investigation of the MG, the MD, and the internal fit within the range of a few micrometers at multiple sites and directions.[16-18] Disadvantages of the technique are radiation artifacts and the low capacity of discrimination in cases of insufficient radiographic contrast.[19,20] In the current study, to improve the contrast between the acrylic die and the resin-ceramic endocrowns, the evaluation procedure was done without cementation. Moreover, all restorations of each group were seated on the same

Table 5: *Mean, SD, and Median Values of the S/V Void Ratio*

Preparation type	S/V	
	N	Mean (SD)
H2	12	39.33 (3.83) ^{A}
H3	12	44.69 (2.89) ^{B}
H4	12	46.99 (2.01) ^{B}

Within a column, the same uppercase superscript letters show mean values with no statistically significant difference ($p > 0.05$*).*

prepared die, keeping the conditions of the samples more standardized. Previous studies have concluded that micro-XCT can be a reliable method for further evaluation of the marginal and internal fit of indirect restorations.[16,18,20,21]

Marginal fit or misfit is defined as a combination of gap and extension errors. The MD is an angular combination of the MG and the over- or under-extension errors.[15] Therefore, in the present study, micro-XCT was used to evaluate the MG and the MD as well.

In this study, significant differences among the groups were identified in MG and MD values. Increase of preparation and intraradicullar extension of 1 and 2 mm in groups H3 and H4, respectively, inversely affected marginal fit of the restorations. There is much controversy in the literature regarding the clinical acceptable MG width. Many authors agree that marginal openings below 120 μm are clinically acceptable,[22-24] whereas a maximum gap width of 100 μm is advocated by other researchers.[25] In the present study, mean and median MG values for all tested groups could be considered within the clinically acceptable range.

Many researchers have criticized the marginal and internal fit of CAD/CAM restorations.[20,26] Continuing developments and improvements in CAD/CAM technology have led to marginal adaptation competing with that of the laboratory-fabricated restorations.[27] The majority of the studies evaluating marginal or internal adaptation of esthetic restorations fabricated from the CAD/CAM systems have focused on onlay or crown restorations made of composite or various all-ceramic systems. Therefore, no direct comparisons of the present results can be made. Moreover, comparisons with studies reporting 2D measurements made from physical sectioning should be made with caution. A wide range of marginal gaps has been reported for CAD/CAM-fabricated esthetic restorations. The type of the preparation design,[28,29] the impression method, the CAD/CAM system and the restorative material,[30] and the testing method seem to have an effect on the marginal and internal adaptation of CAD/CAM-fabricated indirect restorations. There are only limited data evaluating marginal and internal adaptation of endocrown restorations. Cook and Fasbinder[14] evaluated the marginal and internal adaptation of chairside CAD/CAM IPS Empress endocrowns on premolars, using different preparation designs at the marginal area and the internal line angles. MG width ranged from 49 to 102 μm, which is in accordance to the results of the present study, whereas the internal gap widths ranged from 139 to 229 μm, which is higher than those reported here (89.5-155.2 μm).[14] The use of premolars instead of molars, a different type of restorative material, and different testing and measuring techniques between the two studies do not allow for direct comparisons. The reported mean MGs for CEREC I intracoronal restorations range from 191 to 308 μm,[31,32] whereas for CEREC 2 intracoronal restorations, the values varied from 59 to 121 μm.[31-33] A mean MG range of 46-162 μm has been reported for all ceramic and composite crowns fabricated with CEREC 3, whereas an MG width of 201 ± 78 μm and up to 116 μm have been shown for Cerec3 and Cerec 3D onlay restorations, respectively.[25,28,34-36]

The effect of measuring location on the MG and MD was evaluated in the present study. Differences of median MG and MD values in the evaluated landmarks were identified as significant only in a few cases. Further measurements with additional samples might be required to draw a more representative conclusion. In the literature, a wide range of marginal space has been described at different measured locations of various all-ceramic system restorations.[24,37] In a recent clinical study, significantly larger MGs were observed at the distal location of CAD/CAM-fabricated metal-ceramic crowns in conjunction with intraoral digital impressions.[36] The lingual site had a significantly lower marginal discrepancy than the mesial, distal, and buccal sites in some other laboratory investigations.[36]

Internal fit has been advocated as an important factor in the retention and favorable clinical performance of indirect restorations. High bond strength has been achieved in conjunction with cement thickness of 50-100 μm, but when MG was raised to 150 μm or more, a significantly higher washout of the cement was shown.[26] Cement space should be uniform and facilitate seating without compromising resistance or retention forms.[38]

In the present study, S/V void ratio was calculated instead of evaluating the absolute volume void to compensate for the 1 mm in depth added intraradicularly in each preparation type tested. According to the results, H2 showed significantly lower S/V than the other two groups, which did not differ significantly. In addition to S/V ratio, internal fit was assessed by evaluating the gap width in 72 measuring locations per tooth. In all groups tested, median values of gap measured at the axial wall (A) were significantly lower than the pulpal (P) and cervical floor seat (C) area. Compared with marginal

discrepancy widths (MG and MD), internal gap values (A, P, and C) were significantly higher for all three tested groups. Higher internal gap than marginal gap values have been reported in studies evaluating marginal and internal fit of CAD/CAM-fabricated full coverage or onlay esthetic restorations.[24,28,35,39,40] In most cases, internal gap on the occlusal or pulpal region was presented as higher than on other regions. Kokubo and others[24] attributed this difference to the scanning process, preparation height, luting space, convergence angle, and variations between CAD/CAM systems.

Scanning process, software design, milling, and shrinkage effects have an influence on the fitting accuracy of CAD/CAM restoration. Despite the significant improvements in CAD/CAM technology systems, there might be some clinical problems.[20,41] Optical contrasting powder application procedure, camera misalignment, distance between the camera and the scanned surface, and imaging walls with different orientation in relation to the camera are all possible sources of accuracy problems and dimensional errors.[42.43]

Although the manufacturer information states that the CEREC Bluecam has a depth of field up to 15 mm (CEREC AC Operating Manual, Sirona), which is sufficient to capture deep preparations, increasing the preparation depth in the current study negatively affected the accuracy of the captured image. This effect was clearly manifested in H4 group, as internal gap values increased significantly on the axial and pulpal walls (A and P) that were moved further away from the prism, but not on the cervical seats (C1 and C2), which remained at the same distance from the camera. In addition to this, the increased thickness of the scanning powder in the deepest areas of the canals and the corners could have become a negative input for the optical scans.[44]

Questions regarding the efficacy of the systems to accurately scan in depth and record the pulp chamber and part of the canals in endocrown preparations have been raised.[45] Limited optical depth of field may result in a blurred image of the central retention cavity of the endodontic preparation if the adjacent teeth limit the position of the camera head.[1,42,45]

This *in vitro* study simulates the compromised situation of extensive loss of tooth structure, which does not readily allow for the use of the ferrule effect in crown preparation. CAD/CAM-fabricated endocrowns are relatively new treatment modalities, and information about their long-term clinical performance is limited in the literature. High survival rates for Cerec endocrowns on molars were reported in two different studies after 5 and 12 years of evaluation (90.5%), with no significant difference compared with classic shoulder crowns. Both studies reported higher risk of failure for endocrowns fabricated on premolar teeth.[46,47]

Limitations of the present study include the use of one CAD/CAM system and one restorative material and the spray used for digitizing the specimens. The use of other systems or materials might have resulted in different outcomes. Further research is proposed to investigate the marginal and cavity adaptation of endocrowns and the effect of intraradicular extension on the retention of endocrowns, especially in cases of excessive tooth loss, and to determine whether the results of the current study have an effect on the long-term clinical performance of the restorations.

CONCLUSIONS

Within the limitations of the present *in vitro* study, the following conclusions can be drawn:

1. Intraradicular extension of the endocrown preparation negatively affected both the marginal adaptation and the internal fit of the final restoration.
2. Marginal fit of the three groups tested proved to be significantly better than internal fit evaluated by analyzing the internal gap width in various measuring positions.

Acknowledgments

The authors are grateful to the College of Graduate Studies & Research, University of Sharjah, UAE for funding the project and to Mr Petros Tsakiridis (technical assistant, Department of Biomaterials, National and Kapodistrian University of Athens, Greece) for his assistance in the m-XCT evaluation of the samples.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the College of Dental Medicine, University of Sharjah, UAE.

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 6 October 2015)

REFERENCES

1. Chang CY, Kuo JS, Lin YS, & Chang YH (2009) Fracture resistance and failure modes of CEREC endo-crowns and conventional post and core-supported CEREC crowns *Journal of Dental Sciences* **4(3)** 110-117.
2. Biacchi GR, & Basting RT (2012) Comparison of fracture strength of endocrowns and glass fiber post-retained conventional crowns *Operative Dentistry* **37(2)** 130-136.
3. El-Damanhoury H, Haj-Ali R, & Platt J (2015) Fracture resistance and microleakage of endocrowns utilizing three CAD-CAM blocks *Operative Dentistry* **40(2)** 201-210.
4. Pissis P (1995) Fabrication of a metal-free ceramic restoration utilizing the monobloc technique *Practical Periodontics & Aesthetic Dentistry* **7(5)** 83-94.
5. Bindl A, & Mörmann WH (1999) Clinical evaluation of adhesively placed Cerec endocrowns after 2 years—Preliminary results *Journal of Adhesive Dentistry* **1(3)** 255-265.
6. Mörmann WH, Bindl A, Lüthy H, & Rathke A (1998) Effect of preparation and luting system on all-ceramic computer-generated crowns *International Journal of Prosthodontics* **11(4)** 333-339.
7. Mou SH1, Chai T, Wang JS, & Shiau YY (2002) Influence of different convergence angles and tooth preparation heights on the internal adaptation of Cerec crowns *Journal of Prosthetic Dentistry* **87(3)** 248-255.
8. Felton DA, Kanoy BE, Bayne SC, & Wirthman GP (1991) Effect of in vivo crown margin discrepancies on periodontal health *Journal of Prosthetic Dentistry* **65(3)** 357-364.
9. Laurent M, Scheer P, Dejou J, & Laborde G (2008) Clinical evaluation of the marginal fit of cast crowns—Validation of the silicone replica method *Journal of Oral Rehabilitation* **35(2)** 116-122.
10. Federlin M, Krifka S, Herpich M, Hiller KA, & Schmalz G (2007) Partial ceramic crowns: Influence of ceramic thickness, preparation design and luting material on fracture resistance and marginal integrity in vitro *Operative Dentistry* **32(3)** 251-260.
11. Zhang Y, Kim JW, Bhowmick S, Thompson VP, & Rekow ED (2009) Competition of fracture mechanisms in monolithic dental ceramics: Flat model systems *Journal of Biomedical Materials Research Part B Applied Biomaterials* **88(2)** 402-411.
12. Tuntiprawon M, & Wilson PR (1995) The effect of cement thickness on the fracture strength of all-ceramic crowns *Australian Dental Journal* **40(1)** 17-21.
13. Karakaya S, Sengun A, & Ozer F (2005) Evaluation of internal adaptation in ceramic and composite resin inlays by silicon replica technique *Journal of Oral Rehabilitation* **32(6)** 448-453.
14. Cook KT, & Fasbinder DJ (2012) Marginal and internal adaptation of CAD/CAM endocrown preparation designs. *Journal of Dental Research* **91(Spec. Iss A)** 1022.
15. Holmes JR, Bayne SC, Holland GA, & Sulik WD (1989) Considerations in measurement of marginal fit. *Journal of Prosthetic Dentistry* **62(4)** 405-408.
16. Pelekanos S, Koumanou M, Koutayas SO, Zinelis S, & Eliades G (2009) Micro-CT evaluation of the marginal fit of different In-Ceram alumina copings. *European Journal of Esthetic Dentistry* **4(3)** 278-292.
17. Seo D, Yi Y, & Roh B (2009) The effect of preparation designs on the marginal and internal gaps in Cerec3 partial ceramic crowns. *Journal of Dentistry* **37(5)** 374-282.
18. Borba M, Cesar PF, Griggs JA, & Della Bona Á (2011) Adaptation of all-ceramic fixed partial dentures *Dental Materials* **27(11)** 1119-1126.
19. SkyScan (1998-2001) *Desktop X-Ray Microtomograph. Instruction Manual: Skyscan, Version 3 for 1072 Scanners 4NT and 5NT* SkyScan Aartselaar, Belgium.
20. Mously HA, Finkelman M, Zandparsa R, & Hirayama H (2014) Marginal and internal adaptation of ceramic crown restorations fabricated with CAD/CAM technology and the heat-press technique *Journal of Prosthetic Dentistry* **112(2)**:249-256.
21. Rungruanganunt P, Kelly JR, & Adams DJ (2010) Two imaging techniques for 3D quantification of pre-cementation space for CAD/CAM crowns *Journal of Dentistry* **38(12)** 995-1000.
22. McLean JW, & von Fraunhofer JA (1971) The estimation of cement film thickness by an in vivo technique *British Dental Journal* **131(3)** 107-111.
23. Beuer F, Naumann M, Gernet W, & Sorensen JA (2009) Precision of fit: Zirconia three-unit fixed dental prostheses *Clinical Oral Investigations* **13(3)** 343-349.
24. Kokubo Y, Nagayama Y, Tsumita M, Ohkubo C, Fukushima S, & Vult von Steyern P (2005) Clinical marginal and internal gaps of In-Ceram crowns fabricated using the GN-I system *Journal of Oral Rehabilitation* **32(10)** 753-758.
25. Reich S, Gozdowski S, Trentzsch L, Frankenberger R, & Lohbauer U (2008) Marginal fit of heat-pressed vs. CAD/CAM processed all-ceramic onlays using a milling unit prototype. *Operative Dentistry* **33(6)** 644-650.
26. Bayne SC, & Heymann HO (1996) CAD/CAM in dentistry: Present and future applications. *Quintessence International (Current Concepts)* **27(6)** 431-433.
27. Ng J, Ruse D, & Wyatt C (2014) A comparison of the marginal fit of crowns fabricated with digital and conventional methods *Journal of Prosthetic Dentistry* **112(3)** 555-560.
28. Akbar JH, Petrie CS, Walker MP, Williams K, & Eick JD (2006) Marginal adaptation of Cerec 3 CAD/CAM composite crowns using two different finish line preparation designs *Journal of Prosthodontics* **15(3)** 155-163.
29. Souza RO, Özcan M, Pavanelli CA, Buso L, Lombardo GH, Michida SM, Mesquita AM, & Bottino MA (2012) Marginal and internal discrepancies related to margin design of ceramic crowns fabricated by a CAD/CAM system *Journal of Prosthodontics* **21(2)** 94-100.
30. Ramírez-Sebastià A, Bortolotto T, Roig M, & Krejci I (2013) Composite vs ceramic computer-aided design/computer-assisted manufacturing crowns in endodonti-

cally treated teeth: Analysis of marginal adaptation *Operative Dentistry* **38(6)** 663-673.

31. Sturdevant JR, Bayne SC, & Heymann HO (1999) Margin gap size of ceramic inlays using second-generation CAD/CAM equipment *Journal of Esthetic Dentistry* **11(4)** 206-214.
32. 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.
33. Sato K, Matsumura H, & Atsuta M (2002) Relation between cavity design and marginal adaptation in a machine-milled ceramic restorative system *Journal of Oral Rehabilitation* **29(1)** 24-27.
34. Nakamura TL, Dei N, Kojima T, & Wakabayashi K (2003) Marginal and internal fit of Cerec 3 CAD/CAM all-ceramic crowns *International Journal of Prosthodontics* **16(3)** 244-248.
35. Tsitrou EA, Northeast SE, & van Noort R (2007) Evaluation of the marginal fit of three margin designs of resin composite crowns using CAD/CAM *Journal of Dentistry* **35(1)** 68-73.
36. da Costa JB, Pelogia F, Hagedorn B, & Ferracane JL (2010) Evaluation of different methods of optical impression making on the marginal gap of onlays created with Cerec 3D *Operative Dentistry* **35(3)** 324-329.
37. Tamim H, Skjerven H, Ekfeldt A, & Rønold HJ (2014) Clinical evaluation of CAD/CAM metal-ceramic posterior crowns fabricated from intraoral digital impressions *International Journal of Prosthodontics* **27(4)** 331-337.
38. May KB, Russell MM, Razzoog ME, & Lang BR (1998) Precision of fit: The Procera AllCeram crown *Journal of Prosthetic Dentistry* **80(4)** 394-404.
39. Schaefer O, Watts DC, Sigusch BW, Kuepper H, & Guentsch A (2012) Marginal and internal fit of pressed lithium disilicate partial crowns in vitro: A three-dimensional analysis of accuracy and reproducibility *Dental Materials* **28(3)** 320-326.
40. Tidehag P, Ottosson K, & Sjögren G (2014) Accuracy of ceramic restorations made using an in-office optical scanning technique: an in vitro study *Operative Dentistry* **39(3)**:308-316.
41. Anadioti E, Aquilino SA, Gratton DG, Holloway JA, Denry IL, Thomas GW, & Qian F (2015) Internal fit of pressed and computer-aided design/computer-aided manufacturing ceramic crowns made from digital and conventional impressions *Journal of Prosthetic Dentistry* **113(4)** 304-309.
42. Parsell DE, Anderson BC, Livingston HM, Rudd JI, & Tankersley JD (2000) Effect of camera angulation on adaptation of CAD/CAM restorations *Journal of Esthetic Dentistry* **12(2)**:78-84.
43. Todorovic A, Lisjak D, Lazic V, & Spadijer-Gostovic A (2010) Possible errors during the optical impression procedure *Serbian Dental Journal* **57(1)** 30-34.
44. Lehmann KM, Azar MS, Kämmerer PW, Wentaschek S, Ernst NF, Hell ENF, & Scheller E (2011) The effect of optical conditioning of preparations with scan spray on preparation form *Acta Stomatol Croat* 2011;**45(2)** 86-92.
45. Mörmann WH, & Bindl A (2002) All-ceramic, chair-side computer-aided design/computer-aided machining restorations *Dental Clinics of North America* **46(2)** 405-426.
46. Bindl AL, Richter B, Mörmann WH (2005) Survival of ceramic computer-aided design/manufacturing crowns bonded to preparations with reduced macroretention geometry *International Journal of Prosthodontics* **18(3)** 219-224.
47. Otto T, & Mörmann WH (2015) Clinical performance of chairside CAD/CAM feldspathic ceramic posterior shoulder crowns and endocrowns up to 12 years *International Journal of Computerized Dentistry* **18(2)** 147-161.

©Operative Dentistry, 2016, **41-6,** 617-626

Surface Properties and Color Stability of Resin-Infiltrated Enamel Lesions

X Zhao • Y-F Ren

Clinical Relevance

Resin infiltration increases surface hardness of white spot lesions and remains stable under thermocycling challenges, but its surface polish and color stability may be of concern when used in the esthetic zone.

SUMMARY

Objectives: To examine the surface topographies, microhardness, and color stability of resin-infiltrated enamel lesions before and after aging challenges *in vitro* using three-dimensional laser scanning profilometry, surface microhardness testing, spectrophotometry, and scanning electron microscopy.

Methods: Forty human third molars were embedded in epoxy resin, and each tooth was prepared to have two white spot lesions and one sound enamel area. One white spot lesion received resin infiltration and the other was untreated. Ten specimens were subjected to thermocycling for 10,000 cycles, 10 specimens were immersed in coffee solutions, and 10 specimens were placed in water storage. Surface area roughness (Sa), Vickers microhardness (VHN), and CIE L*a*b* color values were measured on sound enamel, resin-infiltrated lesions, and untreated lesions before and after aging. The surface morphology of resin-infiltrated lesions was observed after aging under scanning electron microscopy and compared with 10 specimens that were not subjected to aging challenge.

Results: Resin infiltration increased the surface microhardness of the enamel lesions from 89.3 to 212.0 VHN. The surface microhardness of resin-infiltrated enamel lesions was not significantly affected by aging. The surface roughness of resin-infiltrated lesions (0.32–0.37 μm) was greater than that of sound enamel (0.05–0.06 μm) and untreated lesions (0.12–0.13 μm). Thermocycling and water storage further increased surface roughness of resin-infiltrated surfaces. Resin-infiltrated enamel lesions showed greater discoloration than sound enamel surfaces. Surface microfissures and microcracks were observed on resin-infiltrated enamel lesions after thermocycling.

Conclusions: Surface hardness of enamel lesions increased significantly after resin infiltration and remained stable following thermocycling. Surface roughness and color

Xiaoyi Zhao, DDS, PhD, Department of General Dentistry, Peking University School of Stomatology, Beijing, China; and Department of General Dentistry, University of Rochester Eastman Institute for Oral Health, Rochester, NY, USA

*Yan-Fang Ren, DDS, PhD, MPH, Department of General Dentistry, University of Rochester Eastman Institute for Oral Health, Rochester, NY, USA.

*Corresponding author: 625 Elmwood Ave, Rochester, NY 14620; e-mail: Yanfang_ren@urmc.rochester.edu

DOI: 10.2341/15-319-L

stability of resin-infiltrated enamel lesions were less than ideal and might further deteriorate after aging in the oral environment.

INTRODUCTION

Although frequent application of fluoride is often recommended as the treatment of choice for initial enamel caries on smooth or proximal surfaces, the effectiveness of this approach depends strongly on the patient's oral hygiene practice and is therefore not suitable for noncompliant patients. An alternative therapy to arrest initial caries lesions is infiltration of the pores and microspaces within enamel lesions using a low-viscosity liquid resin.[1] It has been shown that artificial and natural caries lesions on smooth, interproximal, and occlusal surfaces can be successfully infiltrated using this microinvasive technique.[2-4]

In comparison to fluoride therapy, resin infiltration was found to result in greater surface hardness of enamel carious lesions.[5] Resin infiltration was effective, at least in the short-term, in arresting both the smooth surface and interproximal surface enamel lesions in randomized and controlled clinical trials,[6-9] and the procedure was well received by both the practitioners and the patients.[10] As this technique is relatively new, there is a lack of data on its long-term outcomes. In a recent study that followed 45 patients for a period of 12 months, the infiltrated surface showed excellent marginal adaptation but significant discoloration.[11] This finding was further substantiated by an experiment *in vitro*, which found that infiltration resin had the highest staining susceptibility as compared with several resin-based dental bonding and adhesive materials.[12]

As smooth surface white spot lesions are often present in the esthetic zone, color stability of resin-infiltrated surfaces is an important determinant for long-term success. It is generally accepted that all resin-based dental materials degrade to some extent in the oral environment. Water sorption and surface degradation are considered factors associated with discoloration of resin-based dental materials.[13,14] In addition, polishability and surface roughness contribute significantly to color stability and discoloration of this type of material.[15-17]

Although discoloration of infiltration resin has been reported in the aforementioned studies *in vivo* and *in vitro*, the mechanism underlying the color change remains unclear. In contrast to conventional resin-based restorative materials, currently available infiltration resin is composed mainly of hydrophilic triethyleneglycol-dimethacrylate (TEGDMA). Infiltration resin has two major differences from the other resin-based materials: it is an unfilled liquid resin composed of mostly TEGDMA and it does not have a polishing step after its application per the manufacturer's instructions. TEGDMA is important for maintaining the extremely low viscosity that allows penetration of the resin to the demineralized lesion. But it is well known that TEGDMA has a high water sorption rate and is prone to discoloration,[18,19] and a nonpolished surface may mean a rougher surface.[20] Aging challenges under thermal stress might further affect the physical property and color stability of resin-infiltrated enamel surfaces and compromise the long-term outcomes. The aim of the present study was to evaluate the influence of aging challenge on the physical properties and color stability of resin-infiltrated enamel surfaces in vitro.

METHODS AND MATERIALS

A total of 40 freshly extracted permanent third molars were collected from oral surgery and general dentistry clinics following ethic guidelines from the authors' institution. The teeth were cleaned from soft tissues and stored in a refrigerator in 0.1% thymol solution for no more than two months before use.

Sample Preparation

The teeth were sectioned at the cemento-enamel junction using a high-speed handpiece (TF12, Mani, Inc., Tochigi, Japan) with water coolant. The crowns were placed in cylindrical plastic molds (20 mm in diameter and 20 mm in height) with cusps facing down on a flat surface and embedded in epoxy resin. The occlusal surfaces of the embedded crowns were ground flat with 400-grit waterproof SiC paper (Softflex, Matador GmbH, Remscheid, Germany) under water cooling until at least three flat areas of the enamel, each measuring at least 2 mm $\times$ 2 mm, were exposed. The exposed enamel areas were typically the two buccal cusps and one lingual cusp of the molar crown (Figure 1a,b). Exposed enamel surfaces were then polished in sequence with 800-, 1200-, 2400-, and 4000-grit waterproof SiC paper using running tap water as a coolant. The prepared specimens were examined under a stereomicroscope to verify that the enamel surfaces were exposed, with absence of cracks or other surface defects. After preparation, the specimens were stored in 0.1% thymol solution to avoid dehydration.

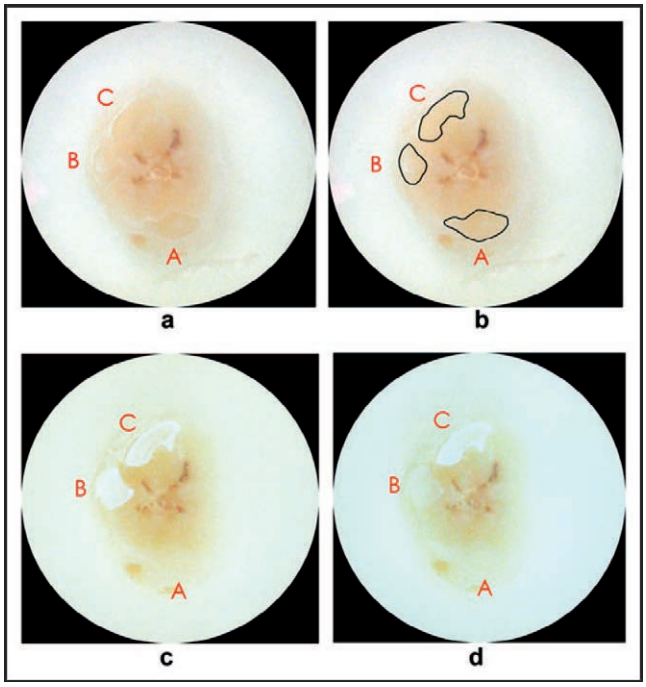


Figure 1. *Sample preparation. (a): Occlusal surface of human third molars were embedded in epoxy resin and ground flat to expose three areas (A, B, and C) of at least 2 mm × 2 mm enamel. (b): The exposed enamel areas were typically the two buccal cusps and one lingual cusp of the molar crown. (c): Two separate white spot lesions (A and B) were created. (d): One white spot lesion B was treated with the ICON infiltration resin and one lesion was untreated.*

Artificial Enamel Caries Lesions

Of the three exposed enamel surface areas, one area was covered with acid-resistant nail polish to serve as a sound enamel control. The other two areas were left exposed. Artificial lesions were created within the two exposed enamel areas by immersing each tooth into a 50-mL aliquot of a Ca/PO_4/acetate solution containing 2.0 mmol/L calcium, 2.0 mmol/L phosphate, and 0.075 mol/L acetate maintained at pH 4.5 and a temperature of 37°C for 48 hours. The artificial white spot lesions created presented depths between 100 and 150 μm and exhibited optical properties of early-stage caries lesions.[21]

The artificial caries model prepared this way has two separate artificial white spot lesions on the same specimen and one sound enamel area as internal control (Figure 1c). The white spot lesion was identified as an opaque and chalky white area on the enamel surface in contrast to the semi-translucent sound enamel.[22] Each specimen was inspected visually to ensure that the white spot lesions were successfully created on the enamel surfaces.

Resin Infiltration of Enamel Surface Lesions

Of the two lesions on the enamel surface of the specimen, one was randomly chosen to receive the ICON (ICON DMG, Hamburg, Germany) resin infiltration treatment, while the other served as the control. A computerized simple randomization scheme was used to select the lesion for treatment (see Supplementary Materials). The resin infiltration treatment followed a protocol described in detail in a previous report (Figure 1d).[23] Briefly, the selected lesion was etched with 37% phosphoric acid gel (Gluma Etch 35 Gel, Heraeus Kulzer GmbH, Hanau, Germany) for five seconds, rinsed with air-water spray for 30 seconds, and air dried for 10 seconds. Pure ethanol was then applied to the lesion surface for 10 seconds, followed by air drying for another 10 seconds to render surface desiccation. The ICON infiltration resin was applied to the enamel caries lesions for three minutes, and resin excess was removed with a cotton roll. The resin-infiltrated surface was then light-cured for 40 seconds. The infiltration resin was applied for a second time as above for one minute and light cured for another 40 seconds. After light curing, the resin surface was polished with 4000-grit aluminum oxide abrasive paper for 20 seconds.

Thermocycling, Staining Challenge, and Water Storage

Following resin infiltration treatments, the specimens were randomly divided into the following four groups using a random list generator: 10 specimens for thermocylcing challenges, 10 specimens for staining challenges in coffee at 37°C, 10 specimens for water storage at 37°C as a control, and 10 specimens for scanning electron microscopy (SEM) to obtain baseline surface morphology data on resin-infiltrated enamel lesions.

For the thermocycling group, the specimens were placed in a thermocycling machine programmed to perform 10,000 cycles in 180 hours (7 days 12 hours) between two water baths at temperatures of 5°C and 55°C, respectively, with a dwell time of 30 seconds at each bath temperature.

For the staining challenge group, the specimens were immersed in coffee solution prepared with 75 g instant coffee (Nescafé, Nestlé, Vevey, Switzerland) in 750 mL boiling water and stored at 37°C for 180 hours. The coffee solution was refreshed every day for seven days.

For the water storage group, the specimens were stored in distilled water at 37°C for 180 hours, the

same duration as the thermocycling and the staining challenge group.

3D Laser Scanning Microscopy

Specimens in the thermocycling and water storage group were subjected to surface roughness testing using a three-dimensional (3D) laser scanning microscope (VK-X100/X200, Keyence, Osaka, Japan) at 3000× magnification before and after the aging challenges. For each area on each specimen, three different locations (95.7993 × 71.8495 μm^2 in size) were randomly chosen and scanned for 3D surface area profiling. Surface roughness was measured in average arithmetic roughness (Sa) values. The mean values of the three measurements for each area were used as the Sa value.

Surface Microhardness Testing

Surface microhardness of sound enamel, resin-infiltrated lesions, and untreated lesions on each specimen in the thermocycling and water storage group was determined using a Shimadzu microhardness tester (HMV-2T, Shimadzu Corporation, Kyoto, Japan) with a Vickers diamond indenter. Three microhardness indentations were performed with 25-g load and 10 seconds dwell time on each area. The Vickers microhardness (VHN) value for each specimen was measured before and after thermocycling or water storage. The mean values of the three indentations for each area were used as the VHN value.

Spectrophotometry

Specimens in the staining challenge group were subjected to color measurement using an Olympus CrystalEye Spectrophotometer (Olympus, Tokyo, Japan) before and after coffee storage. For each area on the specimen, three different sites were measured according to the CIE L*a*b* system, and the mean values were used as the color of the area.

Scanning Electron Microscopy

To qualitatively assess the surface morphology of resin-infiltrated lesions before aging challenges, 10 specimens were examined with an SEM (BCPCAS4800, JEOL, Tokyo, Japan) immediately following infiltration resin treatment. All specimens were coated with a gold layer approximately 10-nm thick before examination, and the scanning was operated at 4000× and 20,000× magnifications with an accelerating voltage of 1.5 kV. All 20 specimens in the thermocycling and the water storage groups were also scanned in the same manner to qualitatively assess the surface morphology of the resin-infiltrated lesions after the 180-hour aging challenges.

Statistical Analysis

The primary outcome measures of the present study were the changes in the surface roughness of the resin-infiltrated enamel surfaces following thermocycling challenges. Our pilot testing showed that the surface roughness of enamel lesions treated with the ICON infiltrant resin was approximately 0.4 with a standard deviation of approximately 0.05. We considered that a 20% difference in surface roughness was clinically significant as it correlated to an increase of ΔE from 2.9 to 3.5.[24] It has been shown that a ΔE smaller than 3.3 is not clinically significant.[25,26] Based on the effect size of 20% difference in surface roughness, we needed nine samples in each group to achieve 90% power at an alpha level of 0.05. We decided to use 10 specimens in each group. Surface roughness, microhardness, and coffee staining data were compared among the three areas (untreated lesions, resin-infiltrated lesions, and sound enamel) using analysis of variance and the post hoc Fisher PLSD test. The surface roughness and microhardness data were compared before and after thermocycling or water storage using paired *t* tests with Bonferroni corrections. Data were analyzed using StatView (SAS Institute Inc., Cary, NC, USA) for two-tailed tests, and a *P* value smaller than 0.05 was considered statistically significant.

RESULTS

Surface Roughness

Representative 3D laser scanning images of sound enamel, resin-infiltrated lesions, and untreated lesions are shown in Figure 2. Surface roughness data before and after thermocycling or water storage are listed in Tables 1 and 2. Before aging challenges, the surface roughness of the untreated enamel lesions was greater than that of the sound enamel but less than that of resin-infiltrated lesions ($p<0.01$; Figure 2; Tables 1 and 2), indicating resin infiltration increased the surface roughness of the enamel lesions. Thermocycling and water storage caused a further increase in surface roughness of the resin-infiltrated surfaces ($p<0.01$) but did not have a significant impact on the sound enamel and untreated lesion surfaces ($p>0.05$; Figure 2; Tables 1 and 2). The average surface roughness (Sa) of the resin-infiltrated surface increased from 0.373 μm to 0.621

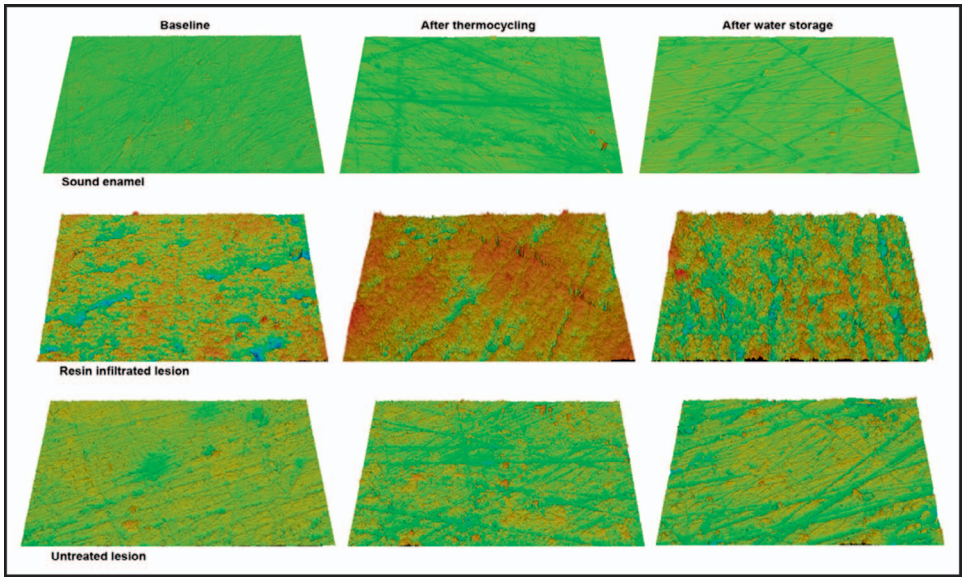


Figure 2. *Three-dimensional laser scanning images of sound enamel (top row), resin-infiltrated lesions (middle row), and untreated lesions (bottom row) at baseline (left column), after thermocycling (center column), and after water storage (right column). Surface roughness of resin-infiltrated lesions are greater than sound enamel and untreated lesions and further increased after thermocycling and water storage.*

μm after thermocycling for 180 hours. Water storage had a similar effect on the resin-infiltrated surfaces but was smaller in magnitude (from 0.317 μm to 0.472 μm on average).

Surface Microhardness

Surface microhardness measured in VHN before and after thermocycling or water storage is listed in Tables 3 and 4. Compared with that of sound enamel, the mean surface microhardness of the artificial carious lesion was reduced by more than threefold. Resin infiltration of the enamel lesions increased the surface microhardness by more than twofold ($p<0.05$; Tables 3 and 4). Surface microhardness of resin-infiltrated enamel lesions decreased slightly after thermocycling and water storage, but this change did not reach the *a priori* level of statistical significance ($p>0.05$).

Surface Staining

The overall color change of the specimens after coffee storage for all experimental sites is shown in Table 5. Color changes happened to all experimental sites (sound enamel, resin-infiltrated lesions, and untreated lesions). Resin-infiltrated surfaces showed significantly higher color alteration ($\Delta E=12.7\pm4.7$) than sound enamel ($\Delta E=4.3\pm0.8$) but much less than untreated lesions ($\Delta E=31.1\pm4.4$; $p<0.05$). Analysis of L*a*b* values showed that color change of resin-infiltrated lesions was largely due to decreased lightness (ΔL; $p<0.05$), while changes in Δa and Δb were not statistically significant as compared with the sound enamel ($p>0.05$). Untreated lesions showed significant increases in Δa and Δb values ($p<0.05$) in addition to decreased lightness.

Surface Morphology Under SEM

As shown in Figure 3, resin-infiltrated surfaces were largely intact and uniform before aging challenges (Figure 3a). Surface microcracks and microfissures appeared on resin-infiltrated lesions following thermocycling challenges (Figure 3b). Minor changes could also be observed following water storage but were much less distinct than those after thermocycling (Figure 3c). At high magnification (20,000×), hydroxyapatite crystals were shown to be embedded in the resin matrix to form a relatively uniform and intact surface (Figure 3d). After aging challenges, microfissures and microcracks could be observed on

Table 1: *Effect of Thermocycling on Surface Roughness (Sa) of Resin-Infiltrated Enamel Lesions (Mean ± SD)*

	Before Thermocycling[a]	After Thermocycling[a]	Paired *t* Test
Sound enamel	0.059±0.004a	0.063±0.006a	NS
Resin-infiltrated lesion	0.373±0.022b	0.621±0.035b	<0.01
Untreated lesion	0.129±0.041c	0.142±0.037c	NS
Analysis of variance	<0.01	<0.01	

[a] *Different letters in the same column denote statistically significant differences between each other with analysis of variance post hoc tests. NS, not significant.*

Table 2: *Effect of Water Storage on Surface Roughness (Sa) of Resin-Infiltrated Enamel Lesions (Mean ± SD)*[a]

	Before Water Storage	After Water Storage	Paired *t* Test
Sound enamel	0.049±0.008a	0.053±0.011a	NS
Resin infiltrated lesion	0.317±0.042b	0.472±0.114b	<0.01
Untreated lesion	0.122±0.039c	0.134±0.031c	NS
Analysis of variance	<0.01	<0.01	

[a] *Different letters in the same column denote statistically significant differences between each other with analysis of variance post hoc tests. NS, not significant.*

the surfaces, especially after thermocycling (Figure 3e,f).

DISCUSSION

The findings of the present study indicate that resin infiltration significantly increases the surface microhardness of enamel lesions and remains stable after thermocycling challenges. However, surface properties of resin-infiltrated enamel lesions may deteriorate with time in the oral environment and result in an increase in surface roughness and discoloration. Microcracks may appear on the resin-infiltrated surfaces after thermocycling challenges, which may further render the surface vulnerable to staining and discoloration.

Water sorption and surface degradation caused by thermocycling may affect the mechanical properties of resin-based dental materials. Surface microhardness of many resin composites decreased following thermocycling challenges.[27,28] Plasticization of the resin matrix by water sorption and hydrolytic breakdown of the resin–filler interface were considered to be the causes of the reduced surface hardness of resin composite materials.[28] However, the surface microhardness of resin-infiltrated enamel lesions was not significantly altered following thermocycling in the present study. Although the TEGDMA-based infiltration resin is unfilled and prone to water sorption and matrix degradation, the microhardness of the resin-infiltrated surfaces found in the present study and other studies ranged from 150 to 240 VHN (median 185 VHN)[5,20,29] which is significantly higher than most of the highly filled resin composites with a range from 40 to 150 VHN and a median of 72 VHN.[28,30] Such high surface hardness is obviously not a function of the resin matrix as polymerized TEGDMA is the softest (26 VHN) among the resin polymers used in dental restorative materials.[31] The infiltration resin was designed to penetrate the porous lesions left after acid etching and to fill the voids and spaces of the demineralized zone in a white spot lesion, thus preventing further demineralization and lesion progression.[32,33] It appears that the infiltration resin was able to encapsulate the hydroxyapatite crystals in the white spot lesion and form a relatively uniform resin-hydroxyapatite complex (Figure 3a,d) that exhibits high surface hardness. Although microcracks and microfissures may occur on the surface of the resin–hydroxyapatite complex, its surface hardness remained stable following thermocycling challenges. A stable resin–hydroxyapatite complex may be the foundation for clinical success of the resin infiltration technology.

Surface roughness of resin-infiltrated carious lesions was reported to be as high as 6.9 μm on average using the ICON infiltration resin,[20] which is considerably higher than the 0.2 μm threshold generally regarded as acceptable for a restorative material to resist plaque accumulation.[34-36] We found that the average surface roughness of resin-infiltrated areas was approximately 0.32 μm to 0.37 μm immediately after treatments, which was generally in agreement with that of Mueller and others.[36] After thermocycling challenge at temperatures between 5°C and 55°C for 10,000 cycles, simulating one-year of clinical service,[37] the surface roughness of resin-infiltrated lesions further deteriorated to 0.62 μm on average (Table 2; Figure 2), signifying a 70% increase compared with baseline. Repeated temperature fluctuations in the oral cavity may

Table 3: *Effect of Thermocycling on Surface Microhardness (VHN) of Resin-Infiltrated Enamel Lesions (Mean ± SD)*[a]

	Before Thermocycling	After Thermocycling	Paired *t* Test
Sound enamel	315.2±31.9a	320.2±30.7a	NS
Resin-infiltrated lesion	212.0±45.6b	202.9±55.2b	NS
Untreated lesion	89.3±24.1c	93.4±29.5c	NS
Analysis of variance	<0.01	<0.01	

[a] *Different letters in the same column denote statistically significant differences between each other with analysis of variance post hoc tests. NS, not significant.*

Table 4: *Effect of Water Storage on Surface Microhardness (VHN) of Resin-Infiltrated Enamel Lesions (mean ± SD)*[a]

	Before Water Storage	After Water Storage	Paired *t* Test
Sound enamel	313.2±25.5a	312.3±17.5a	NS
Resin-infiltrated lesion	219.1±25.2b	209.6±35.6b	NS
Untreated lesion	93.8±25.6c	90.2±23.0c	NS
Analysis of variance	<0.01	<0.01	

[a] *Different letters in the same column denote statistically significant differences between each other with analysis of variance post hoc tests. NS, not significant.*

induce degradation of resin-hydoxyapatite bonds due to differences in thermal expansion coefficients between enamel hydroxyapatite and the infiltration resin.[38,39] Thermal stress may also affect the surface integrity of resin-infiltrated enamel lesions as indicated by the presence of surface microcracks and microfissures after thermocycling challenges (Figure 3). These changes in surface properties may contribute to staining and discoloration of resin-infiltrated surfaces. The findings of the present study are in agreement with recent reports that ICON resin-infiltrated carious lesions were prone to discoloration under staining challenges.[40,41]

Discoloration of resin-based restorative materials may arrive from intrinsic and/or extrinsic stains. Intrinsic stain is associated with the properties of the polymeric networks such as water sorption and the presence of unreacted methacrylate in the resin matrix, while extrinsic stain is caused by external colorants such as those in beverages and foods.[42-44] The ICON infiltration resin is primarily a TEGDMA-based polymer with high penetration efficiencies.[45,46] Compared with other resin polymers commonly used in dental materials, such as UDMA and Bis-GMA, TEGDMA has the highest degree of water sorption owing to the presence of hydrophilic ether linkages.[19,42,47] A high degree of water sorption has long been linked to color stability issues and discoloration of resin-based dental materials.[13,14,18] On the other hand, surface roughness was recognized as the most important extrinsic factor for discoloration of resin-based dental materials.[15-17] Most modern resin composite materials for esthetic restorations could achieve a high glossy finish with surface roughness below the acceptable threshold of 0.2 μm after finishing and polishing.[35] Such a high degree of polishability appears to be difficult to achieve with the infiltration resin, as its surface roughness was not improved even after polishing with the Sof-Lex finishing and polishing system.[36] Therefore, the mechanisms underlying the discoloration of infiltration resin are likely twofold: one is intrinsically associated with its primary constituent TEGDMA, which has a high degree of water sorption, and the other is extrinsically related to a less than ideal surface polish that deteriorates with time in the oral cavity. To ensure long-term success, further research is warranted to improve surface polish and esthetic outcomes of resin infiltration, especially when smooth surface white spot lesions are involved in the esthetic zone.

As TEGDMA is prone to water sorption and may absorb twice as much water as compared with Bis-GMA, TEGDMA-based materials may be more susceptible to degradation than Bis-GMA- or UDMA-based materials.[42,48,49] The presence of water in the resin matrix may increase internal stress, leading to microcracking.[50] It was also shown that water sorption by TEGDMA increases with the elevation of temperatures,[49] which in combination with the thermal expansion and contraction effects of temperature fluctuation may further affect the integrity of the TEGDMA-based infiltration resin. The surface microcracks and microfissures observed on the resin-infiltrated lesions in the present study may be a result of such internal and thermal stresses due to water sorption and thermal expansion and contraction.

Table 5: *Effect of Staining Challenge on Surface Color Change of Resin-Infiltrated Enamel Lesions (Mean± SD)*

	ΔL	Δa	Δb	ΔE
Sound enamel	−3.0±0.8a	0.7±0.4a	2.9±0.7a	4.3±0.8a
Resin-infiltrated lesion	−9.7±3.5b	1.7±1.7a	6.4±5.8a	12.7±4.7b
Untreated lesion	−25.3±4.9c	5.5±2.6c	16.2±5.1c	31.1±4.4c
Analysis of variance	<0.01	<0.01	<0.01	<0.01

[a] *Different letters in the same column denote statistically significant differences with analysis of variance post hoc tests.*

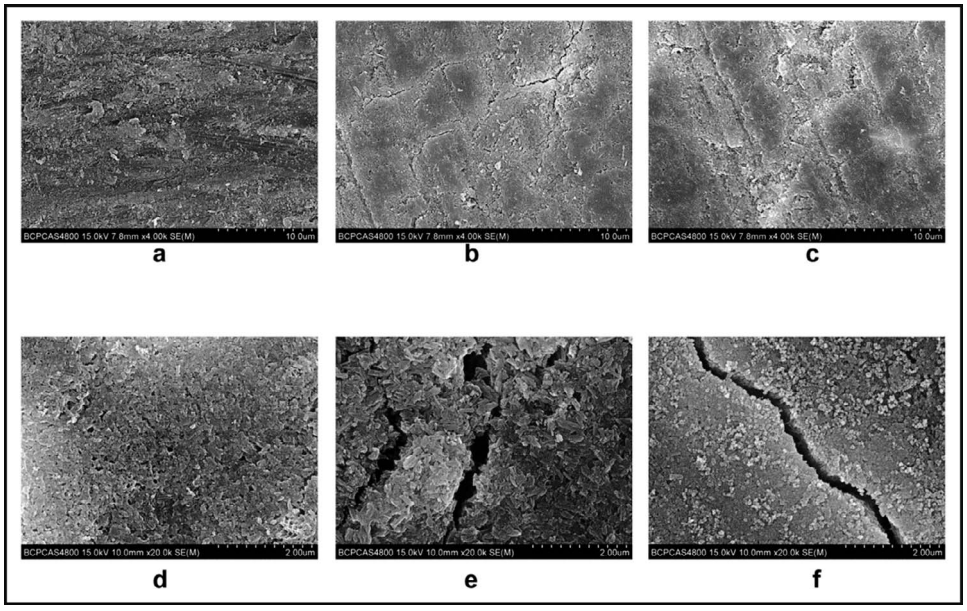


Figure 3. *Scanning electron microscopy images of resin-infiltrated lesions. Top row, at 4000×: (a): immediately after resin infiltration; (b): after thermocycling; (c): after water storage. Bottom row, at 20,000×: (d): immediately after resin infiltration; e: microfissures after thermocycling; f: microcracks after thermocycling.*

CONCLUSION

Within the limitations of this study, we conclude that the surface hardness of resin-infiltrated enamel lesions was high and remained stable following the thermocycling challenges. Surface roughness and color stability of resin-infiltrated enamel lesions was less than ideal and might further deteriorate after aging in the oral environment. Surface microcracks and microfissures could occur on the surface of the resin-hydroxyapatite complex after aging challenges. These changes may render the resin-infiltrated areas susceptible to discoloration.

Acknowledgement

This study was self-funded by the authors' institutions and received no financial support or other incentives from commercial or industrial entities.

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 Rochester.

Conflict of Interest

The authors 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 3 April 2016)

REFERENCES

1. Paris S, Meyer-Lueckel H, Colfen H, & Kielbassa AM (2007) Resin infiltration of artificial enamel caries lesions with experimental light curing resins *Dental Materials Journal* **26(4)** 582-588.
2. Ekstrand KR, Bakhshandeh A, & Martignon S (2010) Treatment of proximal superficial caries lesions on primary molar teeth with resin infiltration and fluoride varnish versus fluoride varnish only: efficacy after 1 year *Caries Research* **44(1)** 41-46.
3. Meyer-Lueckel H. & Paris S (2008) Progression of artificial enamel caries lesions after infiltration with experimental light curing resins. *Caries Research* **42(2)** 117-124.
4. Lausch J, Paris S, Selje T, Dorfer CE, & Meyer-Lueckel H (2015) Resin infiltration of fissure caries with various techniques of pretreatment in vitro *Caries Research* **49(1)** 50-55.
5. Torres CR, Rosa PC, Ferreira NS, & Borges AB (2012) Effect of caries infiltration technique and fluoride therapy on microhardness of enamel carious lesions *Operative Dentistry* **37(4)** 363-369.
6. Senestraro SV, Crowe JJ, Wang M, Vo A, Huang G, Ferracane J, & Covell DA Jr (2013) Minimally invasive resin infiltration of arrested white-spot lesions: a randomized clinical trial *Journal of the Americal Dental Association* **144(9)** 997-1005.
7. Martignon S, Ekstrand KR, Gomez J, Lara JS, & Cortes A (2012) Infiltrating/sealing proximal caries lesions: a 3-year randomized clinical trial *Journal of Dental Research* **91(3)** 288-292.
8. Paris S, Bitter K, Naumann M, Dorfer CE, & Meyer-Lueckel H (2011) Resin infiltration of proximal caries lesions differing in ICDAS codes *European Journal of Oral Sciences* **119(2)** 182-186.
9. Paris S, Hopfenmuller W, & Meyer-Lueckel H (2010) Resin infiltration of caries lesions: an efficacy randomized trial *Journal of Dental Research* **89(8)** 823-826.

10. Altarabulsi MB, Alkilzy M, & Splieth CH (2013) Clinical applicability of resin infiltration for proximal caries *Quintessence International* **44(2)** 97-104.
11. Altarabulsi MB, Alkilzy M, Petrou MA, & Splieth C (2014) Clinical safety, quality and effect of resin infiltration for proximal caries *European Journal of Paediatric Dentistry* **15(1)** 39-44.
12. Rey N, Benbachir N, Bortolotto T, & Krejci I (2014) Evaluation of the staining potential of a caries infiltrant in comparison to other products *Dental Materials Journal* **33(1)** 86-91.
13. Arocha MA, Mayoral JR, Lefever D, Mercade M, Basilio J, & Roig M (2013) Color stability of siloranes versus methacrylate-based composites after immersion in staining solutions *Clinical Oral Investigations* **17(6)** 1481-1487.
14. Nasim I, Neelakantan P, Sujeer R, & Subbarao CV (2010) Color stability of microfilled, microhybrid and nanocomposite resins—an in vitro study *Journal of Dentistry* **38(Supplement 2)** e137-e142.
15. Festuccia MS, Garcia Lda F, Cruvinel DR, & Pires-De-Souza Fde C (2012) Color stability, surface roughness and microhardness of composites submitted to mouthrinsing action *Journal of Applied Oral Science* **20(2)** 200-205.
16. Gonulol N, & Yilmaz F (2012) The effects of finishing and polishing techniques on surface roughness and color stability of nanocomposites *Journal of Dentistry* **40(Supplement 2**) e64-e70.
17. Sirin Karaarslan E, Bulbul M, Yildiz E, Secilmis A, Sari F, & Usumez A (2013) Effects of different polishing methods on color stability of resin composites after accelerated aging *Dental Materials Journal* **32(1)** 58-67.
18. Shintani H, Satou N, Yukihiro A, Satou J, Yamane I, Kouzai T, Andou T, Kai M, Hayashihara H, & Inoue T (1985) Water sorption, solubility and staining properties of microfilled resins polished by various methods *Dental Materials Journal* **4(1)** 54-62.
19. Sideridou I, Tserki V, Papanastasiou G (2003) Study of water sorption, solubility and modulus of elasticity of light-cured dimethacrylate-based dental resins *Biomaterials* **24(4)** 655-665.
20. Taher NM, Alkhamis HA, & Dowaidi SM (2012) The influence of resin infiltration system on enamel microhardness and surface roughness: an *in vitro* study *Saudi Dental Journal* **24(2)** 79-84.
21. Wu J, & Fried D (2009) High contrast near-infrared polarized reflectance images of demineralization on tooth buccal and occlusal surfaces at lambda = 1310-nm *Lasers in Surgery and Medicine* **41(3)** 208-213.
22. Bishara SE, & Ostby AW (2008) White spot lesions: formation, prevention, and treatment *Seminars in Orthodontics* **14(3)** 174-182.
23. Neuhaus KW, Schlafer S, Lussi A, & Nyvad B (2013) Infiltration of natural caries lesions in relation to their activity status and acid pretreatment *in vitro Caries Research* **47(3)** 203-210.
24. Gönülol N, & Yılmaz F (2012) The effects of finishing and polishing techniques on surface roughness and color stability of nanocomposites *Journal of Dentistry* **40(Supplement 2)** e64-e70.
25. Turkun LS, & Turkun M (2004) Effect of bleaching and repolishing procedures on coffee and tea stain removal from three anterior composite veneering materials *Journal of Esthetic & Restorative Dentistry* **16(5)** 290-301.
26. Um CM, & Ruyter IE (1991) Staining of resin-based veneering materials with coffee and tea *Quintessence International* **22(5)** 377-386.
27. Baek CJ, Hyun SH, Lee SK, Seol HJ, Kim HI, & Kwon YH (2008) The effects of light intensity and light-curing time on the degree of polymerization of dental composite resins *Dental Materials Journal* **27(4)** 523-533.
28. Tuncer S, Demirci M, Tiryaki M, Unlu N, & Uysal O (2013) The effect of a modeling resin and thermocycling on the surface hardness, roughness, and color of different resin composites *Journal of Esthetic & Restorative Dentistry* **25(6)** 404-419.
29. Paris S, Schwendicke F, Seddig S, Muller WD, Dorfer C, & Meyer-Lueckel H (2013) Micro-hardness and mineral loss of enamel lesions after infiltration with various resins: influence of infiltrant composition and application frequency *in vitro Journal of Dentistry* **41(6)** 543-548.
30. Choudhary S, & Suprabha B (2013) Effectiveness of light emitting diode and halogen light curing units for curing microhybrid and nanocomposites *Journal of Conservative Dentistry* **16(3)** 233-237.
31. Venter SAdS, Fávaro SL, Radovanovic E, & Girotto EM (2013) Hardness and degree of conversion of dental restorative composites based on an organic-inorganic hybrid. *Materials Research* **16** 898-902.
32. Paris S, & Meyer-Lueckel H (20096) Masking of labial enamel white spot lesions by resin infiltration—a clinical report *Quintessence International* **40(9)** 713-718.
33. Paris S, Meyer-Lueckel H, Colfen H, & Kielbassa AM (2007) Penetration coefficients of commercially available and experimental composites intended to infiltrate enamel carious lesions *Dental Materials* **23(6)** 742-748.
34. Bollen CM, Lambrechts P, & Quirynen M (1997) Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature. *Dental Materials* **13(4)** 258-269.
35. Janus J, Fauxpoint G, Arntz Y, Pelletier H, & Etienne O (2010) Surface roughness and morphology of three nanocomposites after two different polishing treatments by a multitechnique approach *Dental Materials* **26(5)** 416-425.
36. Mueller J, Yang F, Neumann K, & Kielbassa AM (2011) Surface tridimensional topography analysis of materials and finishing procedures after resinous infiltration of subsurface bovine enamel lesions *Quintessence International* **42(2)** 135-147.
37. Gale MS, & Darvell BW (1999) Thermal cycling procedures for laboratory testing of dental restorations *Journal of Dentistry* **27(2)** 89-99.
38. Hahnel S, Henrich A, Burgers R, Handel G, & Rosentritt M (2010) Investigation of mechanical properties of

modern dental composites after artificial aging for one year *Operative Dentistry* **35(4)** 412-419.

39. Borges AF, Santos Jde S, Ramos CM, Ishikiriama SK, & Shinohara MS (2012) Effect of thermo-mechanical load cycling on silorane-based composite restorations. *Dental Materials Journal* **31(6)** 1054-1059.
40. Araujo G, Naufel FS, Alonso R, Lima D, & Puppin-Rontani RM (2015) Influence of staining solution and bleaching on color stability of resin used for caries infiltration *Operative Dentistry* **40(6)** E250-E256.
41. Borges A, Caneppele T, Luz M, Pucci C, & Torres C (2014) Color stability of resin used for caries infiltration after exposure to different staining solutions *Operative Dentistry* **39(4)** 433-440.
42. Ferracane JL (2006) Hygroscopic and hydrolytic effects in dental polymer networks *Dental Materials* **22(3)** 211-222.
43. Barutcigil C, & Yildiz M (2012) Intrinsic and extrinsic discoloration of dimethacrylate and silorane based composites *Journal of Dentistry* **40(Supplement 1**) e57-e63.
44. Ren YF, Feng L, Serban D, & Malmstrom HS (2012) Effects of common beverage colorants on color stability of dental composite resins: the utility of a thermocycling stain challenge model in vitro *Journal of Dentistry* **40(Supplement 1**) e48-e56.
45. Meyer-Lueckel H, Chatzidakis A, Naumann M, Dorfer CE, & Paris S (2011) Influence of application time on penetration of an infiltrant into natural enamel caries *Journal of Dentistry* **39(7)** 465-469.
46. Meyer-Lueckel H, & Paris S (2010) Infiltration of natural caries lesions with experimental resins differing in penetration coefficients and ethanol addition *Caries Research* **44(4)** 408-414.
47. Venz S, & Dickens B (1991) NIR-spectroscopic investigation of water sorption characteristics of dental resins and composites *Journal of Biomedical Materials Research* **25(10)** 1231-1248.
48. Bagheri R, Tyas MJ, & Burrow MF (2007) Subsurface degradation of resin-based composites *Dental Materials* **23(8)** 944-951.
49. Kalachandra S, & Turner DT (1987) Water sorption of polymethacrylate networks: bis-GMA/TEGDM copolymers. *Journal of Biomedical Materials Research* **21(3)** 329-338.
50. Mair LH (1989) Surface permeability and degradation of dental composites resulting from oral temperature changes *Dental Materials* **5(4)** 247-255.

©Operative Dentistry, 2016, **41-6,** 627-633

An In Situ Study of the Influence of Staining Beverages on Color Alteration of Bleached Teeth

AL Briso • TC Fagundes • MO Gallinari • J Moreira
LCAG de Almeida • V Rahal • RS Gonçalves • PH dos Santos

Clinical Relevance

Although overall tooth color change was not affected at the end of at-home bleach treatment, consumption of staining beverages should be avoided since important color dimensions are altered.

SUMMARY

The aim of this study was to evaluate overall color change in bovine tooth fragments submitted to dental bleaching treatment performed simultaneously with the ingestion of beverages containing dyes. For this purpose, tooth fragments assembled into intraoral devices were submitted to at-home dental bleaching using 10% carbamide peroxide (CP) for 14 days and to immersion in staining beverages for 10 minutes daily. The specimens were divided into the following study groups according to bleaching treatment and staining substance (n=12): G I (negative control): no bleaching + distilled water; G II (positive control): bleaching + distilled water; G III: bleaching + coffee; and G IV: bleaching + grape juice. Twelve volunteers used the device continually, except during meals, oral hygiene, dental bleaching, and pigment challenge. Color readings were performed using a spectrophotometer both before the bleaching treatment and after each treatment week. The results were submitted to the normality test.

*André Luiz Fraga Briso, DDS, PhD, associate professor, Restorative Dentistry, Araçatuba Dental School, Universidade Estadual Paulista, São Paulo, Brazil

Ticiane Cestari Fagundes, DDS, PhD, assistant professor, Restorative Dentistry, Araçatuba Dental School, Universidade Estadual Paulista, São Paulo, Brazil

Marjorie de Oliveira Gallinari, DDS, PhD student, Restorative Dentistry, Araçatuba Dental School, Universidade Estadual Paulista, São Paulo, Brazil

Janaína Cardoso Moreira, DDS, MS, Restorative Dentistry, Araçatuba Dental School, Universidade Estadual Paulista, São Paulo, Brazil

Letícia Cunha Amaral Gonzaga de Almeida, DDS, PhD, Restorative Dentistry, Araçatuba Dental School, Universidade Estadual Paulista, São Paulo, Brazil

Vanessa Rahal, DDS, PhD, postdoctoral student, Restorative Dentistry, Araçatuba Dental School, Universidade Estadual Paulista, São Paulo, Brazil

Rafael Simões Gonçalves, DDS, MS, PhD student, Department of Dentistry, Endodontic and Dental Materials, Bauru School of Dentistry, University of São Paulo, Bauru, SP, Brazil

Paulo Henrique dos Santos, DDS, PhD, associate professor, Dental Materials and Prosthodontics, Araçatuba Dental School, Universidade Estadual Paulista, São Paulo, Brazil

*Corresponding author: Department of Restorative Dentistry, Universidade Estadual Paulista, Rua José Bonifácio, 1193 Vila Mendonça, Araçatuba, São Paulo 16015-050, Brazil; e-mail (alfbriso@foa.unesp.br)

DOI: 10.2341/15-306-L

The data obtained were submitted to analysis of variance and the Tukey or Kruskal-Wallis and Dunn tests (α=0.05). All bleached groups showed similar ΔE results at the end of treatment. Staining beverages generated negative ΔL mean values, and the lowest result was obtained in the treatment with coffee after 14 days. The Δa values in the groups that received treatment with staining beverages were higher when compared to the control groups. Dental bleaching associated with the consumption of staining substances may not affect overall tooth color change by the end of the treatment, although the consumption of staining substances did influence the different color dimensions.

INTRODUCTION

Several factors can alter the esthetics of a smile, including changes in the shape, texture, position, and color of teeth. The color and the appearance of teeth involve complex phenomena that are influenced by the type of ambient light, light scattering, translucency, opacity, and brightness of the substrate.[1,2]

Many esthetic treatments have been proposed seeking to improve dental esthetics; however, dental bleaching has gained great popularity by presenting itself as a conservative and effective technique.[1-3] The at-home bleaching technique is the most frequently used treatment for vital teeth due to its efficacy and biosafety and is considered the gold standard treatment among the different bleaching therapies.[4] It is known, however, that dental bleaching products change, although usually temporarily, the microhardness, surface roughness, and enamel surface morphology, increasing tooth porosity.[5-8] Thus, many professionals and manufacturers recommend that patients avoid eating pigment-rich foods during bleaching in order to avoid compromising the esthetic results.[9-11]

Despite the occurrence of these alterations in the enamel, studies have not been conclusive about the increased risk of pigmentation during bleaching,[12] especially considering that most of these investigations have not taken into account the challenging conditions of the oral environment.

In situ studies represent an intermediate step between laboratory experiments and clinical trials. *In situ* studies can more accurately examine the biological influences and protective effects of saliva under experimental conditions.[6,13] Therefore, intraoral models provide a clinical reality approach while preserving the sensitivity of laboratory models since the analysis can be carried out outside the oral cavity, using sensitive and accurate methods.[10]

Furthermore, few studies regarding the evaluation of the effects of bleaching have considered the three color dimensions separately, the value (ΔL), the amount of red and green (Δa), and the amount of yellow and blue (Δb).[3,11,14] These color parameters are related to human eye color perception and participate in the calculation of the overall change in tooth color (ΔE), representing an important factor in obtaining the final result.[1,2]

Since limited information regarding overall change in tooth color using the *in situ* study design is available and these effects have not been thoroughly investigated, the aim of this *in situ* study was to evaluate the different aspects of overall color change in bovine teeth exposed to staining beverages after undergoing dental bleaching using 10% carbamide peroxide. The tested null hypotheses were 1) that staining beverages do not interfere with the ΔE at the end of the bleaching treatment and 2) that staining beverages do not interfere in the three color dimensions, the value (ΔL), the amount of red and green (Δa), and the amount of yellow and blue (Δb) of the bleached specimens at the end of treatment.

METHODS AND MATERIALS

Volunteer Selection

This crossover, randomized, and double-blind *in situ* study was carried out in accordance with the ethical standards of the committee responsible for human experimentation and with the Helsinki Declaration and was approved by the institutional Research and Ethics Committee (03731512.0.0000.5420). Twelve volunteers were selected after anamnesis and clinical exams. The exclusion criteria were use of removable prosthesis, smokers, expectant mothers, mothers who were breast-feeding, use of drugs that affect salivary flow (antidepressants, narcotics, and diuretics), presence of decayed teeth, periodontal disease, and digestive disorders. Following selection, the volunteers received oral and written information regarding the study and, after agreeing to participate, signed an informed consent statement. Subsequently, they received oral hygiene instructions, a list of guidelines, and a case for storing their devices during meals.

The subjects were also instructed to wear a removable palatine intraoral device for 14 consecu-

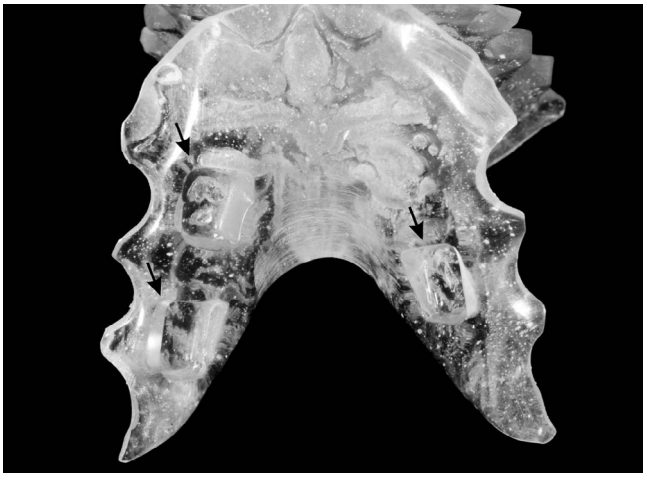


Figure 1. *Intraoral device containing three niches to assemble the experimental samples.*

tive days. Three bovine tooth samples were embedded into each experimental device, one for each study group (n=12). The bleaching treatments and the pigment challenges were performed daily and outside of the oral cavity.

Specimen Preparation

Thirty-six permanent bovine teeth from 24- and 30-month-old steers were selected. The experimental units (enamel/dentin discs) were obtained from bovine incisors. The 4.7-mm-diameter discs were obtained from the middle third of the buccal surface of the teeth. Those that presented with cracks, enamel stains, morphological crown alterations, and/or excessive wear on the incisal edge were excluded. The selected teeth were mechanically cleaned using a scalpel blade, followed by prophylaxis with pumice and water. Afterward, the dentin surface was wet-ground using fine (#400) and extra fine (#600) aluminum oxide sandpaper (T469-SF-Noton, Saint-Gobam Abrasivos Ltd, São Paulo, Brazil), until 1.0 mm (±0.2 mm) of enamel and 1.0 mm of dentin remained. Thereafter, the dentin tissue was impermeabilized with the application of two coats of clear nail polish (Risqué, NIASI, São Paulo, Brazil) so that only the enamel maintained contact with the staining substances.

Intraoral Device

After volunteer selection, impressions were made and working models fabricated. The intraoral palatal devices were made using acrylic resin (Jet, Artigos Odontológicos Clássico Ltd, São Paulo, Brazil) containing three niches of $5 \times 5 \times 4$ mm to hold the experimental samples (Figure 1). The specimens were sterilized in ethylene oxide and randomly placed in the intraoral devices using sticky wax (Kota Ind. e Com. Ltd, São Paulo, Brazil) and positioned 2 mm below the surface of the resin. They were carefully handled to prevent lateral cracks between the block and the wax. A polyethylene screen was fixed in the acrylic covering the specimens to keep the bleaching agent on the enamel samples during treatment.

Experimental Groups

The study groups were divided according to the bleaching treatment and the staining procedure. Before carrying out the experimental procedures, the pH of each product was measured with a previously calibrated digital pH meter (Crison Instruments SA, Barcelona, Spain).

In G I (negative control), no bleaching agent was used, and the samples were immersed in 1 mL of distilled water. In G II (positive control), the volunteers were instructed to deposit 0.04 mL of the 10% carbamide peroxide bleaching product Whiteness Perfect (FGM Produtos Odontológicos Ltd, Santa Catarina, Brazil) on specimens placed in the intraoral device, with the bleaching product remaining in contact with the enamel for 4 hours for each of the 14 days. Bleaching treatment was performed inside the oral cavity, and, following the product application, the specimens were washed in running water. After 1 hour, the samples were immersed in 1 mL of distilled water. In G III (coffee) and IV (grape juice), bleaching treatment using the 10% carbamide peroxide was performed as described for G II. After 1 hour, the volunteers took the device to the researcher for samples to be exposed to the staining beverages. Specimens exposed to coffee (G III) were immersed in 1 mL of coffee infusion at room temperature. The infusion was obtained using 8 g of Nescafe (Nestle SA, Vevay, Switzerland; pH 5.21) dissolved in 50 mL of purified water. Specimens exposed to grape juice (G IV) were immersed in 1 mL of industrialized grape juice at room temperature (Del Valle Mais, Coca-Cola Company, Rio de Janeiro, Brazil; pH 2.59).

For pigment treatments, the samples were carefully removed from the intraoral device and exposed to the appropriate staining beverages for 10 minutes daily. After exposure to these substances, the samples received prophylaxis, were rewashed and repositioned in the intraoral device (Figure 1).

Table 1: *Mean Values (SD) of ΔE in the Different Experimental Conditions and Evaluation Times*[a]

Evaluation Times	G I (Negative Control)	G II (Positive Control)	G III (Coffee)	G IV (Grape Juice)
T0	0.00 (0.0) Ba	0.00 (0.0) Ba	0.00 (0.0) Ba	0.00 (0.0) Ba
T1	1.26 (0.7) Ac	4.26 (1.6) Aa	3.41 (1.8) Aab	2.23 (1.3) Abc
T2	1.96 (0.9) Ab	5.53 (2.5) Aa	4.93 (2.4) Aa	4.22 (2.2) Aab

[a] *Means followed by different letters (uppercase in vertical, lowercase in horizontal) represent significant difference according to statistical analysis ($p<0.05$). One-way analysis of variance and Tukey test were used when comparing evaluation times in positive control and when comparing groups within T2. Kruskal-Wallis and Dunn tests were used for other comparisons.*

Spectrophotometry Analysis

Before each measurement, the specimens were submitted to prophylaxis with a Robinson-type brush to remove excess dye and other impurities that could interfere with the color measurement. Three color measurements were performed at each analysis, using an ultraviolet-visible reflection spectrophotometer (model UV-2450, Shimadzu Corporation, Kyoto, Japan). Arithmetic means were used to perform the statistical analysis.

The spectrophotometer used the CIE L*a*b* color model, established by the Commission Internacionale de l'Eclairage (CIE; International Commission on Illumination), which allows for the specification of color perceptions in three-dimensional models. Readings were taken on the buccal surface of the specimens. The axial "L" indicates the color value and extends from 0 (black) to 100 (perfect white). The coordinate "a" represents the amount of red (positive values) and green (negative values), while the coordinate "b" is the amount of yellow (positive value) and blue (negative values). The system CIE L*a*b* calculates the ΔE between two points using the formula $\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$. Readings were performed before the start of the bleaching treatment and after the pigment application on the seventh day (T1) and the 14th day (T2) of 10% CP application. All baseline parameters had means and standard deviations as follows: L = 67.90 (1.99); a = −1.95 (0.16), and b = 3.56 (0.42).

Statistical Analysis

The results were submitted to the normality test (Shapiro-Wilk). Analysis of variance and Tukey tests were used for ΔE positive control, ΔL negative control, and grape juice when comparing evaluation times within the same group. Similar analyses were performed for Δb in T1 and for ΔE, ΔL, and Δb in T2 when comparing groups within the same evaluation time. Other comparisons did not pass the normality test; therefore, the Kruskal-Wallis and Dunn tests were used. All analyses were performed at a 5% significance level using SigmaPlot 12.0 statistics software.

RESULTS

The statistical analysis showed that the study groups presented considerable differences in ΔE after the first week (T1). All groups presented a continuing increase in the mean value of ΔE but without differences between T1 and T2. The comparison performed between groups showed that, at the seventh day of treatment (T1), the group exposed to grape juice and the negative control group (G IV and G I) had lower ΔE when compared to the positive control group exposed to distilled water and the group exposed to coffee (G II and G III). However, all bleached groups (G II, G III, and G IV) showed similar results at the end of treatment (T2). In general, the positive control had the highest mean ΔE values, unlike the negative control, which presented the lowest mean values until the end of the treatment (Table 1).

When analyzing the ΔL values, Table 2 shows that the bleached groups had similar results to the group that did not receive the bleaching treatment (G I) after the seventh day of treatment (T1). On the other hand, the positive control group (G II) showed the

Table 2: *Mean Values (SD) of ΔL in the Different Experimental Conditions and Evaluation Times*[a]

Evaluation Times	G I (Negative Control)	G II (Positive Control)	G III (Coffee)	G IV (Grape Juice)
T0	0.00 (0.0) ABa	0.00 (0.0) Ba	0.00 (0.0) Aa	0.00 (0.0) Aa
T1	0.51 (0.7) Aab	4.73 (4.1) Aa	−1.90 (2.8) Bb	0.19 (2.1) ABb
T2	−0.80 (1.2) Bb	4.59 (3.6) Aa	−3.13 (3.5) Bb	−2.51 (3.1) Bb

[a] *Means followed by different letters (uppercase in vertical, lowercase in horizontal) represent significant difference according to statistical analysis ($p<0.05$). One-way analysis of variance and Tukey test were used when comparing evaluation times in negative control and grape juice, and when comparing groups within T2. Kruskal-Wallis and Dunn tests were used for other comparisons.*

Table 3: *Mean Values (SD) of Δa in the Different Experimental Conditions and Evaluation Times*[a]

Evaluation Times	G I (Negative Control)	G II (Positive Control)	G III (Coffee)	G IV (Grape Juice)
T0	0.00 (0.0) Ba	0.00 (0.0) Ba	0.00 (0.0) Ba	0.00 (0.0) Ba
T1	0.35 (0.2) Ac	0.13 (0.5) ABc	1.35 (0.3) Aab	0.96 (0.2) Ab
T2	0.58 (0.2) Abc	0.42 (0.3) Ac	1.93 (0.3) Aa	1.26 (0.5) Aab

[a] *Means followed by different letters (uppercase in vertical, lowercase in horizontal) represent significant difference according to statistical analysis ($p<0.05$). Kruskal-Wallis and Dunn tests were used for all comparisons.*

highest luminescence values (ΔL) at T2. It was also noted that the pigment treatments generated negative ΔL mean values and that the lowest value obtained in the treatment was with coffee at T2.

Table 3 shows that the negative and positive control groups had statistically similar Δa values throughout the study. All groups presented a continuing increase in mean value of Δa but without differences between T1 and T2. It was also verified that the Δa values in the groups receiving treatment with staining beverages (G III and G IV) were higher when compared to the control groups (G I and G II).

Table 4 indicates that there were no statistically significant differences in Δb between the evaluation times for all groups.

DISCUSSION

In the present study, bovine teeth were used, as they are easy to obtain and standardize. Bovine teeth have been commonly used in laboratory research since they have low variations of experimental responses due to their uniform composition and are similar to human teeth with respect to morphology and histology.[15]

The recommendation to avoid foods rich in dyes when undergoing bleaching procedures is routine among dental professionals and manufacturers, even though the evidence of the effect of such foods is based on *in vitro* studies.[9,10] In the present study, the *in situ* experimental design was used to account for both the effect of bleaching products and of staining beverages and the action of saliva and thermal/chemical variations in the oral cavity. As such, it was possible to reproduce similar conditions to those found in patients that perform the bleaching treatment, and the current results most likely approximate the potential results that would be observed in a clinical setting.[13]

The overall tooth color change results showed that the exposure of dental fragments to grape juice during at-home dental bleaching resulted in a ΔE that was different from the group that received only the bleaching treatment after seven days. However, all of the bleached study groups presented similar color alterations at the final stage of treatment, accepting the first hypothesis of the study. Similar results were recently reported by Matis and others,[11] who noted that, based on data obtained in previous clinical trials, dark beverages do not negatively influence the overall tooth color change final values. Despite this result, it is been suggested that bleached teeth may be susceptible to staining after whitening treatment.[10] It is also worth mentioning that some studies have indicated that the acidic pH of pigment solutions promotes a loss of mineral in tooth structure,[16,17] favoring the penetration of these solutions into the demineralized structure, thus influencing the overall tooth color change during the bleaching treatment.

Although clinical trials show that at-home dental bleaching requires 15 to 21 days to achieve the best overall tooth color change, ΔE values obtained in G II from T2 were the same as those observed in studies that performed longer treatments.[18-20] It is possible that the present results were obtained faster (in seven days) because of the dimensions of the experimental samples, which were tapered (preserving 1.5 mm of dentin) so that they could be placed into intraoral devices, causing minimal discomfort for the volunteers. It is important to note

Table 4: *Mean Values (SD) of Δb in the Different Experimental Conditions and Evaluation Times*[a]

Evaluation Times	G I (Negative Control)	G II (Positive Control)	G III (Coffee)	G IV (Grape Juice)
T0	0.00 (0.0) Aa	0.00 (0.0) Aa	0.00 (0.0) Aa	0.00 (0.0) Aa
T1	0.39 (1.1) Aa	−0.68 (1.5) Aa	0.22 (1.3) Aa	−0.63 (1.1) Aa
T2	−0.03 (1.6) Aa	−1.18 (1.4) Aa	0.49 (2.2) Aa	−0.63 (2.4) Aa

[a] *Means followed by different letters (uppercase in vertical, lowercase in horizontal) represent significant difference according to statistical analysis ($p<0.05$). One-way analysis of variance and Tukey test were used when comparing groups within T1 and T2. Kruskal-Wallis and Dunn tests were used for other comparisons.*

that in clinical studies, the dentin, which supposedly houses most of the chromophore molecules, presents considerably greater thickness, making overall tooth color changes easier to observe even after 15 days of treatment.[18,19,21,22]

With the aim for an accurate analysis of the overall tooth color changes promoted by the bleaching treatment and the staining substances, three color dimensions were also analyzed—ΔL, Δa, and Δb—since dental bleaching outcome is characterized mainly by alterations in these values.[23,24] G II was found to have a higher value (ΔL) when compared to G III and G IV at all evaluation times. These results corroborate the findings of Attin and others,[3] who demonstrated that bleached specimens simultaneously pigmented with black tea had a darker appearance than the specimens that were only bleached. These data suggest that the use of staining beverages during dental bleaching may darken or delay the desired esthetic result.

A previous report affirms that beverages/foods that are not considered part of a white diet do not negatively affect the bleaching process.[11] However, the present research used the exact bleaching agent methodology and staining substances from the above-mentioned study, simulating a more intense consumption of these beverages.

The Δa analysis showed that specimens treated with coffee and grape juice presented a more reddish color than those from G I and G II due to the dye used in these substances, indicating that the bleaching treatment was not fully effective in breaking down the pigment molecules from these beverages.

On the other hand, negative values were obtained for G I, G II, and G IV for Δb, indicating that they became less yellow, indicating that the bleaching agent was able to bind with the pigments and break their complex chains, making the samples more blue.[19,25]

In general, the CIE L*a*b* analyses indicated that beverages with a high potential for pigmentation did in fact influence the color of the experimental samples submitted to bleaching, allowing us to reject the second null hypothesis.

All of these observations related to the assessment of color indicate that the isolated study of ΔE is not sufficient when analyzing the effectiveness of bleaching therapies in cases where substances with antagonistic effects (bleaching substances and pigments) are used; the study of ΔE should be complemented by analyses of Δa, Δb, and ΔL. The analyses of these data show that the staining substances may affect the outcome of the bleaching treatment.

The present study may contribute to the understanding of the mechanisms of pigmentation during bleaching procedures, although further studies considering some important factors that influence the dental esthetics, such as fluorescence, glow, and other optical properties, are needed to effectively analyze these effects.

CONCLUSION

Under the experimental conditions of this clinical study, it can be concluded that dental bleaching associated with the consumption of staining substances may not affect overall tooth color change by the end of the treatment, although the consumption of staining substances did influence the different color dimensions.

Acknowledgment

The authors acknowledge FAPESP for financial support (grant number 2012/10224-0).

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Institutional Research and Ethics Committee. The approval code for this study is 03731512.0.0000.5420.

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 4 April 2016)

REFERENCES

1. Caneppele TM, Borges AB, & Torres CR (2013) Effects of dental bleaching on the color, translucency and fluorescence properties of enamel and dentin *European Journal of Esthetic Dentistry* **8(2)** 200-212.
2. Dietschi D, Benbachir N, & Krejci I (2010) In vitro colorimetric evaluation of the efficacy of home bleaching and over-the-counter bleaching products *Quintessence International* **41(6)** 505-516.
3. Attin T, Manolakis A, Buchalla W, & Hanning C (2003) Influence of tea on intrinsic colour of previously bleached enamel *Journal of Oral Rehabilitation* **30(5)** 488-494.
4. Haywood VB, & Heymann HO (1989) Nightguard vital bleaching *Quintessence International* **20(3)** 173-176.
5. Markovic L, Jordan RA, Lakota N, & Gaengler P (2007) Micromorphology of enamel surface after vital tooth bleaching *Journal of Endodontics* **33(5)** 607-610.

6. De Arruda AM, dos Santos PH, Sundfeld RH, Berger SB, & Briso AL (2012) Effect of hydrogen peroxide at 35% on the morphology of enamel and interference in the de-remineralization process: An in situ study *Operative Dentistry* **37(5)** 518-525.
7. Lia Mondelli RF, Garrido Gabriel TR, Piola Rizzante FA, Magalhães AC, Soares Bombonatti JF, & Ishikiriama SK (2015) Do different bleaching protocols affect the enamel microhardness? *European Journal of Dentistry* **9(1)** 25-30.
8. Giannini M, Silva AP, Cavalli V, & Paes Leme AF (2006) Effect of carbamide peroxide-based bleaching agents containing fluoride or calcium on tensile strength of human enamel *Journal of Applied Oral Science* **14(2)** 82-87.
9. Berger SB, Coelho AS, Oliveira VA, Cavalli V, & Giannini M (2008) Enamel susceptibility to red wine staining after 35% hydrogen peroxide bleaching *Journal of Applied Oral Science* **16(3)** 201-204.
10. Attia ML, Aguiar FH, Mathias P, Ambrosano GM, Fontes CM, & Liporoni PC (2009) The effect of coffee solution on tooth color during home bleaching applications *American Journal of Dentistry* **22(3)** 175-179.
11. Matis BA, Wang G, Matis JI, Cook NB, & Eckert GJ (2015) White diet: Is it necessary during tooth whitening? *Operative Dentistry* **40(3)** 235-240.
12. Carey CM (2014) Tooth whitening: What we now know *Journal of Evidence Based Dental Practice* **(Supplement 14)** 70-76.
13. Zero DT (1995) In situ caries models *Advances in Dental Research* **9(3)** 214-230.
14. Delfino CS, Chinelatti MA, Carrasco-Guerisoli LD, Batista AR, Fröner IC, & Palma-Dibb RG (2009) Effectiveness of home bleaching agents in discolored teeth and influence on enamel microhardness *Journal of Applied Oral Science* **17(4)** 284-288.
15. Schmeling M, Maia HP, & Baratieri LN (2012) Opalescence of bleached teeth. *Journal of Dentistry* **40(Supplement 1)** e35-e39.
16. Zanet CG, Fava M, & Alves LAC (2011) In vitro evaluation of the microhardness of bovine exposed to acid solutions after bleaching. *Brazilian Oral Research* **25(6)** 526-527.
17. Scatena C, Borsatto MC, Rodrigues-Júnior AL, & Serra MC (2015) Effect of foods and drinks on primary tooth enamel after erosive challenge with hydrochloric acid. *Brazilian Oral Research* **29(1)** 1-7.
18. Almeida LC, Riehl H, Santos PH, Sundfeld ML, & Briso AL (2012) Clinical evaluation of the effectiveness of different bleaching therapies in vital teeth *International Journal of Periodontics and Restorative Dentistry* **32(3)** 303-309.
19. Briso ALF, Fonseca MSM, de Almeida LCAG, Mauro SJ, & dos Santos PH (2010) Color alteration in teeth subjected to different bleaching techniques *Laser Physics* **20(12)** 2066-2069.
20. Bernardon JK, Sartori N, Ballarin A, Perdigão J, Lopes GC, & Baratieri LN (2010) Clinical performance of vital bleaching techniques *Operative Dentistry* **35(1)** 3-10.
21. Sundfeld RH, Sundfeld Neto D, Machado LS, de Oliveira FG, de Alexandre RS, Palo RM, & Sundefeld ML (2014) Dental bleaching with a 10% hydrogen peroxide product: A six-month clinical observation *Indian Journal of Dental Research* **25(1)** 4-8.
22. Briso AL, Lima AP, Gonçalves RS, Gallinari MO, & dos Santos PH (2014) Transenamel and transdentinal penetration of hydrogen peroxide applied to cracked or microabrasioned enamel *Operative Dentistry* **39(2)** 166-173.
23. Matis BA, Cochran MA, Wang G, & Eckert GJ (2009) A clinical evaluation of two in-office bleaching regimens with and without tray bleaching *Operative Dentistry* **34(2)** 142-149.
24. Matis BA, Cochran MA, Eckert GJ, & Matis JI (2007) In vivo study of two carbamide peroxide gels with different desensitizing agents *Operative Dentistry* **32(6)** 549-555.
25. Kawamoto K, & Tsujimoto Y (2004) Effects of the hydroxyl radical and hydrogen peroxide on tooth bleaching *Journal of Endodontics* **30(1)** 45-50.

©Operative Dentistry, 2016, **41-6,** 634-641

Influence of Finishing and Polishing Techniques and Abrasion on Transmittance and Roughness of Composite Resins

PMA Carneiro • TM Ramos • CS de Azevedo • E de Lima
SHJ de Souza • ML Turbino • PF Cesar • AB Matos

Clinical Relevance

Clinicians can choose any of the finishing and polishing systems tested without compromising the light transmittance of the final restoration.

SUMMARY

The aim of this study was to evaluate the influence of finishing and polishing systems and toothbrush abrasion on transmittance (T) and surface roughness (Ra) of three composite resins (Filtek Z350 XT, Tetric N-Ceram, and IPS Empress Direct). Eighteen resin disks (10 mm diameter × 2 mm thick) finished by polyester strips had initial surface smoothness recorded, representing phase 1 (P1). Specimens were divided into three groups (n=6) according to the finishing/polishing instrument used (One-Gloss, TopGloss, and Sof-Lex) to compose phase 2 samples (P2). Then specimens were subjected to 514 cycles of toothbrush simulation using a toothpaste slurry, with a constant load applied to soft bristles, and were then washed (phase 3=P3). After each phase, the specimens were examined by an optical profiler and spectrophotometer to measure Ra and T. Data were analyzed by analysis of variance, Tukey and Pearson tests. T values were statistically influenced by composite resin (p=0.000) and phase of measurement (p=0.000) factors, while the finishing/polishing system used (p=0.741) did not affect T. On the other hand, Ra values were

Paula Mendes Acatauassú Carneiro, DDS, MSc, University of Sao Paulo–School of Dentistry, Operative Dentistry Department, Sao Paulo, Brazil

Thayanne Monteiro Ramos-Oliveira, DDS, MSc, PhD, University of Sao Paulo–School of Dentistry, Operative Dentistry Department, Sao Paulo, Brazil

Cynthia Soares de Azevedo, DDS, MSc, PhD University of Sao Paulo–School of Dentistry, Operative Dentistry Department, Sao Paulo, Brazil

Erick de Lima, DDS, MSc, University of Sao Paulo–School of Dentistry, Biomaterials and Oral Biology Department, Sao Paulo, Brazil

Samira Helena João de Souza, DDS, MSc, University of Sao Paulo–School of Dentistry, Operative Dentistry Department, Sao Paulo, Brazil

Míriam Lacalle Turbino, DDS, MSc, PhD, University of Sao Paulo–School of Dentistry, Operative Dentistry Department, Sao Paulo, Brazil

Paulo Francisco Cesar, DDS, MSc, PhD, University of Sao Paulo–School of Dentistry, Biomaterials and Oral Biology Department, Sao Paulo, Brazil

*Adriana Bona Matos, DDS, MSc, PhD, University of Sao Paulo–School of Dentistry, Operative Dentistry Department, Sao Paulo, Brazil

*Corresponding author: Av. Prof. Lineu Prestes, 2227, Sao Paulo, 05508-000, Brazil: e-mail: bona@usp.br

DOI: 10.2341/15-281-L

statistically affected by the factor finishing/polishing system (p=0.000), but not by composite resin (p=0.100) and phase of measurement (p=0.451). Tetric N-Ceram and Empress Direct presented higher values of roughness when polished by OneGloss, while TopGloss and Sof-Lex showed a lower roughness. It can be concluded that composite resins transmitted more light after dental abrasion. Transmittance of composite resins was not modified by the distinct roughness created by finishing/polishing instruments.

INTRODUCTION

The surface quality of restorations is considered a key factor for clinical success. A smooth surface increases the lifetime of composite resin restorations, optimizing their esthetic appearance and reducing plaque accumulation and surface pigmentation.[1] In this context, the finishing and polishing techniques used are important steps to guarantee long-lasting restorations.[2,3]

The literature shows numerous reports evidencing a wide range of finishing and polishing instruments available for clinicians, such as multilaminated drills, diamond-impregnated rotary instruments, rubber or silicone disks and wheels, and carbide- and aluminum oxide–based abrasive disks.[4] There is no consensus in the literature about which instrument is the most suitable for each type of composite resin; the restorations are classified by filler particles[5] as microfilled, microhybrid, or nanohybrid.[6]

In addition to surface smoothness, esthetic and chromatic characteristics of dental restorations may also be influenced by illumination conditions and optical properties of the restorative material, such as transmittance, light scattering, fluorescence, and opalescence.[7-9] Transmittance, for example, refers to the transmission of light through a body and may occur with different levels of absorption and scattering within the material.[10] The transmittance of the human tooth is higher at the incisal edge, especially in young patients with relatively intact enamel. Thus, the restoration of young teeth is difficult to achieve because of the original optical properties. The filler content of a composite resin is an important factor affecting the transmittance of the final restoration. In addition, the amount, size, and shape of the filler particles directly affect the light scattering ability of the composite resin.[11]

Both surface texture and optical properties are affected by degradation between the organic matrix and the filler, wear of the filler particles, and weakening of the adhesion between the organic matrix and inorganic filler.[12] The literature[13,14] proves that the action of toothbrushes and the use of abrasive toothpastes result in significant wear of the restoration, with consequent increased surface roughness. Even when excellent finishing and polishing procedures are carried out and the restoration surface is very smooth, it is important to remember that the wear rate of restorative materials submitted to tooth brushing is not similar to that of dental enamel.[15] There are several previous works[14,16,17] showing the effect of tooth brushing on the surface roughness of restorative materials, but there are no reports concerning the effect of surface roughness on light transmittance and the final esthetic quality of composite resin restorations.

From a clinical standpoint, it is important to understand whether the changes caused by brushing can affect the optical properties and the roughness of different composite resins polished by different finishing and polishing systems. Hence, the aim of this study was to evaluate the transmittance and surface roughness of three different composite resins as a function of the type of finishing/polishing system used and the phase of measurement of these properties (before or after polishing and brushing).

METHODS AND MATERIALS

Eighteen disk-shaped specimens (10 mm diameter × 2 mm thick) of each composite resin (Table 1) were prepared using a polypropylene matrix. The composite resin was inserted in one single portion, covered with a polyester strip, and photoactivated (Radii-Cal, SDI, Bayswater, Australia; intensity of 800 mW/cm^2) for 40 seconds. The surface smoothness promoted by a polyester strip represented phase 1 (P1). All specimens were stored in distilled water at 37°C for 24 hours.

A 10 × 4 mm square area was delimited on disk surfaces. The disks of each composite resin were subdivided (n=6) according to the type of finishing and polishing system used (Table 2): OneGloss (Shofu Inc, Kyoto, Japan), TopGloss (EDENTA, St. Gallen, Switzerland), and Sof-Lex (3M ESPE, St Paul, MN, USA). The polished specimens were stored in distilled water at 37°C for seven days, representing phase 2 (P2).

Toothbrush simulation was carried out using a tooth brushing machine (MSE-ELQUIP, São Carlos, SP, Brazil) equipped with 10 toothbrushes with soft

Table 1: *Materials Used in the Study*

Composite/Lot No.	Manufacturer	Type	Shape	Filler Content
Filtek® Supreme XT/N367731)	3M ESPE, St Paul, MN, USA	Nanofilled	Translucent (CT)	Nonagglomerated/nonaggregated, 75 nm silica nanofiller
				Agglomerate silica nanocluster (0.6-1.4 μm)
				72.5% by weight
Tetric® N-Ceram/R27050	Ivoclar Vivadent AG, Schaan, Liechtenstein	Nanohybrid	Translucent (T)	Barium glass, ytterbium trifluoride, mixed oxide, and copolymers (0.04-3 μm)
				80%-81% by weight
IPS Empress® Direct/R47920	Ivoclar Vivadent AG, Schaan, Liechtenstein	Nanohybrid	Translucent (Trans 30)	Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide, and copolymers (0.04-3 μm)
				75%-79% by weight

nylon bristles (Oral B 30, Procter & Gamble, São Paulo, SP, Brazil) (Figure 1B) and a toothpaste slurry in a proportion of 1:2 by weight (90 g of Colgate Máxima Proteção Anti-Cáries® [Colgate-Palmolive, Osasco, SP, Brazil] + 180 mL of distilled water). Taking into account that an individual brushes his teeth twice a day for one minute each time, the machine was calibrated to perform 257 cycles per minute for two minutes, comprising a total of 514 cycles. One cycle corresponded to the full movement (back and forth) of the toothbrush. A constant load of 200*g* was applied to the active tips of the brushes' bristles. Brushes were replaced after each 514 cycles, and the samples were randomly brushed. After brushing, specimens were abundantly washed in running tap water, representing phase 3 (P3).

After each of the three phases, specimens were examined in an optical profilometer (Proscan 2100, Scantron, Venture Way, Tauton, UK) and with a spectrophotometer (CM-3700d, Konica Minolta, Aichi Prefecture, Japan) for determination of the surface roughness (Ra) and total transmittance (T), respectively.

Measurement of Surface Roughness

Surface roughness measurements were performed with an optical profilometer (2100, Scantron, Venture Way). Scanning areas were limited to 1 mm^2 in the center of each specimen with the use of a S11/03 sensor. At the X axis, 1000 steps of 0.001 mm were used, while at the Y axis, five steps were used, with a pitch of 0.2 mm. The mean values for Ra were obtained from each experimental group using specific software (Proscan Application software, version 2.0.17).

Measurement of Light Transmittance

The transmittance is given by the ratio (%) of intensity of the incident light and the light transmitted by the specimen for each wavelength. After storage in water, specimens were evaluated by the spectrophotometer Cintra 10 UV-Visible Spectrometer (GBC, Braeside, VIC, Australia). Measurements were made every 5 nm in the wavelength range from 400 to 700 nm, corresponding to the visible light spectrum of the human eye. For each specimen, three readings were recorded at different places and the obtained values were averaged. The numerical values (percentage) of transmittance in each wave-

Table 2: *Composition and Application Time of Finishing and Polishing Systems Used in this Study*

Finishing and Polishing Material/Lot No.	Manufacturer	Material Time	Application Time, s
OneGloss (OG)/281091	Shofu Inc, Kyoto, Japan	Aluminum oxide one-step finisher and polisher	15
TopGloss (TG)/Y09.001	EDENTA Ag, AU, SG, Switzerland	Diamond-impregnated micropolisher	15
Sof-Lex (SL)/1227200585	3M ESPE, St Paul, MN, USA	Aluminum oxide–coated disks (coarse, medium, fine, superfine)	15 for each disk

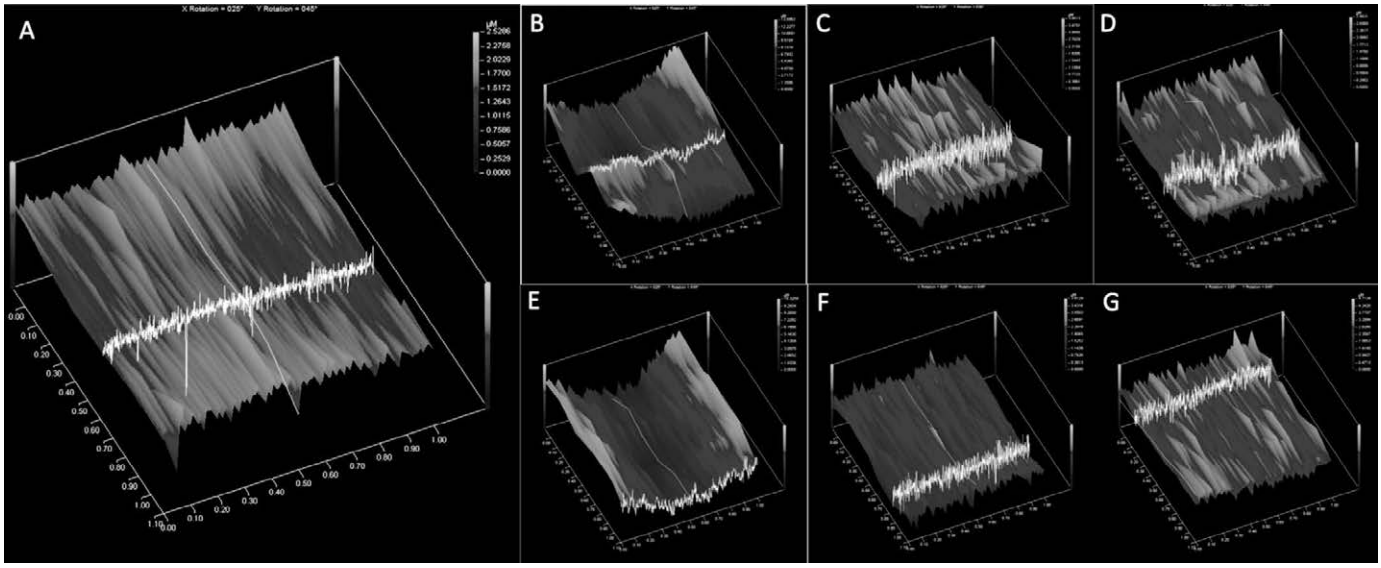


Figure 1. *Representative roughness images from composite Z350 XT: (A) Phase 1—Surface smoothness promoted by Mylar strip. (B, C, D) Phase 2—Surface smoothness promoted by different finishing and polishing systems: OneGloss, TopGloss, and Sof-Lex, respectively. (E, F, G) Phase 3—Surface smoothness after tooth brushing of the specimens.*

length were initially stored as a file on the spectrophotometer software and transferred to Microsoft Excel (Microsoft Excel, version 2007, Microsoft, Seattle, USA) for statistical analysis.

Statistical Analysis

A power analysis was performed using the software Minitab version 16 (Minitab Inc., State College, Pennsylvania) assuming a general full factorial design involving the main factors, an alpha value of 0.05, six specimens per group, and a global standard deviation of 1.71 for transmittance and of 0.48 for roughness. The test resulted in power of 100%. Two-way analysis of variance was used to evaluate both roughness and transmittance data. Means were compared by Tukey test (α=0.05), and regression analysis was used to investigate whether there was a correlation between transmittance and surface roughness values (α=0.05).

RESULTS

Light Transmittance

Table 3 shows the mean values of transmittance obtained for the composite resin specimens used in the study, as a function of the finishing/polishing system and the measurement phase.

The results showed that there was no effect of the polishing system on the total transmittance of the specimens, regardless of the composite resin evaluated. However, the measurement phase significantly affected the transmittance of the specimens.

Statistical differences were observed among the different phases for all composite resins evaluated (p=0.01). For Tetric N-Ceram and Empress Direct, the effect of the measurement phase on transmittance was the same, since the mean values of phases 1 and 2 were similar; however, for these materials, transmittance measurements made after phase 3 were significantly higher in relation to those measures after the two other phases. For Z350 XT, the effect of measurement phase on T was significant. It was observed that after phase 2, the T mean value significantly decreased in relation to the values obtained after phase 1. Also, after phase 3, there was a significant increase in T in relation to the values obtained after phases 1 and 2.

Surface Roughness

Table 4 shows the Ra mean values for composite resins used in the current study, as a function of the finishing/polishing system and measurement phase. Figures 1, 2, and 3 show the roughness patterns of experimental groups.

Tetric N-Ceram and Empress Direct did not have their roughness affected by the measurement phase. Only Z350 XT was affected by measurement phase, as its roughness decreased significantly after phase 2 in relation to the measurement made after phase 1. The roughness of Z350 XT after phase 3 was similar to that obtained after phase 2.

Table 3: *Mean Values and Standard Deviations of Total Transmittance (%)*[a]

	OG (OneGloss)	TG (TopGloss)	SL (Sof-Lex)	Mean
Composite Z350 XT				
P1	46.8±0.9	46.2±1.5	46.4±1.5	46.5 [B]
P2	40.1±0.7	39.9±1.8	39.2±2.9	39.7 [C]
P3	54.8±1.4	56.6±0.8	56.0±1.3	55.8 [A]
Mean	47.23 [a]	47.57 [a]	47.20 [a]	
Composite Tetric N-Ceram				
P1	22.4±0.8	22.2±0.7	21.6±0.6	22.1 [B]
P2	20.6±1.5	20.1±0.5	20.4±0.6	20.4 [B]
P3	27.4±1.0	27.1±1.2	27.1±0.6	27.2 [A]
Mean	23.47 [a]	23.13 [a]	23.03 [a]	
Composite Empress Direct				
P1	34.5±0.9	36.3±1.4	35.7±2.4	35.5 [B]
P2	33.3±1.6	32.9±1.6	31.8±3.2	32.7 [B]
P3	43.4±3.1	44.1±1.6	44.2±3.8	43.9 [A]
Mean	37.07 [a]	37.77 [a]	37.23 [a]	

Abbreviations: P1, phase 1; P2, phase 2; P3, phase 3.
[a] Distinct superscript letters indicate statistically significant differences. Capital letters refer to differences between lines, and lowercase letters refer to the differences between columns.

Table 4: *Mean Values and Standard Deviations of Surface Roughness (μm)*[a]

	OG (OneGloss)	TG (TopGloss)	SL (Sof-Lex)	Mean
Composite Z350 XT				
P1	1.21 ± 0.90	0.86 ± 0.31	1.12 ± 0.94	1.06 [A]
P2	1.26 ± 0.71	0.52 ± 0.22	0.60 ± 0.48	0.79 [B]
P3	1.06 ± 0.34	0.35 ± 0.12	0.72 ± 0.46	0.71 [B]
Mean	1.18 [a]	0.58 [a]	0.81 [a]	
Composite Tetric N-Ceram				
P1	0.82 ± 0.48	0.89 ± 0.24	0.70 ± 0.37	0.80 [A]
P2	1.28 ± 0.46	0.70 ± 0.21	0.51 ± 0.23	0.83 [A]
P3	1.12 ± 0.24	0.51 ± 0.17	0.55 ± 0.28	0.73 [A]
Mean	1.07 [a]	0.70 [b]	0.59 [b]	
Composite Empress Direct				
P1	1.29 ± 0.71	0.89 ± 0.48	0.69 ± 0.60	0.96 [A]
P2	1.54 ± 0.58	0.92 ± 0.81	0.48 ± 0.23	0.98 [A]
P3	1.55 ± 0.55	0.95 ± 0.54	0.60 ± 0.33	1.03 [A]
Mean	1.46 [a]	0.92 [b]	0.59 [b]	

Abbreviations: P1, phase 1; P2, phase 2; P3, phase 3.
[a] Distinct superscript letters indicate statistically significant differences. Capital letters refer to differences between lines, and lowercase letters refer to the differences between columns.

The regression analysis showed no correlation between light transmittance and the surface roughness for the three materials tested: Z350 XT (p=0.538), Tetric N-Ceram (p=0.334), and Empress Direct (p=0.875).

DISCUSSION

The hypothesis that the different finishing and polishing systems tested in this study would influence the total transmittance of the composite resins studied was rejected because regardless of the type of composite resin used (nanohybrid or nanofilled), all polishing systems resulted in the same level of light transmission. According to Lee,[11] one of the main components of a dental composite resin that is able to significantly affect its translucency is the inorganic filler. Therefore, it is possible to infer that the small variation in the inorganic content of the three composite resins used (52% to 59% in volume) might have been responsible in part for the similarity observed in terms of optical behavior as a function of the polishing system. These results have a clinical impact, since they indicate that the clinician can choose any of the tested finishing and polishing systems without compromising the light transmission of the final restoration. In addition, the clinician may choose the most appropriate polisher geometry to polish the different anatomic regions of the restoration.

The hypothesis that the different measurement phases could affect the transmittance of the specimens was accepted. In the case of Z350 XT, the highest transmittance observed for the first measurement (control, P1) in relation to the second measurement (after polishing, P2) is probably due to the use of the Mylar strip during polymerization of the material. The use of such a strip results in an extremely glossy surface, with reduced light scattering. For this material there was also a significant reduction in roughness after P2, which appears to be related to the lower transmittance observed after the polishing procedure.

For Tetric N-Ceram and Empress Direct, the transmittance was not affected by the polishing procedure. This finding may be explained by the fact that the microstructures of these two composite resins are significantly different from that of Z350 XT. While the microstructure of Z350 XT consists exclusively of nanoparticles of silica (20 nm) and zirconia (4 to 11 nm), Tetric N-Ceram and Empress Direct contain glass and barium fillers and are considered nanohybrid composites, with particle sizes ranging from 40 nm up to 3 μm. A previous study[10] demonstrated that direct transmittance of composite resins strongly depends on the composition of the material, particularly the type and size of inorganic particles. Furthermore, it is known that the difference between the refractive index of the

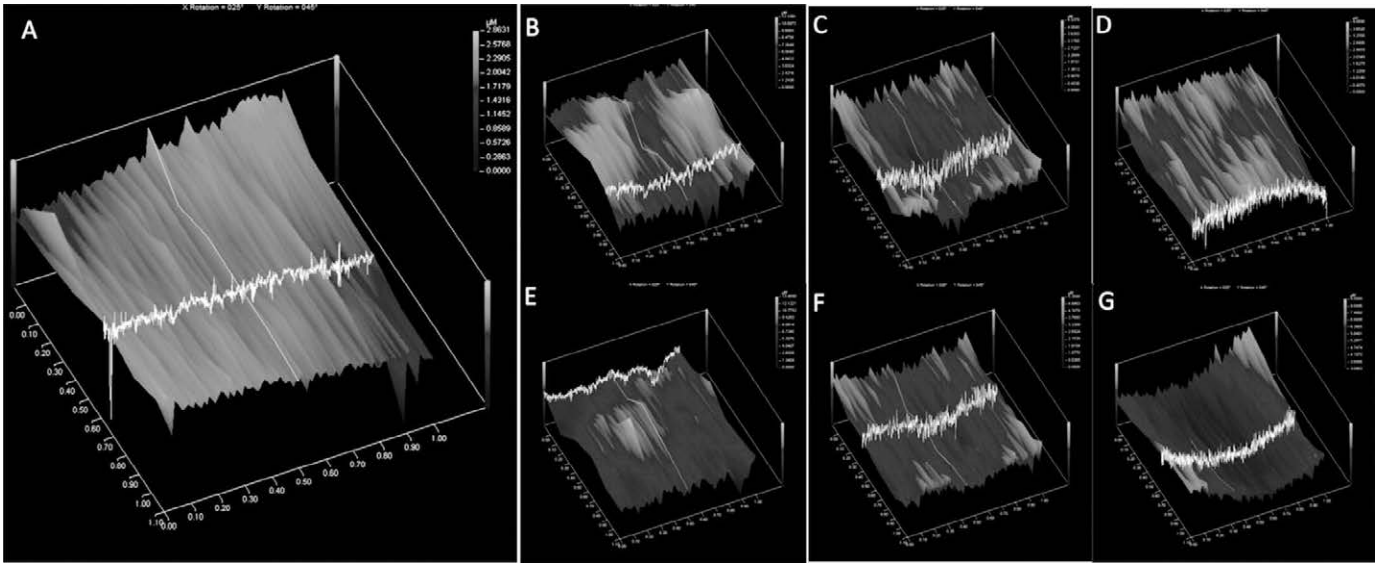


Figure 2. *Representative roughness images from composite Tetric N-Ceram: (A) Phase 1—Surface smoothness promoted by Mylar strip. (B, C, D) Phase 2—Surface smoothness promoted by different finishing and polishing systems: OneGloss, TopGloss, and Sof-Lex, respectively. (E, F, G) Phase 3—Surface smoothness after tooth brushing of the specimens.*

organic matrix and that of the filler influences the final optical properties of the composite resins.[18]

After toothbrush abrasion (P3), all composite resins tested showed significant increases in transmittance. A decrease in transmittance was expected after toothbrush abrasion because theoretically this type of abrasion adds scratches and irregularities to the surface, resulting in greater light scattering and thus lower transmittance. In fact, a previous study[12] showed that resin specimens aged in water had their transmittance reduced as the result of an increase in their opacity. Considering that the toothbrush abrasion carried out in the current investigation is a type of aging protocol, one could expect that the same increase in opacity would occur for the composite resins tested. One possible explanation for the observed increase in transmittance after brushing is the reduction of approximately 1 μm in the thickness of the specimens after they were submitted to the brushing protocol. There is a strong

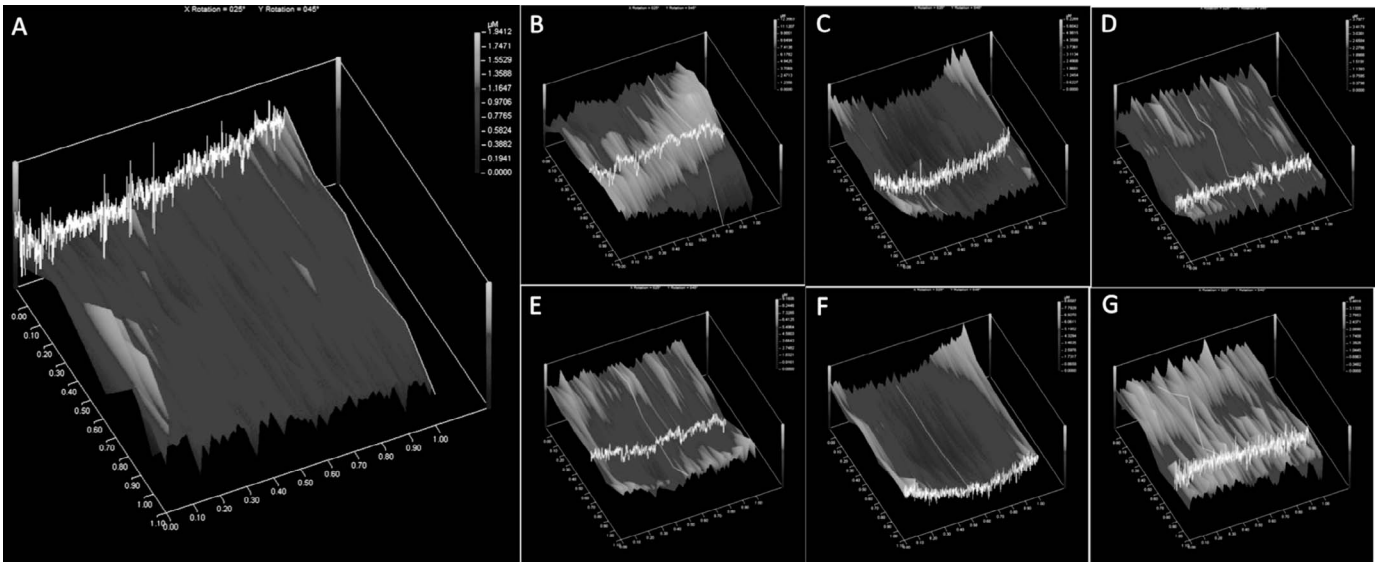


Figure 3. *Representative roughness images from composite Empress Direct: (A) Phase 1—Surface smoothness promoted by Mylar strip. (B, C, D) Phase 2—Surface smoothness promoted by different finishing and polishing systems: OneGloss, TopGloss, and Sof-Lex, respectively. (E, F, G) Phase 3—Surface smoothness after tooth brushing of the specimens.*

correlation between the thickness of the specimen and its transmittance, such that the greater the thickness, the lower the transmittance of light due to the increased scattering within the material structure.[19] From a clinical point of view, these results suggest that with time the daily brushing procedure may result in increased translucency of resin composite restorations, which may represent an undesirable shade mismatch for the restoration. To minimize this problem, a layering technique can be used to produce the restoration. In this way, more opaque colors may be used in deeper regions to decrease the impact of the increased translucency of the surface layer on the final shade of the restoration.[20]

Regarding roughness data, it was observed that the Z350 XT resin was not affected by the type of polishing system. As mentioned earlier, the microstructure of this material contains nanometric particles and clusters that responded similarly to the action of the different polishing systems used in this study. The literature[21,22] showed that the ability to obtain smoother surfaces in composite resins is related to the filler size.

The interaction of the composite resins Tetric N-Ceram and Empress Direct with the polishing system OneGloss resulted in greater surface roughness values compared to the other two polishing systems. OneGloss is composed of a tip impregnated with aluminum oxide particles. These results are in agreement with those of other studies[23,24] indicating that aluminum oxide disk systems promote smoother surfaces compared to impregnated abrasive tips. The duration of the finishing/polishing procedures may justify the higher surface roughness values observed after application of OneGloss. This system is applied to the material surface for only 15 seconds, while the Sof-Lex system, which has a similar composition, is applied for total time of 60 seconds as a result of the sequential application of disks with different abrasiveness. Sof-Lex and TopGloss systems resulted in similar roughness results for Empress Direct and Tetric N-Ceram, which is in accordance with the findings of other studies[21,25] showing that silicon diamond tips can achieve surface roughness results similar to those obtained with disks.

The results of this study indicate that there was no correlation between surface roughness and light transmittance for the materials studied. This result may be related to the fact that the specimens used in the current investigation were relatively thick (2 mm). Based on the results of this study, it is possible to conclude that knowing the microstructure of the composite resin restorative materials and the features of the polishing system is key to predicting the behavior of the restoration in terms of transmittance and roughness. The surface roughness of the restorative materials tested in this study was not affected by tooth brushing abrasion, which may be considered a positive result from the clinical standpoint.

CONCLUSIONS

Based on the results of this study, it can be concluded that

1. Regardless of the type of composite resin used (nanohybrid or nanofilled), all polishing systems resulted in the same final level of light transmission;
2. On the other hand, both polishing and toothbrush abrasion affected the transmittance of the composite resins evaluated; however, these results varied according to the composite microstructure;
3. Neither nanohybrid composite had its roughness affected by polishing or tooth brushing;
4. However, for the nanofilled composite, roughness significantly decreased after polishing and was kept at the same level after tooth brushing.

Conflict of Interest Statement

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 6 April 2016)

REFERENCES

1. Lu H, Roeder LB, Lei L, & Powers JM (2005) Effect of surface roughness on stain resistance of dental resin composites Journal of Esthetic and Restorative Dentistry **17(2)** 102-108.
2. Reis AF, Giannini M, Lovadino JR, & Ambrosano GM (2003) Effects of various finishing systems on the surface roughness and staining susceptibility of packable composite resins Dental Materials **19(1)** 12-18.
3. Türkün LS, & Türkün M (2004) The effect of one-step polishing system on the surface roughness of three esthetic resin composite materials Operative Dentistry **29(2)** 203-211.
4. Jefferies SR (1998) The art and science of abrasive finishing and polishing in restorative dentistry Dental Clinics of North America **42(4)** 613-627.
5. Ferracane JL (2001) Resin composite—State of the art Dental Materials **27(1)** 29-38.
6. Senawongse P, & Pongprueksa P (2007) Surface roughness of nanofill and nanohybrid resin composites after

polishing and brushing Journal of Esthetic and Restorative Dentistry **19(5)** 265-273; discussion 274-275.

7. Joiner A (2004) Tooth colour: A review of the literature Journal of Dentistry **32(Supplement 1)** 3-12.
8. Nakajima M, Arimoto A, Prasansuttiporn T, Thanatvarakorn O, Foxton RM, & Tagami J (2012) Light transmission characteristics of dentine and resin composites with different thickness Journal of Dentistry **40(Supplement 2)** 77-82.
9. Li R, Ma X, Liang S, Sa Y, Jiang T, & Wang Y (2012) Optical properties of enamel and translucent composites by diffuse reflectance measurements Journal of Dentistry **40(Supplement 1)** 40-47.
10. Masotti AS, Onofrio AB, Conceição EN, & Spohr AM (2007) UV-vis spectrophotometric direct transmittance analysis of composite resins Dental Materials **23(6)** 724-730.
11. Lee YK (2008) Influence of filler on the difference between the transmitted and reflected colors of experimental resin composites Dental Materials **24(9)** 1243-1247.
12. Kaizer Mda R, Diesel PG, Mallmann A, & Jacques LB (2012) Ageing of silorane-based and methacrylate-based composite resins: Effects on translucency Journal of Dentistry **40(Supplement 1)** 64-71.
13. Kielbassa AM, Gillman L, Zantner C, Meyer-Lueckel H, Helling E, & Schulte-Monting J (2005) Profilometric and microradiographic studies on the effects of toothpaste and acidic gel abrasivity on sound and demineralized bovine dental enamel Caries Research **39(5)** 380-386.
14. Takahashi R, Jin J, Nikaido T, Tagami J, Hickel R, & Kunzelmann KH (2013) Surface characterization of current composites after toothbrush abrasion Dental Materials **32(1)** 75-82.
15. Yip KH, Smales RJ, & Kaidonis JA (2004) Differential wear of teeth and restorative materials: Clinical implications International Journal of Prosthodontics **17(3)** 350-356.
16. Roselino L de M, Cruvinel DR, Chinelatti MA, & Pires-de-Souza F de C (2013) Effect of brushing and accelerated ageing on color stability and surface roughness of composites Journal of Dentistry **41(Supplement 5)** 54-61.
17. da Silva EM, de Sá Rodrigues CU, Dias DA, da Silva S, Amaral CM, & Guimarães JG (2014) Effect of toothbrushing-mouthrinse-cycling on surface roughness and topography of nanofilled, microfilled, and microhybrid resin composites Operative Dentistry **39(5)** 521-529.
18. Hirabayashi S, & Hirasawa T (1990) Improvements to light transmittance in light-cured composite resins by the utilization of low refractive index dimethacrylates Dental Materials Journal **9(2)** 203-214.
19. Antonson SA, & Anusavice KJ (2001) Contrast ratio of veneering and core ceramics as a function of thickness International Journal of Prosthodontics **14(4)** 316-320.
20. Ikeda T, Murata Y, & Sano H (2004) Translucency of opaque-shade resin composites American Journal of Dentistry **17(2)** 127-130.
21. Gönülol N, & Yilmaz F (2012) The effects of finishing and polishing techniques on surface roughness and color stability of nanocomposites Journal of Dentistry **40(Supplement 2)** 64-70.
22. Salgado VE, Cavalcante LM, Silikas N, & Schneider LF (2013) The influence of nanoscale inorganic content over optical and surface properties of model composites Journal of Dentistry **41(Supplement 5)** 45-53.
23. Barbosa SH, Zanata RL, Navarro MF, & Nunes OB (2005) Effect of different finishing and polishing techniques on the surface roughness of microfilled, hybrid and packable composite resins Brazilian Dental Journal **16(1)** 39-44.
24. Venturini D, Cenci MS, Demarco FF, Camacho GB, & Powers JM (2006) Effect of polishing techniques and time on surface roughness, hardness and microleakage of resin composite restorations Operative Dentistry **31(1)** 11-17.
25. Gedik R1, Hürmüzlü F, Coşkun A, Bektaş OO, & Ozdemir AK (2005) Surface roughness of new microhybrid resin-based composites Journal of the American Dental Association **136(8)** 1106-1012.

©Operative Dentistry, 2016, **41-6,** 642-654

Bacterial Colonization in the Marginal Region of Ceramic Restorations: Effects of Different Cement Removal Methods and Polishing

SMB Pereira • LC Anami • CA Pereira • ROA Souza • KZ Kantorski
MA Bottino • AOC Jorge • LF Valandro

Clinical Relevance

The polishing of the ceramic-dentin marginal region rather than excess cement removal techniques reduces significantly the surface roughness. This procedure has an effect in bacterial adhesion, especially when the excess cement is removed with a microbrush.

SUMMARY

This study evaluated the effects of excess cement removal techniques, with or without subsequent polishing, on biofilm formation and micromorphology in the marginal region of the tooth/restoration. From bovine teeth, 96 dentin blocks (4 × 8 × 2 mm) were produced, molded, and reproduced in type IV gypsum, on which 96 pressed ceramic blocks (Vita PM9, Vita Zahnfabrik; 4 × 8 × 2 mm) were produced via the lost wax technique. The dentin blocks and their respective ceramic blocks were cemented with a self-adhesive resin cement (RelyX U200, 3M ESPE), and cement excess was removed from the margin using four different

Sarina Maciel Braga Pereira, DDS, MSc, PhD, Graduate Program in Restorative Dentistry (Prosthodontic), Univ. Estadual Paulista - UNESP, São José dos Campos, Brazil

Lilian Costa Anami, DDS, MSc, PhD, Graduate Program in Restorative Dentistry (Prosthodontic), Univ Estadual Paulista - UNESP, São José dos Campos, Brazil

Cristiane Aparecida Pereira, MSc, PhD, Department of Biosciences and Oral Diagnosis, Univ Estadual Paulista - UNESP, São José dos Campos, Brazil

Rodrigo Othavio Souza, DDS, MSc, PhD, adjunct professor, Department of Restorative Dentistry (Prosthodontics), Federal University of Rio Grande do Norte, Natal, Brazil

Karla Zanini Kantorski, DDS, MSc, PhD, adjunct professor, Federal University of Santa Maria, Department of Stomatology (Periodontology), Santa Maria, Brazil

Marco Antonio Bottino, DDS, MSc, PhD, professor, Department of Dental Materials and Prosthodontics, Univ Estadual Paulista - UNESP, São José dos Campos, Brazil

Antonio Olavo Cardoso Jorge, DDS, MS, PhD, professor, Department of Biosciences and Oral Diagnosis, Univ Estadual Paulista - UNESP, São José dos Campos, Brazil

*Luiz Felipe Valandro, DDS, MSc, PhD, associate professor, Federal University of Santa Maria, Department of Restorative Dentistry (Prosthodontics), Santa Maria, Brazil

*Corresponding author: R. Floriano Peixoto 1184, Santa Maria, RS, Brazil, 97015-372; email: lfvalandro@hotmail.com

DOI: 10.2341/15-206-L

techniques, followed or not by polishing with silicone rubber tips: MBr, removal with microbrush and photoactivation; MBr-Pol, MBr + polishing; Br, removal with brush and photoactivation; Br-Pol, Br + polishing; Photo-Expl, 5 seconds of initial photoactivation, removal with explorer, and final curing; Photo-Expl-Pol, Photo-Expl + polishing; Photo-SB, 5 seconds of initial photoactivation, removal with scalpel, and final curing; and Photo-SB-Pol, Photo-SB + polishing. After 24 hours, the roughness in the marginal region was analyzed using a profilometer (three measurements on each sample). Micromorphological analyses of the region were performed by stereomicroscope and scanning electron microscopy (SEM). Then the samples were contaminated with sucrose broth standardized suspension with *Streptococcus mutans*, *Staphylococcus aureus*, and *Candida albicans* and incubated for a period of 48 hours. The samples were quantitatively analyzed for bacterial adherence in the marginal region by confocal laser scanning microscopy and counting of colony-forming units (CFUs/mL) and qualitatively analyzed using SEM. Roughness data (Ra) were submitted to two-way analysis of variance, Tukey test at a confidence level of 95%, and Student *t*-tests. CFU, biomass, and biothickness data were analyzed by Kruskal-Wallis, Mann-Whitney, and Dunn tests. The removing technique statistically influenced Ra (MBr, $p=0.0019$; Br, $p=0.002$; Photo-Expl, $p=0.0262$; Photo-SB, $p=0.0196$) when comparing the polished and unpolished groups. The MBr and MBr-Pol technique differed significantly for CFU/mL values ($p=0.010$). There was no significant difference in the amounts of biomass and biothickness comparing polished and unpolished groups and when all groups were compared ($p>0.05$). Different morphological patterns were observed (more regular surface for polished groups). We conclude that margin polishing after cementation of feldspar/pressed ceramic restorations is decisive for achieving smoother surfaces, as the excess cement around the edges can increase the surface roughness in these areas, influencing bacterial adhesion.

INTRODUCTION

In recent years, adhesively cemented ceramic restorations, such as inlays/onlays and veneers, have been used as the main approach for minimally invasive esthetic restorations in anterior and posterior teeth. However, its clinical success is related, among other factors, to the quality of tooth restoration.[1] Factors such as marginal misfit,[2] surface irregularities,[3] and cement excess[3,4] may favor the accumulation of microorganisms,[3] compromising restoration clinical longevity.

The cement retained on the tooth surface can promote plaque accumulation, leading to gingivitis and radicular surface demineralization.[4] In the case of the implant/prosthetic restoration interface, excess cement has a clinical impact, resulting in peri-implantation bone loss and bleeding on probing and resulting in implant loss.[5-11]

Excess cement on the tooth-restoration interface promotes bacteria adhesion due to the presence of surface irregularities.[3] Biofilm establishment in that area promotes periodontal tissues and peri-implant inflammation, compromising esthetics.[12,13] At the same time, biofilm can also promote a deleterious effect on the bond strength between the tooth and the restoration.[14-17]

Due to esthetic contingencies, preparation margins are commonly positioned at the intrasulcular level hindering access to proper excess cement removal. Consequently, a more favorable environment promoting the deleterious effects of biofilm can be seen.[18-25]

Considering the surface roughness effect on microbial adhesion and retention,[19,23,26-32] various excess cement removal protocols for tooth restoration[3,4,33] and implant-restoration interfaces[5-11] have been investigated. Anami and others[3] found that the morphology of the tooth-ceramic interface, according to different protocols of excess cement removal, influenced the *in vitro* adhesion of *Streptococcus mutans*.

However, polishing-associated evidence of different effects of cement removal methods on the morphology of ceramic restoration margins, roughness, and bacterial colonization is scarce. As is known, the polishing of ceramic and polymeric surfaces is performed for surface smoothing by light surface abrasion, which might impact bacterial adherence.

Considering the aforementioned premises, the research questions for this study were the following: Do different resin-cement excess removal techniques affect marginal region morphology and *in vitro* biofilm formation? Does marginal polishing have an effect on surface roughness and *in vitro* biofilm formation after excess cement removal?

Thus, the objective of this study was to assess whether the resin-cement excess removal methods in the marginal area between tooth and ceramic restorations, with or without polishing of the margin region, have an effect on surface roughness and *in vitro* biofilm formation.

METHODS AND MATERIALS

Obtaining Tooth Samples

Ninety-six dentin blocks were obtained from freshly extracted bovine incisors. The teeth were cleaned with periodontal curettes, Robinson brushes, and pumice. Crowns were removed under constant cooling using diamond discs (EDENTA, Au, Switzerland) mounted in a handpiece. The buccal surface was ground with silicon carbide paper (#100) adapted to a polishing device (METASERV 3000, Buehler, Lake Bluff, IL, USA) until an 8 × 4-mm area of dentin had been exposed. Then the dentin was cut into a block of 2-mm thickness. The smear layer was standardized by grinding with silicone carbide paper (#600) under constant cooling.[34,35] The final dimensions of 8 × 4 × 2 mm were verified with a digital caliper (Mitutoyo, São Paulo, Brazil). The blocks remained immersed in distilled water in an oven (Orion 502, Fanem, São Paulo, Brazil) at 37°C until the time of the cementation procedure.

Obtaining Ceramic Samples

In order to enable proper positioning of the ceramic blocks on the dentin surface during cementation, two 1-mm-deep cavities (guides) were made with a spherical diamond bur (EDENTA) on a 4 × 8-mm surface of each dentin block. These guides were made with an adapted device to standardize depth and the insertion axis.

The marked dentin surface of each specimen was molded with impression material (Express, 3M ESPE, St Paul, MN, USA) and a model in plaster type IV (Durone, Dentsply, Petrópolis, Brazil) was obtained on which a wax-up was performed for each specimen with dimensions similar to its respective dentin block. Prior to waxing, VITA In-Ceram interspace varnish (Vita Zahnfabrik, Bad Säckingen, Germany) was applied on dye surfaces, except in marginal areas, to facilitate fitting between blocks.

Ceramic blocks were produced by the lost-wax casting technique, followed by ceramic material injection. For this, the waxing was included in investment, and the ceramic (PM9, Vita Zahnfabrik) was pressure injected (Vita Vacumat 6000 MP, Vita Zanhfabrik) following the manufacturer's instruction. Ceramic samples were removed from coating with the aid of sandblasting.

Table 1: *Experimental Design, Considering Two Study Factors (Cement Removal Technique and Polishing: Presence/Absence) (n=12)*

Groups (Codes)	Study Factors	
	Cement Removal Technique	Polishing[a]
MBr	Microbrush:[b]	—
MBr-Pol	Excess removal with microbrush and photoactivation	With*
Br	Brush:[c]	—
Br-Pol	Excess removal with brush and photoactivation	With*
Photo-Expl	Photoactivation and explorer:	—
Photo-Expl-Pol	Photoactivation for 5 s, excess removal with explorer	With*
Photo-SB	Photoactivation and scalpel blade:	—
Photo-SB-Pol	Photoactivation for 5 s, excess removal with scalpel blade #12	With*

[a] *Polishing with silicon rubber tips (Shofu, San Marcos, CA, USA).*
[b] *Microbrush (KG Sorensen, Cotia, Brazil).*
[c] *Brush (Kota, Cotia, Brazil).*

The corresponding ceramic and dentin blocks were simultaneously polished with silicon carbide paper (#400, #600, #800, #1200, #1500) through a polishing machine (Metaserv 3000, Buehler) under constant cooling. The cementation surfaces of both blocks were not polished. All ceramic blocks were submitted to ultrasonic cleaning in isopropyl alcohol for five minutes (Vitasonic, Vita Zanhfabrik).

Ceramic and tooth blocks were analyzed by a stereomicroscope (10×/50×, Discovery V20, Zeiss, Göttingen, Germany) to ensure the absence of bubbles. Ninety-six sets of ceramic/dentin samples were counted and randomly distributed into eight groups (Table 1) using a specific Internet-based tool (www.randomizer.org), considering two study factors (n=12): excess cement removal technique (in four levels) and polishing (in two levels). The experimental unit of this study was the ceramic/dentin interface.

Cementation of Samples

Dentin blocks were cleaned with pumice and water, and excess water was removed with air jets without drying of the dentin. Ceramic blocks were cleansed with isopropyl alcohol ultrasonic bath for 480 seconds; surfaces were etched with 10% hydrofluoric acid for 60 seconds, washed, and air jet dried. Silane agent (Ceramic Primer, 3M/ESPE) was applied and air jet dried for five seconds.

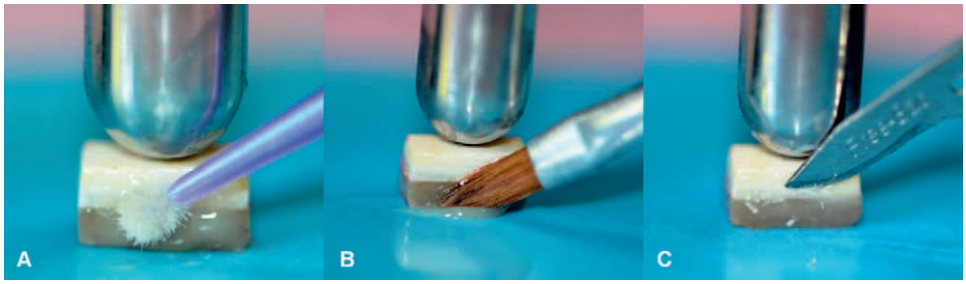


Figure 1. *Photos show cement removal using microbrush (A), brush (B), and blade (C) from the marginal region.*

Self-adhesive resin cement (RelyX U200, 3M ESPE) was used according to the manufacturer's recommendations. The cement was dispensed and applied with the aid of a plastic spatula on the ceramic surface, and it was positioned on its respective dentin block. A 750-g vertical load was used to correctly position the blocks during curing. The excess of resin-cement was removed according to the techniques described in Table 1 (Figure 1).

Microbrushes and brushes were passed through the marginal region in one direction to remove the remaining excess cement (one microbrush/brush for each specimen). The brush was perpendicularly positioned to the cementation line to prevent the input of bristles onto the cementation line and the formation of grooves in the region. The explorer and scalpel blade were used in one lever movement to displace excess cement. Polishing was carried out in dry conditions.

Each side of the set was photoactivated for 40 seconds, and after five minutes at room temperature, the cemented blocks were stored in distilled water at 37°C for 24 hours.

Surface Roughness

Quantitative analysis of surface roughness was performed with a contact profilometer (SJ 400, Mitutoyo, Tokyo, Japan). The samples were positioned with the interfacial area, perpendicular to the tip, which performed three measurements (about 1-mm distance between measurements) following a 3-mm path on each sample. Mean values of roughness arithmetic average (Ra, in μm) were obtained for each sample.

Sterilization

All samples were sterilized by gamma radiation. For this, they were individually wrapped in hot sealed surgical-type paper. The Embrarad Company (Cotia, Brazil) is authorized by ANVISA (Brazilian Health Surveillance Agency, São Paulo, Brazil) and ISO 9001 certified and performed the sterilization process with a gamma radiation camera (cobalt 60) at 20 kGy for six hours.

Morphological Analysis by Scanning Electron Microscopy

One sample of each group was evaluated by scanning electron microscopy (SEM) under 300× magnification (Inspect S50, FEI Company, Brno, Czech Republic) for marginal quality characterization. The samples were fixed in the sampler with a double-sided carbon adhesive tape (SPI, West Chester, PA, USA). The top surface was coated with gold-palladium alloy (Polaron SC 7620 Sputter Coater, Quorum Technologies, Newhaven, UK) (time: 130 s; 10-15 mA current; 130 mTorr vacuum; plating rate: 3.5 nm/min; Pd-Au layer: about 80 A). The SEM was operated at 20 kV.

Biofilm Analysis

Biofilms were formed according to the following methodology: standardized suspensions were prepared using standard strains of *Candida albicans* (ATCC 18804), *Staphylococcus aureus* (ATCC 6538), and *S. mutans* (ATCC 35688). The *C. albicans* strains were subcultured onto Sabouraud dextrose agar (Difco, Detroit, MI, USA). The strains of *S. aureus* and *S. mutans* were picked in brain heart infusion agar (BHI) (Brain Heart Infusion, Difco). The microorganisms were incubated in a bacteriological incubator at 37°C for 24 hours. All tests with *S. mutans* strains were incubated in a bacteriological oven under microaerophilic conditions (5% CO_2). After the incubation period, microorganism colonies were suspended in sterile saline solution (0.9% NaCl) and set in a spectrophotometer (B 582, Micronal, São Paulo, Brazil) for obtaining a standardized suspension containing 10^6 cells/mL. The parameters of optical density and wavelength were, respectively, 0.284 and 530 nm for *C. albicans*, 0.374 and 490 nm for *S. aureus*, and 0.620 and 398 nm for *S. mutans*.

Table 2: *Mean, Standard Deviation, Confidence Interval, Tukey Test Groups, and Student* t*-Test Results for Ra Data (μm) for the Experimental Groups*[a]

Cement Removal Technique	Ra (μm)		
	Polishing		*p*-Value[d]
	Absence[b]	Presence (Pol)[c]	
MBr	4.5 ± 2.2 (2.90-6.16) a	1.8 ± 0.6 (1.37-2.26) A	0.0019
Br	4.6 ± 1.7 (3.38-5.92) a	1.7 ± 0.7 (1.27-2.31) A	0.0002
Photo-Expl	3.9 ± 1.2 (3.12-4.86) ab	2.5 ± 1.3 (1.63-3.56) A	0.0262
Photo-SB	2.7 ± 1.0 (2.06-3.50) bc	1.8 ± 0.4 (1.55-2.21) A	0.0196

[a] *One-way analysis of variance and Tukey tests.*
[b] *Different lowercase letters indicate statistically significant differences ($p<0.05$) in the absence of polishing.*
[c] *Different capital letters indicate statistically significant differences ($p<0.05$) in the presence of polishing.*
[d] *Student* t*-test; $p<0.05$ indicates statistically different means.*

For biofilm formation, the broth proposed by Gybbons and Nygaard[36] was used, which is composed of 20 g trypticase, 2 g NaCl, 3 g K_2HPO_4, 2 g KH_2PO_4, 1 g K_2CO_3, 120 mg $MgSO_4$, 15 mg $MnSO_4$, and 50 g $C_6H_8O_7$ g, dissolved in 1000 mL of distilled water. The broth was sterilized by autoclaving at 121°C for 15 minutes. The sterilized specimens were placed in the first row of 24-well plates (Costar Corning, Corning, NY, USA) with the aid of a sterile tweezers, and 0.1 mL of each microbial suspension was inoculated in each well of the plate for the formation of the multispecies (heterotypic) biofilm. The plates were incubated in a bacteriological incubator maintained at 37°C for 48 hours.

CFU Counting (CFU/mL)—In order to remove the nonadherent microbial cells, 2 mL of broth were substituted by 2 mL of sterile saline solution in each well, and the plate was stirred for five minutes with an orbital shaker (Solab, Piracicaba, Brazil).

The CFU count was performed on 10 samples of each group. For this, specimens were individually placed in falcon tubes containing 10 mL of sterile saline solution and then homogenized for 30 seconds using a 50 W power ultrasonic homogenizer (Sonoplus HD 2200, Electronic Bandelin, Berlin, Germany). The obtained microbial suspension had 10^{-1} dilution factor, and new decimal dilutions were performed (10^{-2}, 10^{-3}, 10^{-4}, 10^{-6}) in sterile saline solution; 0.1-mL aliquots of each dilution were placed into duplicate Petri dishes with selective media for each microorganism as follows: 1) *C. albicans* in Sabouraud dextrose agar with 50 mg/L of chloramphenicol (Union Chemicals, São Paulo, Brazil), 2) *S. aureus* in NaCl BHI agar (Difco), and 3) *S. mutans* in Mitis Salivarius agar (Difco) plus 0.2 IU/mL of bacitracin (Chemical Union, São Paulo, Brazil) and 15% sucrose. The plates were incubated at 37°C for 48 hours, and the plates containing from 30 to 300 colonies were counted for the CFU number.

Since samples were contaminated by a biofilm composed of three microorganisms (*S. mutans*, *C. albicans*, and *S. aureus*), for each sample, the definitive CFU/mL value was obtained by summing each individual CFU/mL value. Moreover, due to data variation, CFU/mL values were converted into $\log_{10}$.

Biofilm Analysis Using SEM—Sixteen samples were fixed using the following protocol: immersion in glutaraldehyde solution at 2.5% (for 1 hour) and subsequent dehydration through a series of passages

Table 3: *Mean, Standard Deviation, Confidence Interval, Dunn Test Groups, and Mann-Whitney Test Results of CFU/mL $\log_{10}$ Values for the Experimental Groups*

Cement Removal Technique	CFU/mL $\log_{10}$		
	Polishing		*p*-Value[c]
	Absence[a]	Presence (Pol)[b]	
MBr	9.35 ± 0.19 (9.21-9.49) ab	9.16 ± 0.09 (9.09-9.24) A	0.010
Br	9.21 ± 0.17 (9.08-9.34) b	9.25 ± 0.11 (9.16-9.33) A	0.424
Photo-Expl	9.33 ± 0.12 (9.23-9.42) ab	9.30 ± 0.16 (9.18-9.41) AB	0.725
Photo-SB	9.57 ± 0.37 (9.31-9.84) a	9.46 ± 0.09 (9.39-9.53) B	0.896

[a] *Comparison between unpolished groups using Kruskal-Wallis and Dunn tests: different lowercase letters indicate statistically significant differences.*
[b] *Comparison between polished groups using the Kruskal-Wallis and Dunn tests: different capital letters indicate statistically significant differences.*
[c] *Comparison between absence and presence of polishing of each cement removal technique using Mann-Whitney test: $p<0.05$ indicates statistical difference.*

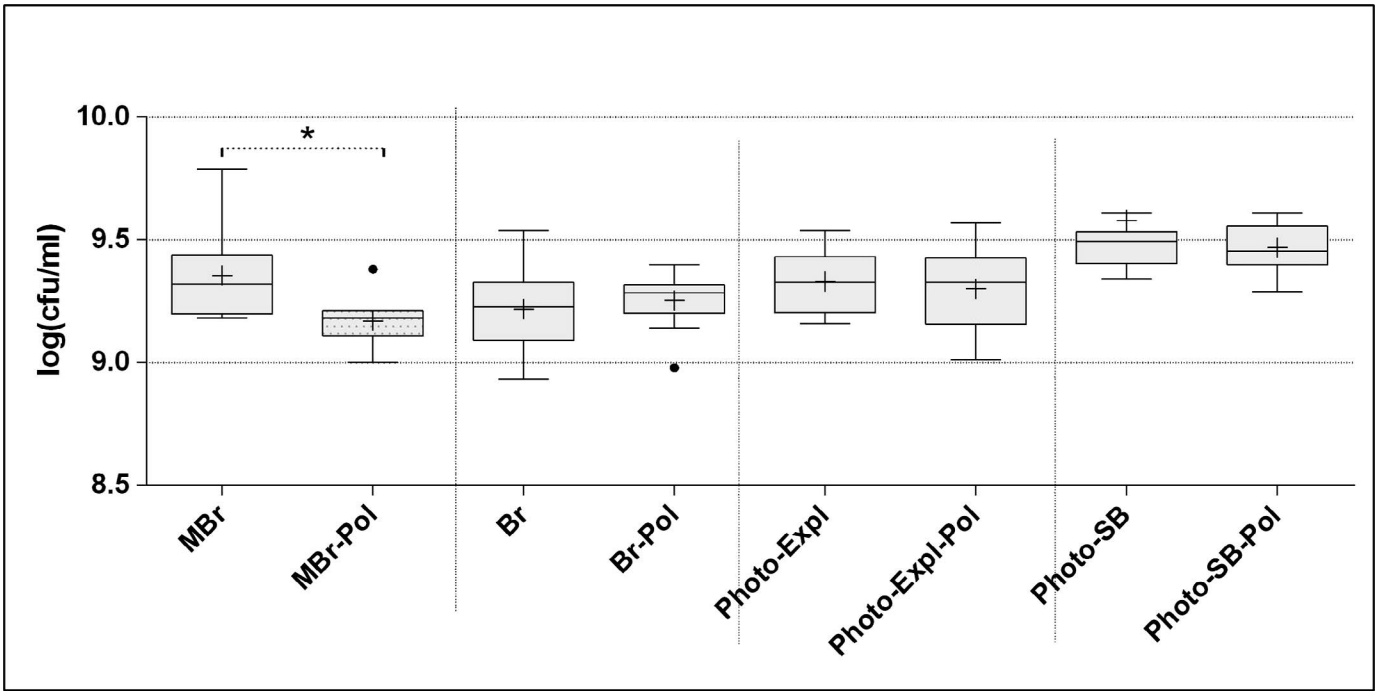


Figure 2. *Box-whisker plots of the CFU/mL log_{10} for the different experimental groups. The means are represented by +, and the medians are represented by the central line. The boxes represent the interquartile ranges. Groups that are statistically significant are denoted by * (Mann-Whitney test).*

with an ethanol concentration increase (50%, 70%, 80%, 90%, and 95% for 20 minutes and 100% for 1 hour).

Samples were gold coated, and SEM (4000×) was operated as previously described. A descriptive analysis of the material formed on the samples was performed.

Biofilm Analysis by Laser Scanning Confocal Microscopy—Ten samples from each experimental group were removed from the culture medium with heterotypic biofilm and then placed on a glass slide with the aid of a sterile tweezers. The Live/Dead Light BAC Bacterial Viability and Counting Kit (Molecular Probes, Eugene, OR, USA) was used for bacterial population viability monitoring, as described by Anami and others.[3]

The contaminated surface of each specimen was analyzed by laser scanning confocal microscopy (LSCM) (LSM 510 META, Zeiss). COMSTAT software was used to characterize the biofilm in terms of mean thickness (μm) and biovolume ($\mu m^3/\mu m^2$).

Data Analysis

After confirming normality assumptions, the roughness data were analyzed by two-way analysis of variance and Tukey and Student *t*-tests with a significance level of 95%. CFU/mL log_{10}, mean thickness, and biovolume were analyzed by nonparametric tests (Kruskal-Wallis, Dunn, and Mann-

Table 4: *Mean, Standard Deviation, Confidence Interval, and Mann-Whitney Test Results of Biovolume Values ($\mu m^3/\mu m^2$) for the Experimental Groups*

Cement Removal Technique	Biovolume		
	Polishing		*p*-Value[c]
	Absence[a]	Presence (Pol)[b]	
MBr	0.027 ± 0.030 (0.01-0.052)	0.021 ± 0.035 (−0.006-0.048)	*p*=0.3176
Br	0.006 ± 0.010 (−0.06-0.019)	0.023 ± 0.034 (−0.005-0.050)	*p*=0.4103
Photo-Expl	0.047 ± 0.095 (−0.05-0.14)	0.015 ± 0.027 (−0.007-0.037)	*p*=0.9038
Photo-SB	0.036 ± 0.063 (−0.00-0.76)	0.020 ± 0.024 (0.001-0.038)	*p*=0.7859

[a,b,c] *No statistically significant difference was detected.*

Table 5: *Mean, Standard Deviation, Confidence Interval, and Mann-Whitney Test Results of Biofilm Thickness Values (μm) for the Experimental Groups*

Cement Removal Technique	Biofilm Thickness		
	Polishing		*p*-Value[c]
	Absence[a]	Presence (Pol)[b]	
MBr	0.060 ± 0.080 (−0.001-0.120)	0.042 ± 0.074 (−0.011-0.094)	*p*=0.2179
Pi	0.003 ± 0.004 (−0.003-0.008)	0.050 ± 0.093 (−0.028-0.127)	*p*=0.6848
Photo-Expl	0.005 ± 0.005 (−0.000-0.009)	0.052 ± 0.092 (−0.025-0.128)	*p*=0.5869
Photo-SB	0.103 ± 0.166 (−0.015-0.221)	0.096 ± 0.171 (−0.035-0.227)	*p*=0.9047

[a,b,c] *No statistically significant difference was detected.*

Whitney) with significance level of 95% (Statistix 8.0, Analytical Software Inc., Tallahassee, FL, USA).

RESULTS

Surface Roughness

The mean and standard deviation data of surface roughness in the experimental groups are shown in Table 2.

Regardless of the cement removal technique, polishing reduced the surface roughness significantly (Table 2). In the absence of polishing, the photoactivation prior to cement removal reduced surface roughness values, particularly when the blade was used to remove the cement. No difference was observed between the cement removal techniques when the polishing was performed (Table 2).

CFU/mL $\log_{10}$

The mean and standard deviation data of CFU/mL $\log_{10}$ in experimental groups are shown in Table 3. Medians and interquartile ranges for CFU/mL $\log_{10}$ are shown in Figure 2.

In the absence of polishing, a statistically significant difference was observed only between Br and Photo-SB groups. When polishing was carried out, cement removal by microbrush or brush resulted in lower biofilm accumulation compared to the blade removal technique. The polishing reduced the counts of CFU/mL for the MBr group (MBr vs MBr-Pol) significantly but had no influence on the other excess cement removal techniques (Table 3).

Biovolume and Biofilm Thickness

The mean and standard deviation data of biovolume and biofilm thickness in the experimental groups are shown in Tables 4 and 5, respectively. Medians and interquartile ranges of biovolume and biofilm thickness data are shown in Figures 3 and 4, respectively.

Biovolume and biofilm thickness were not influenced by the applied cement removal technique

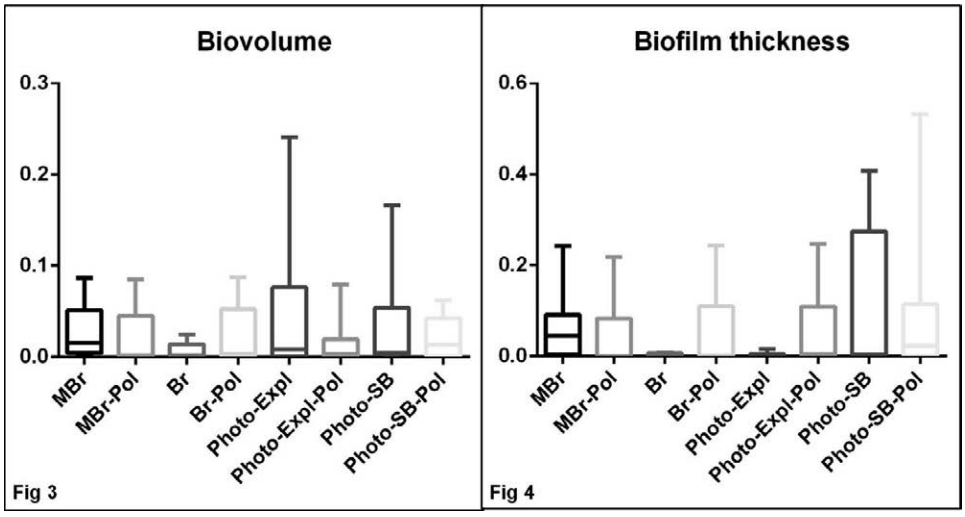


Figure 3. *Graph of median and interquartile ranges for biovolume values for experimental groups.*
Figure 4. *Graph of median and interquartile ranges for biofilm thickness values for experimental groups.*

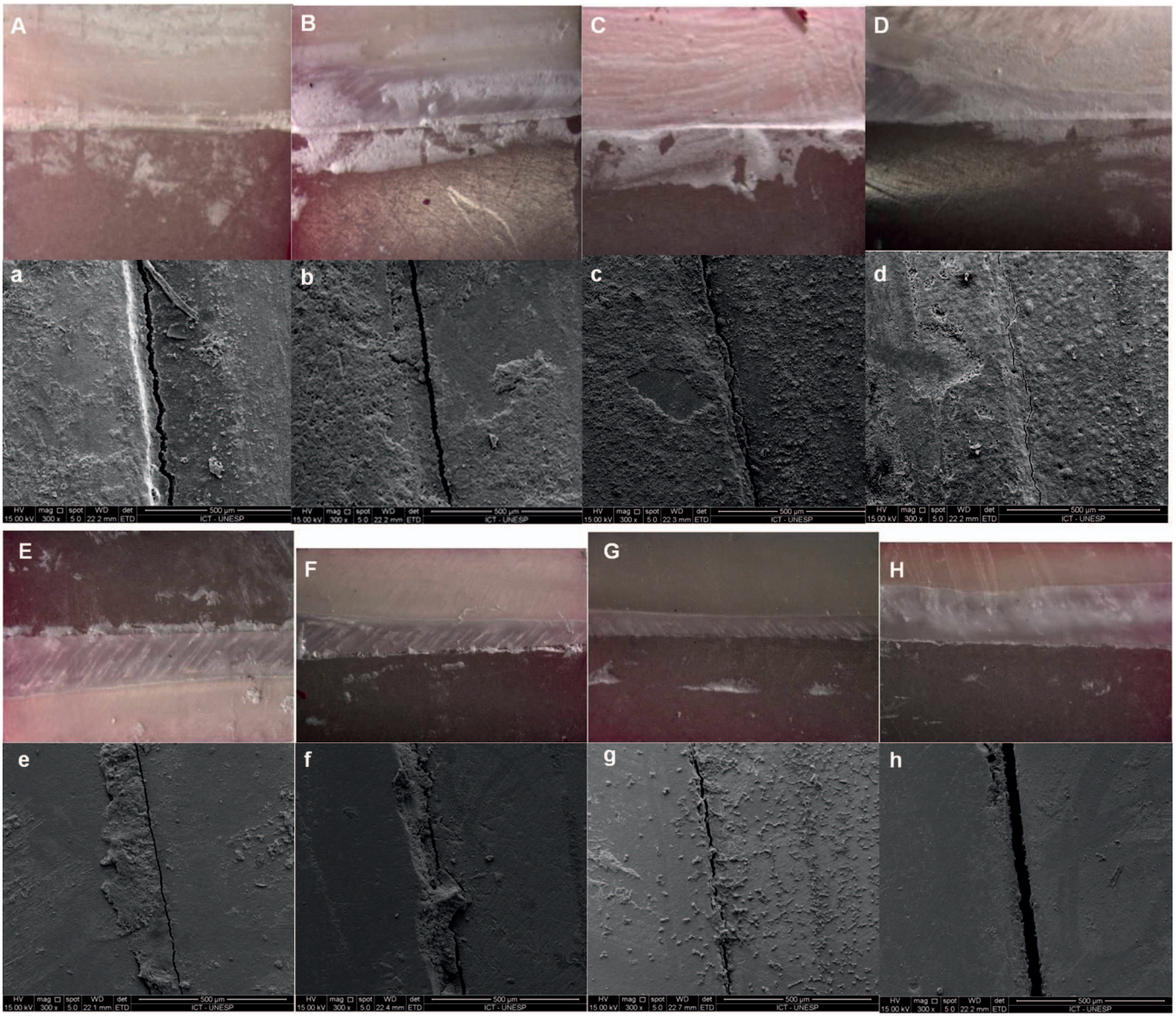


Figure 5. *Stereomicroscopic (A-H) (20×) and SEM (a-h) images (500×) of the marginal region for excess cement removal methods: MBr (A, a), MBr-Pol (B, b), Br (C, c), Br-Pol (D, d), Photo-Expl (E, e), Photo-Expl-Pol (F, f), Photo-SB (G, g), Photo-SB-Pol (H, h).*

(p=0.389 and p=0.249, respectively) or by the polishing (p=0.950 and p=0.723, respectively).

Micromorphological Analysis

The surface characteristics of the region/ceramic interface after the application of different excess cement removal techniques are presented in Figure 5.

The cement had overlaid the tooth and ceramic surface in the marginal region in all groups. In polished samples, the remaining cement appeared to be smoother and with a less rough appearance, especially in the MBr-Pol and Br-Pol groups compared with the unpolished MBr and Br groups (Figure 5Aa,Bb,Cc,Dd). In the margin, resulting from the Photo-Expl and Photo-SB techniques, small irregularities concentrated near the marginal line region were observed (Figure 5Ee,Gg). Grooves are still visible in the enamel (Figure 5H).

Biofilm Analysis by SEM

Figure 6 shows representative micrographs of the heterotypic biofilm on the samples. After 48 hours of *in vitro* biofilm formation, significant adhesion and the presence of colonies of *S. mutans*, *C. albicans*, and *S. aureus* were noted regarding the experimen-

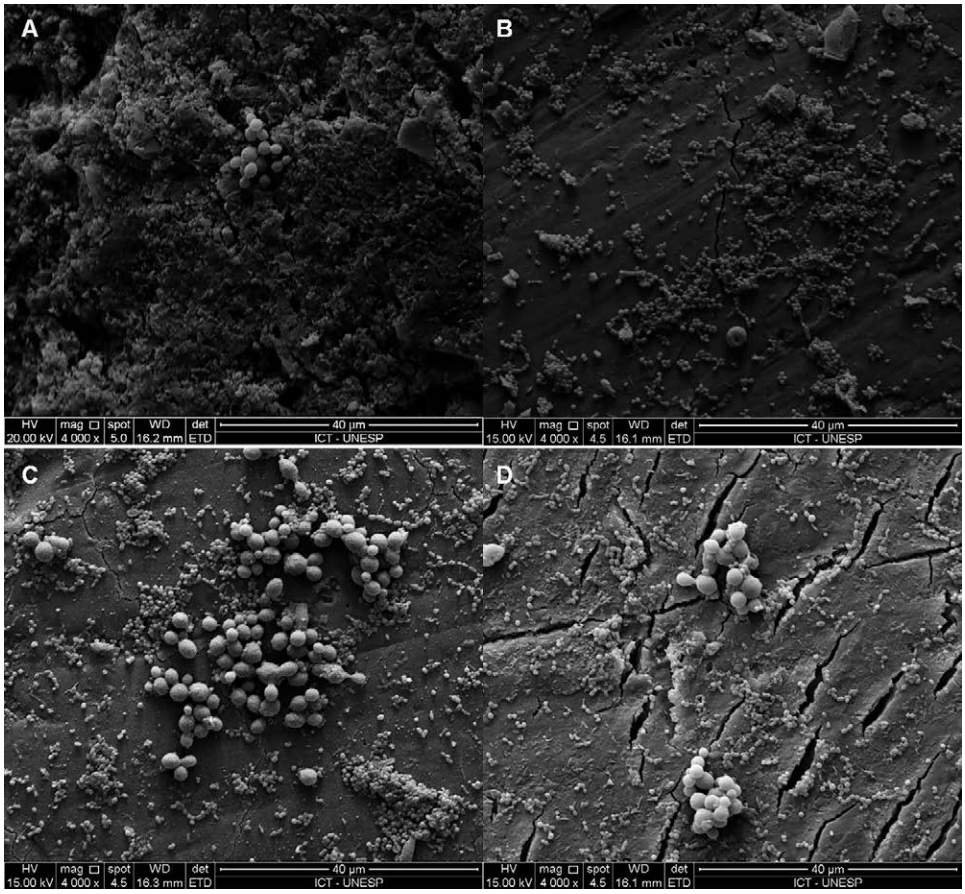


Figure 6. *Representative micrographs (4000×) of the biofilm formed on samples in the Pi-Pol (A), Pi- Pol (B), Photo-Expl-Pol (B), and Photo-Bi-Pol (C) groups.*

tal groups. The time of biofilm formation (48 hours) was not enough to recover the surface of specimens with microorganism colonies.

Biofilm Analysis by LSCM

Figure 7 shows representative LSCM images of the formed biofilm on samples. The three types of microorganisms were found (*S. mutans*, *S. aureus*, and *C. albicans*). Green points in the images correspond to "live" microorganisms and red points to "dead" microorganisms. In Figure 7A,B, it is possible to observe a balance between live and dead microorganisms. Figure 7C,D shows mainly dead microorganisms. These models of biofilm presentation were observed in all experimental groups.

DISCUSSION

This study showed that polishing ceramic and tooth marginal areas provides a significantly smoother surface regardless of the applied cement removal technique. Thus, polishing the margins after cementation is more important than the selected cement removal technique.

Considering the microbiological analysis, polishing significantly reduced the CFU/mL counts for the MBr group (MBr vs MBr-Pol) but had no influence on other excess cement removal techniques. In the LSCM analyses of biofilm thickness and biovolume, no difference was observed between groups despite polishing.

Taking into account the experiments without polishing, photoactivation prior to cement removal reduced surface roughness, particularly when the blade was used over the microbrush or brush. This result probably occurred because the prior five seconds of photoactivation provided partial cement polymerization and possibly facilitated explorer and blade excess removal before final photoactivation. This was evidenced by the stereomicroscope micrographs (Figure 5), through which it was observed that prior photoactivation, associated with explorer or blade to cement removal, resulted in a smoother surface, from a topographical point of view, in all tooth/cement/ceramic regions.

Considering the polishing experiments, the stereomicroscopic micrographs demonstrated that the

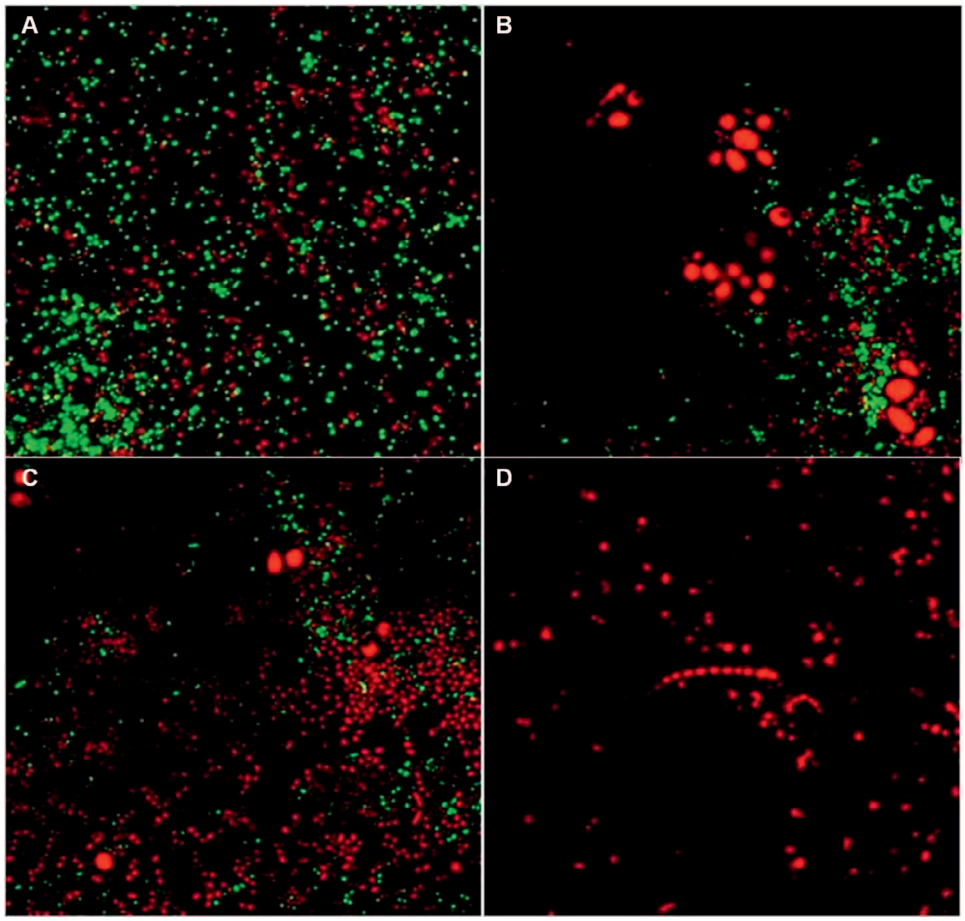


Figure 7. *Representative micrographs of the biofilm formed on the samples in Br-Pol (A) and Photo-Expl-Pol (B-D) groups; the presence of the three types of microorganisms can be verified.*

residual cement was present on the tooth and the ceramic for the microbrush or brush techniques. This did not occur when prior photoactivation was carried out. However, the subsequent polishing provided similar roughness between the techniques despite the presence of residual cement. Contrasting the results of this study, Anami and others[3] demonstrated that polishing, subsequent to cement removal, promoted an increase in surface roughness. The divergent findings may be related to the type of abrasive material for polishing, which can abrade the surface differently, making it rougher or smoother. We used the proper polishing silicone tips indicated for margin restoration.

In the microbial culture analysis, statistical differences were observed between the testing groups (Table 3). In the absence of polishing, the Br group presented a statistically lower number of the CFU/mL $\log_{10}$ than the SB group. However, the Br group showed higher surface roughness values than the SB group. This finding shows that other factors besides surface roughness affect bacterial adhesion.

Importantly, polishing significantly reduced roughness values but did not affect the reduction in microbial adhesion. This can be explained by the fact that biofilm formation time (48 hours) is a rather long time for the roughness effect on microbial adhesion to take place and because of other factors that have an influence on biofilm formation, apart from roughness, such as the material's surface free energy.[37] Undoubtedly, surface roughness has an effect on initial microorganism adherence. Irregularities increase the available adhesion area and protect bacteria from regulatory and control mechanisms of the oral microbiota.[37] Accordingly, biofilm may exhibit more rapid maturation in these areas.[37-40] In addition, evidence that microorganisms remain in surface irregularities despite tooth brushing were presented.[41] Thus, considering frequent subgingival positioning of the tooth-cement-ceramic margin and that microorganism retention in surface irregulari-

ties is greater, smoother margins may have a clinical impact favoring restoration longevity.

The LSCM images allied with the Constat and Matlab programs can be useful in the quantitative analysis[19,31,42] of biofilm formed on the material and provides, among other things, biovolume and biofilm thickness. For biovolume and biofilm thickness values, it was observed that cement removal techniques were statistically similar in both polishing conditions (absence and presence). When comparing the influence of polishing in each technique, no significant difference was observed. Using LSCM, Brentel and others[19] observed differences among the groups and positive correlations between surface roughness/biovolume and roughness/biofilm thickness.

From the SEM and LSCM qualitative analyses,[3,18,19,43] the presence of the three microorganisms (*S. mutans*, *S. aureus*, and *C. albicans*) showed similar behavior of adhesion for the different techniques.

In conclusion, a well-polished surface is important for biofilm formation and definitive esthetic results. Thus, after the excess cement removal technique, polishing has a great importance when considering the evaluated materials and techniques. Clinical trials and/or *in situ* studies could be conducted to assess the clinical behavior of various cement removal techniques and polishing conditions in the marginal region.

CONCLUSIONS

- Marginal surface polishing has a significant influence on surface roughness for all of the cement removal techniques.
- The microbrush polishing technique significantly reduced the CFU/mL $\log_{10}$ values compared with the unpolished condition, although it has not been relevant to other techniques.

Acknowledgement

CAPES (Brazil) Foundation supported this investigation. This article is based on a doctoral thesis submitted to the Graduate Program in Restorative Dentistry, Prosthodontic Unit, Faculty of Odontology, Institute of Science and Technology, Universidade Estadual Paulista—UNESP, São José dos Campos, Brazil, as part of the requirements for the PhD degree (Dr SMB Pereira).

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 3 March 2016)

REFERENCES

1. Liebenberg WH (1999) Interproximal integrity of posterior partial-coverage ceramic restorations: Sequence of delivery and finishing *Quintessence International* **30(12)** 807-818.
2. Cenci MS, Pereira-Cenci T, Cury JA, & Ten Cate JM (2009) Relationship between gap size and dentine secondary caries formation assessed in a microcosm biofilm model *Caries Research* **43(2)** 97-102.
3. Anami LC, Pereira CA, Guerra E, Souza ROA, Jorge AO, & Bottino MA (2012) Morphology and bacterial colonization of tooth/ceramic restoration interface after different cement excess removal techniques *Journal of Dentistry* **40(9)** 742-749.
4. Mansour YF, Pintado MR, & Mitchell CA (2006) Optimizing resin cement removal around esthetic crown margins *Acta Odontologica Scandinavica* **64(4)** 231-236.
5. Korsch M, Robra BP, & Walther W (2015) Cement-associated signs of inflammation: Retrospective analysis of the effect of excess cement on peri-implant tissue *International Journal of Prosthodontics* **28(1)** 11-8.
6. Korsch M, Marten SM, Dötsch A, Jáuregui R, Pieper DH, & Obst U (2015) Effect of dental cements on peri-implant microbial community: Comparison of the microbial communities inhabiting the peri-implant tissue when using different luting cements *Clinical Oral Implants Research* 1-6 In press.
7. Korsch M, Robra BP, & Walther W (2015) Predictors of excess cement and tissue response to fixed implant-supported dentures after cementation *Clinical Implant Dental Related Research* **17(Supplement 1)** e45-e53.
8. Korsch M, & Walther W (2015) Peri-implantitis associated with type of cement: A retrospective analysis of different types of cement and their clinical correlation to the peri-implant tissue *Clinical Implant Dental Related Research* **17(Supplement 2)** e434-443.
9. Korsch M, Obst U, & Walther W (2014) Cement-associated peri-implantitis: A retrospective clinical observational study of fixed implant-supported restorations using a methacrylate cement *Clinical Oral Implants Research* **25(7)** 797-802.
10. Korsch M, Walther W, Marten SM, & Obst U (2014) Microbial analysis of biofilms on cement surfaces: An investigation in cement-associated peri-implantitis *Journal Applied Biomaterials & Functional Materials* **12(2)** 70-80.
11. Wilson TG Jr (2009) The positive relationship between excess cement and peri-implant disease: A prospective clinical endoscopic study *Journal of Periodontology* **80(9)** 1388-1392.
12. Christensen RP, & Ploeger BJ (2010) A clinical comparison of zirconia, metal and alumina fixed-prosthesis frameworks veneered with layered or pressed ceramic: A three-year report *Journal American Dental Association* **141(11)** 1317-1329.

13. Lange RT, & Pfeiffer P (2009) Clinical evaluation of ceramic inlays compared to composite restorations *Operative Dentistry* **34(3)** 263-272.

14. Mutluay MM, Zhang K, Ryou H, Yahyazadehfar M, Majd H, Xu HH, & Arola D (2013) On the fatigue behavior of resin-dentin bonds after degradation by biofilm *Journal of Mechanical Behavior of Biomedical Materials* **18(February)** 219-231.

15. Schmidlin PR, Siebenmann J, Kocher P, Seemann R, Attin T, & Bindl A (2008) Effects of de- and remineralization of dentin on bond strengths yielded by one-, three-, and four-step adhesives *Journal of Adhesive Dentistry* **10(2)** 119-126.

16. Silva EM, Almeida GS, Poskus LT, & Guimaraes JG (2012) Influence of organic acids present in the oral biofilm on the microtensile bond strength of adhesive systems to human dentin *Journal of Biomedical Materials Research: Part B Applied Biomaterials* **100(3)** 735-741.

17. Zhang K, Cheng L, Imazato S, Antonucci JM, Lin NJ, Lin-Gibson S, Bai Y, & Xu HH (2013) Effects of dual antibacterial agents MDPB and nano-silver in primer on microcosm biofilm, cytotoxicity and dentine bond properties *Journal of Dentistry* **41(5)** 464-474.

18. Azevedo SM, Kantorski KZ, Valandro LF, Bottino MA, & Pavanelli CA (2012) Effect of brushing with conventional versus whitening dentifrices on surface roughness and biofilm formation of dental ceramics *General Dentistry* **60(3)** 123-130.

19. Brentel AS, Kantorski KZ, Valandro LF, Fucio SB, Puppin-Rontani RM, & Bottino MA (2011) Confocal laser microscopic analysis of biofilm on newer feldspar ceramic *Operative Dentistry* **36(1)** 43-51.

20. Carlen A, Nikdel K, Wennerberg A, Holmberg K, & Olsson J (2001) Surface characteristics and in vitro biofilm formation on glass ionomer and composite resin *Biomaterials* **22(5)** 481-487.

21. Frojd V, Chavez de Paz L, Andersson M, Wennerberg A, Davies JR, & Svensater G (2011) In situ analysis of multispecies biofilm formation on customized titanium surfaces *Molecular Oral Microbiology* **26(4)** 241-252.

22. 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. 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* **9(1)** 121-147.

23. Kantorski KZ, Scotti R, Valandro LF, Bottino MA, Koga-Ito CY, & Jorge AO (2009) Surface roughness and bacterial adherence to resin composites and ceramics *Oral Health Preventive Dentistry* **7(1)** 29-32.

24. Kawai K, Urano M, & Ebisu S (2000) Effect of surface roughness of porcelain on adhesion of bacteria and their synthesizing glucans *Journal of Prosthetic Dentistry* **83(6)** 664-667.

25. Lima FG, Romano AR, Correa MB, & Demarco FF (2009) Influence of microleakage, surface roughness and biofilm control on secondary caries formation around composite resin restorations: An in situ evaluation *Journal of Applied Oral Science* **17(1)** 61-65.

26. Aykent F, Yondem I, Ozyesil AG, Gunal SK, Avunduk MC, & Ozkan S (2010) Effect of different finishing techniques for restorative materials on surface roughness and bacterial adhesion *Journal of Prosthetic Dentistry* **103(4)** 221-227.

27. Al-Marzok MI, & Al-Azzawi HJ (2009) The effect of the surface roughness of porcelain on the adhesion of oral *Streptococcus mutans Journal of Contemporary Dental Practice* **10(6)** 017-024.

28. Kantorski KZ, Scotti R, Valandro LF, Bottino MA, Koga-Ito CY, & Jorge AO (2008) Adherence of *Streptococcus mutans* to uncoated and saliva-coated glass-ceramics and composites *General Dentistry* **56(7)** 740-747.

29. Scotti R, Zanini Kantorski K, Scotti N, Monaco C, Valandro LF, & Bottino MA (2006) Early biofilm colonization on polished- and glazed-zirconium ceramic surface: Preliminary results *Minerva Stomatology* **55(9)** 493-502.

30. Tanner J, Robinson C, Soderling E, & Vallittu P (2005) Early plaque formation on fibre-reinforced composites in vivo *Clinical Oral Investigations* **9(3)** 154-160.

31. Auschill TM, Arweiler NB, Brecx M, Reich E, Sculean A, & Netuschil L (2002) The effect of dental restorative materials on dental biofilm *European Journal of Oral Science* **110(1)** 48-53.

32. Rimondini L, Fare S, Brambilla E, Felloni A, Consonni C, Brossa F, & Carrassi A (1997) The effect of surface roughness on early in vivo plaque colonization on titanium *Journal of Periodontology* **68(6)** 556-562.

33. Akgüngör G, Aydin M, Sen D, & Issever H (2008) Clinical efficacy of the Vector system in excess cement removal during fixed prosthodontic treatment *Quintessence International* **39(4)** 139-146.

34. Burrow MF, Nopnakeepong U, & Phrukkanon S (2002) A comparison of microtensile bond strengths of several dentin bonding systems to primary and permanent dentin *Dental Materials* **18(3)** 239-245.

35. Ogata M, Harada N, Yamaguchi S, Nakajima M, & Tagami J (2002) Effect of self-etching primer vs phosphoric acid etchant on bonding to bur-prepared dentin *Operative Dentistry* **27(5)** 447-54.

36. Gybbons RJ, & Nygaard M (1968) Synthesis of insoluble dextran and its significance in the formation of gelatinous deposits by plaque-forming streptococci *Archives of Oral Biology* **13(10)** 1249-1262.

37. Quirynen M, Marechal M, Busscher HJ, Weerkamp AH, Darius PL, & van Steenberghe D (1990) The influence of surface free energy and surface roughness on early plaque formation: An in vivo study in man *Journal of Clinical Periodontology* **17(3)** 138-144.

38. Quirynen M, van der Mei HC, Bollen CM, Schotte A, Marechal M, Doornbusch GI, Naert I, Busscher HJ, & van

Steenberghe D (1993) An in vivo study of the influence of the surface roughness of implants on the microbiology of supra- and subgingival plaque *Journal of Dental Research* **72(9)** 1304-1309.

39. Gatewood RR, Cobb CM, & Killoy WJ (1993) Microbial colonization on natural tooth structure compared with smooth and plasma-sprayed dental implant surfaces *Clinical Oral Implants Research* **4(2)** 53-64.
40. Quirynen M, & Bollen CML (1995) The influence of surface roughness and surface-free energy on supra- and subgingival plaque formation in man. A review of the literature *Journal of Clinical Periodontology* **22(1)** 1-14.
41. Scotti R, Kantorski KZ, Monaco C, Valandro LF, Ciocca L, & Bottino MA (2007) SEM evaluation of in situ early bacterial colonization on a Y-TZP ceramic: A pilot study *International Journal of Prosthodontics* **20(4)** 419-422.
42. Staudt C, Horn H, Hempel DC, & Neu TR (2004) Volumetric measurements of bacterial cells and extracellular polymeric substance glycoconjugates in biofilms *Biotechnology and Bioengineering* **88(5)** 585-592.
43. Pereira SM, Kantorski KZ, Brentel AS, Valandro LF, & Bottino MA (2008) SEM analysis of the in situ early bacterial colonization on two novel feldspathic ceramics submitted to different types of glazing *Journal of Contemporary Dental Practice* **9(2)** 49-56.

©Operative Dentistry, 2016, **41-6,** 655-665

Filler Content, Surface Microhardness, and Rheological Properties of Various Flowable Resin Composites

S Jager • R Balthazard • A Dahoun • E Mortier

Clinical Relevance Statement

Using a flowable resin composite with high filler content, high initial surface microhardness, low viscosity, and good spreadability may be a satisfactory clinical compromise between mechanical properties and ease of use for the restoration of low-volume cavities.

SUMMARY

Objectives: The objectives of this study were to determine the filler content, the surface microhardness (at baseline and after immersion in water for 2 years), and the rheological properties of various flowable resin composites.

Methods: Three flowable resin composites (Grandioso Heavy Flow [GHF], Grandio Flow [GRF], Filtek Supreme XTE Flow [XTE]), one pit and fissure sealant resin composite (Clin-Pro [CLI]), and three experimental flowable resin composites with the same matrix and a variable filler content (EXPA, EXPB, EXPC) were tested. The filler content was determined by calcination. The Vickers surface microhardness was determined after polymerization and then after immersion in distilled water at 37°C for 7, 60, 180, 360, and 720 days. The rheological measurements were performed using a dynamic shear rheometer.

Results: The determined filler contents differed from the manufacturers' data for all the materials. The materials with the highest filler content presented the highest microhardness, but filler content did not appear to be the only influencing parameter. With respect to the values recorded after photopolymerization, the values were maintained or increased after 720 days compared with the initial microhardness values, except for GHF. For the values measured after immersion for 7 days, an increase in microhardness was observed for all the materials over time. All the materials were non-Newtonian, with shear-thinning behavior. At all the shear speeds, GRF presented a lower viscosity to GHF and XTE.

Conclusions: GRF presented a low viscosity before photopolymerization, associated with high filler content, thereby providing a good compromise between spreadability and mechanical properties after photopolymerization.

*Stéphanie Jager, DDS, PhD, Faculty of Dentistry, Department of Pediatric Dentistry, Nancy, France

Rémy Balthazard, DDS, PhD, Faculty of Dentistry, Department of Conservative Dentistry, Nancy, France

Abdesselam Dahoun, Professor, Si2M Department, UMR CNRS 7198, Jean Lamour Institute, Nancy, France

Eric Mortier, DDS, PhD, Faculty of Dentistry, Department of Conservative Dentistry, Nancy, France

*Corresponding author: 96 avenue de Lattre de Tassigny, Nancy, 54000, France; e-mail: stephaniejager999@gmail.com

DOI: 10.2341/16-031-L

INTRODUCTION

Preventive dentistry, adhesive dentistry, and ultraconservative dentistry have all developed significantly in recent decades and are now an integral part of the treatment arsenal available to practitioners. In the past 15 or so years, the emergence of flowable resin composites has further expanded the range of options available to dentists.[1,2] The relative ease of use of these low-viscosity resins, their capacity to spread and take shape in small occlusal or cervical cavities, as well as their ability to penetrate into pits and fissures mean that they have been adopted by numerous dental practitioners.[3-5] Inside the warm, moist environment of the mouth, materials which are subject to abrasion and numerous mechanical stresses, particularly flexion and compression, need to meet a broad range of specifications to ensure long-term clinical success. From this point of view, *in vitro* study of the physical and mechanical properties of materials is essential since it helps evaluate the capacity of materials to meet said specifications. One of the most useful properties to assess is the surface microhardness because this is closely correlated with resistance to compression and resistance to abrasion,[6-9] parameters that are particularly important, especially when the materials are used in low thicknesses, as is frequently the case for low-viscosity "flowable" resin composites. For the latter, it is important to specify that to make a resin composite flowable, manufacturers can primarily intervene on two levels. The first option consists of increasing the proportion of viscosity-lowering monomers to counter the high viscosity of high-molecular-weight monomers such as bisphenol A glycidyl dimethacrylate (Bis-GMA). These viscosity-lowering monomers are small, low molecular weight aliphatic monomers; triethylene glycol dimethacrylate (TEGDMA) is very frequently used for this purpose, for example. The second option consists of reducing the filler content in terms of volume compared with the matrix volume. Obviously, the two options can be employed simultaneously to varying degrees. These specific composition characteristics will therefore influence the initial viscosity of these resin composites, as well as their physical and mechanical properties once they are photopolymerized.

The literature available concerning the microhardness of resin composites is relatively abundant, but the study protocols used are highly variable, making comparison between them difficult.[10-15] In addition, to date, no study has described the evolution of microhardness after more than 6 months of immersion in a medium simulating the oral cavity, meaning that its long-term evolution has been little reported. Furthermore, the notion of resin viscosity remains somewhat vague, with the term *flowable* simply an indication used by manufacturers to cover a group of nonetheless very different materials.

The objectives of our study were as follows: 1) to determine the filler content of various flowable resin composites, 2) to determine the initial surface microhardness, 3) to monitor the evolution in surface microhardness following immersion in distilled water for 2 years, and finally, 4) to determine the rheological properties of various flowable resin composites. To perform this study, three commercially available flowable resin composites and one ultra-flowable resin pit and fissure sealant, along with three experimental flowable resin composites, were used.

METHODS AND MATERIALS

The compositions of the products used are presented in Table 1. Three flowable resin composites (Grandio Flow and GrandioSo Heavy Flow, Voco, Cuhaven, Germany; Filtek Supreme XTE flow, 3M ESPE Dental Products, St Paul, MN, USA), one ultra-flowable resin pit and fissure sealant material (Clinpro Sealant, 3M ESPE Dental Products), and three experimental flowable resin composites with the same matrix and a variable filler content were used. The experimental resin composites were manufactured at our request by Voco. The decision to include experimental materials among the materials studied was guided by a desire to exclude the "matrix" parameter to record the influence of the filler content alone. The decision to include an ultra-flowable pit and fissure sealant resin among the materials studied was guided by a desire to record the potential impact of the absence of fillers, since ClinPro (CLI) is described as unfilled by the manufacturer.

In total, 56 disc-shaped specimens were prepared; eight disks of each material were made. The samples were prepared using a Teflon mold (15 mm in diameter by 1 mm in thickness). For each sample, the material was injected into the mold using an applicator nozzle, remaining in contact with one wall of the mold to minimize air inclusion. A glass slide with a thickness of 1.10 mm was then firmly applied and held on the mold using a clamp to compress the material and eliminate any excess. Polymerization was performed using a light-emitting diode lamp

Table 1: *Materials Used in This Study*

Code	Material (Manufacturer)	Main Components	Shade	Batch No.
GHF	Grandio So Heavy Flow (Voco D-27457 Cuxhafen Germany)	*Monomers:* Bis-GMA, Bis-EMA, TEGDMA *Fillers: (83 wt.% = 68 vol.%):* glass ceramic (average particle size: 1 μm), functionalized SiO_2 nanoparticles (from 20 to 40 nm)	A3	1234322
GRF	Grandio Flow (Voco D-27457 Cuxhafen Germany)	*Monomers:* Bis-GMA, HEDMA, TEGDMA *Fillers: (80.2 wt.% = 65.7 vol.%):* nano-hybrid inorganic fillers	A3	1233159
XTE	Filtek Supreme XTE Flow (3M/ESPE Dental Products St Paul, MI, USA)	*Monomers:* Bis-GMA, TEGDMA, Procrylat resins *Fillers: (65 wt.% =46 vol.%):* ytterbium trifluoride (from 0.1 to 5.0 μm), nonagglomerated/nonaggregated surface modified 20-nm silica fillers, nonagglomerated/nonaggregated surface-modified 75-nm silica fillers, surface-modified aggregated zirconia/silica fillers (composed of 20-nm and 4- to 11-nm zirconia particles with an average cluster particle size of 0.6 to 10 μm)	A3	N412976
CLI	Clinpro Sealant (3M/ESPE Dental Products, St Paul, MI, USA)	*Monomers:* Bis-GMA, TEGDMA *Fillers:* unfilled	—	N401833
EXPA	Experimental RBC (Voco D-27457, Cuxhafen, Germany)	*Monomers:* Bis-GMA (33wt.%), UDMA (33 wt.%), TEGDMA(33 wt.%) *Fillers: (65 wt.%):* silanized glass, silica (7 μm)	A3	—
EXPB	Experimental RBC (Voco D-27457, Cuxhafen, Germany)	*Monomers:* Bis-GMA (33wt.%), UDMA (33 wt.%), TEGDMA(33 wt.%) *Fillers: (55 wt.%):* silanized glass, silica (7 μm)	A3	—
EXPC	Experimental RBC (Voco D-27457, Cuxhafen, Germany)	*Monomers:* Bis-GMA (33 wt.%), UDMA (33 wt.%), TEGDMA (33 wt.%) *Fillers: (45 wt.%):* silanized glass, silica (7 μm)	A3	—

Abbreviations: Bis-EMA, ethoxylated bisphenol-A glycol dimethacrylate; Bis-GMA, bisphenol-A glycidylmethacrylate; HEDMA, hexanediol dimethacrylate; RBC, resin-based composite; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

(Elipar Freelight 2, 3M ESPE Dental Products). The light tip was first directed over the center of the sample for 40 seconds and then irradiated eight peripheral overlapping sectors for 20 seconds each. The samples were then carefully removed from the molds. The side opposite the one already photopolymerized was also exposed in the same way. Each sample was thus photopolymerized for a total of 400 seconds. Excess material was carefully removed using a scalpel blade.

Determination of the Filler Content Mass by Calcination

Three samples of each of the materials taken using the protocol described previously were weighed on a precision balance (Précisa, XB220A, Dietikon, Switzerland) to determine their initial mass. The samples were then placed in a chamber furnace with a power of 10 kW^2 (Nabertherm, LH30/14, Lilienthal, Germany) and heated to a temperature of 900°C for one hour. They were gradually returned to room temperature simply by stopping the heating cycle. Calcining the materials led to elimination of the resin matrix, leaving only the fillers in the form of an agglomerated residual powder. The calcination residue was then weighed. The filler content by mass, as a percentage, was determined using the formula (residue mass/initial mass of samples before calcination) × 100.

Microhardness Assessment

Five samples of each of the materials prepared using the protocol described above were tested. The initial microhardness of the samples was determined immediately after preparation using a microhardness tester (Matsuzawa MXT 50, Matsuzawa Seiki Co Ltd, Tokyo, Japan). A 200-g weight was applied for 20 seconds to the side to be polymerized first through the glass slide. The diagonals of the square imprint left by the diamond pyramid tip were measured after positioning cursors on a monitor (Ryokosha Co Ltd, Tokyo, Japan) coupled to the indentation hardness meter, with a magnification of 400×. The Vickers microhardness was obtained using the following formula: $VHN = (1854.4 \times W)/d^2$ (where VHN is the Vickers hardness number expressed in kg/mm^2, W is the weight in g, and d is the length of the diagonal in μm). Three measurements at different randomly selected points were performed per sample. After measurement, the samples were immediately stored in distilled water and placed in an oven at 37°C (Firlabo, Meyzieu, France). After 7 days, 60 days (2 months), 180 days (6 months), 360 days (1 year), and 720 days (2 years), each sample was briefly rinsed with distilled water, carefully wiped using absorbent paper, and left in air for 15 seconds before the microhardness was tested again at three different randomly chosen points, all on the same side. The distilled water storage

Table 2: *Filler Content by Mass as a Percentage for the Different Materials Tested*

Material	Wt.% (SD)
GHF	78.6 (0.1)
GRF	77.3 (0.1)
XTE	58.9 (0.2)
CLI	26.5 (0.8)
EXPA	63.1 (1.6)
EXPB	53.7 (0.2)
EXPC	43.6 (0.1)

medium was changed every 15 days for two years. The results were statistically analyzed using Kruskal-Wallis tests followed by paired multiple comparisons (Bonferroni adjustment; $\alpha=0.05$).

Rheological Properties

A dynamic shear rheometer (Physica MCR 301, Anton Paar GmbH, Graz, Austria) connected to a temperature control system (TC30, Anton Paar GmbH) was used to determine the rheological properties of the materials tested. A parallel plate viscometer module with a diameter of 25 mm was used. The gap between the plates was 1 mm. The temperature in the chamber was controlled at 23°C. Given the thixotropic behavior of resin composites, a preshear test was performed at a speed of 1 s^{-1} over 360° before a sweep test at a constant frequency of 0.1 $rad.s^{-1}$ for 7200 seconds. Then the sweep test was performed at an angular frequency of 100 to 0.01 $rad.s^{-1}$, making it possible to determine, due to sinusoidal strain, the viscosity (Pa.s) and the elastic storage modulus G′ (Pa) as a function of the shear frequency imposed.

RESULTS

The results of the determination of the filler content using the calcination method are presented in Table 2. The filler contents can be ranked in decreasing order as follows: Grandioso Heavy Flow (GHF), Grandio Flow (GRF), Filtek Supreme XTE Flow (XTE), and CLI for the commercially available materials and EXPA, EXPB, and EXPC for the experimental materials.

The results for determination of microhardness are presented in Tables 3 and 4. Before immersion, the commercially available materials tested presented significantly different microhardnesses ($p<0.0001$), with, in decreasing order, GHF, GRF, XTE, then CLI. For GHF and GRF, a significant decrease in the values was observed after seven days of immersion ($p<0.0001$), which is not the case for XTE and CLI. After 2 years of immersion, CLI and XTE present, on average, hardness values that are significantly higher than the initial values ($p<0.0001$), whereas GRF presents average hardness values that are not significantly different from the initial values and GHF presents average values that are significantly lower than the initial values ($p<0.0001$).

Irrespective of the immersion time, the experimental materials tested presented significantly different microhardness ($p<0.0001$), with, in decreasing order, EXPA, EXPB, and EXPC. After two years of immersion, the three experimental materials presented, on average, hardness values that were significantly higher than the initial values ($p<0.0001$).

The curves for complex viscosity and storage modulus as a function of shear frequency for the commercially available materials tested are presented in Figures 1 and 2; those for the experimental materials tested are presented in Figures 3 and 4. For all materials, the viscosity decreased as the shear frequency increased. Irrespective of the shear frequency, the viscosity can be ranked in decreasing order as follows: GHF, GRF, XTE, and CLI for the commercially available materials and EXPA, EXPB, and EXPC for the experimental materials. For all the materials, the storage modulus increased as the frequency increased. Irrespective of the shear fre-

Table 3: *Evolution of Vickers Microhardness Mean Values (Standard Deviation) for the Different Commercially Available Materials Tested as a Function of Their Immersion Time in Distilled Water*[a]

Material	Immediately Postcure	Immersion Time					
	J0	J7	J60	J120	J180	J360	J720
GHF	68.0^{aA} (1.9)	58.0^{aB} (1.7)	61.7^{aC} (1.1)	62.9^{aC} (1.6)	62.7^{aC} (1.27)	63.5^{aC} (2.6)	63.3^{aC} (1.4)
GRF	45.2^{bA} (2.3)	40.9^{bB} (1.4)	41.8^{bA} (1.0)	42.0^{bA} (1.3)	42.37^{bA} (1.1)	42.4^{bA} (1.0)	43.5^{bA} (1.3)
XTE	36.3^{cA} (2.0)	37.9^{bA} (1.2)	38.6^{cA} (1.0)	39.9^{bA} (1.4)	38.9^{cA} (0.8)	41.0^{bB} (0.6)	41.7^{bB} (0.7)
CLI	9.5^{dA} (0.6)	9.5^{cA} (0.5)	10.4^{dB} (0.2)	10.9^{cB} (0.3)	10.9^{dB} (0.2)	11.6^{cC} (0.1)	12.3^{cC} (0.2)

[a] *In each column, results with the same superscript lowercase letter are not statistically different. In each row, results with the same superscript uppercase letter are not statistically different.*

Table 4: *Evolution of Vickers Microhardness Mean Values (Standard Deviation) for the Different Experimental Materials Tested as a Function of Their Immersion Time in Distilled Water*[a]

Material	Immediately Postcure	Immersion Time					
	J0	J7	J60	J120	J180	J360	J720
EXPA	32.2^{aA} (1.3)	31.3^{aA} (0.8)	34.8^{aB} (0.6)	34.4^{aB} (0.7)	34.1^{aB} (0.7)	34.8^{aB} (1.0)	34.7^{aB} (0.4)
EXPB	23.6^{bA} (0.8)	24.4^{bA} (1.2)	24.6^{bA} (0.3)	25.6^{bA} (0.6)	25.2^{bA} (0.3)	26.0^{bB} (0.4)	27.2^{bC} (0.3)
EXPC	18.9^{cA} (0.6)	19.3^{cA} (0.4)	21.5^{cB} (0.3)	21.5^{cB} (0.5)	21.6^{cB} (0.5)	21.6^{cB} (0.3)	22.3^{cC} (0.3)

[a] *In each column, results with the same superscript lowercase letter are not statistically different. In each row, results with the same superscript uppercase letter are not statistically different.*

quency, the storage modulus can be ranked in decreasing order as follows: XTE, GHF, GRF, and CLI for the commercially available materials and EXPA, EXPB, and EXPC for the experimental materials.

DISCUSSION

It is widely accepted that the higher the filler content of a resin composite, the better its mechanical behavior and, consequently, the greater the potential durability of the restoration made using

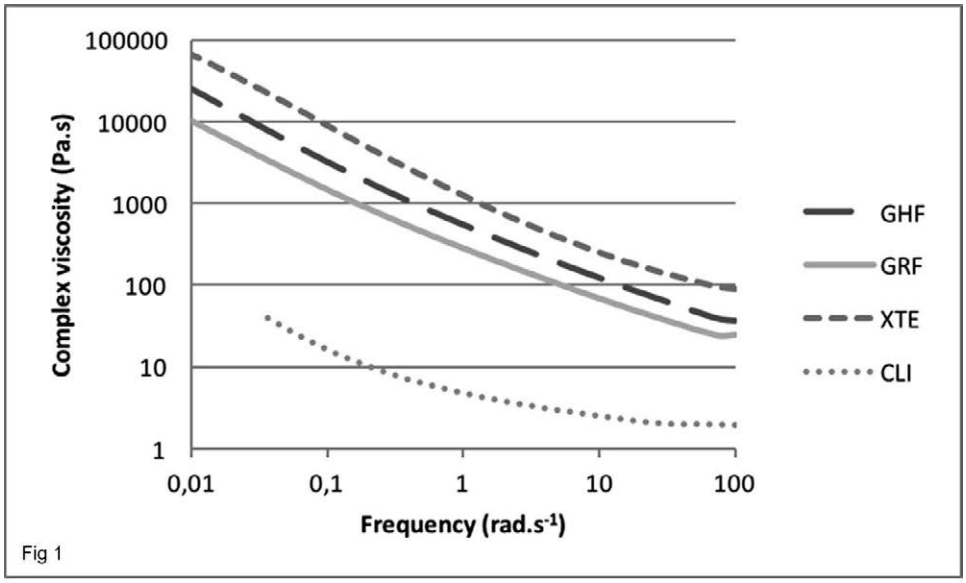


Figure 1. *Complex viscosity of the commercially available materials as a function of shear frequency.*

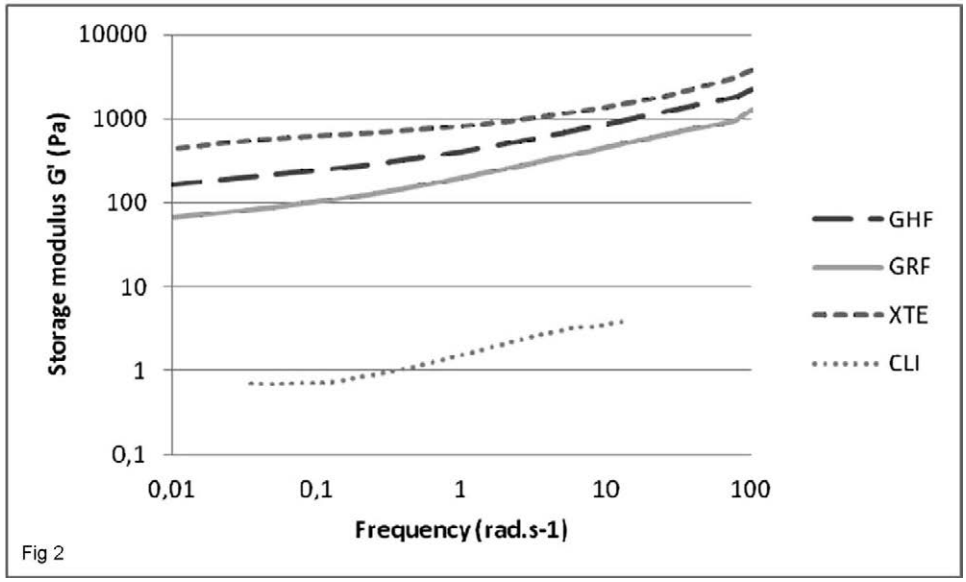


Figure 2. *Storage modulus G′ of the commercially available materials as a function of shear frequency.*

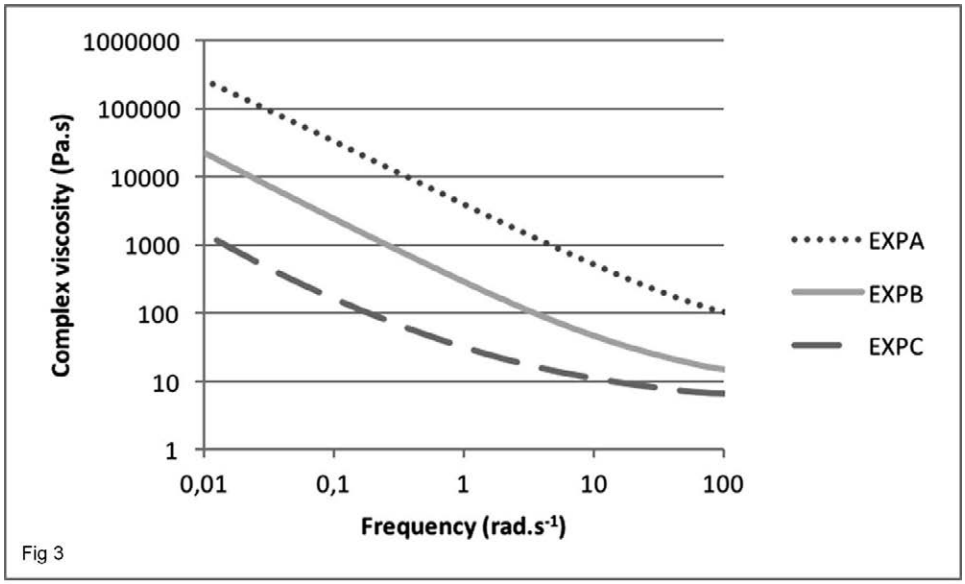


Figure 3. *Complex viscosity of the experimental materials as a function of shear frequency.*

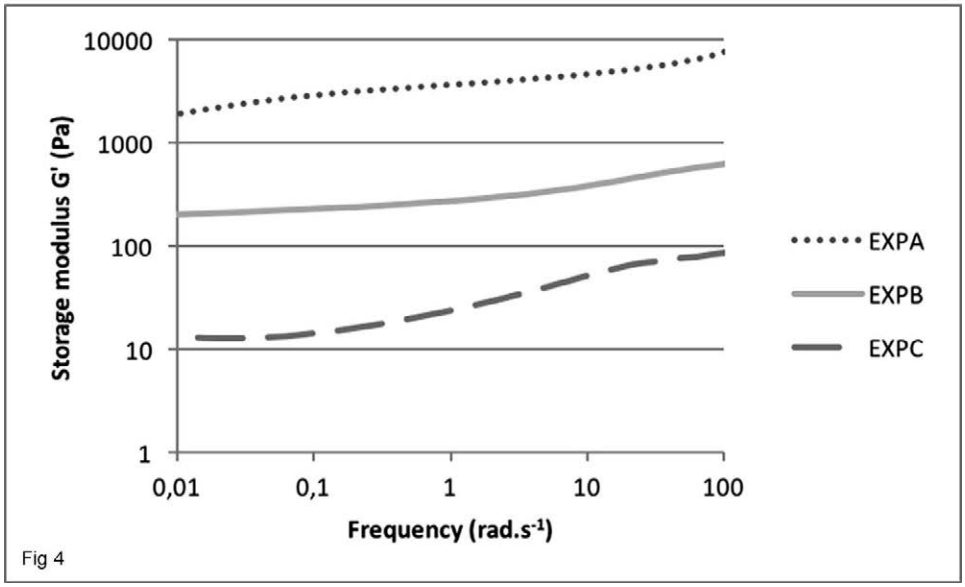


Figure 4. *Storage modulus G′ of the experimental materials as a function of shear frequency.*

said resin composite.[16-18] As a result, the filler content is sometimes highlighted by manufacturers as a commercial argument, and resin composites with a very high filler content have emerged.[18,19] But to make a resin composite more flowable, one of the methods commonly used by manufacturers consists in reducing the filler content, thereby potentially affecting its mechanical properties. Nonetheless, resin composites described as both flowable and having high filler content have appeared on the market.

Our values for filler content by weight do indeed indicate high values for GHF and GRF, of 78.6% and 77.3%, respectively. It is no surprise that, in contrast, CLI, intended as a pit and fissure sealant only, presents the lowest filler content, at 26.5 wt.%. The values obtained in our study do not entirely match the data supplied by manufacturers. Yet the calcination method used here, with heating to 900°C for one hour, effectively eliminates the organic portion of resin composites and appears to be a validated method, like thermogravimetric analysis, for determination of filler content.[20,21] The difference in value can probably be explained by the fact that manufacturers assess the filler content after silanization of the fillers. Silanization, which is essential for coupling between fillers and the matrix, involves industrial processes specific to each manufacturer, and the silane quantity used, as well as the thickness of the silane layer coating the fillers, depend on the methods used. If it is taken into account by the manufacturer when assessing the

filler mass, this silane quantity increases the values, while, since it is eliminated during calcination, it is not taken into account in the measurements performed in our study. Similarly, the combination of small-sized mineral fillers in a prepolymerized resin matrix for the purposes of forming clusters of fillers with a higher diameter may explain the differences between our values and the manufacturer's data. In fact, the organic part of these fillers will be eliminated during calcination, whereas the mineral part will persist.[21] This destruction of the organic fraction of the fillers probably largely explains the difference in the values observed for XTE, described as containing 20 nm silica and 4 to 11 nm zirconia fillers grouped into clusters of 0.6 to 10 μm. For CLI, we determined a filler content of approximately 26 wt%. Surprisingly, the instructions for use accompanying CLI describe it as unfilled. However, oddly, the technical product profile available on request from the manufacturer specifies the presence of silanized amorphous silica fillers of 0.016 μm at a concentration of 6%, with no mass or volume indication relative to these data. It should be noted that the presence of TiO_2, which gives the material a white color after photopolymerization and is resistant to temperatures of 900°C, can also increase the mass of the residue after calcination, in line with the difference in values that exists between our measurements and the manufacturer's data. It therefore appears that, due to a lack of standardization in filler content determination methods, the values communicated by manufacturers should be regarded with a certain degree of caution.

Because it is a good indirect indicator of the mechanical performance of materials, surface microhardness is regularly used in studies.[15] In our study, the results obtained before immersion in distilled water for each of our materials—either commercially available or experimental—are consistent with the literature: the higher the filler content of a resin composite, the higher its surface microhardness.[22] It is no surprise that EXPA, with a filler content of 65 wt%, has the highest initial hardness of the experimental materials. Nor is it surprising that CLI, a material with an indication limited to pit and fissure sealing and with very low filler content, presents an initial surface microhardness that is about seven times lower than GHF, the material with the highest filler content in our study. However, given the nonlinear relationship observed between it and the values obtained, the filler content parameter alone is not the only explanation. For example, GHF and GRF, which have almost the same filler contents, present significant differences in terms of surface microhardness. This can probably be explained by the nature and proportion of the monomers included in the polymer network. The degree of cross-link density and the three-dimensional complexity appear to be factors with a major influence on the mechanical behavior of the polymer network.[23] Hence, small aliphatic monomers, such as TEGDMA, promote the formation of dense molecular networks;[24] BisGMA potentially leads to a lower conversion rate and a lower cross-link density,[25,26] but, conversely, due to its intrinsically rigid molecular structure and capacity to form hydrogen bonds between the monomers, it appears to be a pillar that supports the three-dimensional configuration of the polymer structure.[25,27] BisEMA, which is devoid of hydroxyl radicals but has a high molecular weight, potentially reduces the cross-link density of the polymer.[24] The BisGMA/TEGDMA combination, due to the presence of three ether bonds on the latter, appears to have a synergistic effect that increases the polymer network density.[27] Each monomer and each group therefore involves different properties and different molecular architectures.[20,28] But the absence of data supplied by manufacturers concerning the exact proportions of the different monomers makes interpretation of the current results complicated. What mainly emerges is that the filler content—even when very high—is not, in itself, the only parameter affecting the hardness of the material.

Our measurements carried out on experimental materials also demonstrate that there is no linear relationship between the filler content and the initial microhardness. Yet these three materials present the same matrix composition in terms of nature and composition. This means that another factor must be involved. At equivalent volume, the higher the filler content of a material, the greater the proportion of matrix bound to the fillers. This matrix in contact with the fillers probably does not have the same mechanical properties as the matrix resin not in contact with the fillers. A matrix region doubtless exists with optimized mechanical properties ensuring a transition between the hard filler and the softer matrix.[18] The lower the filler content of the material, the less it will present this optimized matrix zone, relatively speaking, helping to explain the absence of a proportional relationship between the filler content and microhardness.

In our study, it is observed that immersion in distilled water at 37°C leads to variations in the microhardness values of the materials tested. Fol-

lowing the long-term immersion of the materials in distilled water at 37°C, two phenomena generating opposite consequences in terms of microhardness are liable to occur. Initially, the water molecules will have a plasticizing effect, inserting themselves between the polymer network chains. The resin matrix will then swell, and the frictional forces between the chains will decrease.[29] Furthermore, tensile stresses are generated at the resin-filler interfaces, straining the bonds in the inorganic component and increasing the frictional forces between filler and matrix resin, facilitating pull out of fillers.[12,30] Conversely, storage of the samples at 37°C can promote new monomer conversions or additional postcuring cross-linking reactions in the resin phase over time.[31-33] In terms of microhardness, these two phenomena have completely opposing consequences, with water promoting a reduction while heat promotes an increase: the variations in values recorded at the different immersion time points are the complex result of various interacting factors. It is essential to note that the postpolymerization induced by the rise in temperature is dependent on the conversion rate initially obtained before immersion and the three-dimensional structure of the polymer network. Indeed, for postpolymerization to occur, there needs to be the possibility of movement for nonsaturated chains. Hence, the higher the initial conversion rate, the less postpolymerization will occur.[34,35] The high initial hardness associated with a high filler content is also a factor that may potentially restrict molecular mobility within the polymer network initially developed.[15] Thus, for example, a marked reduction in microhardness values is clearly observed following one week of immersion for GHF and GRF, whereas that is not the case for XTE or CLI. In addition, in a recent study,[36] the extremely high conversion rate for GRF immediately after photopolymerization was demonstrated, in line with our observations. For the experimental materials, the potential impact of a high filler content on molecular mobility can also be observed in view of the values recorded for EXPA compared with EXPB and EXPC. For these materials with the highest filler content, it can therefore be seen that the effect of water is more dominant than the effect of temperature. It should also be highlighted that, at an equivalent matrix volume, a filled material has been shown to be capable of absorbing more water than an unfilled material due to the capillary penetration routes offered by the silanized interfaces between the fillers and the matrix, which are liable to become hydrolyzed, thus transporting and holding water.[37]

Over time, compared with the values measured after one week of immersion, a relatively steady increase in microhardness is observed for all the materials. For GHF, for which the values were initially significantly reduced after one week of immersion, the potential mobility of the chains and the formation of new bonds are not sufficient to compensate for the effect of the water and restore postpolymerization microhardness values. After two years, the values for GRF and EXPA are close to the initial postpolymerization values. For the materials with a lower filler content—XTE and CLI or EXPB and EXPC—after two years of immersion, the possibility of forming new bonds between the molecular chains has become more important than the effect of water.

To date, no other study has monitored the microhardness of materials over such a long period. For our materials and in our experimental conditions, microhardness values are shown to be maintained, or even increased, after two years compared with initial values, except in the case of GHF, indirectly reflecting preservation of the mechanical properties in the medium term. Obviously, the superimposition and interdependence of the various factors influencing microhardness make interpretation of the results complicated. However, it appears that a very high initial microhardness, combined with a high filler content, is not an adequate guarantee to protect against the detrimental effects of water in terms of plasticization.

It is important to specify that in our protocol, the samples are photopolymerized for a total of 400 seconds, with 200 seconds of light exposure per surface. This method probably optimized the conversion rate compared with the photopolymerization usually performed in clinical situations and restricts the possibilities of postpolymerization.[38] Conversely, the distilled water chosen as the immersion medium here is known to cause polymer structure changes similar to those found with artificial saliva,[39,40] but less marked than with ethanol,[10,15] coffee, or sweetened fizzy drinks, for example.[13,41]

With respect to the rheological properties, it can be noted that, before photopolymerization, all the materials tested in our study are non-Newtonian fluids (ie, they present a nonconstant viscosity that depends on the shear applied). In addition, for all the materials, a reduction in said apparent viscosity is observed, with an increase in the velocity gradient (increase in angular frequency): they therefore present shear-thinning behavior. This can probably be explained by a progressive alignment of the

molecules in the thickness of the material under the effect of the shear rate, promoting their relative slip. A second potential explanation is a change in the structure of the material as a result of rupture of the Van der Waals bonds joining nonphotopolymerized monomers. The nature and proportion of the various monomers obviously play an important role in viscosity. BisGMA, with its two hydroxyl radicals per molecule, increases the viscosity of monomer mixtures, promoting the formation of hydrogen bonds between the monomers.[27] Therefore, a mixture of monomers containing BisEMA, a molecule with an identical structure to BisGMA except for the hydroxyl groups that have been substituted, presents a lower viscosity than a mixture of BisGMA.[28] The low density of molecular stacking due to the intrinsic structure of BisGMA and the resulting high free volume also contribute to the higher viscosity of monomer mixtures containing a high proportion of BisGMA.[28] Conversely, TEGDMA, a small molecule with no aromatic cycle or hydroxyl radicals, helps lower the viscosity of monomer mixtures.

Examination of the results obtained for our experimental materials, with equivalent filler and matrix types, indicates the influence of filler contents on rheological behavior: here, the higher the material's filler content, the more viscous it is, irrespective of the shear rate. The results for CLI, with its low filler content in comparison to the other commercially available materials, are also in line with this. However, examination of the results for GRF, GHF, and XTE clearly reveals the impact of other parameters apart from filler content alone, in view of the absence of correlation between the latter and rheological behavior. The type and shape of fillers, along with the quality of silanization, probably have a greater influence here than the filler content itself, which would therefore be consistent with the observations of a recent study.[42] Examination of Figures 1 and 2 shows that, excluding CLI, GRF has the lowest viscosity at all the frequencies and the lowest storage modulus at all the frequencies, with, nonetheless, a steep slope at said low frequencies: these data reflect its excellent spreadability inside small cavities or in pits and fissures, whereas it presents a filler content by mass that is almost 20% higher than that of XTE (data not presented: calculation of the slopes of the curves at low frequencies clearly indicates the steepest slope for GRF). In other words, its low viscosity was not obtained to the detriment of its filler content. GRF therefore offers what appears to be a very interesting compromise between a low viscosity and a low elastic behavior at low shear frequencies and high filler content.

It nonetheless remains true that the spreadability of a material in a small cavity is subject to influences other than viscosity alone: surface tension after etching of dental tissues and the adhesive system used also play a role.[42]

CONCLUSION

For the materials tested, with the limitations related to the experimental conditions and the number of samples prepared, it appears that:

- The filler content values communicated by manufacturers have to be regarded with a certain amount of caution.
- The surface microhardness of materials seems to be affected by numerous interdependent factors, one of the main ones appearing to be the filler content; however, at equivalent filler contents, the polymer structure appears to be important.
- An immersion period of two years in distilled water at 37°C seems to have variable effects on the surface microhardness of the materials; a very high initial microhardness, combined with a high filler content is probably not an adequate guarantee to protect against the detrimental effects of water in terms of plasticization.
- A low viscosity and a low elastic behavior at low shear frequency before photopolymerization can be combined with a high filler content, thereby appearing to offer a satisfactory compromise between the spreadability of the material and its microhardness after photopolymerization.

Conflict of Interest

The authors 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 April 2016)

REFERENCES

1. Prager MC (1997) Using flowable composites in direct posterior restorations *Dentistry Today* **16(7)** 62-69.
2. Labella R, Lambrechts P, Van Meerbeck B, & Vanherle G (1999) Polymerization shrinkage and elasticity of flowable composites and filled adhesives *Dental Materials* **15(2)** 128-137.
3. Papacchini F, Goracci, Sadek FT, Monticelli F, Garcia-Godoy F, & Ferrari M (2005) Microtensile bond strength to ground enamel by glass-ionomers, resin-modified glass ionomers, and resin composites used as pit and fissure sealants *Journal of Dentistry* **33(6)** 459-467.

4. Corona SAM, Borsatto MC, Garcia L, Ramos RP, & Palma-Dibb RG (2005) Randomized, controlled trial comparing the retention of a flowable restorative system with a conventional resin sealant: one year follow-up *International Journal of Paediatric Dentistry* **15(1)** 44-50.
5. Pardi V, Sinhoreti MAC, Pereira AC, Ambrosano GMB, & Meneghim M (2006) *In vitro* evaluation of microleakage of different materials used as pit-and-fissure sealants *Brazilian Dental Journal* **17(1)** 49-52.
6. Ferracane JL (1985) Correlation between hardness and degree of conversion during the setting reaction of unfiled dental restorative resins *Dental Materials* **1(1)** 11-14.
7. Asmussen E, & Peutzfeld A (2003) Influence of specimen diameter on the relationship between subsurface depth and hardness of a light-cured resin composite *European Journal of Oral Sciences* **111(6)** 543-546.
8. Watts DC (2005) Reaction kinetics and mechanics in photo-polymerized networks *Dental Materials* **21(1)** 27-35.
9. Yan YL, Kim YK, Kim KH, & Kwon TY (2010) Changes in degree of conversion and microhardness of dental resin cements *Operative Dentistry* **35(2)** 203-210.
10. Aguiar FHB, Braceiro ATB, Ambrosano GMB, & Lovadino JR (2005) Hardness and diametral tensile strength of a hybrid composite resin polymerized with different modes and immersed in ethanol or distilled water media *Dental Materials* **21(12)** 1098-1103.
11. Chinelatti MA, Thomazatti Chimello DY, Pereira Ramos R, & Palma-Dibb RG (2006) Evaluation of the surface hardness of composite resins before and after polishing at different times *Journal of Applied Oral Science* **14(3)** 188-192.
12. de Moraes RR, Machado Marimon JL, Jochims Schneider LF, Coelho Sinhoreti MA, Correr-Sobrinho L, & Bueno M (2008) Effects of 6 months of aging in water on hardness and surface roughness of two microhybrid dental composites *Journal of Prosthetic Dentistry* **17(4)** 323-326.
13. Yanikoğlu N, Duymuş ZY, & Yilmaz B (2009) Effects of different solutions on the surface hardness of composite resin materials *Dental Materials Journal* **28(3)** 344-351.
14. Flury S, Peutzfeld 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
15. Alshali RZ, Salim NA, Satterthwaite JD, & Silikas N (2015) Post-irradiation hardness development, chemical softening, and thermal stability of bulk-fill and conventional resin-composites *Journal of Dentistry* **43(2)** 209-218.
16. Abe Y, Lambrechts P, Inoue S, Braem MJA, Takeuchi M, Vanherle G, & Van Meerbeck B (2001) Dynamic elastic modulus of "packable" composites *Dental Materials* **17(6)** 520-525.
17. Sabbagh J, Vreven J, & Leloup G (2002) Dynamic and static modulus of elasticity of resin-based materials *Dental Materials* **18(1)** 64-71.
18. Ilie N, & Hickel R (2009) Investigations on mechanical behavior of dental composites *Clinical Oral Investigations* **13(4)** 427-438.
19. Hanhel S, Dowling AH, El-Safty S, & Fleming GJP (2012) The influence of monomeric resin and filler characteristics on the performance of experimental resin-based (RBCs) derived from a commercial formulation *Dental Materials* **28(4)** 416-423.
20. Kim KH, Ong JL, & Okuno O (2002) The effect of filler loading and morphology on the mechanical properties of contemporary composites *Journal of Prosthetic Dentistry* **87(6)** 642-649.
21. Sabbagh J, Ryelandt L, Bacherius L, Biebuyck JJ, Vreven J, Lambrechts P, & Leloup G (2004) Characterization of the inorganic fraction of resin composites *Journal of Oral Rehabilitation* **31(11)** 1090-1101.
22. Czasch P, & Illie N (2013) In vitro comparison of mechanical properties and degree of cure of a self-adhesive and four novel flowable composites *Journal of Adhesive Dentistry* **15(3)** 229-236.
23. McCabe JF, & Walls AWG (2009) *Applied Dental Materials* **9th edition** Blackwell Publishing Ltd, Oxford, UK.
24. Cook WD, & Moopnar M (1990) Influence of chemical structure on the fracture behavior of dimethacrylate composite resins *Biomaterials* **11(4)** 272-276.
25. Floyd CJ, & Dickens SH (2006) Networkstructure of Bis-GMA- and UDMA- based resin systems *Dental Materials* **22(12)** 1143-1149.
26. Sideriou 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.
27. Lemon MT, Jones MS, & Stansbury JW (2007) Hydrogen bonding interactions in methacrylate monomers and polymers *Journal of Biomedical Materials Research Part A* **83(3)** 734-746.
28. Gonçalves F, Kawamo Y, Pfeifer C, & Braga RR (2009) Influence of BisGMA, TEGDMA and BisEMA contents on viscosity, conversion and flexural strength of experimental resins and composites *European Journal of Oral Science* **117(4)** 442-446.
29. Ferracane JL, Berge HX, & Condon JR (1998) *In vitro* aging of dental composites in water-effect of degree of conversion, filler volume, and filler/matrix coupling *Journal of Biomedical Materials Research Part A* **42(3)** 465-472.
30. Söderholm KJ (1981) Degradation of glass filler in experimental composites *Journal of Dental Research* **60(11)** 1867-1875.
31. Watts DC, Amer O, & Combe E (1987) Surface hardness development in light-cured composites *Dental Materials* **3(5)** 265-269.
32. Mohamad D, Young R, Mann A, & Watts DC (2007) Post-polymerization of dental resin composites evaluated with nanoindentation and micro-Raman spectroscopy. *Archives of Orofacial Sciences* **2(1)** 26-31.

33. Marghalani HY (2010) Post-irradiation Vickers microhardness development of novel resin composites *Journal of Materials Research* **13(1)** 81-87.

34. Jacobsen PH, & Darr AH (1997) Static and dynamic moduli of composite restorative materials *Journal of Oral Rehabilitation* **24(4)** 265-273.

35. Tarumi H, Imazato S, Ehara A, Kato S, Ebi N, & Ebisu S (1999) Post-irradiation polymerization of composites containing bis-GMA and TEGDMA *Dental Materials* **15(4)** 238-242.

36. Alshali RZ, Silikas N, & Satterhwaite JD (2013) Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals *Dental Materials* **29(9)** 213-217.

37. Venz S, & Dickens B (1991) NIR-spectroscopic investigation of water sorption characteristics of dental resins and composites. *Journal of Biomedical Materials Research* **25(10)** 1231-1248.

38. Koliniotou-Kubia E, & Jacobsen PH (1990) The effect of irradiation time on the physical properties of light cured resins *Clinical Materials* **6(1)** 21-26.

39. Yap AU, Tan SH, Wee SS, Lee CW, Lim EL, & Zeng KY (2001) Chemical degradation of composite restoratives *Journal of Oral Rehabilitation* **28(11)** 1015-1021.

40. Turssi CP, Hara AT, Serra MC, & Rodrigues AL Jr (2002) Effect of storage media upon the surface micromorphology of resin-based restorative materials. *Journal of Oral Rehabilitation* **29(9)** 864-871.

41. Karaman E, Tuncer D, Firat E, Ozdemir OS, & Karahan S (2014) Influence of different staining beverages on color stability, surface roughness and microhardness of silorane and methacrylate-based composite resins *Journal of Contemporary Dental Practice* **15(3)** 319-325.

42. Beun S, Bailly C, Devaux J, & Leloup G (2012) Physical, mechanical and rheological characterization of resin-based pit and fissure sealants compared to flowable resin composites *Dental Materials* **28(4)** 349-359.

©Operative Dentistry, 2016, **41-6,** 666-671

Premolar Axial Wall Height Effect on CAD/CAM Crown Retention

C Gillette • R Buck • N DuVall • S Cushen • M Wajdowicz • H Roberts

Clinical Relevance

Although not applicable for all clinical situations, CAD/CAM adhesive technology may compensate for shorter occlusogingival axial wall height in premolar full-coverage, all-ceramic restorations.

SUMMARY

Objective: To evaluate the significance of reduced axial wall height on retention of adhesively luted, all-ceramic, lithium disilicate premolar computer-aided design/computer-aided manufacturing (CAD/CAM) crowns based on preparations with a near ideal total occlusal convergence of 10°.

Methods: Forty-eight recently extracted premolars were randomly divided into four groups (n=12). Each group received all-ceramic CAD/CAM crown preparations featuring axial wall heights of 0, 1, 2, and 3 mm, respectively, all with a 10° total occlusal convergence. Scanned preparations were fitted with lithium disilicate all-ceramic crowns that were luted with a self-etching resin cement. Specimens were tested to failure at a 45° angle to the tooth long axis with failure load converted to megapascals (MPa) based on the measured bonding surface area. Mean data were analyzed using analysis of variance/Tukey's post hoc test (α=0.05).

Results: Lithium disilicate crowns adhesively luted on preparations with 0 axial wall height demonstrated significantly less failure resistance compared with the crowns luted on preparations with axial wall heights of 1 to 3 mm. There was no failure stress difference between preparations with 1 to 3 mm axial wall height.

Conclusions: Under conditions of this study, adhesively luted lithium disilicate bicuspid crowns with a total occlusal convergence of 10° demonstrated similar failure resistance independent of axial wall height of 1 to 3 mm. This study provides some evidence that adhesion combined with an ideal total occlusal convergence may compensate for reduced axial wall height.

Christopher Gillette, DMD, MS, Deputy Director, AEGD-1, Wright-Patterson AFB, OH, USA

Richard Buck, DMD, MS Director, Prosthodontics, 48th Dental Squadron, RAF Lakenheath, GB

Nicholas DuVall, DDS, MS, Deputy Director, AEGD-2, Air Force Postgraduate Dental School, Keesler Air Force Base, Biloxi, MS, USA

Sarra Cushen, DDS, MS, Director, Prosthodontics, Air Force Postgraduate Dental School, Keesler AFB, MS, USA

Michael Wajdowicz, DDS, Director AEGD-2, Air Force Postgraduate Dental School, Keesler AFB, MS, USA

*Howard Roberts, DMD, MS, Director, Graduate Dental Research, AGED-2, Air Force Postgraduate Dental School, Keesler Air Force Base, Biloxi, MS, USA

*Corresponding author: 606 Fisher St., Keesler AFB, MS 39534; e-mail: howard.roberts@us.af.mil

DOI: 10.2341/16-086-L

INTRODUCTION

Full-coverage restorations, either metal or ceramic, have tooth preparation guidelines that include

degree of total occlusal convergence (TOC), axial wall height, and specific intracoronal features.[1-3] Specifically, a 3 mm occlusocervical (OC) axial wall height is recommended for adequate retention of premolar crowns.[1,3] These guidelines were formulated in the era of aqueous-based luting agents before the advent of resin cements. At that time, full-coverage restorations relied largely on preparation retention and resistance features as aqueous-based cements could only provide macromechanical retention by filling the space between the restoration intaglio surface and the prepared tooth surface. The increased clinical use of all-ceramic full-coverage restorations provided the impetus for the development of adhesively bonded resin cements that are touted to provide macro- and micromechanical retention as well as chemical bonding to selected materials.[4,5] Finish lines, intracoronal features, and TOC degree are preparation elements that the clinician usually has significant control over. However, the axial wall may be compromised due to disease or trauma and ideal axial wall heights may not be within the clinician's control, especially in situations where crown-lengthening surgery is not feasible. The purpose of this study was to evaluate if adhesion technology can compensate for reduced axial wall height in premolar all-ceramic crowns luted to preparations containing a 10° TOC. The null hypothesis was that there would be no difference in failure stress between preparations containing 0, 1, 2, or 3 mm axial wall height.

METHODS AND MATERIALS

Human premolar teeth that contained no restorations or caries were used in this study. All teeth, collected and used under the guidance of the local Institutional Review Board, were obtained from local oral and maxillofacial surgery clinics and had been removed per routine clinical indications for orthodontic expediency.

Forty-eight freshly extracted premolar teeth were randomly assigned to one of four groups (n=12) with the occlusal surfaces removed to 1 mm below the marginal ridge with a slow-speed, water-cooled diamond saw (Buehler, Lake Bluff, IL, USA). The sectioned teeth were then mounted in autopolymerizing denture base methyl-methacrylate resin (Diamond D, Keystone Industries, Cherry Hill, NJ, USA). Preparations were accomplished following manufacturer's recommendations for lithium disilicate all-ceramic crowns (IPS e.max CAD, Ivoclar Vivadent, Amherst NY, USA) by a single operator using a high-speed electric dental handpiece (EA-51LT, Adec, Newburg, OR, USA) equipped with a diamond bur (8845KR.31.025, Brassler USA, Savannah, GA, USA) under continuous water coolant spray. Preparation features and TOC of 10° was standardized as much as possible with the handpiece placed in a fixed lathe arrangement. Teeth in the four groups received occlusal reduction resulting in OC preparation heights of 0, 1, 2, and 3 mm, respectively. The 0 mm axial wall height group received a buccal lingual groove preparation featuring the approximate width and half depth of a no. 8 round bur across the total occlusal surface. This feature was required to allow the correct restoration alignment to the preparation, and the orientation of this groove was designed not to add resistance features to the preparation as it was parallel to the testing force vectors. All preparations underwent final review and any necessary refinement by a board-certified prosthodontist. The prepared tooth surface area was then measured using a digital recording microscope (KH-7700, Hirox USA, Hackensack, NJ, USA) that allowed for the determination of bonding surface area.

The specimens were restored by one operator using a computer-aided design/computer-aided manufacturing (CAD/CAM) acquisition device (Cerec AC, version 4.2.4.72301/Cerec MC XL, Sirona Dental Systems, Charlotte, NC, USA) according to the manufacturer's instructions and/or recommendations. All specimens were scanned using a standardized template to allow the establishment of suitable clinically relevant restoration contours. The occlusal table was established at the same height regardless of axial wall height and had an occlusal thickness not less than 2 mm. The restorations were milled from a lithium disilicate ceramic material (IPS e.max CAD) followed by crystallization and glaze (IPS e.max CAD Crystall Glaze Spray, Ivoclar-Vivadent) following the manufacturer's protocol in a dental laboratory ceramic furnace (Programat P700, Ivoclar-Vivadent).

The milled restorations were adjusted and seated to the preparations using a disclosing agent (Occlude, Pascal International, Bellevue, WA, USA), after which the restoration was steam cleaned and dried. The restoration's intaglio surface was then prepared with a 5% hydrofluoric acid-etch solution (IPS Ceramic Etching Gel, Ivoclar-Vivadent) for 20 seconds, rinsed with water spray, and dried with oil-free compressed air. A coat of silane agent (Monobond Plus, Ivoclar Vivadent) was applied to the etched surface using a monobrush following manufacturer's instructions. After 60 seconds of reaction time, the silane agent was air-dried using oil-free compressed air.

Table 1: *Mean Failure Load (N) and Stress (MPa) (n=12)*[a]

Mean Preparation Axial Wall Height (mm)	Failure Load (N)	Failure Stress (MPa)
0	148.3 (70.1) A	2.89 (1.1) A
1	374.8 (150.9) B	6.35 (2.5) B
2	499.7 (117.5) BC	7.16 (1.6) B
3	622.4 (142.1) C	7.52 (1.7) B

[a] *Groups identified with same letter are statistically similar within each column (Tukey, α=0.05)*

Table 2: *Failure Mode Analysis*

Failure Mode	Occlusocervical Axial Wall Height			
	0 mm	1 mm	2 mm	3 mm
Cohesive ceramic	0	0	1	4
Adhesive crown/ tooth material	12	12	2	2
Tooth fracture	0	0	9	6

The tooth surface was prepared for cementation by cleaning with a pumice and water slurry, rinsed, and dried using oil-free compressed air. A self-adhesive resin cement (RelyX Unicem, 3M ESPE, St Paul, MN, USA) was placed into the intaglio surface of the ceramic restoration and then seated on the preparation using digital finger pressure. Restorations were tack cured for 1 second using a visible light curing unit (Bluphase G2, Ivoclar Vivadent). After the excess cement was removed, the restoration was light cured at the buccal, lingual, and proximal marginal areas for a total of 80 seconds. The specimens were stored under dark conditions at 37°C ± 1°C and 98% ± 1% humidity.

Twenty-four hours after cementation, each specimen was placed into a vise fixture on a universal testing machine (RT-5, MTS Corporation, Eden Prairie, MN, USA) with the long axis of the tooth at a 45° angle to the vertical axis of the testing fixture. The testing fixture consisted of a 3 mm-diameter hardened, stainless steel piston with a 0.5-m radius of curvature as described by Kelly and others.[6] Specimens were loaded on the facial cusps at a rate of 0.5 mm per minute until failure; failure load was recorded in Newtons with a resultant failure stress calculated based on preparation surface area. Failure mode for each specimen was determined by visual examination under 20× magnification (KH-7700, Hirox USA) as well as microtomography (MicroCT) (Skyscan 1172, Bruker MicroCT, Kontich, Belgium) at a resolution of 13.6 μm using 100 kV energy with a 0.4° step size. Individual images were combined into a three-dimensional (3D) image using recombination software (nRecon, Bruker MicroCT) and analyzed with a volume-rendering 3D software (CTVox, Bruker MicroCT).

Mean failure load and stress were first evaluated using the Shapiro-Wilk test and Bartlett's test to ascertain normal distribution and homogenous variance of the data. Analysis of variance identified a difference within the groups followed by the Tukey's *post hoc* test. Statistical analysis was performed with a computer-based program (SPSS 20, IBM SPSS, Chicago, IL, USA) with a 95% level of confidence (α=0.05).

RESULTS

The failure results are listed in Table 1. When based on failure load, the preparations with 3 mm axial preparation height exhibited significantly greater failure resistance than the preparations containing 0 and 1 mm axial wall heights. There was no difference in failure load between the 2 and 3 mm axial wall height groups. When bonded surface area was taken into consideration, there was no significant difference in the failure stress between preparations of 1, 2, and 3 mm axial height.

The failure analysis mode results can be seen in Table 2.

All restorations in the 0 and 1 mm axial height preparation groups experienced restoration debonding without any tooth or crown material failure. The major failure mode for the 2 and 3 mm axial height preparation groups was root fracture that did not involve the tooth preparation.

DISCUSSION

Proponents of CAD/CAM dentistry anecdotally promote that adhesive technology may compensate for loss of preparation features required when luting castings with aqueous-based cements. This current study attempted to evaluate whether the adhesion involved with all-ceramic premolar CAD/CAM restorations could compensate for loss of OC axial wall height using a standardized TOC angle of 10°. This could identify possible advantages in clinical situations where tooth structure loss might alleviate the need for elective endodontic or surgical periodontal procedures to gain adequate tooth structure for a full-coverage restoration.

The tooth preparations were standardized as much as possible with one operator using a lathe-type arrangement. The TOC of 10° was chosen as Goodacre and others[1] recommend a TOC between

Table 3: *Mean Tooth Preparation Parameters (n=12)*

Group (Axial Wall Height)	Axial Wall Height (mm)	Total Occlusocervical Convergence (°)	Surface Area (mm^2)
0 mm	–	–	50.2 (18.5)
1 mm	1.16 (0.07)	10.3 (0.8)	61.0 (8.33)
2 mm	2.10 (0.05)	9.61 (0.51)	71.0 (13.56)
3 mm	3.1 (0.04)	10.19 (0.9)	83.1 (6.22)

10° and 20°; those authors concluded that TOCs <10° are rarely clinically achieved. The mean preparation parameters are listed in Table 3.

The use of a digital recording microscope (KH 7700, Hirox USA) allowed the confirmation of tooth preparation parameters that also allowed the measurement of preparation surface area that could be available for adhesive bonding.

The total preparation convergence was determined by taking the mean of the four convergence measurements (facial/lingual, mesial/distal). Under the conditions of this study, TOC convergence was standardized at 10°. The surface area determination allowed the calculation of failure stress, which is uncommon in the dental scientific literature and may compensate for the disparities in tooth size inherent with the usual failure load reporting. Preparation surface area within the 2 and 3 mm groups was similar to that reported in an evaluation of preparation stone dies in a commercial dental laboratory.[7] Preparation surface area within each group was fairly consistent, as covariance ranged from 7% to 18% among the preparations that contained axial wall height. The surface area variability increased within the 0 mm axial wall height group, as surface area was largely dependent on tooth size. Failure load results found that the 3 mm preparation axial wall height was found to be significantly greater not only than the 0 mm group but also the 1 mm axial wall height group. Failure stress determination contrasted the more traditional failure load results in that no difference in failure stress was noted between OC preparation heights of 1, 2, and 3 mm, and all three were significantly greater than the preparations with no axial wall height. Failure stress, which is based on preparation surface area, may normalize failure load results, but more evaluation is incumbent in this area before definitive judgments can be proffered. Regardless of analysis, the null hypothesis was rejected. The results should be considered with the understanding that the preparation TOC of 10° in this study may be considered very conservative and may not be routinely achieved in the clinical environment.[8] Furthermore, it should be noted that all OC axial wall height preparations demonstrated failure loads greater than that reported for the normal human bite strength,[9,10] and follow-up fatigue load/stress studies are planned.

Failure mode analysis revealed that mode of failure depended largely on the preparation axial wall height. The 0 and 1 mm OC axial wall height groups failed predominately by adhesive failure of the resin cement with occasional minor reparable tooth material fracture. One-third of the OC 3 mm axial wall height group displayed cohesive ceramic fracture, but tooth fracture was the leading failure mode of both the 2 and 3 mm OC axial wall height groups. Microtomographic analysis found that these fractures were mostly initiated near the lingual margins and the base of the lingual preparation wall (Figures 1 and 2).

This study is one of the first to evaluate the effect of premolar OC axial wall height using CAD/CAM adhesive technology. Ersu and others[11] found that OC axial wall height affected zirconia coping retention, which led them to reinforce the philosophy that for preparations with <3 mm of OC axial wall height consideration should be given to endodontic therapy with post and core fabrication to gain additional retention and resistance features. However, that study differs from the present work in that stainless steel copings served as the foundation preparation material. Leong and others,[12] in their study involving maxillary premolars, found no difference in fatigue strength between 2 and 3 mm OC axial wall heights when a resin luting agent was used, even with a 20° TOC. The failure loads in this study were lower than those reported by Attia and Kern,[13,14] who studied two other leucite-reinforced ceramics in two separate studies. However, the conditions of their research applied forces along the specimen long axis, and preparation parameters were more conservative; they used a 5 mm OC axial wall height with an even more conservative TOC of 6°.[13,14] Lastly, failure loads of this study are also less than that reported by Good and others[15] who also used a TOC of 6° and an earlier leucite-reinforced ceramic. Although the failure loads may seem to be less than those in other reports, the authors maintain that the conditions of the present study may more closely represent those encountered in clinical practice.

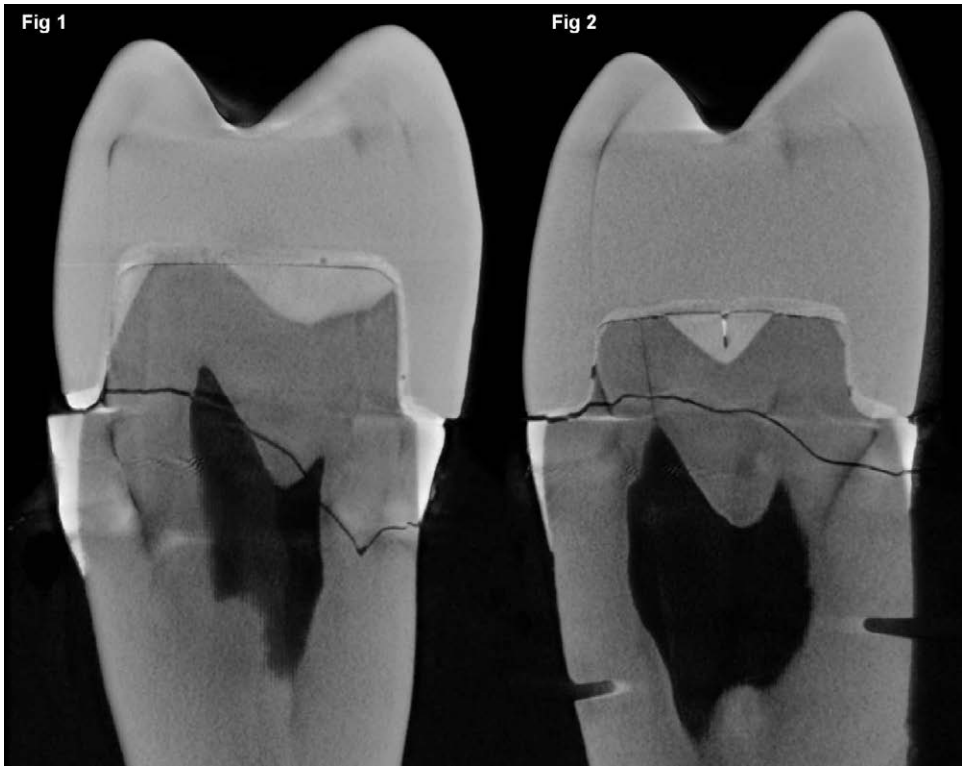


Figure 1. *MicroCT image slice showing 3-mm occlusocervical axial height failure.*
Figure 2. *MicroCT image slice showing 2-mm occlusocervical axial height failure.*

CONCLUSIONS

Under the conditions of this study, premolars restored with adhesively luted, CAD/CAM fabricated, lithium disilicate crowns based on a 10° TOC displayed similar failure stress resistance with OC axial wall heights of 1, 2, and 3 mm. This study provides some evidence that adhesion may compensate for less than ideal axial wall height in all-ceramic premolar preparations with a conservative total occlusal convergence.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the 81 Medical/Dental Group. The approval code for this study is: FKE20140016N.

Disclaimer

Any opinions expressed in this work are of the authors only and do not represent the official opinion of the United States Air Force, the Department of Defense, or the United States government. The authors have no commercial interest in any of the products or processes described and mention of such does not imply endorsement.

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 July 2016)

REFERENCES

1. Goodacre CJ, Campagni WV, & Aquilino SA (2001) Tooth preparations for complete crowns: An art form based on scientific principles *Journal of Prosthetic Dentistry* **85(10)** 363-376.
2. Wiskot HW, Nicholls JI, & Belser UC (1996) The relationship between abutment taper and resistance of cemented crowns to dynamic loading *International Journal of Prosthodontics* **9(2)** 117-130.
3. Wiskot HW, Nicholls JI, & Belser UC (1997) Effect of tooth preparation height and diameter on the resistance of complete crowns to fatigue loading *International Journal of Prosthodontics* **10(3)** 207-215
4. Rosenstiel SF, Land MF, & Crispin BJ (1998) Dental luting agents: A review of the current literature *Journal of Prosthetic Dentistry* **(80)3** 280-301.
5. Hikita K, Van Meerbeek B, De Munck J, 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
6. Kelly RJ, Rungruanganunt P, Hunter B, & Vailati F. Development of a clinically validated bulk failure test for ceramic crowns (2010) *Journal of Prosthetic Dentistry* **104(4)** 228-38
7. Tiu J, Al-Amleh B, Waddell JN, & Duncan WJ (2015) Reporting numeric values of complete crowns. Part 2: Retention and resistance theories *Journal of Prosthetic Dentistry* **114(1**) 75-80.

8. Tiu J, Al-Amleh B, Waddell JN, & Duncan WJ (2015) Reporting numeric values of complete crowns. Part 1: Clinical preparation parameters *Journal of Prosthetic Dentistry* **114(1)** 67-74
9. Sinn DP, DeAssis EA, & Throckmorton GS (1996) Mandibular excursions and maximum bite forces in patients with temporomandibular joint disorders *Journal of Oral and Maxillofacial Surgery* **54(6)** 671-679
10. Ferrario VF, Sforza C, Serrao G, Dellavia C, & Tartaglia M (2004) Single tooth bite forces in healthy young adults *Journal of Oral Rehabilitation* **31(1)** 18-22
11. Ersu B, Narin D, Aktas G, Yuzugullu B, & Canay S (2012) Effect of preparation taper and height on strength and retention of zirconia crowns. *International Journal of Prosthodontics* **25(6)** 582-584
12. Leong EWJ, Tan KBC, Nicholls JI., Chua EK, Wong KM, & Neo JCL (2009) The effect of preparation height and luting agent on the resistance form of cemented cast crowns under load fatigue. *Journal of Prosthetic Dentistry* **102(3)** 155-164
13. Attia A, & Kern M (2004) Influence of cyclic loading and luting agents on the fracture load of two all-ceramic crown systems *Journal of Prosthetic Dentistry* **92(6)** 551-556
14. Attia A, & Kern M (2004) Fracture strength of all-ceramic crowns luted using two bonding methods *Journal of Prosthetic Dentistry* **91(9)** 247-252
15. Good M-L, Orr JF, & Mitchell CA (2008) *In vitro* study of mean loads and modes of failure of all-ceramic crowns cemented with light-cured or dual-cured luting cement, after 1 and 30 d of storage *European Journal of Oral Sciences* **116(1)** 83-88

©Operative Dentistry, 2016, **41-6,** 672-674

Letter To The Editor

Dear Editor,

We read with great interest the following manuscripts published in the *Operative Dentistry*: Carvalho LD, Bernardon JK, Bruzi G, Andrada MAC & Vieira LCC (2013) Hypoplastic enamel treatment in permanent anterior teeth of a child 38(4) 363-368 and Reston EG, Corba DV, Ruschel K, Tovo MF & Barbosa AN (2011) Conservative approach for esthetic treatment of enamel hypoplasia 36(3) 340-343. We want to personally thank the authors for writing such great clinical papers. While they are generally well written case reports, we have concerns about this articles that needs to be addressed.

1. We disagree with the diagnostic provided by both authors. Developmental defects of enamel are classified in quantitative (hypoplasia) or qualitative (diffuses/demarcated opacities) alterations, based on their macroscopic appearance.[1] Disturbances during the maturation stage of amelogenesis result in opacities and the defect involving an alteration in the translucency, vary in degree. Hypoplasia results of disturbances during the secretion stage of amelogenesis and the defect is associated with a reduced localized thickness.[1-2] Therefore, the figures in the manuscripts show an enamel with demarcated opacities, not a hipoplastic enamel.

2. Our suggestion is to revise the differential diagnosis. The presented images may characterize a condition known as Molar-Incisor Hipomineralization (MIH), a genetic alteration related to disturbances in the maturation stage of enamel[3], not considered in the cited case reports. MIH is a qualitative defect of enamel that affects permanent first molars, with or without the involvement of permanent incisors[4]. The defects range from white-yellow or yellow-brown demarcated opacities to hypomineralised desintegrated enamel[4].

We hope these considerations will be addressed.

Sincerely,

Marina de Deus Moura de Lima, Heloísa Clara Santos Sousa & Lúcia de Ftima Almeida de Deus Moura
Department of Pathology and Dentistry Clinic, School od Dentistry, Universidade Federal do Piauí - UFPI, Teresina, Brazil

1. Federation Dental International (1992) A review of the developmental defects of enamel index (DDE Index) - Commission on Oral Health, Research & Epidemiology: Report of an FDI Working Group *International Dental Journal* **42** 411-426.
2. Hu JC, Chun YH, Al Hazzazzi T & Simmer JP (2007) Enamel formation and amelogenesis imperfecta *Cells Tissues Organs* **186(1)** 78-85.
3. Vieira AR & Kup E (2016) On the Etiology of Molar-Incisor Hypomineralization *Caries Research* **50(2)** 166-169.
4. Weerheijm KL, Jalevik B & Alaluusua S (2001) Molar-incisor hypomineralisation *Caries Research* **35** 390-391.

Response from the Authors

Dear Editor Dr. Platt and Doctors Marina de Deus Moura de Lima, Heloísa Clara Santos Sousa & Lúcia de Fátima Almeida de Deus Moura,

I would like to thank the interest and suggestions on our manuscript, published in 2013, entitled "Hypoplastic enamel treatment in permanent anterior teeth of a child "(1)

The diagnosis provided for the clinical case reported was based on the characteristics of the lesions on the teeth submitted to the treatment, considering mainly the origin of the lesion (trauma of the deciduous teeth during the enamel mineralization of the permanent teeth) and following other published papers showing similar lesions. (2,3) However, we have to agree with some of the considerations suggested: in the case reported, the alterations were qualitative and affected basically the degree of opacity. Considering there was no structure loss and according to the Developmental Defects of Enamel (DDE) index, should be classified as demarcated opacities instead of hypoplastic spots. (4–6)

Even with the possibility of a misdiagnosis, it was clear that was not a case of Molar-incisor Hypomineralization (MIH) as suggested (7–9), once the lesions etiology was detected in the patient medical-dental history (trauma) and clinical evaluation, which showed that the molars were not affected.

We also would like to clarify that, even if the stains had received a different classification, the treatment conducted to the altered enamel, with previous dental bleaching for the reduction of the brownish characteristic of the lesions, in function of the lesions depth, followed by the composite restoration, would be kept, considering that "esthetic" was the determinant factor for the decision of restoring the teeth.

Based on that, we are sure this discussion will be able to contribute to an improvement in the scientific approach of the paper.

Sincerely,

Dr. Luana Dutra de Carvalho and co-authors

1. Carvalho LD, Bernardon JK, Bruzi G, Andrada M a. C, Vieira LCC. Hypoplastic enamel treatment in permanent anterior teeth of a child. Oper Dent. 2013 Aug;38(4):363-8.
2. Bernardon JK, Gondo R, Baratieri LN. Minimally invasive restorative treatment of hypoplastic enamel in anterior teeth .pdf. Am J Esthet Dent. 2011 Fall;1(1):1-24.
3. Reston E, Corba D, Ruschel K, Tovo M, Barbosa A. Conservative Approach for Esthetic Treatment of Enamel Hypoplasia. Oper Dent. 2011 May 1;36(3):340-3.
4. Wong HM. Aetiological factors for developmental defects of enamel. 2014 [cited 2016 Jul 4]; Available from: http://hub.hku.hk/handle/10722/200426
5. Seow WK. Clinical diagnosis of enamel defects: pitfalls and practical guidelines. Int Dent J. 1997 Jun;47(3):173-82.
6. A review of the developmental defects of enamel index (DDE Index). Commission on Oral Health, Research & Epidemiology. Report of an FDI Working Group. Int Dent J. 1992 Dec;42(6):411-26.
7. Weerheijm KL. Molar incisor hypomineralisation (MIH). Eur J Paediatr Dent Off J Eur Acad Paediatr Dent. 2003 Sep;4(3):114-20.
8. Hysi D, Kuscu OO, Droboniku E, Toti C, Xhemnica L, Caglar E. Prevalence and aetiology of Molar-Incisor Hypomineralisation among children aged 8-10 years in Tirana, Albania. Eur J Paediatr Dent Off J Eur Acad Paediatr Dent. 2016 Mar;17(1):75-9.
9. Silva MJ, Scurrah KJ, Craig JM, Manton DJ, Kilpatrick N. Etiology of molar incisor hypomineralization - A systematic review. Community Dent Oral Epidemiol. 2016 Apr 28;

Dear Editor Dr Platt,

First of all, we thank our colleagues, Dr Marina de Deus Moura de Lima, Dr Heloísa Clara Santos Sousa, and Dr Lúcia de Fátima Almeida de Deus Moura, for their interest and qualified comments on our paper (1).

In response to their comments, we hereby offer clarifications as follows.

1. Even though the scope of our study was to describe a minimally invasive technique for the removal of intrinsic enamel stains and enamel discoloration, the terminology used to describe the clinical findings is in line with the relevant literature. The references quoted by the readers in their letter and in our original paper clearly use the same terms to describe the clinical features of the developmental defects addressed in the case report.

Figure 1 shows the anterior teeth (incisors and canines), evidencing a defect associated with reduced localized thickness, characterizing hypoplasia. Even though tooth # 9 shows a greater area of diffuse opacity, we chose to classify both this tooth and tooth # 8 as presenting hypoplasia, because this pathology was the most relevant one in that situation (2,3).

Hypoplasia is shown in detail and enlarged in Figure 1, as can also be seen in the original report.

2. With regard to the differential diagnosis of molar-incisor hypomineralization (MIH) for our case, we highlight the unequivocal involvement of the canine (tooth # 6) in the process behind the systemic abnormalities that affected tooth development.

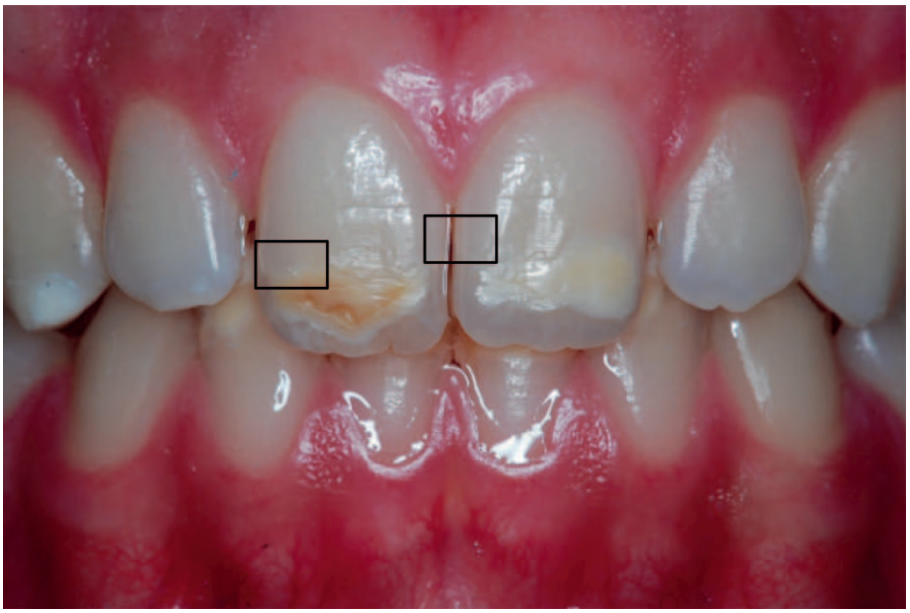


Figure 1. *Areas with hypoplastic characteristics in the patient's anterior teeth.*

There are very rare indications in the literature suggesting that canines could be involved in cases of MIH (4), as the processes of matrix apposition and mineralization in molars, incisors, and canines are not totally simultaneous. We believe that, in our case, canine involvement rules out the diagnosis of MIH.

Still regarding this differential diagnosis, when searching the PubMed database using the keywords molar incisor hypomineralization and MIH, we found that 75% of the papers available are dated 2010 or later. This fact, combined with the other arguments here presented, may suggest that at the time of publication of our original report (2011), the diagnosis of MIH was restricted to very specific cases, and therefore this condition was not included in the literature review conducted at the time. Still, it is relevant to note that the technique described in our report is one option among the treatment possibilities applicable to MIH (5).

Finally, while we acknowledge the importance of a standardized nomenclature for defects resulting from odontogenic abnormalities, it is important to mention that it was not the primary objective of the study to detail the etiology of the case. The conciseness required when writing case reports also contributed to our focus on the treatment technique described.

EG Reston
DDS, MSD, PhD, School of Dentistry, Professor and Head of the Graduate Program, Lutheran University of Brazil, Canoas, Brazil

MF Tovo
DDS, MSD, PhD, School of Dentistry, Associate Professor, Lutheran University of Brazil, Canoas, Brazil

References

1. Reston EG, Corba DV, Ruschel K, Tovo MF, Barbosa AN (2011) Conservative approach for esthetic treatment of enamel hypoplasia *Operative Dentistry* **36(3)** 340-343.
2. World Health Organization (1997) *Oral health surveys: basic methods* 4th ed WHO, Geneva.
3. Federation Dental International (1992) A review of the developmental defects of enamel index (DDE Index) - Commission on Oral Health, Research & Epidemiology: Report of an FDI Working Group *International Dental Journal* **42(6)** 411-426.
4. Bhaskar S, Hegde S (2014) Molar-incisor hypomineralization: prevalence, severity and clinical characteristics in 8- to 13-year-old children of Udaipur, India *Journal of the Indian Society of Pedodontics and Preventive Dentistry* **32(4)** 322-329.
5. William V, Messer LB, Burrow MF (2006) Molar incisor hypomineralization: review and recommendations for clinical management *Pediatric Dentistry* **28(3)** 224-232.

©Operative Dentistry, 2016, **41-6,** 675-676

Faculty Positions

CARIOLOGY AND OPERATIVE DENTIST
Department of Cariology and Comprehensive Care
NEW YORK UNIVERSITY COLLEGE OF DENTISTRY

The New York University College of Dentistry, Department of Cariology and Comprehensive Care, seeks full time tenure track or non-tenure depending on qualifications and history of research and teaching experience in Cariology and Operative Dentistry. Applicants must possess either a dental degree, or Ph.D., or both and should have a proven track record in funded research; and should be published with a record of lectures and seminars presented both nationally and internationally. Responsibilities include; research and teaching at the undergraduate and graduate level. Salary and benefits are highly competitive. Academic rank will be commensurate with experience. Individuals with a NY State dental license will be eligible to participate in our faculty practice.

Please submit a letter of interest along with current curriculum vitae to **Mark S. Wolff, DDS, Ph.D., Professor and Chairman, Department of Cariology and Comprehensive Care, New York University College of Dentistry, 345 East 24th Street, 10 Floor, New York, NY 10010-4086**. *NYU appreciates all responses, but can only respond to qualified applicants*.

NYU is an Equal Opportunity/Affirmative Action Employer.

College of Dental Medicine – Illinois [CDMI]: Research Faculty Position

Introduction: Midwestern University, initially founded in 1900, is a healthcare educational institution offering professional doctoral degrees in osteopathic medicine, dentistry, pharmacy and other health sciences programs on its two (2) campuses in Illinois and Arizona. The Midwestern University College of Dental Medicine – Illinois (CDMI) is located on our Downers Grove / Illinois Campus with 105 acres boasting innovative facilities, nestled serenely in a wooded setting 25 miles west of downtown Chicago. The total enrolment of the University is over 6,000 students with approximately 550 full time faculty dedicated to providing inter-professional education and the development of leaders in a humanistic and scholarly environment.

Position Description: CDMI is seeking applications for a full-time, tenure-track, faculty position with a focus on Dental Research at the rank of Assistant or Associate Professor. This full-time faculty member will take a lead role in developing and fostering research endeavors among the faculty and students at CDMI. The faculty member will participate in the pre doctoral dental program, mentor faculty members and dental students in their research endeavors, and conduct an active research program. It is expected that the selected candidate will also establish an externally funded research program in educational, basic science or clinical research. The faculty member will be responsible for application of expertise in innovative ways that benefit collaborators from the campus, the community and improves research productivity. All faculty at CDMI are expected to participate in the teaching mission of Midwestern University, to participate in scholarly activity and to demonstrate commitment to service in the larger community.

Qualifications: Candidates must possess a PhD or equivalent degree and an established record of extramural funding with quality scholarly and research productivity in the field of craniofacial, oral, dental, and/or related research. Preference will be given to candidates with a DDS or DMD degree.

The successful candidate will be an individual with excellent communication and interpersonal skills, with the ability to engage newer, clinical faculty in the realm of dental research.

Academic rank and salary will be commensurate with training and experience.

Interested individuals should submit a letter of application and interest, curriculum vitae, and three

professional references, plus a one- to two-page summary of a research plan that includes the engagement of clinical faculty, to:

College of Dental Medicine - Illinois
Midwestern University
Attention: Dr. Bruno Jham
555, 31st Street, Auditorium Building 594
Downers Grove, IL 60515
Email: bjham@midwestern.edu

Applications can also be made on-line at www.midwestern.edu.

Midwestern University is an Equal Opportunity/Affirmative Action employer that does not discriminate against an employee or applicant based upon race, color, religion, gender, national origin, disability, or veterans status, in accord with 41 C.F.R. 60-1.4(a), 250.5(a), 300.5(a) and 741.5(a).

©Operative Dentistry, 2016, **41-6,** 677-678

Reviewers

Operative Dentistry, Inc. would like to thank our conscientious team of Reviewers for their hard work, tenacity, and dedication in the furthering of operative dentistry around the word. These individuals dedicate innumerable hours in reading, re-reading, and critiquing manuscripts. Submitted articles, accepted for publication or not, all benefit from these reviewers who help authors present their hard work, as well as verifying that the work we publish is scientifically accurate, clinically relevant, and professionally uplifting. We cannot thank these individuals enough for their contributions, but we do want to publically recognize them.

Be it known here, and throughout the world, that the following named individuals have contributed real and invaluable service to the profession of Dentistry by volunteering their time and talents to the cause of peer-review for Operative Dentistry, Inc.

The time of service listed below is for the 2016 publication year.

Adcook, Richard , United States Navy
Aguilera, Fatima , University of Granada
Ajlouni, Khaldoun , Texas A&M Health Science Center - Baylor
Ando, M , Indiana University
Armstrong, Daniel , Portland ME USA
Armstrong, Steve , University of Iowa
Bailey, Clifton , United States Air Force
Bardwell, David , Tufts University
Barkmeier, Wayne , Creighton University
Beatty, Mark , University of Nebraska
Bernardon, Jussara , Federal University of Santa Catarina
Berzins, David , Marquette University
Blank, Lawrence , University of Maryland
Botero, Tatiana , University of Michigan
Brackett, William , Augusta University
Broome, James , University of Alabama - Birmingham
Brunton, Paul , University of Leeds
Buck, Richard , United States Air Force
Burrow, Michael , University of Melbourne
Certosimo, Fred , Virginia Commonwealth University
Chan, Daniel , University of Washington
Chen, Liang , Bisco Inc.
Chutinan, Supattriya , Harvard University
Cook, N. Blaine , Indiana University
Cruz, Adriana , Fluminense Federal University
De Peralta, Tracy , University of Michigan
Delgado, Alex , University of Florida
Dennison, Joseph , University of Michigan
Diefenderfer, Kim , Indiana University
Durski, Marcelo , University of Louisville
Eichmiller, Fred , Delta Dental of Wisconsin
El-Mowafy, Omar , University of Toronto
Espinosa, Sofia , Universidad Intercontinental
Faria-e-Silva, Andre , Federal University of Sergipe
Fasbinder, Dennis , University of Michigan
Ferracane, Jack , Oregon Health & Science University
Flury, Simon , University of Bern
Foxton, Richard , King's College London
Frazier, Kevin , Augusta University
Fugaro, Jessica , Bainbridge, WA USA
Fugaro, Orlando , Bainbridge, WA USA
Furness, Alan , Augusta University
Gallusi, Gianni , Università degli Studi di Roma Tor Vergata
Galvis, Diana , Rutgers University
Geraldeli, Saulo , University of Florida
Gokçe, Bülent , Ege University
Gorthy, Jeanette , United States Navy
Gureckis, Kevin , University of Texas Health Science Center-SA
Hachmeister, Kevin , Melbourne, FL USA
Hermesch, Charles , University of Texas Health Science Center-SA
Hilton, Thomas , Oregon Health & Science University
Holleron, Barry , San Antonio, TX USA
Jain, Poonam , Southern Illinois University - Edwardsville
Jessup, Jeffrey , United States Air Force
Johnson, William , University of Nebraska
Jones, Gordon , United States Navy
Kazemi, Reza , University of Connecticut
Kelly, J Robert , University of Connecticut
Kilinc, Evren , Nova Southeastern University
Kofford, Kelly , Texas A&M Health Science Center - Baylor
Kooistra, Scott , United States Navy
Kunnilathu, Abraham , University of Western Ontario
Kwon, So Ran , Loma Linda University
Lafuente, David , University of Costa Rica
Laswell, Harold R. , University of Kentucky

Online Only Articles

On occasion we receive manuscripts that we would like to publish, but do not have the page room to include in the print journal. For the full article, please go to www.jopdentonline.org or enter the provided address into your address bar.

Effect of Intracanal Irrigants on Bond Strength of Fiber Posts Cemented With a Self-adhesive Resin Cement

MS Barreto • RA Rosa • VG Seballos • E Machado • LF Valandro • OB Kaizer • MVR Só • CAS Bier

Clinical Relevance: Saline solution or sodium hypochlorite associated with ultrasonic activation seems to be an adequate solution for root canal cleaning before fiber post cementation. Chelating agents may cause decreased bond strengths when cementing with self-adhesive resin cement.

doi: http://dx.doi.org/10.2341/15-246-L

Multidisciplinary Treatment of Complicated Crown-Root Fractures: A Case Study

IL Stojanac • BV Bajkin • MT Premovic • BD Ramic • LM Petrovic

Clinical Relevance: The present clinical report describes the successful therapeutic treatment of complicated crown-root fractures and demonstrates the necessity of a multidisciplinary approach during the therapy of traumatized teeth.

doi: http://dx.doi.org/10.2341/15-080-T

Relined Fiberglass Post: Effect of Luting Length, Resin Cement, and Cyclic Loading on the Bond to Weakened Root Dentin

NC de Souza • ML Marcondes • DFF da Silva • GA Borges • LH Burnett Júnior • AM Spohr

Clinical Relevance: Under mechanical stresses, the luting length is important for the retention of relined fiberglass posts luted to weakened roots with conventional or self-adhesive resin cements.

doi: http://dx.doi.org/10.2341/15-233-L

Influence of the Compliance and Layering Method on the Wall Deflection of Simulated Cavities in Bulk-fill Composite Restoration

Y-J Kim • R Kim • JL Ferracane • I-B Lee

Clinical Relevance: Both conventional and bulk-fill composites showed lower wall deflection when incrementally filled. Restoration by bulk filling with high viscous bulk-fill composites cannot achieve the reduction in the wall deflection of simulated cavities comparable to those obtained with incremental layering of conventional universal composites.

doi: http://dx.doi.org/10.2341/15-260-L

Degradation Potential of Bulk Versus Incrementally Applied and Indirect Composites: Color, Microhardness, and Surface Deterioration

M El Gezawi • D Kaisarly • H Al-Saleh • A ArRejaie • F Al-Harbi • KH Kunzelmann

Clinical Relevance: Covering bulk fills with silorane-based composites might be a better practice for delaying surface degradation.

doi: http://dx.doi.org/10.2341/15-195-L

This page was intentionally left blank.

This page was intentionally left blank.

This page was intentionally left blank.

©Operative Dentistry, 2016, **41-6,** e159-e167

Effect of Intracanal Irrigants on Bond Strength of Fiber Posts Cemented With a Self-adhesive Resin Cement

MS Barreto • RA Rosa • VG Seballos • E Machado
LF Valandro • OB Kaizer • MVR Só • CAS Bier

Clinical Relevance

Saline solution or sodium hypochlorite associated with ultrasonic activation seems to be an adequate solution for root canal cleaning before fiber post cementation. Chelating agents may cause decreased bond strengths when cementing with self-adhesive resin cement.

SUMMARY

Objective: The aim of this study was to evaluate the effect of five intracanal irrigants on bond strength of fiber posts cemented with newer self-adhesive resin cement.

Methods: A total of 60 extracted, single-rooted human premolars, sectioned at 14 mm, were prepared with the ProTaper Universal system with a size F3 instrument and filled with an F3 master cone and AH Plus. The root canal filling was partially removed, leaving 4 mm of apical gutta-percha. Specimens were randomly assigned to five groups (n=12), according to the solution used for dentin surface treatment before fiber post cementation, as follows: EDTA 17% (EDTA); QMix (QM); SmearClear (SC); 2.5% sodium hypochlorite (NaOCl), and 0.9% saline solution (SS). Ultrasonic activation was performed (three times, 20 seconds each), and root canals were dried with paper points. Fiber posts were cemented with RelyX U200. In one specimen per group, rhodamine B dye was mixed with RelyX U200 to provide adequate fluorescence for confocal laser scanning microscopy (CLSM) assessment. Specimens were

Mirela Sangoi Barreto, DDS, MSD, private practitioner, Santa Maria, Brazil

Ricardo Abreu da Rosa, DDS, MSD, PhD, private practitioner, Santa Maria, Brazil

Vivian Ghem Seballos, DDS, MSD, graduate student in Oral Sciences (Prosthodontics), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil

Eduardo Machado, DDS, MSD, private practitioner, Santa Maria, Brazil

*Luiz Felipe Valandro, MSD, PhD, associate professor, Graduate Program in Oral Science (Prosthodontics), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil

Osvaldo Bazzan Kaizer, MSD, PhD, adjunct professor, Graduate Program in Oral Science (Prosthodontics), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil

Marcus Vinícius Reis Só, MSD, PhD, adjunct professor, Graduate Program in Dentistry (Endodontic), Faculty of Odontology, Federal University of Rio Grande do Sul, Porto Alegre, Brazil

Carlos Alexandre Souza Bier, MSD, PhD, adjunct professor, Graduate Program in Oral Science (Endodontics), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil

*Corresponding author: R. Floriano Peixoto, 1184, 97015-372, Rio Grande do Sul State, Santa Maria, Brazil; e-mail: lfvalandro@hotmail.com

DOI: 10.2341/15-246-L

transversally sectioned and three slices were obtained, one for each root third. Next, a push-out test was performed. A stereomicroscope and CLSM were used to analyze the failure modes and to illustrate the pattern of infiltration of RelyX U200 into dentinal tubules, respectively. Bond strength means were calculated, and analysis of variance and Bonferroni tests were used for statistical analysis.

Results: SS showed the highest mean bond strength values (11.5±5.3), superior to QM (5.1±3.1) and SC (5.1±3.3). NaOCl presented intermediary bond strength values (9.7±5.0), similar to EDTA (7.7±2.9) and SS. QM and SC showed the lowest mean bond strength ($p<0.05$). Adhesive failures between cement/dentin were predominant (53.9%).

Conclusion: SS and NaOCl associated with ultrasonic activation seem to be adequate solutions for root canal cleaning before fiber post cementation with self-adhesive resin cement, whereas chelating solutions, such as EDTA, QM, and SC, cause a decrease in bond strength.

INTRODUCTION

Endodontically treated teeth may exhibit pronounced coronal destruction, and the amount of residual coronal dentin can influence the clinical survival of posts and restorations.[1] The minimally invasive approach of associating adhesive techniques and posts with similar mechanical characteristics to dentin seems to contribute to a better clinical prognosis for endodontically treated teeth.[1,2] A direct relationship between fracture resistance and the amount of remaining tooth structure have been reported.[3] Some studies[4] have shown that the presence of substantial remaining coronal tooth structure reduces the risk of failure. Due to this fact, fiber post cementation is a viable alternative to restore weakened teeth.

Glass fiber posts were introduced as an alternative to cast metal posts, because glass fiber presents mechanical properties similar to dentin,[4,6] such as elastic moduli, which improves the distribution of functional loads to the root canal, instead of concentrating it, when compared with cast posts,[7] which might affect the risk and type of root fracture.[8] Several other factors led to the use of glass fiber posts, such as esthetic advantages, low costs, and being a simpler and less time-consuming technique.[6]

In terms of fiber post cementation, self-adhesive resin cements, such as RelyX U200 and RelyX U100 (3M ESPE, St Paul, MN, USA), have been introduced to reduce the sensitivity of pretreatment steps and to prevent application errors of cementation procedures. This factor may increase bond strength, as compared with a conventional three-step system.[9] According to Rodrigues and others[10] and Amaral and others,[11] self-adhesive resin cements are equally effective alternatives to conventional resin cement.

Ideally, a post cement system will provide a tight seal impermeable to oral bacteria; however, debonding failures have been reported.[4] Several variables may be associated with these failures, such as the action of irrigant solutions on dentin collagen (sodium hypochlorite [NaOCl], hydrogen peroxide, chlorhexidine [CHX]); the peculiar conditions of root canal dentin and the type of agent used to condition the substrate; the polymerization stress of resin cement; and the chemical and physical properties of the posts.[12]

To enable satisfactory adhesion of posts to root dentin, the smear layer has to be removed.[13] It consists of an agglomeration of dentin, irrigant solutions, and organic tissues poorly adhered to the root canal walls.[14] The smear layer is able to hinder penetration and adaptation of self-adhesive resin sealers inside dentinal tubules,[13] which may decrease bond strength.

In this sense, chelating solutions play an important role in removing debris and the smear layer.[15] Ethylenediaminetetraacetic acid 17% (EDTA) is the most widely used chelating solution because it enables dissolution of the inorganic portion of dentin and the smear layer.[16] On the other hand, EDTA can cause erosion of root canal dentin[17] and presents reduced antimicrobial action.[18]

With the aim of increasing antimicrobial activity without producing dentin erosion, new irrigants have been proposed. EDTA-based formulations have been developed as final-rinse solutions, such as SmearClear (SC) and QMix (QM).[19] SC (SybronEndo, West Collins, Orange, CA, USA) presents EDTA and cetrimide in its formulation, whereas QM (Dentsply Tulsa Dental, Tulsa, OK, USA) contains EDTA, CHX, and a surfactant agent. This one-step final rinse is supposed to combine the antimicrobial and substantivity properties of CHX with the smear-layer-removing properties of EDTA.[20]

Despite the reported literature, few studies have evaluated the effect of irrigating and chelating solutions on bond strength to root dentin. Moreover,

information related to fiber posts cemented with RelyX U200 is still limited. Therefore, the main goal of this *ex vivo* study was to evaluate the effect of different, ultrasonically activated intracanal irrigants on the bond strength between root dentin and fiber posts cemented with self-adhesive resin cement.

The null hypothesis tested was that different irrigant solutions would have no influence on the bond strength.

METHODS AND MATERIALS

Experimental Design

First, the sample size was calculated, using the BioEstat 5.0 program (Fundação Mamirauá, Belém, Brazil), considering parameters that were based on Rosa and others:[21] minimum difference between groups of 1.65 MPa; standard deviation of 1.1 MPa; power of 80%; for five treatments. The program recommended 12 samples per group as the sample size.

Roots (N=60) were randomly allocated (http://www.random.org) to five groups (n=12), considering one factor (irrigant solutions) at five levels. The main outcome was push-out bond strength and the experimental unit was the root. The operators were blinded to the applications of intracanal solutions, post cementations, push-out tests, and failure analyses.

Tooth Selection

This study was submitted to and approved by the Ethical Committee of the Federal University of Santa Maria (No. 855,457). A total of 60 single-rooted human mandibular premolars with similar dimensions were selected and stored in a 0.9% saline solution at 4°C until use. Periapical radiographs were performed to confirm the presence of one root canal. All roots were observed at 8× magnification with a stereomicroscope (Zeiss Stemi SV6; Carl Zeiss, Jena, Germany) to exclude those with external cracks, incomplete root formation, root resorption, or coronal root canal diameter greater than 2 mm, as measured with a digital caliper (Starrett 727; Starrett, Itu, Brazil).

Specimens were decoronated at the cervical root third to standardize a remaining root length of 14 mm, using a diamond blade (Komet, Santo André, SP, Brazil) under cool water.

Root Canal Preparation

Canal patency was established with a size 10 K-file (Dentsply Maillefer, Chemin du Verger, Ballaigues, Switzerland), followed by PathFile 1, 2, and 3 (Dentsply Maillefer) instruments. The working length was set at 1 mm from the apex. Root canals were prepared by using the ProTaper Universal System (Dentsply Maillefer). Initially, the cervical and middle portions of the roots were prepared by using S1, SX, and S2 instruments. Later, S1, S2, F1, F2, and F3 files were sequentially used for all of the working lengths. Each canal was irrigated with 2 mL of a freshly prepared 2.5% NaOCl (Asfer, São Paulo, Brazil) between each instrument change. Specimens were irrigated with 5 mL of 17% EDTA (Biodinâmica, Ibiporã, Brazil) for three minutes and subsequently rinsed with 2 mL of NaOCl. Next, they were dried by using size 30 paper points (Dentsply Maillefer).

Root Canal Filling

AH Plus (Dentsply Maillefer) was mixed according to the manufacturer's instructions and placed in working length by using a 400-rpm lentulo spiral (Dentsply Maillefer) for five seconds. The single-cone technique was performed by using F3 (Dentsply Maillefer) main gutta-percha cones, coated with sealer and placed into root canals to the working length. The excess gutta-percha in the coronal portion was removed with a flame-heated plugger, and the access cavity was sealed with Filtek Z350 (3M ESPE) composite resin. Roots were stored for one week at 37°C and 100% humidity to allow the sealers to set.

Post Space Preparation

Root canal filling was partially removed using sizes 1, 2, 3, and 4 Largo drills (Dentsply Malleifer), alternating with 0.9% saline solution irrigation, in 10 mm length, leaving 4 mm of apical gutta-percha. Post space preparation was completed using the Exacto Translúcido Angelus N2 (Angelus, Londrina, Brazil) bur at 10 mm. Periapical radiographs were performed to confirm removal of the root filling.

Apical root portions were included in a chemically cured acrylic resin (Dencrilay Dencril, Pirassununga, Brazil) block. The specimens were fixed on a surveyor, with the long axes of the teeth and the resin block parallel to each other and perpendicular to the ground.

Irrigation Protocols

As mentioned, specimens were randomly assigned to five groups (n=12), according to final flushing after post space preparation, as follows: EDTA (EDTA

17%); QM (QMix); SC (SmearClear); NaOCl (2.5% NaOCl), and SS (0.9% saline solution).

EDTA, QM, SC, NaOCl, and SS were delivered into root canals using Ultradent syringes (Ultradent Products Inc, South Jordan, UT, USA) and 30G EndoEzeTip needles (Ultradent Products Inc).

The irrigation protocols for all experimental and control groups were as follows: 1) root canals were rinsed with 1 mL of the corresponding irrigant; 2) ultrasonic activation with a size 20/0.1 ultrasonic tip (Capelli e Fabris Ind, Santa Rosa do Viterbo, Brazil), attached to an NAC Plus ultrasonic device (Adiel LTDA, São Paulo, Brazil) was performed for 20 seconds, without touching root canal walls. This procedure was repeated twice and the irrigant was renewed;[22] 3) a final continuous irrigation with 2 mL of 0.9% saline solution was performed in all groups.

Specimens were dried with paper points. Exacto Translúcido N2 (Angelus) glass fiber posts were cleaned with ethyl alcohol 70%, coated with silane (Angelus) and put out to dry for five minutes, until solvent evaporation.

Rhodamine B dye in a ratio of 0.1%[23] was mixed with RelyX U200 (3M ESPE) in one specimen per group, to provide the fluorescence that enabled CLSM assessment. This procedure allowed the illustration of the patterns of self-adhesive resin cement distribution into the dentinal tubules.

RelyX U200 was mixed according to the manufacturer's specification, inserted into the root canal using Automix tips (3M ESPE), and immediately after, the fiber post was inserted by manual pressure. The cement was light-cured for 40 seconds using a previously calibrated LED light-curing unit (Radii Cal; SDI, Melbourne, Australia), maintaining the light guide tip of the light-curing unit placed perpendicular to the post. A single operator performed all procedures. The coronal access was sealed with composite resin (Filtek Z350; 3M ESPE). Roots were stored for one week at 37°C.

Sample Preparation for CLSM Analysis

To illustrate the pattern of distribution of RelyX U200 inside dentinal tubules of each group, one specimen per group was prepared for CLSM.

A cutting machine (Extec Labcut 1010, Enfield, CT, USA) was used for sectioning transversally the roots, providing three slices, one for each root third. For CLSM assessment, surfaces were polished with Arotec paste (Arotec, Cotia, Brazil) to eliminate dentin debris generated during the cutting procedures. The coronal surface of one sample per group was examined with the Olympus FluoView Confocal Laser 1000 Microscope (Olympus Corporation, Tokyo, Japan). The absorption and emission wavelengths for rhodamine B were 540 nm and 494 nm, respectively. Dentin samples were analyzed using the 10× oil lens.

Push-out Test

The first cervical slice (approximately 1 mm thick) was discarded due to excess cement, which could influence the adhesive resistance. Three other slices per specimen (thickness: 2±0.3 mm) were obtained. Each slice was positioned on a metallic device with a central opening (Ø=3 mm) larger than the canal diameter. The most coronal portion of the specimen was placed downward. For the push-out test, a metallic cylinder (Ø extremity=0.8 mm) induced a load on the post in an apical to coronal direction, without applying any pressure on the cement and/or dentin.

The push-out test was performed in a universal testing machine (Emic DL-2000; Emic, Sao Jose dos Pinhais, Brazil) at a speed of 1 mm/min. Bond strength values (σ) in MPa were obtained as follows: $\sigma = f/a$, where f = load for specimen rupture (N) and a = bonded area (mm^2). To determine the bonded interface area, this formula was used: $A = 2\pi g\,(R^1 + R^2)$, where $\pi = 3.14$, g = slant height, R^1 = smaller base radius, and R^2 = larger base radius. To determine the slant height, the following calculation was used: $g^2 = (h^2 + [R^2 - R^1]^2)$, where h = section height. R^1 and R^2 were obtained by measuring the internal diameters of the smaller and larger base, respectively, which corresponded to the internal diameter between the root canal walls. The diameters and h were measured using a digital caliper (Starrett 727, Starrett).[2,24]

Failure Mode Analysis

Dentin slices were analyzed first in a stereomicroscope (Zeiss Stemi SV6), and some samples were selected for scanning electron microscopy (JEOL 6060, JEOL, Tokyo, Japan) in order to categorize and illustrate the failure modes, respectively. The failure modes were categorized as follows: Ac/d = predominant adhesive at cement/dentin interface failure; Ac/p = predominant adhesive at cement/post interface failure; CC = cement cohesive failure; DC = dentin cohesive failure; PC = post cohesive failure. Specimens presenting cohesive fracture of the fiber post or dentin were excluded from the study given

Table 1: *Mean of Bond Strength Values and Failure Modes Distribution After Push-Out Test for the Irrigants Used Prior to the Post Cementation*

Groups	Bond Strength	Failure					
		Ac/d	Ac/p	DC	PC	CC	Total
SS	11.5 ± 5.3 [a]	19	4	13	—	—	36
NaOCl	9.7 ± 5.0 [ab]	18	8	10	—	—	36
QM	5.1 ± 3.1 [c]	16	15	5	—	—	36
SC	5.1 ± 3.3 [c]	18	6	12	—	—	36
EDTA	7.7 ± 2.9 [bc]	26	2	8	—	—	36
	Total	97 (53.9%)	35 (19.5%)	48 (26.6%)	0 (0%)	0 (0%)	180 (100%)

Abbreviations: Ac/d, predominant adhesive at cement/dentin interface failure; Ac/p, predominant adhesive at cement/post interface failure; CC, cement cohesive failure; DC, dentin cohesive failure; NaOCl, sodium hypochlorite; PC = post cohesive failure; QM, QuickMix; SC, SmearClear; SS, 0.9% saline solution.
** Different superscript letters identify statistically significant differences at the* $p < 0.05$ *level.*

that these types of failures did not represent real push-out bond strength.

Data Analysis

The κ test was used to analyze the intraexaminer agreement regarding failure modes. The mean of bond strength distribution was checked with the Shapiro-Wilk test. One-way analysis of variance (ANOVA) and Bonferroni tests (SPSS 12.0; SPSS Inc., Chicago, IL, USA) were used for statistical analysis. The significance level was set at 5%.

RESULTS

The κ value was 0.84. After the push-out test, some dentin cohesive failures were observed and those specimens were excluded from the bond strength calculations.

One-way ANOVA revealed a significant difference among the groups (p=0.0009). SS showed the highest mean bond strength values, superior to QM and SC (p<0.05). NaOCl presented intermediary bond strength values, similar to EDTA and SS (p>0.05). QM and SC showed the lowest mean bond strength (p<0.05) (Table 1).

Table 1 also presents failure mode distribution. Adhesive failures between cement/dentin (Ac/d) were predominant (53.9%), followed by adhesive failures between cement/post (Ac/p) (19.5%), whereas dentin cohesive (DC) failures represented 26.6% of the specimens. Post cohesive failures (PC) and cement cohesive failures (CC) were not observed. Figure 1 represents the failure modes.

Figure 2 shows the penetration patterns of RelyX U200 into dentinal tubules. Images A (EDTA), C (NaOCl), and E (SS) showed the penetration of RelyX U200 into dentinal tubules. SS presented the most homogeneous penetration into dentinal tubules, along the entire perimeter of the root canal. Images B (QM) and D (SC) showed RelyX U200 limited to the root canal perimeter and not in the dentinal tubules.

DISCUSSION

This investigation indicated that the tested intracanal irrigants promoted different push-out bond strengths; thus, the null hypothesis was rejected. Higher means of bond strength were found for the SS and NaOCl groups, whereas SC and QMix presented the lowest means and EDTA presented intermediary mean values.

This study tested the RelyX U200 resin cement, and the manufacturer recommends the use of NaOCl flushing prior to fiber post cementation; however, the criteria used for choosing this irrigant is not clear. At the moment, no study supports this indication, nor is the NaOCl concentration clear.

It is known that NaOCl acts upon organic components of the dentin and improves the penetration of monomers into the dentinal structure.[25] However, NaOCl presents the potential for collagen degradation, which could affect bond strength to root dentin.[12] A negative correlation was found between dentin exposure time to NaOCl and bond strength.[26] Zhang and others[27] stated that a long period of dentin exposure to a high concentration of NaOCl is able to reduce the bond strength and could result in root fracture. However, in this study the high bond strength values achieved in the NaOCl group may be explained because an intermediary concentration was used (2.5%) just for 60 seconds, contributing to root canal cleaning, reversing possible bond strength loss caused by chelating solutions used during root canal treatment, not resulting in collagen degradation.

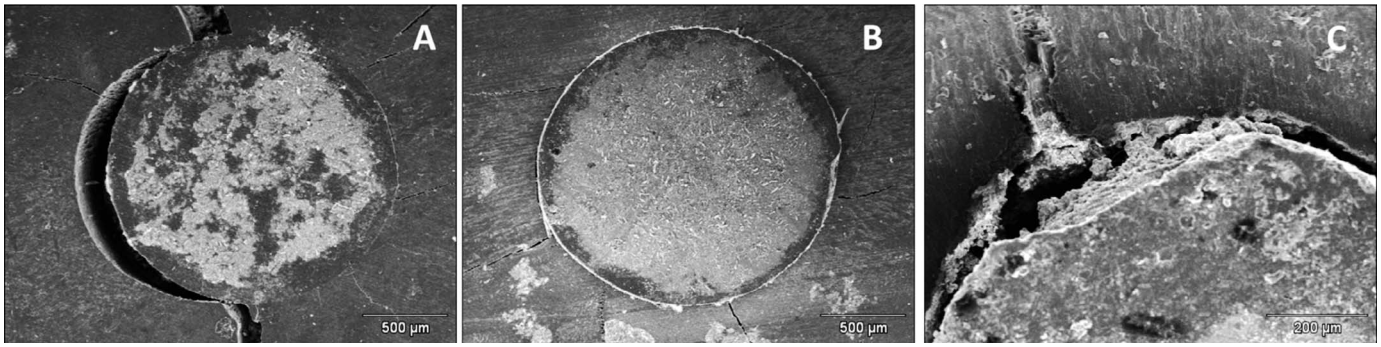


Figure 1. *Scanning electron microscopy photomicrographs of fiber post/self-adhesive resin cement and self-adhesive resin cement/root dentin interfaces. Failure modes are illustrated as follows: (A): Dentin cohesive (DC) failure (50×). (B): Predominant adhesive at cement/dentin (Ac/d) failure (50×). (C): Predominant adhesive at cement/post interface (Ac/p) failure (150×).*

Saline solution presents no antimicrobial and chelating properties. In endodontics, it is frequently associated with CHX 2% gel, for root canal preparation. SS is not able to dissolve organic tissue; however, its ultrasonic activation resulted in higher bond strength values. Such results are also associated with a nondeleterious effect of SS over root dentin. The cleaning efficacy of passive ultrasonic irrigation (PUI) implies the effective removal of dentin debris, microorganisms (planktonic or in biofilm), and organic tissue from the root canal.[22] The cleaning effect of PUI associated with SS can be attributed to cavitation and microstreaming effects of PUI to root canal walls.[22] In this sense, PUI was used to enhance the irrigating and chelating potential of the solutions, exposing dentinal tubules for cement penetration, which could contribute to higher bond strength values. When self-adhesive resin cement is used, a chelating solution plays an important role in smear layer removal,[28] given that no previous acid etching is indicated in the resin cementation technique. This acid etching in a conventional three-step system is responsible for smear layer removal. Rodrigues and others[10] observed that 37% phosphoric acid etching prior to self-adhesive resin cement did not improve the bond strength values.

Chelating agents are widely used in endodontic routines, especially EDTA. It is in a salt obtained

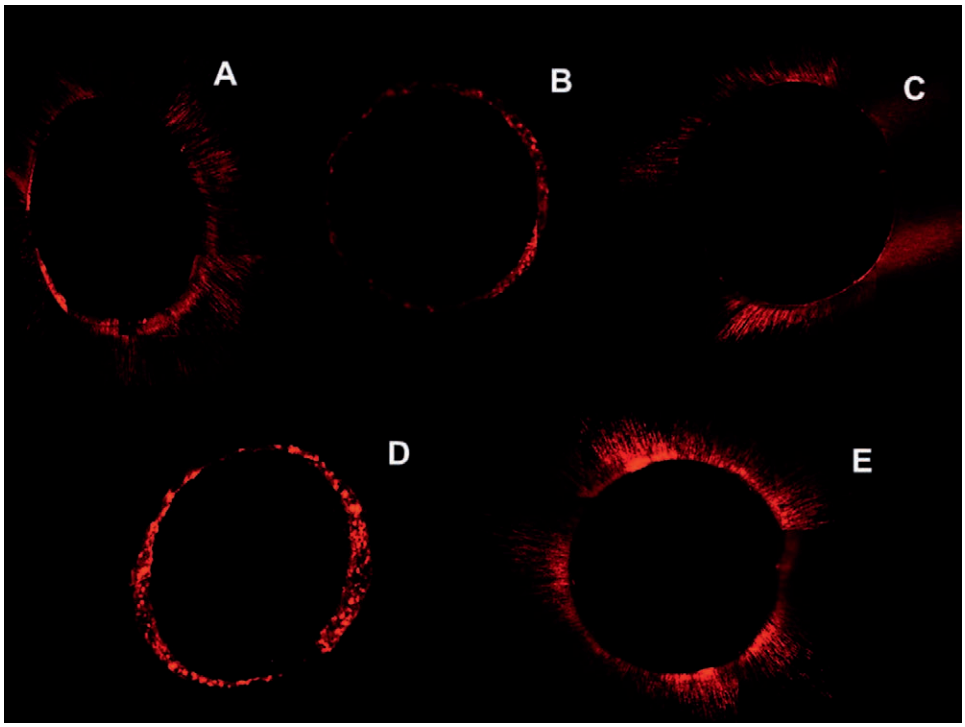


Figure 2. *Qualitative analysis under CLSM (10×) of self-adhesive resin cement penetration into dentin tubules. (A): EDTA; (B): QM; (C): NaOCl; (D): SC; (E): SS. Red: RelyX U200 stained with rhodamine 0.1%. Note that in B and D, the self-adhesive resin cement is confined to the root canal perimeter with absence of tubule penetration.*

from a weak acid that promotes chelation of dentin calcium ions. In an alkaline pH, EDTA selectively removes the hydroxyapatite and noncollagenous protein.[16] On the other hand, the adhesive properties of RelyX U200 depend on a chemical interaction between its acidic monomers and the amount of calcium hydroxyapatite presented in root dentin, providing micromechanical retention.[10,11] Therefore, excessive removal of calcium hydroxyapatite is deleterious for the bond strength of self-adhesive resin cements to root dentin. Intermediary bond strength values were achieved in the EDTA group in this study, contrasting with the findings of Gu and others.[28] These authors stated that EDTA effectively removed the smear layer and increased the bond strength when compared with NaOCl and SS. However, Gu and others[28] activated the EDTA manually for 60 seconds. In the current study, the use of chelating solutions during root canal treatment associated with ultrasonic activation as a dentin pretreatment may lead to dentin erosion and may explain the bond strength reduction.

According to the manufacturer's brochure, QM is a proprietary blend of 2% CHX, EDTA, and a surfactant. SC, in turn, contains EDTA, cetrimide, and a special surfactant. Cetrimide presents low detergent capacity and high antimicrobial activity. The presence of a surfactant aims to enhance the wettability of the irrigant, enabling penetration into root canal anfractuosities. However; Ulusoy and others[29] showed that the presence of surfactants in SC did not improve its efficiency in removing the smear layer. In addition, information related to the influence of SC and QM as irrigants on bond strength of fiber post to root dentin is still poor. An interaction among the three different substances in the formulations of SC and QM and, as mentioned, the presence of EDTA in the formulation, may be responsible for the lower bond strength values obtained in these groups.

In endodontics, CLSM is used to determine the degree of adaptation and penetration of the root canal filling to dentin walls and into dentinal tubules, respectively.[18] Rhodamine dye must be incorporated with cement in a ratio of 0.1%. It did not interfere with the physical-chemical proprieties of endodontic sealers[23,30]; however, it is not clear whether this dye is able to affect the behavior of resin cements used in fiber post cementation. In this study, CLSM was used to illustrate the penetration pattern of self-adhesive resin cement into dentinal tubules. Figure 2 shows that SS, NaOCl, and EDTA presented self-adhesive resin penetration into dentinal tubules. In the QM and SC groups, resin cement remained restricted to root canal space. Considering bond strength and CLSM findings, it can be stated that groups presenting self-adhesive resin penetration into dentinal tubules resulted in higher bond strength values. For instance, SS showed more evident and homogeneous penetration of resin cement into dentinal tubules and also resulted in the greatest bond values. However, as a limitation, just one specimen per group was prepared for CLSM; this finding is suggestive but not conclusive. More studies have to be conducted to establish the relationship between depth-continuity of self-adhesive resin cement penetration and bond strength outcomes.

When new products are investigated, *in vitro* mechanical tests should be conducted to examine experimental variables and test behavioral conditions. Thus, push-out tests are recommended to determine the bond strength of fiber posts to root dentin[31] because they are able to distribute stress more homogeneously; produce less variability in mechanical testing results, fewer pretest failures, and lower standard deviation.[32]

Predominant failures of bonding occur at the cement-dentin interface, which represents a critical interface.[33] In the current study, adhesive failure between cement/dentin (Ac/d) was predominant (53.9%), as expected, followed by adhesive failure between cement/post (Ac/p) (19.5%). Dentin cohesive (DC) failures represent 26.6% of the specimens. Bonding to root canal dentin might be a challenge due to the anatomy of the root, handling characteristics of the adhesive systems, and adhesive procedures.[5] In a root canal, the factor cavity configuration is critical, increasing the stress polymerization of resin cements.[31] The force of polymerization shrinkage into a root canal may be greater than the adhesion of the cement to dentin, resulting in gaps that affect the adhesive interface and may compromise the restoration longevity.[34]

Some limitations of this current study can be depicted as follows: no aging condition (mechanical or thermal cycling) was performed on the tooth with crown restoration, which could depict more realistic clinical behavior. Also, a control group without PUI was not performed. Thus, the isolated action of PUI cannot be stated.

CONCLUSIONS

SS and NaOCl 2.5% associated with PUI promoted the highest bond strength values of RelyX U200.

However, intracanal chelating solutions, such as EDTA, QM, and SC, decreased the bond strength of RelyX U200.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethical Committee of the Federal University of Santa Maria, Brazil. The approval code for this study is 855.457.

Conflict of Interest

The authors 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 4 April 2016)

REFERENCES

1. Sarkis-Onofre R, Jacinto C, Boscato N, Cenci MS, & Pereira-Cenci T (2014) Cast metal vs glass fibre posts: A randomized controlled trial with up to 3 years of follow up *Journal of Dentistry* **42(5)** 582-587.
2. Valandro LF, Baldissara P, Galhano GA, Melo RM, Mallmann A, Scotti R, & Bottino MA (2007) Effect of mechanical cycling on the push-out bond strength of fiber posts adhesively bonded to human root dentin *Operative Dentistry* **32(6)** 579-588.
3. Gonçalves LAA, Vansan LP, Paulino SM, & Sousa Neto MD (2006) Fracture resistance of weakened roots restored with a transilluminating post and adhesive restorative materials *Journal of Prosthetic Dentistry* **96(5)** 339-344.
4. Ferrari M, Vichi A, Fadda GM, Cagidiaco MC, Tay FR, Breschi L, Polimeni A, & Goracci C (2012) A randomized controlled trial of endodontically treated and restored premolars *Journal of Dental Research* **91(7)** 72-78.
5. Ferrari M, Manocci F, Vichi A, Cagidiaco MC, & Mjor IA (2000) Bonding to root canal: Structural characteristics of the substrate *American Journal of Dentistry* **13(5)** 255-260.
6. Malferrari S, Monaco C, & Scotti R (2003) Clinical evaluation of teeth restored with quartz fiber–reinforced epoxy resin posts *International Journal of Prosthodontics* **16(1)** 39-44.
7. Barjau-Escribano A, Sancho-Bru JL, Forner-Navarro L, Rodríguez-Cervantes PJ, Pérez-Gónzález A, & Sánchez-Marín FT (2006) Influence of prefabricated post material on restored teeth: Fracture strength and stress distribution *Operative Dentistry* **31(1)** 47-54.
8. Wandscher VF, Bergoli CD, Limberger IF, Ardenghi TM, & Valandro LF (2014) Preliminary results of the survival and fracture load of roots restored with intracanal posts: Weakened vs nonweakened roots *Operative Dentistry* **39(5)** 541-555.
9. Amaral M, Rippe MP, Bergoli CD, Monaco C, & Valandro LF (2011) Multi-step adhesive cementation versus one-step adhesive cementation: Push-out bond strength between fiber post and root dentin before and after mechanical cycling *General Dentistry* **59(5)** 185-191.
10. Rodrigues RF, Ramos CM, Francisconi PA, & Borges AF (2015) The shear bond strength of self-adhesive resin cements to dentin and enamel: An *in vitro* study *Journal of Prosthetic Dentistry* **113(3)** 220-227.
11. Amaral M, Santini MF, Wandscher V, Amaral R, & Valandro LF (2009) An *in vitro* comparison of different cementation strategies on the pull-out strength of a glass fiber post *Operative Dentistry* **34(4)** 443-451.
12. Renovato SR, Santana FR, Ferreira JM, Souza JB, Soares CJ, & Estrela C (2013) Effect of calcium hydroxide and endodontic irrigants on fibre post bond strength to root canal dentine *International Endodontic Journal* **46(8)** 738-746.
13. Monaco C, Ferrari M, Miceli GP, & Scotti R (2003) Clinical evaluation of fiber-reinforced composite inlay FPDs *International Journal of Prosthodontics* **16(3)** 319-325.
14. Teixeira CS, Felippe MC, & Felippe WT (2005) The effect of application time of EDTA and NaOCl on intracanal smear layer removal: An SEM analysis *International Endodontic Journal* **38(5)** 285-290.
15. Castagna F, Rizzon P, da Rosa RA, Santini MF, Barreto MS, Duarte MA, & Só MV (2013) Effect of passive ultrasonic instrumentation as a final irrigation protocol on debris and smear layer removal—A SEM analysis *Microscopy Research and Technique* **76(5)** 496-502.
16. Carvalho RM, Tay F, Sano H, Yoshiyama M, & Pashley DH (2000) Long-term mechanical properties of EDTA-demineralized dentin matrix *Journal of Adhesive Dentistry* **2(3)** 193-199.
17. Qian W, Shen Y, & Haapasalo M (2011) Quantitative analysis of the effect of irrigant Solution sequences on dentin erosion *Journal of Endodontics* **37(10)** 1437-1341.
18. Ordinola-Zapata R, Bramante CM, Cavenago B, Graeff MS, Gomes de Moraes I, Marciano M, & Duarte MA (2012) Antimicrobial effect of endodontic solutions used as final irrigants on a dentine biofilm model *International Endodontic Journal* **45(2)** 162-168.
19. Aranda-Garcia AJ, Kuga MC, Vitorino KR, Chávez-Andrade GM, Duarte MA, Bonetti-Filho I, Faria G, & Só MV (2013) Effect of the root canal final rinse protocols on the debris and smear layer removal and on the push-out strength of an epoxy-based sealer *Microscopy Research and Technique* **76(5)** 533-537.
20. Morgental RD, Singh A, Sappal H, Kopper PM, Vier-Pelisser FV, & Peters AO (2013) Dentin inhibits the antibacterial effect of new and conventional endodontic irrigants *Journal of Endodontics* **39(3)** 406-410.
21. Rosa RA, Barreto MS, Moraes RA, Broch J, Bier CA, Só MV, Kaizer OB, & Valandro LF (2013) Influence of endodontic sealer composition and time of fiber post cementation on sealer adhesiveness to bovine root dentin *Brazilian Dental Journal* **24(3)** 241-246.
22. Van der Sluis L, Versluis M, Wu MK, & Wesselink PR (2007) Passive ultrasonic irrigation of the root canal: A review of the literature *International Endodontic Journal* **40(6)** 415-426.

23. D'Alpino PH, Pereira JC, Svizero NR, Rueggeberg FA, & Pashley DH (2006) Use of fluorescent compounds in assessing bonded resin-based restorations: A literature review *Journal of Dentistry* **34(9)** 623-634.

24. Bottino MA, Baldissara P, Valandro LF, Galhano GA, & Scotti R (2007) Effects of mechanical cycling on the bonding of zirconia and fiber posts to human root dentin *Journal of Adhesive Dentistry* **9(3)** 327-331.

25. Demiryürek EO, Külünk S, Saraç D, Yüksel G, & Bulucu B (2009) Effect of different surface treatments on the pushout bond strength of fibre post to root canal dentin *Oral Surgery Oral Medicine Oral Pathology Oral Radiology and Endodontics* **108(2)** 74-80.

26. Santos JN, Carrilho MR, De Goes MF, Zaia AA, Gomes BP, Souza-Filho FJ, & Ferraz CC (2006) Effect of chemical irrigants on the bond strength of a self-etching adhesive to pulp chamber dentin *Journal of Endodontics* **32(11)** 1088-1090.

27. Zhang K, Tay FR, Kim YK, Mitchell JK, Kim JR, Carrilho M, Pashley DH, & Ling JQ (2010) The effect of initial irrigation with two different sodium hypochlorite concentrations on the erosion of instrumented radicular dentin *Dental Materials* **26(6)** 514-523.

28. Gu XH, Mao CY, Liang C, Wang HM, & Kern M. (2009) Does endodontic post space irrigation affect smear layer removal and bonding effectiveness? *European Journal of Oral Sciences* **117(5)** 597-603.

29. Ulusoy OI, & Gorgul G (2013) Effects of different irrigation solutions on root dentine microhardness, smear layer removal and erosion *Australian Endodontic Journal* **39(2)** 66-72.

30. Ordinola-Zapata R, Bramante CM, Graeff MS, Del Carpio Perochena A, Vivan RR, Camargo EJ, Garcia RB, Bernardineli N, Gutmann JL, & De Moraes IG (2009) Depth and percentage of penetration of endodontic sealers into dentinal tubules after root canal obturation using a lateral compaction technique: A confocal laser scanning microscopy study *Oral Surgery Oral Medicine Oral Pathology Oral Radiology and Endodontology* **108(3)** 450-457.

31. Soares CJ, Santana FR, Castro CG, Santos-Filho PC, Soares PV, Qian F, & Armstrong SR (2008) Finite element analysis and bond strength of a glass post to intraradicular dentin: Comparison between microtensile and push-out tests *Dental Materials* **24(10)** 1405-1411.

32. Goracci C, Grandini S, Bossù M, Bertelli E, & Ferrari M (2007) Laboratory assessment of the retentive potential of adhesive posts: A review *Journal of Dentistry* **35(11)** 827-835.

33. Zicari F, Couthino E, De Munck J, Poitevin A, Scotti R, Naert I, & Van Meerbeek B (2008) Bonding effectiveness and sealing ability of fiber-post bonding *Dental Materials* **24(7)** 967-977.

34. Goracci C, Tavares AU, Fabianelli A, Monticelli F, Raffaelli O, Cardoso PC, Tay F, & Ferrari M (2004) The adhesion between fiber posts and root canal walls: Comparison between microtensile and push-out bond strength measurements *European Journal of Oral Science* **112(4)** 353-361.

©Operative Dentistry, 2016, **41-6,** e168-e173

Multidisciplinary Treatment of Complicated Crown-Root Fractures: A Case Study

IL Stojanac • BV Bajkin • MT Premovic • BD Ramic • LM Petrovic

Clinical Relevance

The present clinical report describes the successful therapeutic treatment of complicated crown-root fractures and demonstrates the necessity of a multidisciplinary approach during the therapy of traumatized teeth.

SUMMARY

Traumatic dental injuries usually occur among children and adolescents, with maxillary central incisors as the most often affected teeth. Complicated crown-root fractures are particularly challenging for esthetic and functional rehabilitation and often require a multidisciplinary approach. A 21-year-old male patient came to the Dental Clinic due to fractured maxillary incisors caused by trauma during a sporting activity. Clinical examination revealed horizontal fractures of teeth 7, 8, and 9, initiating in the labial cervical third and extending subgingivally on the palate, with exposed pulp tissues. On provisional repositioning and splinting the fragments, root canal treatment was performed. Definitive repositioning was accomplished by raising a full-thickness gingival flap, using fiber-reinforced composite posts, by an endodontist and an oral surgeon. Reattachment was accomplished under surgical conditions to ensure precise positioning of fragments by exposing the palatal aspect of the fracture lines and providing a dry operating field. Definitive composite resin veneers were performed after seven days.

Igor Lj Stojanac, DDS, PhD, assistant professor, Faculty of Medicine, University of Novi Sad, Clinic of Dentistry, Novi Sad, Serbia

Branislav V Bajkin, DDS, PhD, assistant professor, Faculty of Medicine, University of Novi Sad, Clinic of Dentistry, Novi Sad, Serbia

Milica T Premovic, DDS, PhD student, Faculty of Medicine, University of Novi Sad, Clinic of Dentistry, Novi Sad, Serbia

*Bojana D Ramic, DDS, PhD student, Faculty of Medicine, University of Novi Sad, Clinic of Dentistry, Novi Sad, Serbia

Ljubomir M Petrovic, DDS, PhD, full professor, Faculty of Medicine, University of Novi Sad, Clinic of Dentistry, Novi Sad, Serbia

*Corresponding author: Hajduk Veljkova 3, Novi Sad, 21000, Serbia; e-mail: bojana.ramic@mf.uns.ac.rs

DOI: 10.2341/15-080-T

PURPOSE

The etiology of traumatic dental injuries (TDIs) includes oral, environmental, and human factors that are additionally divided into intentional and unintentional.[1] Thirteen to 39% of all dental injuries are sports related, with males traumatized twice as often as females. The exposed position of upper central incisors in the dental arch makes them the most frequently traumatized teeth in both primary and permanent dentitions.[2,3] Basketball players are very susceptible to TDIs because of the frequent hand or elbow contact with other players.[4] A study

by Kumamoto[5] showed that the highest incidence of orofacial injuries created during sports activities was observed in basketball, baseball and unorganized football when athletes didn't wear mouthguards. Wearing a custom-made mouthguard can help prevent orofacial sport-related injuries and provide significant protection if an injury occurs.

TDIs have a great influence on a patient's quality of life since they cause esthetic, functional, and phonetic problems. Psychosocial aspects of the patient are particularly noticed if the fractured teeth are visible. Therefore, TDI requires rapid reaction of the patient and the therapist in order to achieve the best treatment option in the least traumatic way. Complete rehabilitation of traumatized teeth often requires a multidisciplinary approach, which can include oral and maxillofacial surgeons, endodontists, pediatric dentists, orthodontists, prosthodontists, and periodontists.

Crown-root fractures involve enamel, dentin, and cementum; occur below the gingival margin; and are classified as complicated and uncomplicated according to the pulp tissue involvement.[1] The unstable position of the fractured coronal fragment, loss of tooth vitality, invasion of biological width, and problems fitting fragments together make definitive restorations of crown-root fractures difficult and challenging.[6,7]

The management of dental trauma has to be planned carefully, including a detailed history of the patient and careful clinical and radiological examination. The correct diagnosis and most suitable treatment options for the patient include factors such as the type, direction, and intensity of the trauma; involvement of the pulp tissue or biological width; stage of root growth; mobility of the tooth; diastasis of the fragments; and the knowledge and skills of the dentist.[8,9]

The following clinical report describes the management of complicated crown-root fractures, including endodontic treatment, surgically raising a full-thickness gingival flap, adhesive reattachment of fragments using fiber-reinforced composite posts, and esthetic veneering of fractured teeth.

DESCRIPTION OF TECHNIQUE

A 21-year-old male patient was referred to the Dental Clinic of Vojvodina due to intense pain of fractured teeth caused by trauma during a basketball game the day before.

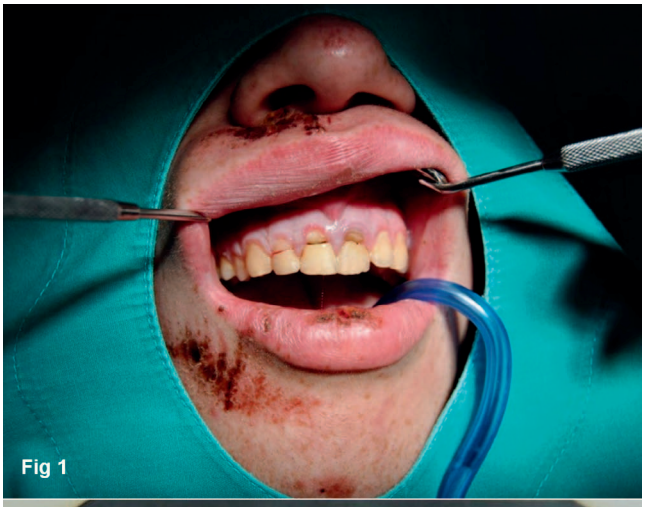
A maxillofacial surgeon examined the patient, performed debridement of the wounds, determined that there were no fractures to the facial bones, prescribed antibiotic therapy, and referred the patient to the Department of Restorative Dentistry and Endodontics.

A clinical examination revealed horizontal fractures of the maxillary right lateral incisor and both maxillary central incisors, labially in the cervical third and extending subgingivally on the palate, with exposed pulp tissue (Figure 1). The crown fragments were highly mobile but still in place, fixed slightly on the palatal side by periodontal ligament fibers, while the root fragments were stationary and stable. Adjacent teeth had no sign of trauma. Radiographs confirmed the presence of horizontal fractures for teeth 7, 8, and 9 (Figure 2).

On presenting all treatment options, to include both risks and benefits, the patient opted for reattachment of the fragments using glass fiber posts under surgical conditions. At first, the fragments were temporarily repositioned and splinted using a composite-resin splint, from teeth 6 to 11, to stabilize the position of the teeth and facilitate root canal treatment (Figure 3). On cleaning and shaping (ProTaper, Dentsply, Maillefer, Switzerland), the canals were treated with calcium hydroxide for seven days. Definitive obturation was performed using gutta-percha points and AH plus sealer (Dentsply DeTrey, Konstanz, Germany) (Figure 4). The patient was instructed on oral hygiene maintenance and nutrition.

Considering that the fracture lines on the palatal aspect were deeply subgingival, definitive repositioning of fragments was performed two days later after raising a full-thickness gingival flap, a procedure that was performed by an endodontist and an oral surgeon. The envelope flap was elevated from the canine to the canine both buccally and palatally. The subgingival palatal fracture lines were exposed in this manner to control bleeding and ensure precise fragment repositioning. The crown fragments were gently separated from the periodontal tissue and cleaned. The previously prepared access cavities were extended to ensure a straight-line passage of posts. The fragments were fitted once more to ensure adaptation, followed by immersion of the fragments in a saline solution in order to keep them moist and prevent dehydration (Figure 5).

The post space was prepared using a Largo Peeso Reamer and Precision Drill for the X-Post No. 3, reduced to an adequate length, and cleaned with alcohol (Dentsply Post&Core System, Dentsply DeTrey). The canal spaces and surrounding tooth issue, as well as the crown fragments, were etched, rinsed, and dried in order to be prepared for the reattachment



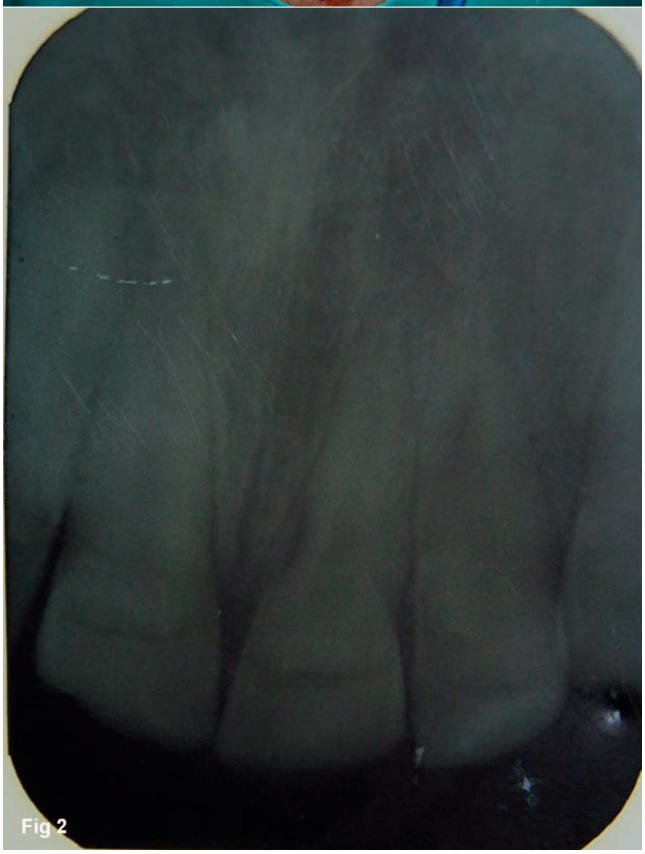
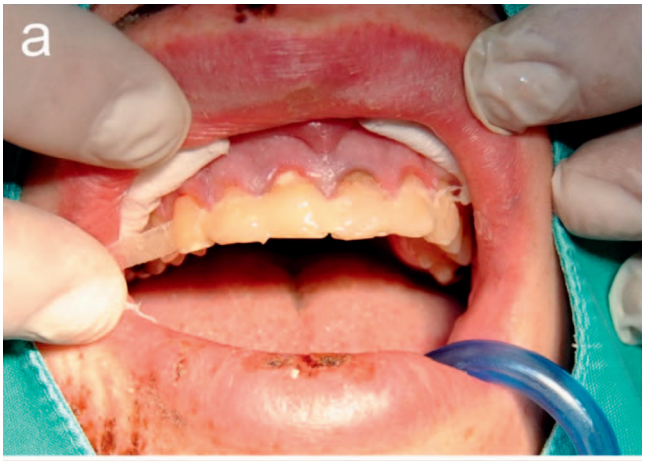
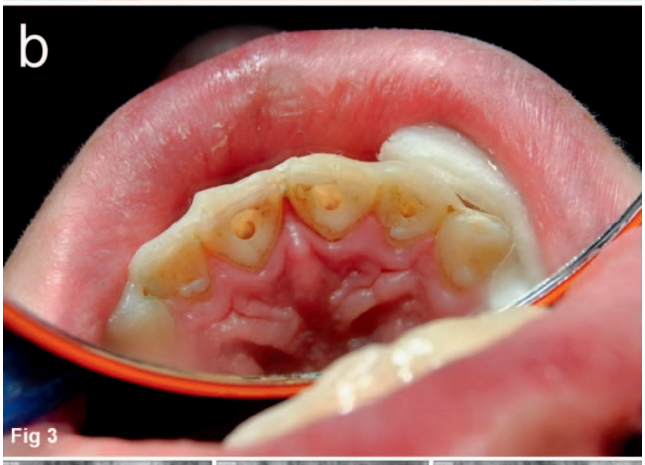


Figure 1. *Intraoral view of the patient.*
Figure 2. *Preoperative retroalveolar radiograph of fractured teeth.*





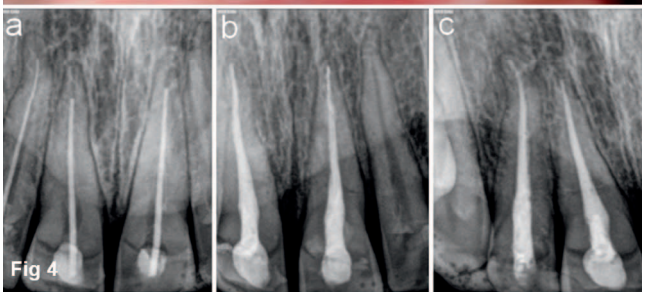
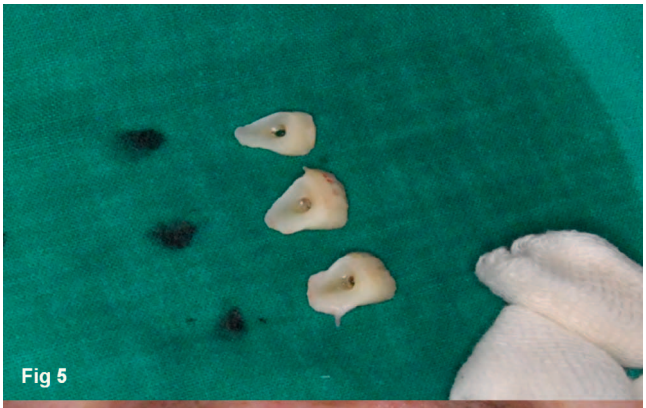
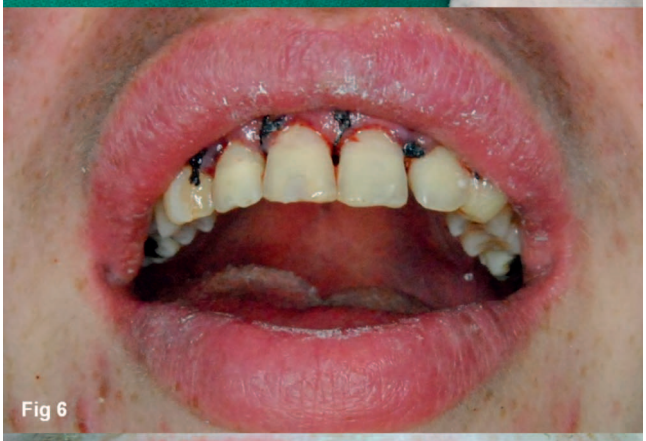


Figure 3. *(a): Temporarily repositioned and splinted teeth. (b): Prepared access cavities for root canal treatments.*
Figure 4. *(a): Retroalveolar radiograph on working length determination. (b, c): Definitive root canal tretaments.*

procedures. An XP Bond/SCA mixture was applied into the canal, the X-post, and the surfaces of the fractured fragments and gently dried, followed by application of Core-X flow into the canal. The post was set and stabilized, the crown fragment was repositioned, and the entire unit was light cured for 40 seconds from each side (Radii Plus, SDI, Bayswater, Australia). The same procedure was repeated for the adjacent teeth. Sterile gauze was placed beneath the flap with additional suction used to achieve a dry operating field. On completion of the reattachment procedure, the flap was repositioned, fixed, and sutured using black-silk suture 3-0 (Figure 6).

Preparation for composite resin veneers was kept strictly in enamel except at the fracture line area, where the preparation was deeper. The teeth were etched with 37% phosphoric acid for 20 seconds (Super Etch, SDI) and thoroughly rinsed and dried, and the dentin bonding agent (Stae, SDI) was





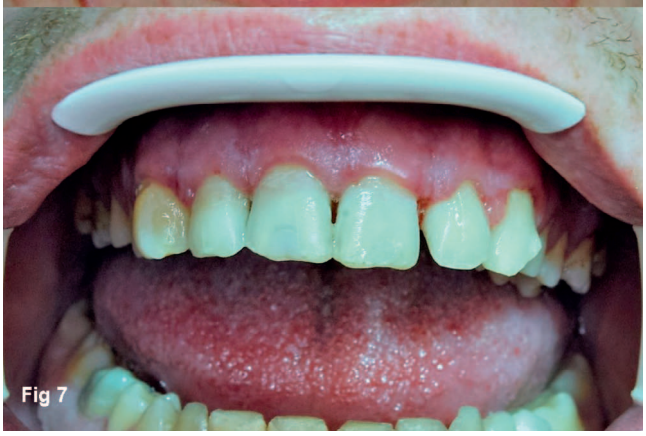


Figure 5. *Separated and cleaned coronal fractured fragments.*
Figure 6. *Repositioned and sutured lifted gingival flap and temporarily made composite resin veneers.*
Figure 7. *Clinical view on complete treatment.*

applied and light cured for 10 seconds. Nanohybrid composite resin (Ice, SDI), shade A2, was applied, contoured for a natural look, and polymerized for 20 seconds for each layer. Removal of the excess of material in the area of the gingival sulcus was performed using a No. 3 10-blade SafeEnd series carbide finishing bur (SS White Burs, Inc, Lakewood, NJ, USA) with a noncutting tip, designed to trim without damaging the gingival tissue. The remaining area was finished and polished with a No. 7 10-blade and a No.7 20-blade SafeEnd series carbide bur, respectively (SS White Burs). After seven days, the sutures were removed, composite resin veneers were checked, and additional polishing was performed (Figure 7).

On completion of the restorative procedures, the patient was instructed on the importance of maintaining adequate oral hygiene, implementation of precautions, and the necessity of using a mouthguard during sports activity. The patient had no complaints at follow-up examinations.

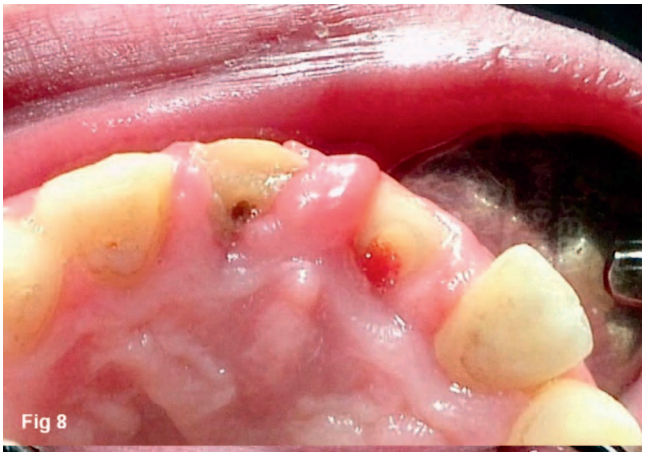
Seven months later, the patient was referred to the Dental Clinic due to fractured previously restored teeth 8 and 9, which again occurred during a basketball game. Tooth 7 had no sign of trauma (Figures 8 and 9). Considering that the patient did not heed instructions about necessity of wearing a mouthguard, two previously reconstructed teeth were indicated for extraction.

POTENTIAL PROBLEMS

The esthetic, functional, and biological rehabilitation of traumatic injured teeth is a real challenge for every dentist, often requiring an interdisciplinary/multidisciplinary approach. Various treatment modalities have been proposed for crown-root fractures: coronal fragment removal followed by prosthetic restoration, fragment reattachment, gingivectomy and osteotomy (crown lengthening), orthodontic extrusion of a root fragment, forced surgical extrusion, and extraction followed by implants or fixed partial denture.[10,11] Techniques that shorten and simplify treatment, that provide natural-looking restorations, and that ensure long-term success rates are preferred.[12]

The reattachment procedure is not convenient if the fracture line is positioned subgingivally, a dry operating field is difficult to achieve, and the whole treatment can be finished unsuccessfully. Therefore, the reattachment procedures in the present case were performed after raising a full-thickness gingival flap, under surgical conditions, in order to expose the fracture line, control bleeding, and ensure the precise repositioning of fragments.

Periodontal structures should be preserved as much as possible; therefore, repositioning must be done gently and atraumatically in order to provide desirable wound healing and acceptable biological restorations. Complications such as gingival inflammation, clinical attachment loss, and bone loss are frequent, especially in patients who do not comply with instructions given by the therapist.[1,13]



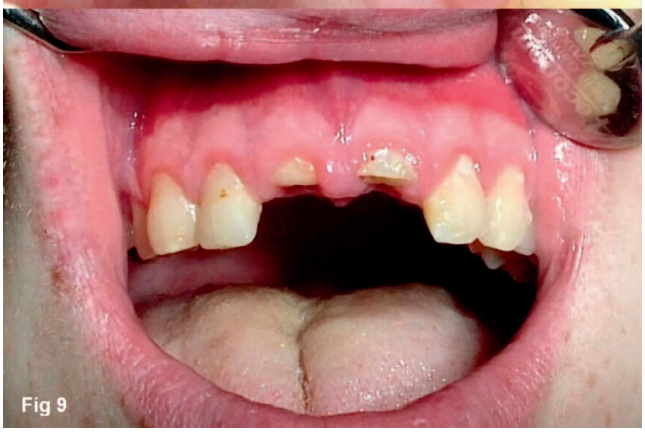


Figure 8. *Intraoral view of the fractured previously reattached dental structures.*
Figure 9. *Extraoral view of the fractured previously reattached dental structures.*

Treatments of choice for converting subgingival fractures into supragingival fractures include some of the following treatment options. Gingivectomy and osteotomy (crown lengthening) are simple and rapid procedures, but they are not indicated in esthetic areas.[14-16] Orthodontic extrusion is favorable for maintaining periodontal health and preserves the crown-root ratio, but the procedure is time consuming, requiring five weeks to achieve 2-3 mm of extrusion and a retention period of about 8-10 weeks.[11,14,17] Surgical extrusion of the tooth is a less time consuming procedure, but there is a risk of root resorption and compromising the integrity of the periodontal ligament.[14]

During the reattachment of separated fragments, they should be kept out of the mouth for as little time as possible so that they can remain moist and prevent dehydration; in this way, they maintain an adequate bond strength and their original esthetic appearance.[18] In the present case, the fractured fragments were preserved in a saline solution until reattachment (approximately 10 minutes), which was necessary to prepare and set the posts.

SUMMARY OF ADVANTAGES AND DISADVANTAGES

Advances in the development of adhesive materials and restorative techniques have made the selection of reattachment procedure the most preferred treatment option for tooth fracture whenever the coronal tooth fragment is intact and available and precise repositioning between fragments is possible. Although technically sensitive, the reattachment procedure has significant advantages: it is a conservative approach and a rapid procedure, it immediately restores the natural appearance and function of the tooth, and it results in positive emotional and social responses from the patient.[7,9]

Tooth-colored glass fiber posts were used to fix the tooth fragments, enhance retention, and strengthen the remaining tooth structure. Glass fiber posts were chosen to reinforce the fractured teeth due to their excellent esthetics, ability to bond to tooth structure, stiffness and elasticity similar to dentin, high durability, and fracture resistance.[19,20]

Composite resin veneers were used to strengthen the area of the fracture line and make the teeth esthetically pleasing. A thin layer of composite resin was applied, respecting the original tooth form and color and providing natural-looking restorations. Minimally invasive direct composite restorations are more preferable than more extensive indirect restorations.

The present clinical report describes the successful therapeutic treatment of complicated crown-root fractures and demonstrates the necessity of using a multidisciplinary approach during the therapy of traumatized teeth. Additionally, it emphasizes the importance of wearing a mouthguard during sports activity in order to prevent TDIs as well as preserve previously reattached dental structures.

Acknowledgement

This research was supported by a grant from the Ministry of Science of Serbia, Grant No. 174005, for research on the viscoelasticity of fractional type and optimization of shape in rod theory.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Faculty of Medicine, University of Novi Sad, Clinic of Dentistry, Serbia.

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 April 2016)

REFERENCES

1. Glendor U, Marcenes W, & Andreasen JO (2007) Classification, epidemiology and etiology In: Andreasen JO, Andreasen FM, Andersson L (eds) *Textbook and Color Atlas of Traumatic Injuries to the Teeth* Wiley-Blackwell, Oxford 217-254.
2. Olin WH (1996) Dentistry and sport: Meeting the needs of our patients *Journal of the American Dental Association* **127(6)** 809-818.
3. Altun C, Cehreli ZC, Güven G, & Acikel C (2009) Traumatic intrusion of primary teeth and its effects on the permanent successors: A clinical follow-up study *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology and Endodontics* **107(4)** 493-498.
4. Cohenca N, Roges RA, & Roges R (2007) The incidence and severity of dental trauma in intercollegiate athletes *Journal of the American Dental Association* **138(8)**1121-1126.
5. Kumamoto DP (1996) Sports dentistry at the state level. *Journal of the American Dental Association* **127(6)**:816.
6. Cvek M, Tsilingaridis G, & Andreasen JO (2008) Survival of 534 incisors after intra-alveolar root fracture in patients aged 7-17 years *Dental Traumatology* **24(4)** 379-387.
7. Stojanac I, Ramic B, Premovic M, Drobac M, & Petrovic L (2013) Crown reattachment with complicated chisel-type fracture using fiber-reinforced post *Dental Traumatology* **29(6)** 479-482.
8. Macedo GV, Diaz PI, De O Fernandes CA, & Ritter AV (2008) Reattachment of anterior teeth fragments: A conservative approach *Journal of Esthetic and Restorative Dentistry* **20(1)** 5-20.
9. Sargod SS, & Bhat SS (2010) A 9 year follow-up of a fractured tooth fragment reattachment *Contemporary Clinical Dentistry* **1(4)** 243-245.
10. Trushkowsky RD (1998) Esthetic, biologic, and restorative considerations in coronal segment reattachment for a fractured tooth: A clinical report *Journal of Prosthetic Dentistry* **79(2)** 115-119.
11. Olsburgh S, Jacoby T, & Krejci I (2002) Crown fractures in the permanent dentition: Pulpal and restorative considerations *Dental Traumatology* **18(3)** 103-115.
12. Murchison DF, Burke FJ, & Worthington RB (1999) Incisal edge reattachment: Indications for use and clinical technique *British Dental Journal* **186(12)** 614-619.
13. Pama-Benfenati S, Fugazzotto PA, Ferreira PM, Ruben MP, & Kramer GM (1986) The effect of restorative margins on the postsurgical development and nature of the periodontium. Part II. Anatomical considerations. *International Journal of Periodontics and Restorative Dentistry* **6(1)** 64-75.
14. Kulkarni VK, Sharma DS, Banda NR, Solanki M, Khandelwal V, & Airen P (2013) Clinical management of a complicated crown-root fracture using autogenous tooth fragment: A biological restorative approach *Contemporary Clinical Dentistry* **4(1)** 84-87.
15. Turgut MD, Gönül N, & Altay N (2004) Multiple complicated crown-root fracture of a permanent incisor *Dental Traumatology* **20(5)** 288-292.
16. Wang Z, Heffernan M, & Vann WF Jr (2008) Management of a complicated crown-root fracture in a young permanent incisor using intentional replantation *Dental Traumatology* **24(1)** 100-103.
17. Fidel SR, Fidel-Junior RA, Sassone LM, Murad CF, & Fidel RA (2011) Clinical management of a complicated crown-root fracture: A case report *Brazilian Dental Journal* **22(3)** 258-262.
18. Capp CI, Roda MI, Tamaki R, Castanho GM, Camargo MA, & de Cara AA (2009) Reattachment of rehydrated dental fragment using two techniques *Dental Traumatology* **25(1)** 95-99.
19. Krishna A, Malur MH, Swapna DV, Benjamin S, & Deepak CA (2012) Traumatic dental injury—An enigma for adolescents: A series of case reports *Case Reports in Dentistry* ID 756526 doi:10.1155/2012/756526.
20. Qualtrough AJ, & Mannocci F (2003) Tooth-colored post systems: A review *Operative Dentistry* **28(1)** 86-91.

©Operative Dentistry, 2016, **41-6,** e174-e182

Relined Fiberglass Post: Effect of Luting Length, Resin Cement, and Cyclic Loading on the Bond to Weakened Root Dentin

NC de Souza • ML Marcondes • DFF da Silva • GA Borges • LH Burnett Júnior • AM Spohr

Clinical Relevance

Under mechanical stresses, the luting length is important for the retention of relined fiberglass posts luted to weakened roots with conventional or self-adhesive resin cements.

SUMMARY

This study evaluated the effects of luting length of the post, the resin cement, and cyclic loading on pull-out bond strength of fiberglass posts relined with composite resin in weakened roots. The canals of 80 bovine incisors were endodontically treated and weakened with diamond burs. The teeth were randomly divided into eight groups (n=10) according to the luting procedures of the relined fiberglass post (RFP): In groups 1, 2, 3, and 4, the RFPs were luted with RelyX ARC, and in groups 5, 6, 7, and 8 they were luted with RelyX U200. In groups 1, 3, 5, and 7, the RFPs were luted at a length of 5 mm, and in groups 2, 4, 6, and 8 they were luted at a length of 10 mm. Specimens from groups 3, 4, 7, and 8 were submitted to cyclic loading. Specimens were subjected to a pull-out bond strength test in a universal testing machine. The results (MPa) were analyzed by three-way analysis of variance and the Tukey post hoc test (α=0.05). Six human upper anterior teeth were used to analyze the bond interface by confocal laser scanning microscopy (CLSM). The pull-out bond strength of RFPs luted with RelyX U200 was statistically higher than that of RelyX ARC. Cyclic loading influenced the bond strength only for the luting length of 5 mm. CLSM analysis revealed the formation of resin cement tags for both materials. Luting length is an important factor in retaining RFPs in weakened roots when they are subjected to

Niélli C. de Souza, MS, Restorative Dentistry, Pontifical Catholic University of Rio Grande do Sul, Rio Grande do Sul, Brazil

Maurem L. Marcondes, PhD, Restorative Dentistry, Pontifical Catholic University of Rio Grande do Sul Rio Grande do Sul, Brazil

Diego F.F. da Silva, MS, Restorative Dentristry, Pontifical Catholic University of Rio Grande do Sul, Rio Grande do Sul, Brazil

Gilberto Antonio Borges (Gilberto Antonio Borges), PhD, Dental Materials, University of Uberaba, Minas Gerais, Brazil

Luiz Henrique Burnett Júnior, PhD, Restorative Dentistry, Pontifical Catholic University of Rio Grande do Sul, Rio Grande do Sul, Brazil

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

*Corresponding author: Porto Alegre, Rio Grande do Sul 90619-900, Brazil; email: anaspohr@terra.com.br

DOI: 10.2341/15-233-L

cyclic loading, and RelyX U200 resulted in greater bond strengths to the root canal in comparison with RelyX ARC.

INTRODUCTION

Fiberglass posts have a modulus of elasticity close to that of the remaining tooth structure and distribute the stress more evenly over the tooth in comparison with conventional cast post-core systems, thus reducing the risk of root fractures.[1,2] Prefabricated fiberglass posts do not always adapt perfectly to the root canals, and with compromised frictional retention, the cement is the only thing responsible for retention. Debonding of the post is the main reason for the failure of teeth that are restored with fiberglass posts.[3,4] Thus, one of the techniques proposed is to use fiberglass posts relined with composite resin, especially for the treatment of large root canals.[5]

Among the factors that determine the degree of post retention, the selection of the luting agent has been widely studied, and resin cements have shown positive results with respect to their mechanical properties and adhesive capacities.[6,7] Among the many resins that are available, the self-adhesive resin cements RelyX U100 and RelyX U200 have been shown to have greater bond strength values to root dentin compared with other materials.[8,9] However, many dentists still use resin cements requiring an adhesive system.

Another factor that may influence the retention of fiberglass posts is the length at which they are luted. Adhesion to the root canal walls is more difficult in the apical third as a result of the difficulty involved in controlling moisture and the ability to effectively cure the adhesive/resin cement.[10,11] As a solution to this problem, luting posts at shorter lengths into the canal has been proposed to eliminate the problem of polymerization at the apical region.[12] A recent study[13] has shown promising data for fiberglass posts luted with resin cements up to 5 mm deep into the canal. However, little is known about the performance of relined fiberglass posts luted with different resin cements in different lengths of luting after mechanical cycling, justifying additional studies to evaluate these issues.

Therefore, the aim of the study was to evaluate the pull-out bond strength between relined fiberglass posts and root dentin using two different luting lengths and two different resin cements with and without cyclic loading. The dentin-resin interface was also assessed by confocal laser scanning microscopy (CLSM). This study was conducted using the following null hypotheses: 1) the luting length, 2) the resin cement, and 3) and the cyclic loading do not influence the pull-out bond strength between the relined fiberglass post and weakened root dentin.

METHODS AND MATERIALS

Eighty permanent bovine incisors, extracted at the age of two years, with similar root sizes and lengths were selected. The teeth were cleaned of gross debris and stored in distilled water at 4°C. The water was changed every week, and the teeth were used within three months. The crowns of the bovine incisors were removed below the cemento-enamel junction with a low-speed diamond disc, and the roots were trimmed to a length of 16 mm. A step-back preparation technique was used for the endodontic treatment. The teeth were instrumented at a working length of 1 mm from the apex to a #55 master apical file. A step-back technique was performed with stainless-steel K-files #60 to #80 and Gates Gliden drills #4 to #5. All enlargement procedures were followed by irrigation with a 2.5% sodium hypochlorite solution. The prepared root canals were filled with gutta-percha cones using the lateral condensation technique and Sealer-26 resin sealer (Dentsply, Petrópolis, RJ, Brazil). After the endodontic treatment, the roots were stored at 100% relative humidity at 37°C for 48 hours, and the teeth were randomly divided in eight groups (n=10) (Figure 1).

The gutta-percha was removed with a heated Rhein instrument until it reached the set length of 5 mm (groups 1, 3, 5, and 7) or 10 mm (groups 2, 4, 6, and 8). To obtain standardized weakened canals, they were enlarged using a Largo drill #5 and high-speed conical diamond burs #4138 (larger and smaller diameter of 1.8 mm and 1.2 mm, respectively) and #4137 (larger and smaller diameter of 2.5 mm and 1.8 mm, respectively) (KG Sorensen, São Paulo, SP, Brazil) with water irrigation to a predetermined length (5 or 10 mm) that was controlled with silicone stops. The roots were embedded in a metallic cylinder with self-cured acrylic resin.

The fiberglass posts n.3 (Reforpost; Angelus, Londrina, Brazil) were etched with 37% phosphoric acid for 15 seconds, followed by silane application (Angelus, Londrina, PR, Brazil). A layer of the bond adhesive Scotchbond Multi-Purpose (3M ESPE, St Paul, MN, USA) was applied and light-cured for 20 seconds. The posts were then covered with composite resin Z350 (3M ESPE, St Paul, MN, USA) and inserted into the root canals that were previously lubricated with hydrosoluble gel (K-Y gel, Johnson &

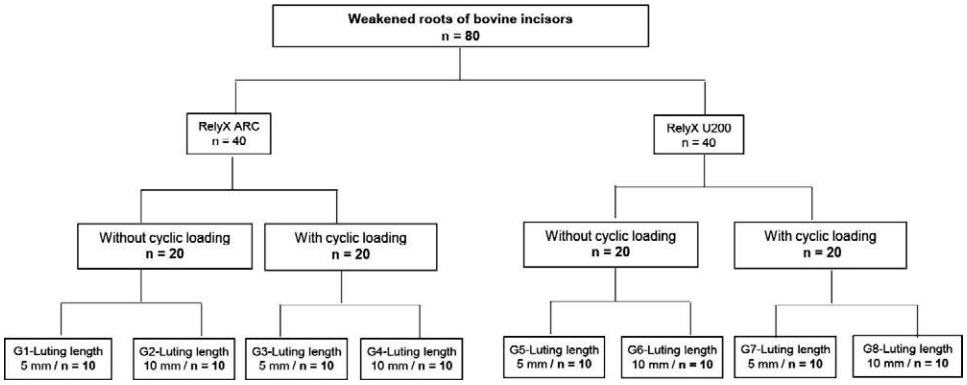


Figure 1. *Schematic diagram of the experimental groups and the sample size according to the resin cement, cyclic fatigue loading, and luting length.*

Johnson, São José dos Campos, SP, Brazil), light-cured for 20 seconds with the curing unit Radii Cal (SDI, Australia) with light intensity of 1000 mW/cm^2, removed from the canal, and light-cured again for 20 seconds. The canals were rinsed for 30 seconds to remove the hydrosoluble gel, followed by drying with absorbing paper points. The resin cements used in the present study and the luting procedures are described in Table 1. In groups 1, 2, 3, and 4, the relined fiberglass posts were luted with RelyX ARC, and in groups 5, 6, 7, and 8 they were luted with RelyX U200. In each relined post, a small notch was made in the composite resin of the coronal portion with a diamond bur to adapt the plunger of the mechanical cycling machine to the specimen.

The same luting procedures were performed for the two luting lengths, and the samples were stored at 100% relative humidity at 37°C for 24 hours. Specimens from groups 3, 4, 7, and 8 were first submitted to cyclic loading of 50 N at 45° using 1,000,000 cycles at 1 Hz in distilled water,[14] followed by the pull-out test. Specimens from groups 1, 2, 5, and 6 were subjected only to the pull-out test.

The pull-out test was performed at a cross-head speed of 0.5 mm/min using a universal testing machine (EMIC, São José dos Campos, PR, Brazil) with a 500-N cell load. The force required to dislodge each relined post was recorded in Kgf and converted to MPa using the following calculation:

$$A = \pi(R + r)\sqrt{h^2 + (R + r)^2}$$

where A denotes area, R indicates the larger radius of diamond bur 4137, r indicates the smaller radius of diamond bur #4137, and h indicates height.

Statistical analysis was performed by applying a three-way analysis of variance followed by the Tukey post hoc test at a 95% confidence level.

For the CLSM analysis, six human maxillary anterior single-rooted teeth were obtained after approval was obtained from the ethics committee (30904114.4.0000.5336). After disinfection with 0.5% chloramine for 48 hours, the crowns were removed with a diamond disc, and the length of the roots was standardized to 15 mm long. The endodontic treatment, the weakening of the roots at the 10-mm length, and the method for relining the fiberglass post were the same as those described for bovine teeth. Luting with RelyX ARC and RelyX U200 was performed in three teeth for each of the resin cements, as described in Table 1. Fluorescein isothiocyanate–dextran (Sigma Aldrich, St Louis, MO, USA) was incorporated into each bottle of Adper Scotchbond Multi-Purpose Plus adhesive system (activator, primer, and catalyst) (40 mg/mL). The dye was mixed directly into the supplied bottle using a mixing device (Vortex Machine, Scientific Industries, New York, NY, USA) for two hours to completely dissolve the dye. Rhodamine B (Sigma) was added to the base resin cement paste and mixed to obtain a paste of uniform shade (0.32 mg/mg).[15] After luting and storage in water at 37°C for 24 hours, 1-mm-thick slices were cut from the apical, middle, and cervical thirds of the root using a diamond disc mounted in a low-speed laboratory cutting machine (Labcut 1010, Extec Corp, London, UK) under cooling. The sections were stored in dark containers and then observed under the CLSM. The CLSM images (LSM 5, Zeiss, Jena, Germany) were obtained in dual fluorescence mode using 20×, 40×, and 63× objectives. An argon laser at 488 nm and He-Ne laser at 543 nm provided excitation energies. The sizes of the recorded images were 187×187 μm^2 with a resolution of 1024×1024 pixels.

RESULTS

Analysis of variance showed that the resin cement (p=0.0001) and cyclic loading (p=0.0001) had a

Table 1: *Chemical Composition and Bonding Procedures for the Luting Resin Cements Investigated in the Present Study*

Resin Cement/ Manufacturer	Composition	Adhesive Strategy Curing	Dentin Pretreatment	Luting Agent Application
RelyX ARC/3M ESPE, St Paul, MN, USA	Paste A: Bis-GMA, TEGDMA, zirconia silica, pigments, amines and photoinitiator system; Paste B: Bis-GMA, TEGDMA, zirconia silica, benzoyl peroxide	Conventional dual-cure resin cement	The canal walls were etched with 35% phosphoric acid for 15 s, rinsed for 15 s, and gently air-dried. Excess water was removed from the canal with absorbent paper points. The Scotchbond Multipurpose Plus Activator was applied into the root canal with a microbrush of compatible size and air-dried for 5 s. Afterward, the Scotchbond Multipurpose Plus Primer, followed by Catalyst, were applied and air-dried.	The dual-cured resin cement RelyX ARC was mixed and placed over the relined post, which was inserted into the root canal with light pressure. The excess luting material was removed and light-cured for 40 s on the occlusal surface with a Radii curing light.
RelyX U200/3M ESPE, St Paul, MN, USA	Base paste: glass powder treated with silane, 2-propenoic acid, 2-methyl 1,1′-(1-[hydroxymetil]-1,2-ethanodlyl) ester dimethacrylate, TEGDMA, silica-treated silane, glass fiber, sodium persulfate and per-3,5,5-trimethyl hexanoate *t*-butyl; Catalyst paste: glass powder treated with silane, substitute dimethacrylate, silica-treated silane, sodium *p*-toluenesulfonate, 1-benzyl-5-phenyl-acid barium, calcium, 1,12-dodecane dimethacrylate, calcium hydroxide, and titanium dioxide	Self-adhesive resin cement	The root canal was rinsed with water; Excess water was removed from the canal with absorbent paper points.	The mixing tip with endo tip was attached on RelyX U200 Automix syringe. Application of RelyX U200 Automix cement directly into the root canal. The relined posts were inserted, excess cement was removed, and the remaining cement cured for 40 s on the occlusal surface with a Radii curing light.

Abbreviations: Bis-GMA, bisphenol A glycidyl ether dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

significant effect on the pull-out bond strength, while the luting length was not significant ($p=0.328$). The interaction between cyclic loading and luting length was significant ($p=0.0001$). However, the interaction between resin cement and cyclic loading ($p=0.123$), the interaction between the resin cement and luting length ($p=0.301$), and the interaction among all three factors ($p=0.225$) were not significant. Comparisons using the Tukey test are shown in Tables 2 and 3. The mean pull-out bond strength for RelyX U200 (6.47 MPa) was significantly greater than that of RelyX ARC (5.51 MPa) ($p<0.05$). For a luting length of 5 mm, the pull-out bond strength of the specimens without cyclic loading (6.98 MPa) was statistically superior to that of the specimens with cyclic loading (4.78 MPa) ($p<0.05$). For a length of 10 mm, there was no significant difference between the specimens without (6.17 MPa) and with (6.03 MPa) cyclic loading.

In the CLSM images, the formation of adhesive tags and resin cement tags for RelyX ARC (Figure 2) was observed, and resin cement tags for RelyX U200 (Figure 3) were visible. The resin cement tags in both were visible in the cervical and middle thirds of the

Table 2: *Pull-out Bond Strength Means (MPa) and Standard Deviations Between the Resin Cements*[a]

Resin Cement	n	Mean	Standard Deviation
RelyX U200	40	6.47 A	1.34
RelyX ARC	40	5.51 B	1.18

[a] *Different letters indicate statistically different means according to Tukey test ($p<0.05$).*

Table 3: *Pull-out Bond Strength Means (MPa) and Standard Deviations Between the Different Luting Lengths Without and With Cyclic Fatigue Loading*[a]

Luting Length mm	Cyclic Fatigue Loading	n	Mean	Standard Deviation
5	Without	20	6.98 A	1.18
	With	20	4.78 B	1.32
10	Without	20	6.17 A	0.88
	With	20	6.03 A	0.99

[a] *Different letters indicate statistically different means according to Tukey test ($p<0.05$).*

root canals. Hybrid layer formation was observed in the cervical (Figure 2b), middle, and apical thirds for RelyX ARC.

DISCUSSION

The first null hypothesis was rejected because the pull-out bond strength for RelyX U200 was significantly greater than that for RelyX ARC. Other studies[16,17] have also shown better results for self-adhesive resin cements compared to conventional resin cements.

The RelyX U200 is a self-adhesive resin cement, which eliminates the need for an adhesive system. The bond mechanism of this resin cement to dentin appears to be more chemical than micromechanical in nature. This bond is established by the specific multifunctional phosphoric-acid methacrylates, which are ionized at the time of mixing and react with the hydroxyapatite in the mineralized tissues of the tooth.[18] At the beginning of the reaction, the resin cement has a very low pH.[19] During the polymerization reaction, the acidic monomers interact with the fillers in the resin cement and with the hydroxyapatite in the tooth, neutralizing the reaction and raising the pH. The water that is formed in this neutralization reaction contributes to the cement's initial hydrophilicity, which provides improved adaptation to the tooth structure and moisture tolerance.[20] Subsequently, water is reused by the acidic functional groups during the cement reaction with ion-releasing basic filler particles, resulting in a hydrophobic matrix.[20] Despite the low pH, the interaction with root dentin is superficial, and no evident hybrid layer is observed.[21]

The resin cement RelyX ARC needs an adhesive system, which is responsible for the bond to the root dentin. In the present study, the Scotchbond Multipurpose Plus adhesive system was used in the sequence (etching with phosphoric acid, activator, primer, and catalyst) to make the adhesive a dual material, allowing for self-cure polymerization in regions not irradiated by the LED unit.[22] The bond mechanism of this adhesive system to dentin functions through the formation of a hybrid layer.[23] Because there are several operative steps for using this system, there is greater technical sensitivity compared to that associated with self-adhesive resin cements. However, the bond strength difference between the RelyX U200 and RelyX ARC was 0.96 MPa in the present study, and it is questionable whether this difference is clinically significant.

The relined fiberglass post decreases the thickness of the resin cement in the root canal and exerts pressure on the adaptation of resin cement against the dentinal walls.[24,25] As a result of the thixotropic behavior of RelyX U200, the application of pressure decreases its viscosity and improves its adaptation to the cavity walls.[26] Consequently, the formation of resin cement tags in the cervical and middle thirds of the root canal can be observed in CLSM images. For RelyX ARC, there was penetration of both the adhesive and the resin cement into the dentinal tubules, forming adhesive tags and resin cement tags. It can be speculated that the luting pressure generated by the relined post pushes the resin cement and the adhesive against the walls of the canal, favoring penetration into the dentinal tubules. In addition, CLSM images demonstrated hybrid layer formation of the green-labeled adhesive in the cervical, middle, and apical thirds. Similar images of the hybrid layer were obtained in another study[27] when an etch-and-rinse adhesive system was applied in the root canal.

The null hypothesis that the luting length does not affect the pull-out bond strength was accepted because this factor was not significant. The luting of the post at the 5-mm length allows the LED light to reach the resin cement more effectively, resulting in a greater degree of conversion and,

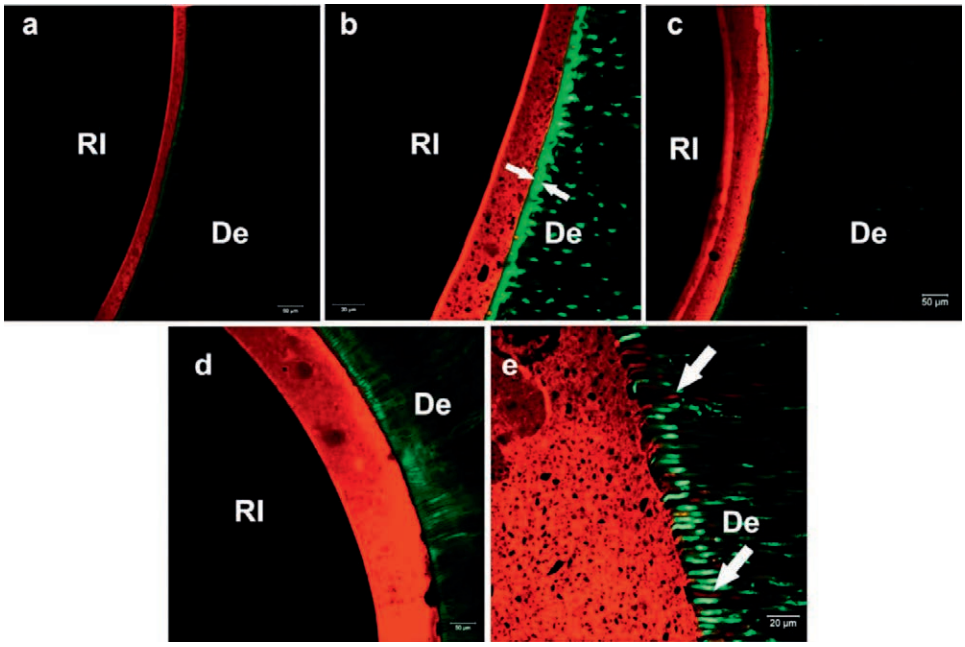


Figure 2. *CLSM image of the interface between a relined fiberglass post and root dentin using RelyX ARC resin cement. Green color: Adper Scotchbond Multi-Purpose Plus Adhesive (activator, primer, and catalyst). Red color: resin cement RelyX ARC. RI: relined fiberglass post; De: dentin. (a) Cervical third (20×); (b) cervical third (40×) showing the hybrid layer formation of the green-labeled adhesive (arrows); (c) middle third (20×); (d) apical third (20×); (e) higher magnification (63×) of the middle third showing the presence of tags from both the adhesive system and the resin cement. Penetration into the dentinal tubules is identified by the arrows.*

consequently, better mechanical properties of the resin cement. Furthermore, there is better penetration of the cement into the root canals at shorter lengths, thus minimizing the formation of bubbles or voids and better controlling the moisture of the substrate.[11,28]

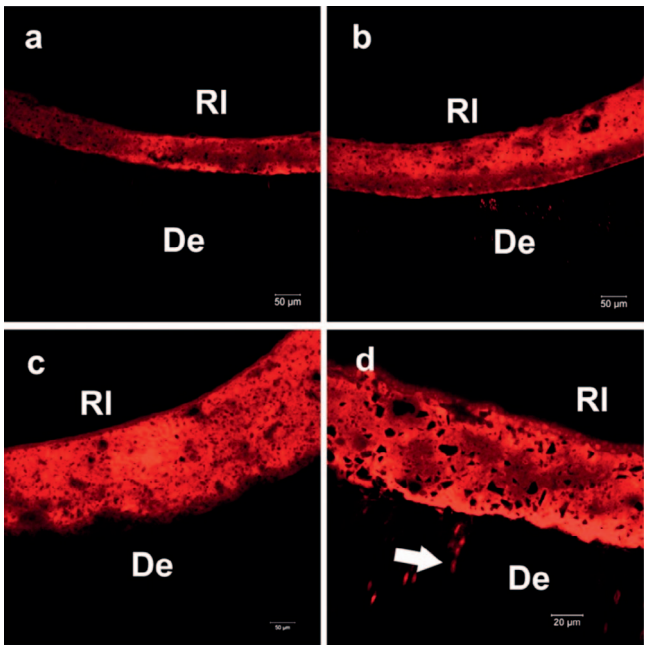


Figure 3. *CLSM image of the interface between the relined fiberglass post and root dentin using RelyX U200. Red color: resin cement. RI: relined fiberglass post; De: dentin. (a) Cervical third (20×); (b) middle third (20×); (c) apical third (20×); (d) higher magnification (63×) of the middle third demonstrates the penetration of the resin cement into the dentinal tubules forming resin tags (arrows).*

In the present study, the interaction between the luting length and cyclic loading was significant. The relined fiberglass posts with a length of 5 mm had weaker bond strength after cyclic loading. Therefore, cyclic loading accelerated the degradation of the bond between the resin cement and the root dentin at the 5-mm length. However, it is important to note that there was no failure of luting during cyclic loading, regardless of the luting length. This probably occurred because of the juxtaposition of the relined fiberglass post to the root dentin, favoring the formation of a thinner resin cement film. Moreover, the fiberglass post and the composite resin have a modulus of elasticity that is close to that of dentin (18 to 20 GPa), so the forces applied to the dental structure are dissipated throughout the fiberglass post.[29] For the 10-mm luting length, there was no significant difference in the pull-out bond strength between specimens with and without cyclic loading, suggesting greater clinical performance of treatments using relined fiberglass posts in weakened roots with 10-mm luting lengths. It has been demonstrated[30] that sliding friction contributes significantly to fiberglass post retention in root canals, with sliding friction being directly proportional to the contact area.

The third null hypothesis was rejected because cyclic loading influenced the pull-out bond strength

of relined fiberglass posts to root dentin. The interface between the restorative material and the tooth structure can undergo degradation over time as a result of hydrolysis, temperature changes, and crack propagation when the restoration is retained mechanically.[14] The hostile environment simulated in cyclic loading can lead to catastrophic failure of the restoration over time.[31] Cyclic loading in a wet environment is an aging method that simulates clinical conditions, predicting the clinical behavior of materials or restorative techniques in the oral environment.[31] For dental evaluations, the minimum number of 10^6 cycles, which simulates one year in service, should be applied to restorations when the desire is to approximate the performance of a material relative to clinically relevant service.[14] As the cyclic loading was applied directly on the relined fiberglass post, because there was no restoration, the load of 50 N was selected, which corresponds to approximately half of the maximal bite force for the anterior dentition.[32,33]

The pull-out bond strength test was conducted because it is the only feasible method of evaluating the influence of different luting lengths. Studies indicate the pull-out test as the *in vitro* test that is able to assess precisely the bond strength between the fiberglass post and the root dentin, because this test better distributes the stresses in the post-dentin interface, being a reliable test.[34] The use of bovine teeth in the pull-out test is justified because of difficulty in obtaining a large number of anterior human teeth and because of the morphological and histological resemblance to human teeth of bovine teeth.[35] However, a few human teeth were used for the CLSM analysis. CLSM is a method that allows samples to be studied without vacuum in a humid environment, and this method allows visualizing different components through the use of dyes. In the present study, two dyes, rhodamine B and fluorescein, were used because they have different characteristics.[36,37] Rhodamine B is a molecule added to the resin cement, and Fluorescein is added to the adhesive system components (activator, primer, and catalyst).[38] The same proportion (0.1%) was used for both dyes. Bitter and others[39] showed that fluorescein inserted into the adhesive showed up without diffusing into the red caused by rhodamine B in resin cements, marking a clear distinction between the dyes.

As a result of the limitations of *in vitro* studies, randomized clinical trials are necessary to generate better evidence on luting protocols with relined fiberglass posts.

CONCLUSIONS

In accordance with the results obtained in this study, it was concluded that

- The luting length is an important factor in retaining relined fiberglass posts in weakened roots when subjected to cyclic loading;
- The self-adhesive resin cement RelyX U200 resulted in greater bond strengths to the root canal in comparison with the conventional resin cement RelyX ARC; and
- Resin cement tags were formed in the cervical and middle thirds of the root canals when relined fiberglass posts were luted with RelyX ARC and RelyX U200.

Acknowledgement

The authors thank Ricardo Vaz Breda from the Institute of Biomedical Research of Pontifical Catholic University of Rio Grande do Sul (PUCRS) for assisting with the images of confocal laser scanning microscopy.

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 of Pontifical Catholic University of Rio Grande do Sul. The approval code for this study is 30904114.4.0000.5336.

Conflict of Interest

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

(Accepted 11 May 2016)

REFERENCES

1. Clavijo VG, Reis JM, Kabbach W, Silva AL, Oliveira Júnior OB, & Andrade MF (2009) Fracture strength of flared bovine roots restored with different intraradicular posts *Journal of Applied Oral Science* **17(6)** 574-578.
2. Schmitter M, Hamadi K, & Rammelsberg P (2011) Survival of two post systems—Five-year results of a randomized clinical trial *Quintessence International* **42(10)** 843-850.
3. Monticelli F, Grandini S, Goracci C, & Ferrari M (2003) Clinical behavior of translucent-fiber posts: A 2-year prospective study *International Journal of Prosthodontics* **16(6)** 593-596.
4. 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.
5. Grandini S, Sapio S, & Simonetti M (2003) Use of anatomic post and core for reconstructing an endodonti-

cally treated tooth: A case report *Journal of Adhesive Dentistry* **5(3)** 243-247.

6. Gaston BA, West LA, Liewehr FR, Fernandes C, & Pashley DH (2001) Evaluation of regional bond strength of resin cement to endodontic surfaces *Journal of Endodontics* **27(5)** 321-324.
7. Bouillaguet S, Troesch S, Wataha JC, Krejci I, Meyer JM, & Pashley DH (2003) Microtensile bond strength between adhesive cements and root canal dentin *Dental Materials* **19(3)** 199-205.
8. Amaral M, Rippe MP, Bergoli CD, Monaco C, & Valandro LF (2011) Multi-step adhesive cementation versus one-step adhesive cementation: Push-out bond strength between fiber post and root dentin before and after mechanical cycling *General Dentistry* **59(5)** e185-e191.
9. Nova V, Karygianni L, Altenburger MJ, Wolkewitz M, Kielbassa AM, & Wrbas KT (2013) Pull-out bond strength of a fiber-reinforced composite post system luted with self-adhesive resin cements *Journal of Dentistry* **41(11)** 1020-1026.
10. Ferrari M, Mannocci F, Vichi A, Cagidiaco MC, & Mjör IA (2000) Bonding to root canal: Structural characteristics of the substrate *American Journal of Dentistry* **13(5)** 255-260.
11. Vichi A, Grandini S, Davidson CL, & Ferrari M (2002) An SEM evaluation of several adhesive systems used for bonding fiber posts under clinical conditions *Dental Materials* **18(7)** 495-502.
12. McLaren JD, McLaren CI, Yaman P, Bin-Shuwaish MS, Dennison JD, & McDonald NJ (2009) The effect of post type and length on the fracture resistance of endodontically treated teeth *Journal of Prosthetic Dentistry* **101(3)** 174-182.
13. Zicari F, Van Meerbeek B, Scotti R, & Naert I (2012) Effect of fiber post length and adhesive strategy on fracture resistance of endodontically treated teeth after fatigue loading *Journal of Dentistry* **40(4)** 312-321.
14. Wiskott HWA, Nicholls JL, & Belser UC (1995) Stress fatigue: Basic principles and prosthodontics implications *International Journal of Prosthodontics* **8(2)** 105-116.
15. Arrais CA, Miyake K, Rueggeberg FA, Pashley DH, & Giannini M (2009) Micromorphology of resin/dentin interfaces using 4th and 5th generation dual-curing adhesive/cement systems: A confocal laser scanning microscope analysis *Journal of Adhesive Dentistry* **11(1)**15-26.
16. Leme AA, Coutinho M, Insaurralde AF, Scaffa PM, & da Silva LM (2011) The influence of time and cement type on push-out bond strength of fiber posts to root dentin *Operative Dentistry* **36(6)** 643-648.
17. 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 canal: A systematic review and meta-analysis of in vitro studies *Operative Dentistry* **39(1)** E31-E44.
18. Gerth HUV, Dammaschke T, Züchner H, & Schäfer E (2006) Chemical analysis and bonding reaction of RelyX Unicem and Bifix composites—A comparative study *Dental Materials* **22(10)** 934-941.
19. Saskalauskaite E, Tam LE, & McComb D (2008) Flexural strength, elastic modulus, and pH profile of self-etch resin cements *Journal of Prosthodontics* **17(4)** 262-268.
20. Radovic I, Monticelli F, Goracci C, Vulicevic ZR, & Ferrari M (2008) Self-adhesive resin cements: A literature review *Journal of Adhesive Dentistry* **10(4)** 251-256.
21. Cantoro A, Goracci C, Vichi A, Mazzoni A, Fadda GM, & Ferrari M (2011) Retentive strength and sealing ability of new self-adhesive resin cements in fiber post luting *Dental Materials* **27(10)** e197-e204.
22. Arrais CA, Giannini M, & Rueggeberg FA (2009) Effect of sodium sulfinate salts on the polymerization characteristics of dual-cured resin cement systems exposed to attenuated light-activation *Journal of Dentistry* **37(3)** 219-227.
23. Sarr M, Kane AW, Vreven J, Mine A, Van Landuyt KL, Peumans M, Lambrechts P, Van Meerbeek B, & De Munck J (2010) Microtensile bond strength and interfacial characterization of 11 contemporary adhesives bonded to bur-cut dentin *Operative Dentistry* **35(1)** 94-104.
24. Grandini S, Goracci C, Monticelli F, Borracchini A, & Ferrari M (2005) SEM evaluation of the cement layer thickness after luting two different posts *Journal of Adhesive Dentistry* **7(3)** 235-240.
25. Schetini DFF, Ferreira FJR, Amaral FLB, Miranda ME, & Turssi CP (2014) Root canal flare: Effect on push-out strength of relined posts *International Journal of Adhesion and Adhesives* **55** 139-144.
26. De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, & Van Meerbeek B (2004) Bonding of an auto-adhesive luting material to enamel and dentin *Dental Materials* **20(10)** 963-971.
27. Bitter K, Paris S, Mueller J, Neumann K, & Kielbassa AM (2009) Correlation of scanning electron and confocal laser scanning microscopy analyses for visualization of dentin/adhesive interfaces in the root canal *Journal of Adhesive Dentistry* **11(1)** 7-14.
28. Faria e Silva AL, Arias VG, Soares LE, Martin AA, & Martins LR (2007) Influence of fiber-post translucency on the degree of conversion of a dual-cured resin cement *Journal of Endodontics* **33(3)** 303-305.
29. Plotino G, Grande NM, Bedini R, Pameijer CH, & Somma F (2007) Flexural properties of endodontic posts and human root dentin *Dental Materials* **23(9)** 1129-1135.
30. Goracci C, Fabianelli A, Sadek FT, Papacchini F, Tay FR, & Ferrari M (2005) The contribution of friction to the dislocation resistance of bonded fiber posts *Journal of Endodontics* **31(8)** 608-612.
31. Grandini S, Chieffi N, Cagidiaco MC, Goracci C, & Ferrari M (2008) Fatigue resistance and structural integrity of different types of fiber posts *Dental Materials* **27(5)** 687-694.
32. Helkimo E, Carlsson GE, & Helkimo M (1977) Bite force and state of dentition *Acta Odontologica Scandinavica* **35(6)** 297-303.
33. Pereira-Cenci T, Pereira LJ, Cenci MS, Bonachela WC, & Del Bel Cury AA (2007) Maximal bite force and its

association with temporomandibular disorders *Brazilian Dental Journal* **18(1)** 65-68.

34. Castellan CS, Santos-Filho PC, Soares PV, Soares CJ, & Cardoso PE (2010) Measuring bond strength between fiber post and root dentin: A comparison of different tests *Journal of Adhesive Dentistry* **12(6)** 477-485.
35. Schilke R, Lisson JA, Bauss O, & Geurtsen W (2000) Comparison of the number and diameter of dentinal tubules in human and bovine dentine by scanning electron microscopic investigation *Archives of Oral Biology* **45(5)** 355-361.
36. Wang Y, & Spencer P (2004) Exploring the nature of acid-resistant hybrid layer with wet bonding *Operative Dentistry* **29(6)** 650-655.
37. Bitter K, Gläser C, Neumann K, Blunck U, & Frankenberger R (2014) Analysis of resin-dentin interface morphology and bond strength evaluation of core materials for one stage post-endodontic restorations *PLoS One* **9(2)** e86294.
38. Aguiar TR, Andre CB, Arrais CAG, Bedran-Russo AK, & Giannini M (2012) Micromorphology of resin–dentin interfaces using self-adhesive and conventional resin cements: A confocal laser and scanning electron microscope analysis *International Journal of Adhesive and Adhesion* **38** 69-74.
39. Bitter K, Paris S, Pfuertner C, Neumann K, & Kielbassa AM (2009) Morphological and bond strength evaluation of different resin cements to root dentin *European Journal of Oral Science* **117(3)** 326-333.

©Operative Dentistry, 2016, **41-6,** e183-e194

Influence of the Compliance and Layering Method on the Wall Deflection of Simulated Cavities in Bulk-fill Composite Restoration

Y-J Kim • R Kim • JL Ferracane • I-B Lee

Clinical Relevance

Both conventional and bulk-fill composites showed lower wall deflection when incrementally filled. Restoration by bulk filling with high viscous bulk-fill composites cannot achieve the reduction in the wall deflection of simulated cavities comparable to those obtained with incremental layering of conventional universal composites.

SUMMARY

The aim of this study was to investigate the effects of the layering method and compliance on the wall deflection of simulated cavities in bulk-fill and conventional composite restorations and to examine the relationships between the wall deflection and the polymerization shrinkage, flexural modulus, and polymerization shrinkage stress of composites. Six light-cured composites were used in this study. Two of these were conventional methacrylate-based composites (Filtek Z250 and Filtek Z350 XT Flowable [Z350F]), whereas four were bulk-fill composites (SonicFill, Tetric N-Ceram Bulk-Fill, SureFil SDR Flow [SDR], and Filtek Bulk-Fill). One hundred eighty aluminum molds simulating a mesio-occluso-distal cavity (6 W×8 L×4 D mm) were prepared and classified into three groups with mold wall thicknesses of 1, 2, and 3 mm. Each group was further subdivided according to the composite layering method (bulk or incremental layering). Linear variable differential transformer probes were used to measure the mold wall deflection of each composite (n=5) over a period of 2000 seconds (33.3 minutes). The polymerization shrinkage, flexural modulus, and polymerization shrinkage stress of the six composites were also measured. All groups with bulk filling exhibited significantly higher deflection compared with groups with incremental layering. The deflection decreased as mold wall thickness increased. The highest and lowest polymerization shrinkage stresses were recorded

Yu-Jin Kim, DDS, MSD, Department of Conservative Dentistry, School of Dentistry, Seoul National University, Seoul, Republic of Korea

Ryan Kim, BDS, PhD, Gwanak Dental Hospital, Seoul National University, Seoul, Republic of Korea

Jack L. Ferracane, PhD, Restorative Dentistry, Oregon Health & Science University, Portland, OR, USA

*In-Bog Lee, DDS, PhD, Department of Conservative Dentistry, School of Dentistry, Seoul National University, Seoul, Republic of Korea

*Corresponding author: 28, Daehak-Ro 5-gil, Jongro-Gu, Chongro-gu, Seoul 110-749, Republic of Korea; inboglee@snu.ac.kr

DOI: 10.2341/15-260-L

for Z350F (5.07 MPa) and SDR (1.70 MPa), respectively. The correlation between polymerization shrinkage and the mold wall deflection decreased with increasing wall thickness. On the other hand, the correlation between flexural modulus and the mold wall deflection increased with increasing wall thickness. For all groups, wall deflection correlated strongly with polymerization shrinkage stress.

INTRODUCTION

The polymerization shrinkage stress of dental composites may compromise the bond integrity and cause enamel cracking and cusp deflection.[1] Therefore, minimizing polymerization shrinkage stress of composites is still a major challenge for dental clinicians when placing composite restorations.

Incremental layering can reduce the effects of the configuration-factor, thereby allowing more flow of the composite from the free surface, which also reduces the volume of composite being cured, maximizes the degree of conversion, and increases the adaptation to cavity walls.[2-4] Although incremental layering does have apparent benefits, the process of multiple layering and curing is time consuming; moreover, the effectiveness of this strategy in reducing polymerization shrinkage stress and cusp deflection has been questioned.[5-8] However, a number of studies have reported considerably reduced cusp deflection by using incremental layering compared with bulk filling.[4,9,10]

To predict polymerization shrinkage stress in the clinical situation, experiments must be designed in a way that mimics the tooth/composite interface.[11] Cusp deflections are well described to be closely related with polymerization shrinkage stress.[12] Moreover, cusp deflections have been extensively investigated using a variety of techniques and instruments, including the strain gage[13] and the linear variable differential transformer (LVDT) methods.[9]

Cusp compliance is an important factor that affects cusp deflection. Compliance is defined as a change in dimension of a system to unit force and has opposite meaning to stiffness. Thus, to obtain clinically relevant results, cusp compliance should be similar to that observed in clinical situations. Several studies have reported that teeth with cavities exhibit relatively high compliance.[9,14,15] The stress values measured in low compliance systems have ranged from 4 to 25 MPa,[3,16-18] whereas values obtained in high compliance systems have barely exceeded 5 MPa.[19,20]

In this study, aluminum blocks with a differing thickness of mold wall were used for reducing the substrate variation. The elastic modulus of aluminum is 68.5 GPa, which is within the range of tooth enamel (84.1 GPa) and dentin (18.5 GPa).[10] In a previous study, the cusp compliance of natural teeth with mesio-occluso-distal (MOD) cavities (1.5 W $\times$ 2 D and 3 W $\times$ 2 D mm) was 2.96 and 3.32 μm/N, respectively, which is about three to four times more than that of aluminum blocks.[21] Therefore, although the aluminum block does not exactly replicate the natural tooth, this experimental design enables the investigation of the cusp deflection under the conditions with minimized variables.

Recently, many bulk-fill composites have been introduced as alternatives to conventional composites. These composites are intended to be placed and bulk-cured in one increment, up to 4 to 5 mm in depth, either with or without a superficial capping layer. The rheological properties of these composites can be varied by modifying the filler content, monomer type, or by adding modulators to slow the polymerization rate.[22-24] However, little information is available regarding the polymerization kinetics of these composites. Moreover, no study to date has investigated the effect of cusp compliance and layering method on cusp deflection of bulk-fill composite materials or the relationship between cusp deflection and the polymerization shrinkage kinetics of these composites.

The aim of this study was to investigate the effects of the layering method and compliance on the wall deflections of simulated cavities in bulk-fill vs conventional composite restorations. In addition, the relationships between the wall deflection and the polymerization shrinkage, flexural modulus, and polymerization shrinkage stress of various composites were also examined.

METHODS AND MATERIALS

Materials

Six light-cured composites were examined in this study. Each composite was categorized as conventional or bulk-fill and high-viscosity or low-viscosity (flowable) composite according to its use and viscosity. Two were conventional methacrylate-based composites, a high-viscosity (Filtek Z250 [Z250]) and a flowable (Filtek Z350 XT Flowable [Z350F]) composite. The four bulk-fill composites included two high-viscosity composites (SonicFill

Table 1: *Brand Name, Type, Composition, and Manufacturer of Each Composite Used in This Study*

Composite (code, shade, batch no.)	Type	Composition	Manufacturer
Filtek Z250 (Z250, A2, N482264)	C, H	Bis-GMA, Bis-EMA, TEGDMA, UDMA 0.01-3.5 μm Zr/silica particles (82 wt%/60 vol%)	3M ESPE, St. Paul, MN, USA
SonicFill (SF, A2, 5026722)	B, H	Bis-GMA, TEGDMA, EBPDMA, silica, glass, oxide (83.5 wt%/69 vol%)	Kerr, Orange, CA, USA
Tetric N-Ceram Bulk-Fill (TNB, IVA, S09719)	B, H	Bis-GMA, UDMA Ba-glass filler, ytterbium trifluoride, Mixed oxide prepolymer (79-81 wt%/-)	Ivoclar Vivadent, Schaan, Liechtenstein
Filtek Z350 XT Flowable (Z350F, A2, N50234)	C, F	Bis-GMA, Bis-EMA, TEGDMA 5-20 nm Zr/silica nano-particles, 0.6-1.4 μm nano-clusters (65 wt%/-)	3M ESPE, St. Paul, MN, USA
SureFil SDR Flow (SDR, Universal shade, 130630)	B, F*	Modified UDMA, TEGDMA, EBPDMA Ba-Al-F-B-Si glass, St-Al-F-Si glass (68 wt%/45 vol%)	Dentsply, Konstanz, Germany
Filtek Bulk-Fill Flowable (FB, A2, N540884)	B, F*	Bis-GMA, UDMA, Bis-EMA, Procrylat resins Zr/silica, ytterbium trifluoride (64.5 wt%/42.5 vol%)	3M ESPE, St. Paul, MN, USA

Abbreviations: B, bulk-fill; Bis-EMA, bisphenol-A polyethylene glycol diether dimethacrylate; Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; C, conventional composite; EBPDMA, ethoxylated bisphenol-A-dimethacrylate; F, flowable; H, high-viscosity; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
** Bulk-fill composites requiring a 2-mm capping layer as recommended by manufacturers.*

[SF]/Tetric N-Ceram Bulk-Fill [TNB]) and two flowable composites (SureFil SDR Flow [SDR]/ Filtek Bulk-Fill [FB]). The brand names, types, compositions, and manufacturers of the composites are listed in Table 1. An LED light unit (Elipar S10, 3M ESPE, St. Paul, MN, USA) was used for curing; the light irradiance exiting the tip (9.9 mm in diameter) was 1200 mW/cm^2.

Measurement of Deflection of Aluminum Mold Wall

One hundred eighty aluminum molds simulating an MOD cavity (6 W × 8 L × 4 D mm) were prepared and allocated into three groups with varying thicknesses of the wall of aluminum mold (1, 2, and 3 mm; Figure 1A). The inside wall of each cavity was air abraded with 50 μm Al_2O_3 powder, rinsed with water, and air dried. Then, the inside of the cavity was coated twice

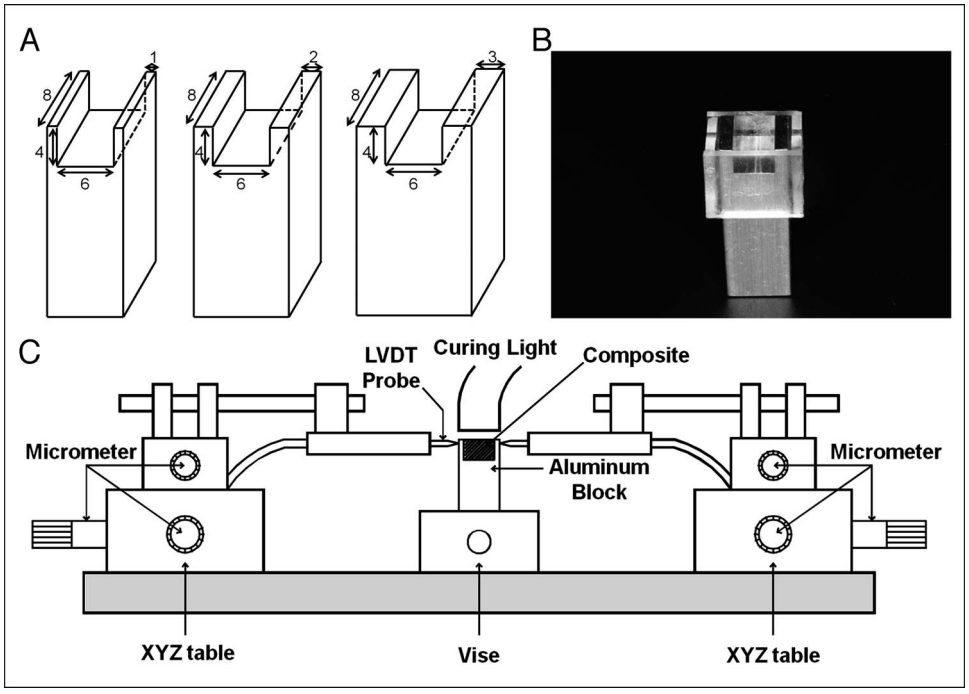


Figure 1. *(A) Dimensions (mm) of aluminum blocks with varying mold wall thicknesses: left, 1 mm; center, 2 mm; right, 3 mm. (B) Acrylic cap placed over the aluminum block. (C) Instrument for measuring mold wall deflection.*

with a metal primer (Z-Prime Plus, Bisco, Schaumburg, IL, USA) and dried. A thin layer of Scotchbond Multipurpose Adhesive (3M ESPE) was applied and light cured for 10 seconds.

An acrylic cap (Figure 1B) with two notches on the top of the lateral wall was fabricated and placed on top of the aluminum block to prevent the composite from being pushed out of the mold during layering. The acrylic cap was also used to place the LVDT probes precisely 1 mm below the upper surface of mold wall through the notches of the acrylic cap. The inner surface of the acrylic cap was lubricated with petroleum jelly to prevent the composite from adhering. The required weight of composite to fill the aluminum mold was calculated from the density of the composite and the volume of the mold, and the appropriate amount of composite was weighed before use.

The groups with different mold wall thickness were further subdivided according to composite layering method (bulk vs incremental layering). Before mounting the specimen in the mold wall deflection measurement instrument, the composite for bulk filling or the first layer of incremental filling was placed in the mold. In the bulk-filling group, the composite was light cured from the upper surface for 20 seconds, the mesially tilted upper side for 20 seconds, the distally tilted upper side for 20 seconds, and again the upper surface for 20 seconds (total 80 seconds to be consistent with the energy delivery for the incremental layering group). For the incremental layering group, the composite was filled in four horizontal increments approximately 1 mm thick. Each layer was light cured from the upper surface for 20 seconds (total 80 seconds) for maximum polymerization to minimize possible bias that could be caused by incomplete curing of composites. Five aluminum blocks were allocated for each subgroup (bulk or incremental) of each composite.

The displacement of the mold wall was measured in real time at 25 ± 0.5°C throughout the curing process using two LVDT probes (AX-1, Solartron Metrology, West Sussex, United Kingdom), each with a sensitivity exceeding 0.1 μm over a range of ±1 mm (Figure 1C). The displacement values measured by the two LVDT probes were stored on a computer using a data acquisition board (PCI-6024, National Instruments, Austin, TX, USA) and software (LabVIEW, National Instruments). Measurement of mold wall deflection was initiated 20 seconds prior to light irradiation to obtain a baseline and continued for up to 2000 seconds (33.3 minutes), at a rate of 2 data points/s. The displacements of both sides were added to obtain the total amount of deflection (n=5).

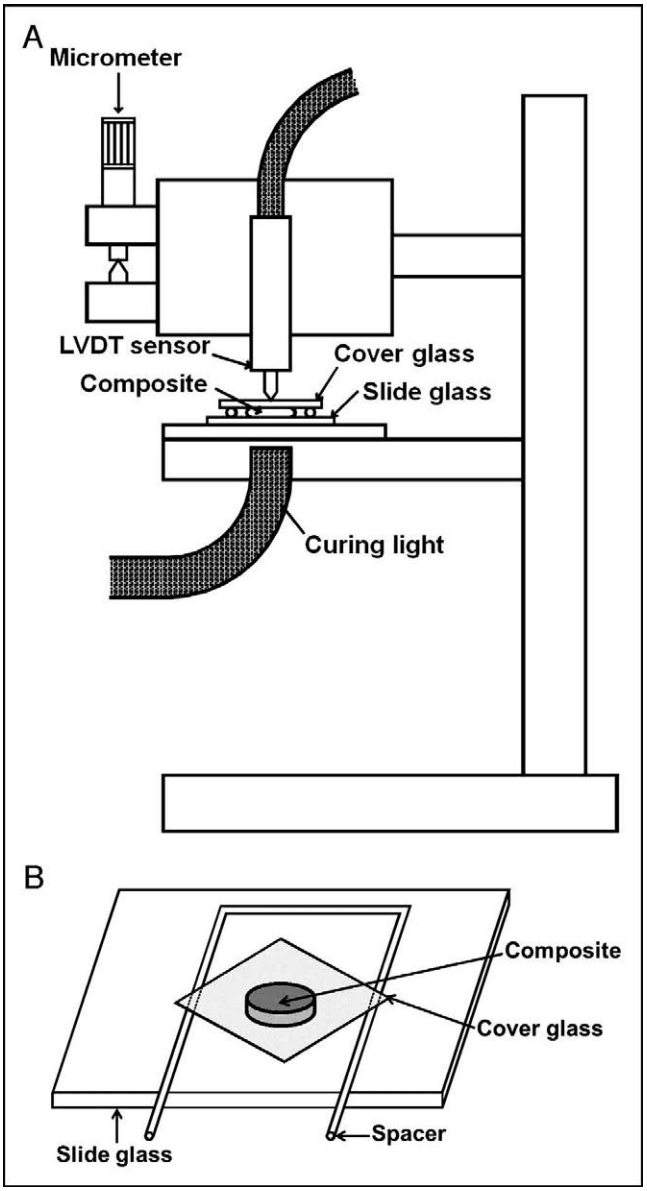


Figure 2. *(A) Instrument for measuring polymerization shrinkage using the modified bonded disc method. (B) Specimen preparation.*

Measurement of Axial Polymerization Shrinkage

Axial polymerization shrinkage was measured with the modified bonded disc method (Figure 2).[25] Briefly, the designated amount of composite was pressed between a slide glass and a flexible cover glass (Marienfeld-Superior, Lauda-Königshofen, Germany). A metal wire spacer was used to make 0.5-mm-thick specimens. The tip of an LVDT probe was placed on the cover glass at the center of the

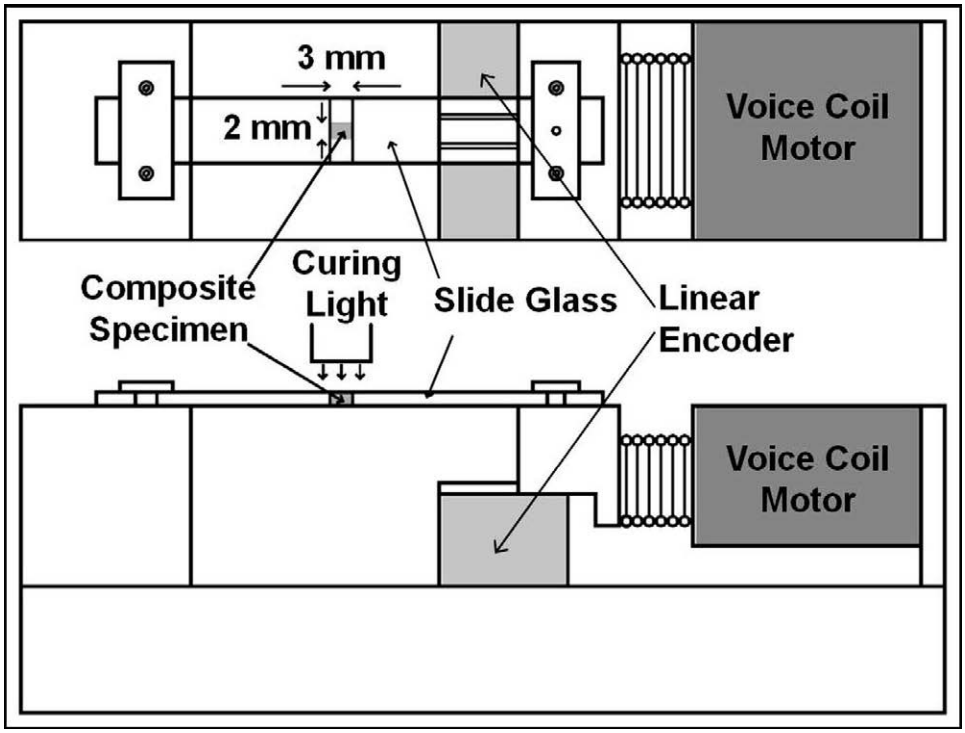


Figure 3. *Instrument for measuring polymerization shrinkage stress using a voice coil motor with feedback mechanism.*

disc-shaped composite specimen; this point was set to zero. Baseline data were obtained for 10 seconds and then the curing light was turned on for 40 seconds. The axial shrinkage data were stored on a computer at a rate of 10 data points/s for 600 seconds (n=5). The thickness of the light-cured specimen was measured using a micrometer. The axial polymerization shrinkage (%) was calculated using the following equation:

$$\text{Axial polymerization shrinkage}(\%) = \frac{100 \times \text{shrinkage}}{(\text{cured specimen thickness} + \text{final shrinkage})}$$

The shrinkage rate (%/s) and time at the peak shrinkage rate (seconds) were also obtained.

Measurement of Flexural Modulus

Bar-shaped specimens were generated by compressing the composite between a Teflon mold (3 W × 3 T × 30 L mm) and a slide glass. The specimens were divided into five parts and light cured with overlapping exposures of 40 seconds each (total 200 seconds). The cured specimens were polished and stored in dry conditions for 1 day in the dark at room temperature (25±1°C). The width and thickness of each specimen was measured with a micrometer; flexural modulus was measured using the three point bending method with a universal testing machine (LF Plus, Lloyd Instruments, West Sussex, United Kingdom) at a crosshead speed of 0.5 mm/min (supporting span length=20 mm; n=5).

Measurement of Polymerization Shrinkage Stress

A custom-made instrument with a voice coil motor (MGV52-20-0.5, Akribis Systems, Singapore) was used to measure the polymerization shrinkage stress (Figure 3). Briefly, a slide glass was fixed to a movable stage, which was connected to the voice coil motor. Another slide glass was fixed to an immobile stage on the opposite side of the motor. As the composite between the two slide glasses contracted due to polymerization, the slide fixed to the movable voice coil motor was pulled to the opposite slide, which was fixed on the immobile stage. This deviation was then detected by the linear encoder. Immediately, a servo amplifier provided electrical current to the voice coil motor to offset this deviation. Therefore, the distance between the two slide glasses was maintained. This feedback mechanism continued, with the servo electrical current staying proportional to the polymerization shrinkage stress. Calibration analysis revealed a linear relationship between the shrinkage force and the servo current.

The end surfaces of two 1-mm-thick slide glasses were sandblasted with 50-μm Al_2O_3 particles and covered with adhesive tape. A 2-mm-wide window

was created on the taped surface, thus exposing the glass surface, which was treated with silane (Monobond S, Ivoclar Vivadent, Schaan, Liechtenstein) and a bonding agent (Scotchbond Multipurpose Adhesive, 3M ESPE) and light cured for 10 seconds. The two slide glasses were aligned 3 mm apart from one another and then fixed on the movable and immobile stages of the instrument. The volume of the composite specimen between the two slide glasses was 6 mm^3. After the composite was placed between the slides, baseline data were obtained for 10 seconds, and the composite was irradiated with a curing light for 40 seconds. Measurements were made for each composite, at a rate of 10 data points/s, for 10 minutes (n=5).

Measurement of the Compliance of Aluminum Mold Wall

The aluminum block was fixed on a metal base and a weight loaded on the block 0.5 mm from the tip of the mold (Figure 4). Additional weight was applied onto the mold wall in increments of 1 to 5 kg. The mold wall displacements were measured using an LVDT probe, and the compliances were obtained from the measured load-displacement curves (n=3).

Statistical Analysis

Data were analyzed using SPSS software (version 21.0). Multiple-way analysis of variance (ANOVA) and Tukey's post hoc test were used to compare the deflection groups. The polymerization shrinkage, flexural modulus, and polymerization shrinkage stress of the composites were compared using one-way ANOVA and Tukey's post hoc tests. Pearson's correlation analysis was conducted to investigate the relationships between deflection and the polymerization shrinkage, flexural modulus, and polymerization shrinkage stress of the composites. All tests were conducted at α=0.05.

RESULTS

Mold Wall Deflection

The mold wall deflections of Z250 fillings of different thicknesses and layering methods as a function of time are shown in Figure 5. The majority of the deflection was observed within 500 seconds and gradually increased thereafter. The deflection occurred in a stepwise manner in the incremental layering group; moreover, deflection decreased slightly at the initiation of each period of light curing and increased thereafter. The mean deflections (micrometers) at 2000 seconds (33.3 minutes)

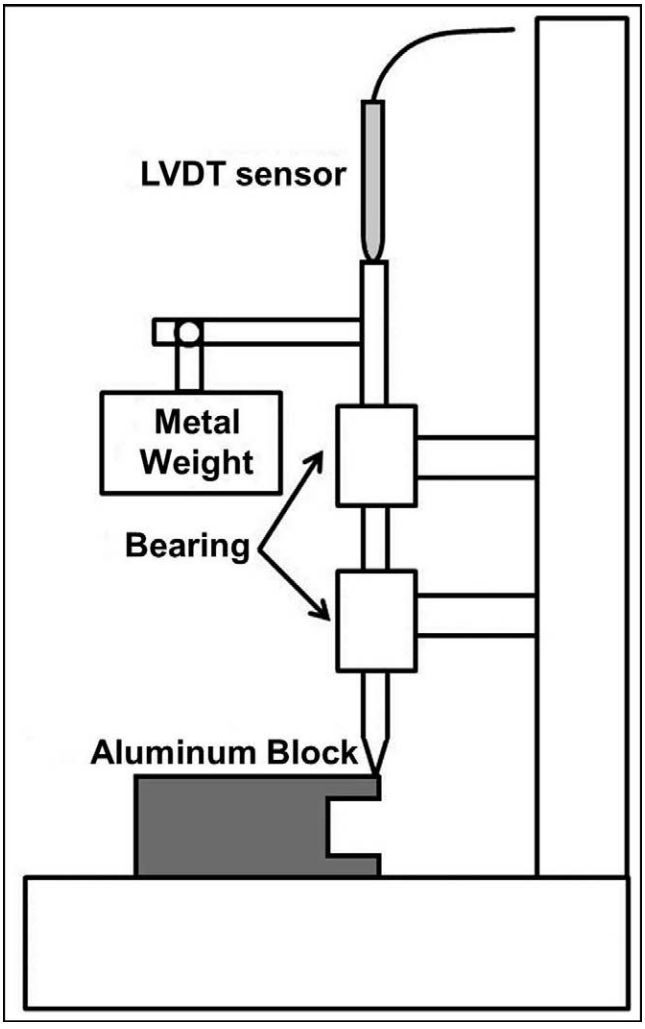


Figure 4. *Instrument for measuring the compliance of the aluminum blocks.*

for each composite are presented in Table 2. The highest and lowest deflections were obtained using Z350F bulk filling/1-mm mold wall thickness (51.0 μm) and SDR incremental layering/3-mm mold wall thickness (3.8 μm), respectively. The deflection (micrometers) and reduction (%) from bulk to incremental layering for each subgroup are presented in Figure 6. Mold wall thickness, layering method, and composite brand all yielded statistically significant differences ($p<0.05$) in the deflection. All groups with bulk filling exhibited significantly higher deflection compared with groups with incremental layering ($p<0.05$). Deflection decreased with increasing mold wall thickness ($p<0.05$).

Axial Polymerization Shrinkage

The Z350F composite demonstrated the highest polymerization shrinkage (3.52%), followed by FB

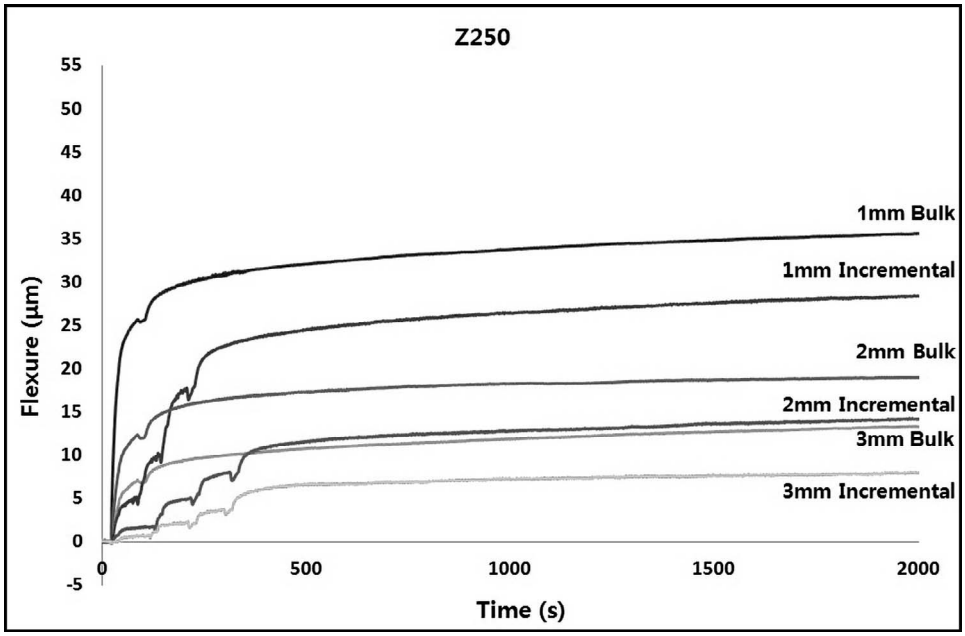


Figure 5. *The deflection (μm) of the Z250 composite with varying mold wall thicknesses and layering methods as a function of time.*

(3.17%), SDR (2.88%), Z250 (2.18%), and TNB (2.11%), whereas SF showed the lowest shrinkage (2.08%; Table 3). No significant differences were observed between Z250, TNB, and SF with respect to polymerization shrinkage ($p>0.05$). The polymerization shrinkage rates (%/s) and times at the peak shrinkage rate are shown in Table 3. The maximum rate of polymerization shrinkage was highest for SDR (0.64 %/s) and lowest for SF (0.34 %/s). The time at the peak shrinkage rate (seconds) was longest (2.11 seconds) for Z350F and shortest (1.11 seconds) for TNB.

Flexural Modulus

The flexural modulus (GPa) of each composite is presented in Table 3. Z250 showed the highest flexural modulus (9.20 GPa), followed by SF, TNB, Z350F, SDR, and FB (4.63 GPa). With the exception of Z350F and SDR ($p=0.888$), the composites exhibited significantly different flexural modulus values ($p<0.05$).

Polymerization Shrinkage Stress

The highest and lowest polymerization shrinkage stresses were recorded for Z350F (5.07 MPa) and SDR (1.70 MPa), respectively (Table 3). No significant differences were observed between Z250, TNB, and SF ($p>0.05$).

Compliance of Aluminum Mold Wall

The mold wall compliances with 1-, 2-, and 3-mm thicknesses were 0.81, 0.22, and 0.13 μm/N, respectively. The compliance decreased with increasing mold wall thickness.

Correlation Analysis

The correlation analysis results are presented in Table 4. For the 1-mm-thick mold, the deflection and polymerization shrinkage showed strong and moderate correlations in bulk ($r=0.706$) and incremental layering ($r=0.446$) groups, respectively. Meanwhile, for the 3-mm-thick mold, the deflection and flexural modulus were moderately correlated ($r=0.376$) in the bulk-filling group. The correlation between polymerization shrinkage and the deflection decreased as mold wall thickness increased. On the other hand, the correlation between flexural modulus and deflection increased with increasing mold wall thickness. The deflection for all groups correlated strongly with the polymerization shrinkage stress ($r=0.785$-0.969) and the product of shrinkage and modulus ($r=0.657$-0.780).

DISCUSSION

In this study, deflection was successfully simulated via micromechanical bonding of the composite to an aluminum block with a simulated cavity. Micromechanical bond strength of the composite to the aluminum surface was sufficient to produce measurable deflection as detected by LVDT probes. This idea is further supported by the lack of debonding spikes in the deflection curves. Therefore, our experimental design effectively simulated cusp de-

Table 2: *Mean Deflection (μm) for Each Group at 2000 Seconds (33.3 Minutes)*

Composite	Layering method	Aluminum mold wall thickness		
		1 mm	2 mm	3 mm
			Deflection, μm	
Filtek Z250	Bulk	35.6 (1.2)^{B,a}	19.0 (0.6)^{B,c}	13.4 (2.0)^{B,d}
	Incremental	28.4 (0.9)^{C,D,b}	14.2 (0.7)^{C,d}	8.1 (0.8)^{C,e}
SonicFill	Bulk	31.0 (0.6)^{C,a}	19.5 (1.7)^{B,c}	12.7 (1.4)^{B,d}
	Incremental	26.6 (1.9)^{D,b}	13.6 (1.2)^{C,d}	9.4 (0.7)^{C,e}
Tetric N-Ceram Bulk-Fill	Bulk	27.2 (0.7)^{D,a}	14.2 (0.7)^{C,c}	8.7 (0.7)^{C,d}
	Incremental	23.1 (0.8)^{E,b}	7.3 (0.5)^{D,e}	4.6 (0.1)^{D,f}
Filtek Z350 XT Flowable	Bulk	51.0 (2.2)^{A,a}	27.6 (2.0)^{A,b}	15.9 (1.1)^{A,d}
	Incremental	48.2 (1.2)^{A,a}	21.0 (1.7)^{B,c}	11.8 (0.3)^{B,e}
SureFil SDR Flow	Bulk	28.4 (1.5)^{C,D,a}	10.8 (1.1)^{D,c}	4.8 (0.2)^{D,e}
	Incremental	15.1 (0.6)^{F,b}	7.0 (0.8)^{D,d}	3.8 (0.3)^{D,e}
Filtek Bulk-Fill Flowable	Bulk	36.3 (2.0)^{B,a}	14.2 (1.3)^{C,c}	9.5 (0.3)^{C,d}
	Incremental	23.4 (0.8)^{E,b}	8.9 (0.7)^{D,E,d}	4.8 (0.8)^{D,e}

Identical uppercase letters: no significant difference among groups of the same wall thickness ($p>0.05$). Identical lowercase letters: no significant difference among groups of the same composite ($p>0.05$). Numbers in parentheses are standard deviations ($n=5$).

flection without the variability associated with natural teeth.

Bulk-fill composites can be classified into two types according to their viscosity and delivery method. Some low-viscosity bulk-fill composites (SDR and FB) necessitate a 2-mm capping layer with a conventional hybrid composite because of their low filler content and decreased abrasion resistance.[22] Another group of bulk-fill composites having high viscosity and filler content (SF and TNB) showed mechanical strength comparable to hybrid conventional composite, so they do not need to be capped with an additional layer.

The present study compared the polymerization shrinkage and related properties of four bulk-fill composites with those of two conventional composites. The polymerization shrinkages of flowable composites (Z350F, SDR, and FB) were higher than those of high-viscosity composites (Z250, SF, and TNB). However, the flexural modulus values of the composites exhibited the opposite trend as for polymerization shrinkage. These are expected based on the difference in filler amount.

Shrinkage stress can be directly influenced by instrument compliance.[26] In the shrinkage stress measuring system used in this study, the dimensional change of the composite specimen during polymerization was not measured at the very end of each glass slide, so it could be considered that this system was not fully rigid. Therefore, the use of a feedback mechanism minimized the compliance of the instrument, but did not totally eliminate it.

Polymerization shrinkage stress, as determined by both polymerization shrinkage and flexural modulus, showed complex results. Z350F exhibited the highest shrinkage stress value because it showed the highest shrinkage strain. Furthermore, Z250 showed the second highest shrinkage stress, perhaps because it had the highest flexural modulus. The bulk-fill flowable composites (SDR and FB) exhibited lower polymerization shrinkage stress due to their lower flexural modulus values, even though they exhibited higher polymerization shrinkage than the bulk-fill high-viscosity composites.[23,24] In contrast to the Z350F conventional flowable composite, the SDR contains the patented, modified urethane dimethacrylate monomer (849 g/mol). This monomer has a relatively high molecular weight, resulting in reduced polymerization shrinkage and stress by decreasing the number of reactive sites per unit volume. Meanwhile, FB excluded the monomer TEGDMA (286 g/mol), which has approximately half the molecular weight of the commonly added dimethacrylates such as Bis-GMA (512 g/mol).[27]

The polymerization shrinkage stress of each bulk-fill composite varied according to the viscosity of the material. The bulk-fill flowable composites (SDR and FB) exhibited lower polymerization shrinkage stress compared with the high-viscosity bulk-fill composites (SF and TNB). These results could be explained by the differences in filler loading, which result in different rheological properties. In the present study, a positive correlation between the flexural modulus and filler fraction was observed. The filler fractions of the bulk-fill composites according to the manufac-

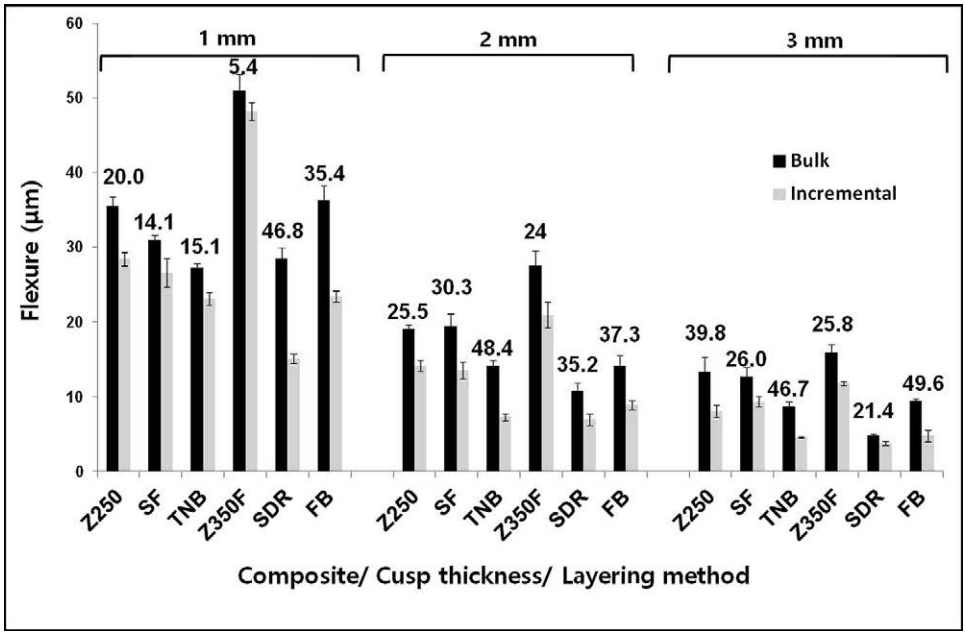


Figure 6. *Mean deflection (μm) and reduction in deflection from bulk to incremental layering (%) for each composite according to mold wall thickness and layering method. The number above each bar indicates the reduction (%) in deflection from bulk to incremental layering.*

turer's information are as follows: SF (83.5 wt%/69 vol%), TNB (79-81wt%/-), SDR (68.0 wt%/45 vol%), and FB (64.5 wt%/42.5 vol%); as expected, this order corresponds with that of the flexural modulus values.[16]

The deflection curves demonstrated slight reductions when the curing light was turned on, due to the thermal expansion effect created by the heat from the curing light (Figure 5). After the light curing unit was turned off, this expansion was counteracted by the ongoing polymerization shrinkage. In the bulk filling group, the thermal expansion effect could be observed only at the beginning of the final light curing, however, in the incremental layering group, four definite reductions in deflection due to thermal expansion effects could be clearly observed.[4]

Mold wall deflection decreased as wall thickness increased (compliance decreased) in all composite groups. However, considering the stiffness (inverse of compliance) of the wall, the thicker mold wall produced the higher stress. Incremental layering significantly reduced deflection compared with bulk filling for both conventional and bulk-fill composites (Table 2). These findings are in agreement with previous studies.[4,9,10] However, another study measured cusp deflection by using different curing techniques in natural teeth filled with conventional (Filtek Supreme Plus, 3M ESPE: bulk and incremental curing) or bulk-fill composites (X-tra fil, VOCO: bulk, incremental, and bulk/transtooth-illumination curing) and reported contradictory results.[28] The authors found no difference in cusp deflection between filling techniques within the same materials. These contradictory results may be due to that they used very thin cusp thickness with high compliance.

Table 3: *Polymerization Shrinkage (%), Maximum Shrinkage Rate (%/s), Time at Peak Shrinkage Rate (s), Flexural Modulus (GPa), and Polymerization Shrinkage Stress (MPa) of each Composite*

Composite	Polymerization shrinkage, %	Maximum shrinkage rate, %/s	Time at peak shrinkage rate, s	Flexural modulus, GPa	Polymerization shrinkage stress, MPa
Filtek Z250	2.18 (0.06)^{d}	0.35 (0.02)^{d}	1.56 (0.06)^{c}	9.20 (0.21)^{a}	2.88 (0.13)^{b}
SonicFill	2.08 (0.07)^{d}	0.34 (0.02)^{d}	1.79 (0.41)^{a,b,c}	7.97 (0.44)^{b}	2.73 (0.10)^{b}
Tetric N-Ceram	2.11 (0.02)^{d}	0.44 (0.03)^{c}	1.11 (0.11)^{d}	6.68 (0.25)^{c}	2.82 (0.13)^{b}
Filtek Z350 XT Flowable	3.52 (0.04)^{a}	0.64 (0.02)^{a}	2.11 (0.07)^{a}	5.79 (0.11)^{d}	5.07 (0.42)^{a}
SureFil SDR	2.88 (0.13)^{c}	0.64 (0.01)^{a}	1.67 (0.08)^{b,c}	5.62 (0.20)^{d}	1.70 (0.16)^{d}
Filtek Bulk-Fill	3.17 (0.03)^{b}	0.52 (0.05)^{b}	1.94 (0.08)^{a,b}	4.63 (0.18)^{e}	2.28 (0.19)^{c}

Identical superscript letters signify that no significant differences were observed among the designated materials within a single column ($p>0.05$). Numbers in parentheses are standard deviations ($n=5$).

Table 4: *Correlations Between the Deflection and the Polymerization Shrinkage, Flexural Modulus, and Polymerization Shrinkage Stress of Composites*

Measured variables	Polymerization shrinkage stress	Deflection					
		1 mm		2 mm		3 mm	
		Bulk	Incremental	Bulk	Incremental	Bulk	Incremental
Polymerization shrinkage	0.393*	0.706**	0.446*	0.282	0.328	0.099	0.17
Flexural modulus	0.033	−0.186	0.048	0.227	0.234	0.376*	0.341
Shrinkage × modulus	0.602**	0.661 **	0.678**	0.71**	0.78**	0.657**	0.727 **
Polymerization shrinkage stress	1	0.832 **	0.969**	0.885**	0.868**	0.785**	0.817 **

*Numbers are Pearson's correlation coefficients (*p<0.05; **p<0.01).*

The six composites used in this study can be classified into three groups according to the level of shrinkage stress they produced: conventional flowable with high stress (Z350F), high viscous bulk-fill (SF and TNB) and conventional (Z250) with moderate stress, and bulk-fill flowable with low stress (SDR and FB). Conventional flowable (Z350F) and bulk-fill flowable (SDR) composites showed the highest and lowest deflections, respectively. On the other hand, both bulk-fill (SF and TNB) and conventional (Z250) composites with moderate stress, which are of high viscosity, exhibited comparable deflections.

Within the composites with moderate stress, the deflection by bulk filling with either TNB or SF was equal to ($p>0.05$) or higher than ($p<0.05$) that by incremental layering with the conventional composite (Z250; Table 2). Therefore, bulk filling of a high viscosity bulk-fill composite with moderate stress does not appear to offer any advantages over incremental layering of a high viscosity conventional composite with moderate stress. Interestingly, TNB always exhibited the lowest deflection among the three composites with moderate stress because of its lowest flexural modulus, even though the three composites exhibited similar polymerization shrinkages.

Unlike the moderate stress groups with high viscosity, bulk filling of bulk-fill flowable composites with low stress (SDR and FB) yielded lower deflections than incremental layering of conventional flowable composites with high stress (Z350F). Despite its low flexural modulus, greater deflection was always observed with FB compared with SDR. This finding may be due to the significantly higher polymerization shrinkage and stress of FB.

The reduction (%) of deflection from bulk to incremental layering was largest for SDR (46.8%), TNB (48.4%), and FB (49.6%) for mold wall thicknesses of 1, 2, and 3 mm, respectively (Figure 6). Thus, bulk-fill composites were more effective in reducing mold wall deflection by incremental layering compared with conventional composites. The reduction of deflection achieved with incremental layering increased as mold wall thickness increased for Z250, Z350F, and FB. Moreover, with the exception of SDR, all composites showed greater reduction of deflection by incremental layering in 3-mm-thick mold wall compared with 1-mm-thick mold wall. In general, reduction of deflection by incremental layering was enhanced in thick mold walls (low compliance; Figure 6). In addition, as the mold wall thickness increased from 1 to 3 mm, the deflection by flowable composites with low modulus decreased more than in high-viscosity composites (Table 2).

The correlation between polymerization shrinkage and mold wall deflection decreased with increasing mold wall thickness. On the other hand, the correlation between flexural modulus and deflection increased with increasing mold wall thickness (Table 4). This result is supported by a previous study of the effect of instrument compliance on polymerization shrinkage stress,[29] which found that shrinkage strain was the major factor in determining stress when instrument compliance was high, whereas shrinkage strain and modulus played equal roles in determining the polymerization shrinkage stress when instrument compliance was restricted. In clinical situations, composites with high shrinkage are likely to produce greater cusp deflection in high compliance cavities, such as a large MOD cavity. In contrast, both the elastic modulus and shrinkage determine the polymerization shrinkage stress in low compliance cavities such as an occlusal cavity.

CONCLUSION

Both conventional and bulk-fill composites showed lower mold wall deflection when incrementally filled. As the mold wall thickness increased, the effect of

incremental layering on the reduction in mold wall deflection was enhanced. In addition, restoration by bulk filling with high viscous bulk-fill composites with moderate stress did not achieve reduction in wall deflection comparable to those obtained with incremental layering with conventional universal composites. When the compliance was high, polymerization shrinkage was the main factor that influenced deflection. In lower compliance cavities, both the flexural modulus and the polymerization shrinkage determined the deflection.

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 April 2016)

REFERENCES

1. Moorthy A, Hogg C, Dowling A, Grufferty B, Benetti AR, & Fleming G (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.
2. 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.
3. Feilzer A, De Gee A, & Davidson C (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.
4. Kwon Y, Ferracane JL, & 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.
5. Versluis A, Douglas W, Cross M, & Sakaguchi R (1996) Does an incremental filling technique reduce polymerization shrinkage stresses? *Journal of Dental Research* **75(3)** 871-878.
6. Jafarpour S, El-Badrawy W, Jazi H, & McComb D (2012) Effect of composite insertion technique on cuspal deflection using an in vitro simulation model *Operative Dentistry* **37(3)** 299-305.
7. Rees J, Jagger D, Williams D, Brown G, & Duguid W (2004) A reappraisal of the incremental packing technique for light cured composite resins *Journal of Oral Rehabilitation* **31(1)** 81-84.
8. Abbas G, Fleming G, Harrington E, Shortall A, & Burke F (2003) Cuspal movement and microleakage in premolar teeth restored with a packable composite cured in bulk or in increments *Journal of Dentistry* **31(6)** 437-444.
9. Lee MR, Cho BH, Son HH, Um CM, & Lee IB (2007) Influence of cavity dimension and restoration methods on the cusp deflection of premolars in composite restoration *Dental Materials* **23(3)** 288-295.
10. Park JK, Chang JH, Ferracane JL, & Lee IB (2008) How should composite be layered to reduce shrinkage stress: Incremental or bulk filling? *Dental Materials* **24(11)** 1501-1505.
11. Bouillaguet S, Gamba J, Forchelet J, Krejci I, & Wataha J (2006) Dynamics of composite polymerization mediates the development of cuspal strain *Dental Materials* **22(10)** 896-902.
12. 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.
13. Meredith N, & Setchell DJ (1997) In vitro measurement of cuspal strain and displacement in composite restored teeth *Journal of Dentistry* **25(3)** 331-337.
14. Fleming G, Hall D, Shortall A, & Burke F (2005) Cuspal movement and microleakage in premolar teeth restored with posterior filling materials of varying reported volumetric shrinkage values *Journal of Dentistry* **33(2)** 139-146.
15. Palin WM, Fleming GJ, Nathwani H, Trevor Burke F, & Randall RC (2005) In vitro cuspal deflection and microleakage of maxillary premolars restored with novel low-shrink dental composites *Dental Materials* **21(4)** 324-335.
16. Condon JR, & Ferracane JL (2000) Assessing the effect of composite formulation on polymerization stress *Journal of the American Dental Association* **131(4)** 497-503.
17. Alster D, Feilzer AJ, de Gee AJ, & Davidson CL (1997) Polymerization contraction stress in thin resin composite layers as a function of layer thickness *Dental Materials* **13(3)** 146-150.
18. Braga RR, Hilton TJ, & Ferracane JL (2003) Contraction stress of flowable composite materials and their efficacy as stress-relieving layers *Journal of the American Dental Association* **134(6)** 721-728.
19. Bouschlicher MR, Vargas MA, & Boyer DB (1997) Effect of composite type, light intensity, configuration factor and laser polymerization on polymerization contraction forces *American Journal of Dentistry* **10(2)** 88-96.
20. Miguel AD, & De la Macorra J (2001) A predictive formula of the contraction stress in restorative and luting materials attending to free and adhered surfaces, volume and deformation *Dental Materials* **17(3)** 241-246.
21. Lee SH, Chang JH, Ferracane JL, & Lee IB (2007) Influence of instrument compliance and specimen thickness on the polymerization shrinkage stress measurement of light-cured composites *Dental Materials* **23(9)** 1093-1100.
22. Ilie N, & Hickel R (2011) Investigations on a methacrylate-based flowable composite based on the SDR™ technology *Dental Materials* **27(4)** 348-355.
23. Sabbagh J, Vreven J, & Leloup G (2002) Dynamic and static moduli of elasticity of resin-based materials *Dental Materials* **18(1)** 64-71.
24. 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.

25. Lee IB, Cho BH, Son HH, Um CM, & Lim BS (2006) The effect of consistency, specimen geometry and adhesion on the axial polymerization shrinkage measurement of light cured composites *Dental Materials* **22(11)** 1071-1079.

26. Fok AS (2013) Shrinkage stress development in dental composites—An analytical treatment *Dental Materials* **29(11)** 1108-1115.

27. Cramer N, Stansbury J, & Bowman C (2011) Recent advances and developments in composite dental restorative materials *Journal of Dental Research* **90(4)** 402-416.

28. Campodonico CE, Tantbirojn D, Olin PS, & Versluis A (2011) Cuspal deflection and depth of cure in resin-based composite restorations filled by using bulk, incremental and transtooth-illumination techniques *Journal of the American Dental Association* **142(10)** 1176-1182.

29. Min SH, Ferracane JL, & Lee IB (2010) Effect of shrinkage strain, modulus, and instrument compliance on polymerization shrinkage stress of light-cured composites during the initial curing stage *Dental Materials* **26(10)** 1024-1033.

©Operative Dentistry, 2016, **41-6,** e195-e208

Degradation Potential of Bulk Versus Incrementally Applied and Indirect Composites: Color, Microhardness, and Surface Deterioration

M El Gezawi • D Kaisarly • H Al-Saleh • A ArRejaie • F Al-Harbi • KH Kunzelmann

Clinical Relevance

Covering bulk fills with silorane-based composites might be a better practice for delaying surface degradation.

SUMMARY

This study investigated the color stability and microhardness of five composites exposed to four beverages with different pH values. Composite discs were produced (n=10); Filtek Z250 (3M ESPE) and Filtek P90 (3M ESPE) were applied in two layers (2 mm, 20 seconds), and Tetric N-Ceram Bulk Fill (TetricBF, Ivoclar Vivadent) and SonicFill (Kerr) were applied in bulk (4 mm) and then light cured (40 seconds, Ortholux-LED, 1600 mW/cm^2). Indirect composite Sinfony (3M ESPE) was applied in two layers (2 mm) and cured (Visio system, 3M ESPE). The specimens were polished and tested for color stability; ΔE was calculated using spectrophotometer readings. Vickers microhardness (50 g, dwell time=45 seconds) was assessed on the top and bottom surfaces at baseline, 40 days of storage, subsequent repolishing, and 60 days of immersion in distilled water (pH=7.0), Coca-Cola (pH=2.3), orange juice (pH=3.75), or anise (pH=8.5) using scanning electron microscopy (SEM). The materials had similar ΔE values (40 days, $p>0.05$), but TetricBF had a significantly greater ΔE than P90 or SF (40 days). The ΔE was less for P90

Moataz El Gezawi, BDS, MDSc, DDSc, Restorative Dental Sciences Department, College of Dentistry, University of Dammam, Dammam, Saudi Arabia

*Dalia Kaisarly, BDS, MDSc, PhD, Department of Conservative Dentistry and Periodontology, LMU Munich, Munich, Germany; Biomaterials Department, Faculty of Oral and Dental Medicine, Cairo University, Cairo, Egypt

Hamad Al-Saleh, Restorative Dental Sciences Department, College of Dentistry, University of Dammam, Dammam, Saudi Arabia

Aws ArRejaie, BDS, DScD, FRCD, Prosthetic Dental Science, College of Dentistry, King Saud University, Saudi Arabia

Fahad Al-Harbi, BDS, MSD, FACP, DScD, Department of Substitutive Dental Sciences, College of Dentistry, University of Dammam, Dammam, Saudi Arabia

Karl-Heinz Kunzelmann, PhD, Department of Conservative Dentistry and Periodontology, LMU Munich, Munich, Germany

*Corresponding author: Goethestr. 70, Munich 80336, Germany; e-mail: kaisarly@dent.med.uni-muenchen.de

DOI: 10.2341/15-195-L

and TetricBF than for Z250, SonicFill, and Sinfony (60 days). Repolishing and further immersion significantly affected the ΔE ($p<0.05$) except for P90. All composites had significantly different top vs bottom baseline microhardnesses. This was insignificant for the Z250/water, P90/orange juice (40 days), and Sinfony groups (40 and 60 days). Immersion produced variable time-dependent deterioration of microhardness in all groups. Multivariate repeated measures analysis of variance with *post hoc* Bonferroni tests were used to compare the results. ΔE and microhardness changes were significantly inversely correlated at 40 days, but this relationship was insignificant at 60 days (Pearson test). SEM showed degradation (40 days) that worsened (60 days). Bulk-fill composites differ regarding color-stability and top-to-bottom microhardness changes compared with those of other composites. P90 showed better surface degradation resistance. In conclusion, bulk-fill composites are not promising alternatives to incremental and indirect composites regarding biodegradation.

INTRODUCTION

Exposure to the oral environment, including a number of beverages, can induce different models of physical or chemical degradation in the structure of resin composite restorations, adversely affecting surface integrity and color stability.[1-9]

Many researchers have studied physical degradation evidenced by material loss or uptake as a result of sorption, extraction, dissolution, and mineralization, accompanied by physical changes, such as softening, stress cracking, and fatigue fracture. Less research has been conducted on the chemical degradation of resin composites. Bourbia[10] stated that chemical degradation might be oxidative, thermolytic, photolytic, radiolytic, or solvolytic in nature, of which solvolysis by hydrolytic degradation was the most frequently studied.

The chemistry, structural composition, and degree of conversion of composites play a major role in the material's mechanical, biological, and physical properties, including water sorption, hydrophilicity, and wear resistance, having consequences on clear surface integrity and color stability.[4] The degree of conversion of the resin matrix depends on the polymerization system and the curing condition. The layering technique for the placement of light-cured resin composites has been used to ensure uniform optimum properties at varying depths of the material. The depth of cure is a function of the material composition and the curing light parameters.[5,6,11,12] Measuring the microhardness at the top and bottom of the material is one of the methods used to assess the cure depth and hence the material performance.[2,4,6,11]

Color match and translucency, surface luster, roughness, and staining are among the criteria for clinically ranking and identifying whether repairs are possible vs whether the composite must be remade.[7,13] Extrinsic discoloration of the resin composite can be caused by the adsorption of colorants from food and beverages. Surface roughness due to wear and chemical degradation can adversely affect color stability and surface gloss, leading to extrinsic discoloration.[14] However, bulk discoloration is related to the resin matrix, filler particles, and photoinitiators.[1,2]

The extraoral high-intensity light curing of indirect composites is advocated in order to achieve a high degree of polymerization with optimum material properties, including a greater resistance to intrinsic discoloration.[1,15] Indirect resin composites are used to overcome the deficiencies of direct composites with different curing techniques and modifications in the resin matrix and inorganic filler content.[16]

Recently, bulk-fill composites have been introduced to improve the performance and shorten the clinical procedure of composite application. This group of materials allows for the placement of a 4-6-mm-thick increment relative to the conventional fill composite with a maximum increment thickness of 2 mm. These materials incorporate modifications in the resin matrix and photoinitiator chemistry, as well as filler particle technology, with a diversity of reports regarding their clinical performance, physical and mechanical properties, and degradation potential. In the study of bulk-fill composites, there is a high level of concern regarding the depth of the cure and the development of polymerization contraction stresses.[12,17]

The silorane resin matrix that polymerizes by ring opening has been employed to reduce polymerization contraction and contraction stresses without compromising the mechanical properties. Due to the hydrophobic siloxane backbone, silorane-based composites were reported to have lower water sorption than experimental ormocer- and methacrylate-based composites.[4-6,12]

The literature indicates that storage for 40 and 60 days is considered to be an extensive testing

period.[18,19] Because polishing is crucial in determining surface luster and color, aging by immersion in different beverages followed by repolishing has been considerably studied to relate the results to the influence of extrinsic vs intrinsic discoloration.[18,20] However, the resistance of the repolished surface to future degradation on further exposure to staining beverages present in the common diet has not been previously studied.

The objective of this study was to assess the influence of the immersion of composite discs in common beverages and repolishing followed by further immersion on the color stability, microhardness, and surface micromorphology. The effect was studied in bulk vs incremental dimethacrylate-based and silorane-based composites as well as in indirect polyfunctional-resin-matrix composites indicating their relative degradation potential. The null hypothesis was that bulk-fill composites behave similarly to incremental and indirect composites regarding their color, stability, and microhardness and that repolishing followed by further immersion does not improve color stability nor microhardness.

METHODS AND MATERIALS

Five composite materials were used for the fabrication of composite discs: Filtek Z250, coded as "Z250" (3M ESPE, St Paul, MN, USA), used as a control, and silorane-based composite Filtek P90, coded as "P90" (3M ESPE), were applied in two increments of 2 mm each.[21] Bulk-fill composites included Tetric N-Ceram Bulk Fill, coded as "TetricBF" (Ivoclar Vivadent, Schaan, Liechtenstein), and SonicFill (Kerr, Orange, CA, USA),[21] in addition to the indirect resin composite Sinfony (3M ESPE), as shown in Table 1. Custom-made molds of 7 mm in diameter and 4 mm in depth made from custom-made polyvinyl-siloxane impression material (Express STD Putty, 3M ESPE) were used in the preparation of 45 discs of each composite material with a total of 225 composite discs. An overview of the study outline is displayed in Figure 1.

For Z250 and P90, two subsequent horizontal increments of 2 mm each were inserted using a plastic instrument and light cured for 20 seconds using a high-intensity LED light-curing device of 1600 mW/cm^2 (Ortholux, 3M Unitek Orthodontic Products, Monrovia, CA, USA) with a wavelength of 430-480 nm and a peak at 455 ± 10 nm. A radiometer (Bluephase Meter, Ivoclar Vivadent) was used to read the power output of the curing unit immediately after each respective use. Light curing of the superficial layer was carried out after pressing the surface using a Mylar strip with a glass slide cover. The tip of the light guide was kept in contact with the glass slide (1.2-mm thickness) in order to maintain a constant distance from the specimen. The bulk-fill composites, TetricBF and SonicFill, were inserted in bulk (4 mm) and light cured for 40 seconds. Sinfony indirect composite was applied in two successive horizontal layers (2 mm), and each layer was preliminarily light cured for 15 seconds using an In-lab light curing device (Visio Alfa Light Curing Unit, 3M ESPE). A final 14-minute cure (Visio Beta Vario Light Curing Unit, 3M ESPE) was performed per the manufacturer's instructions.[15,16,22]

A V-shaped notch was cut at the edge of the top side of each sample with a diamond inverted cone to visually identify the top side. The specimens were stored in distilled water in darkness for 24 hours at 37°C.[17]

After 24 hours, the specimens were polished with 12.7-mm aluminum oxide discs (Sof-Lex, 3M ESPE) in order of decreasing grit size. Each size was used for 10 seconds at 10,000 rpm for coarse and medium discs and at 30,000 rpm for fine and superfine discs using an electric hand piece (K4, KaVo, Leutkirch, Germany).[23] The specimen was moistened with water before polishing with each disc. The polishing was performed by one investigator.

From each composite material, one specimen was randomly selected for scanning electron microscopy (SEM) at baseline. The remaining 44 specimens of each composite material were then divided into four groups (n=11) according to the immersion medium: distilled water (Al-Kawther Industries Co Ltd, Jeddah, Saudi Arabia), pH = 7.0; Coca-Cola (Coca-Cola Bottling Company of Saudi Arabia, Riyadh, Saudi Arabia), pH = 2.3; orange juice (Nada Fresh Orange Juice, Al-Khobar, Saudi Arabia), pH = 3.75; or anise (Aniseed, Alattar, Damascus, Syria), pH = 8.5. The anise was prepared by adding three teabags of anise to 100 mL of boiling water and soaking for five minutes, followed by cooling. The pH values of the immersion media were measured by a pH meter (Model 215, Denver Instrument, Bohemia, NY, USA) before immersion, and the media was replaced with fresh samples every 24 hours.

Before immersion in the respective media, 10 specimens of each group were tested for color and microhardness at baseline, while the eleventh specimen of each group was randomly selected only for SEM after 40 days of immersion. Color and

Table 1: *Materials Used in This Study and Their Composition*

Material	Classification	Composition	Batch Number	Manufacturer
Filtek Z250	Methacrylate-based microhybrid composite	Dimethacrylate (Bis-EMA)	N479326	3M ESPE
		Urethane dimethacrylate (UDMA)		
		Bisphenol A diglycidyl ether dimethacrylate (Bis-GMA)		
		Triethylene glycol dimethacrylate (TEGDMA)		
		Silica		
		Zirconia		
		Pigments		
		Camphorquinone initiator system		
Filtek P90	Silorane-based microhybrid composite	Silorane	N281586	3M ESPE
		Quartz		
		Yttrium fluoride		
		Pigments		
		Initiator system		
Tetric N-Ceram Bulk Fill	Nano-optimized resin composite (dimethacrylate)	Barium aluminum silicate glass filler of a mean particle size of 0.4-0.7 μm; ytterbium fluoride and mixed oxides of a mean particle size of 160- 200 nm; Ivocerin, a germanium-based initiator and a special shrinkage stress reliever	R65898	Ivoclar Vivadent
SonicFill	Sonic-activated resin composite (Bis-GMA, TEGDMA, Bis-EMA, SIMA)	83.5 wt% filler content	4252654	Kerr
Sinfony	Mixture of aliphatic and cycloaliphatic monomers with no TEGDMA or Bis-GMA	Borosilicate glass, quartz, silica (50-1 nm)	492345	3M ESPE
		45% by volume		
		Camphorquinone initiator		

Vickers microhardness measurements and SEM images were taken at baseline and 40 and 60 days after immersion in the media.

Color Stability

The color was measured by one investigator using a spectrophotometer (Color-Eye 7000A, Gretag Macbeth LLC, New Windsor, NY, USA) according to the Commission International de l'Eclairage L*a*b* color system against a white background. Color measurements were performed for each specimen twice, and the average was calculated and used for the analysis.[24] Measurements were performed at baseline (n=10 per study group), then the specimens were stored in the different immersion media for 40 days. After 40 days, each specimen was removed from the storage media, rinsed with distilled water, and dried with absorbent paper before the second color testing. Each specimen was repolished using the same polishing procedure previously described. Afterward, the specimens were stored in the respective media for 20 more days and before a final test.[18] The magnitude of the total color difference, which is represented by a single number (ΔE), was determined using the formula $\Delta E = [(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{1/2}$ after 40 and 60 days of immersion. Values of $\Delta E \geq 3.3$ were considered clinically unacceptable.[18]

Vickers Microhardness Testing

After the color measurements were taken, the tops and bottoms of the same specimens were measured for Vickers microhardness. The surfaces were dried and positioned under the indenter of a microhardness tester (Micromet 6040, Buehler, Lake Bluff, IL, USA), and a load of 50 g was applied through the indenter with a dwell time of 45 seconds.[24-26] After each measurement, the specimen was immersed in the respective medium in a separate and numbered vial. Microhardness values were measured at baseline, after 40 days before the repolishing procedure, and after 60 days of immersion. Color and microhardness measurements were recorded and analyzed using each respective specimen's own values at the different study levels.

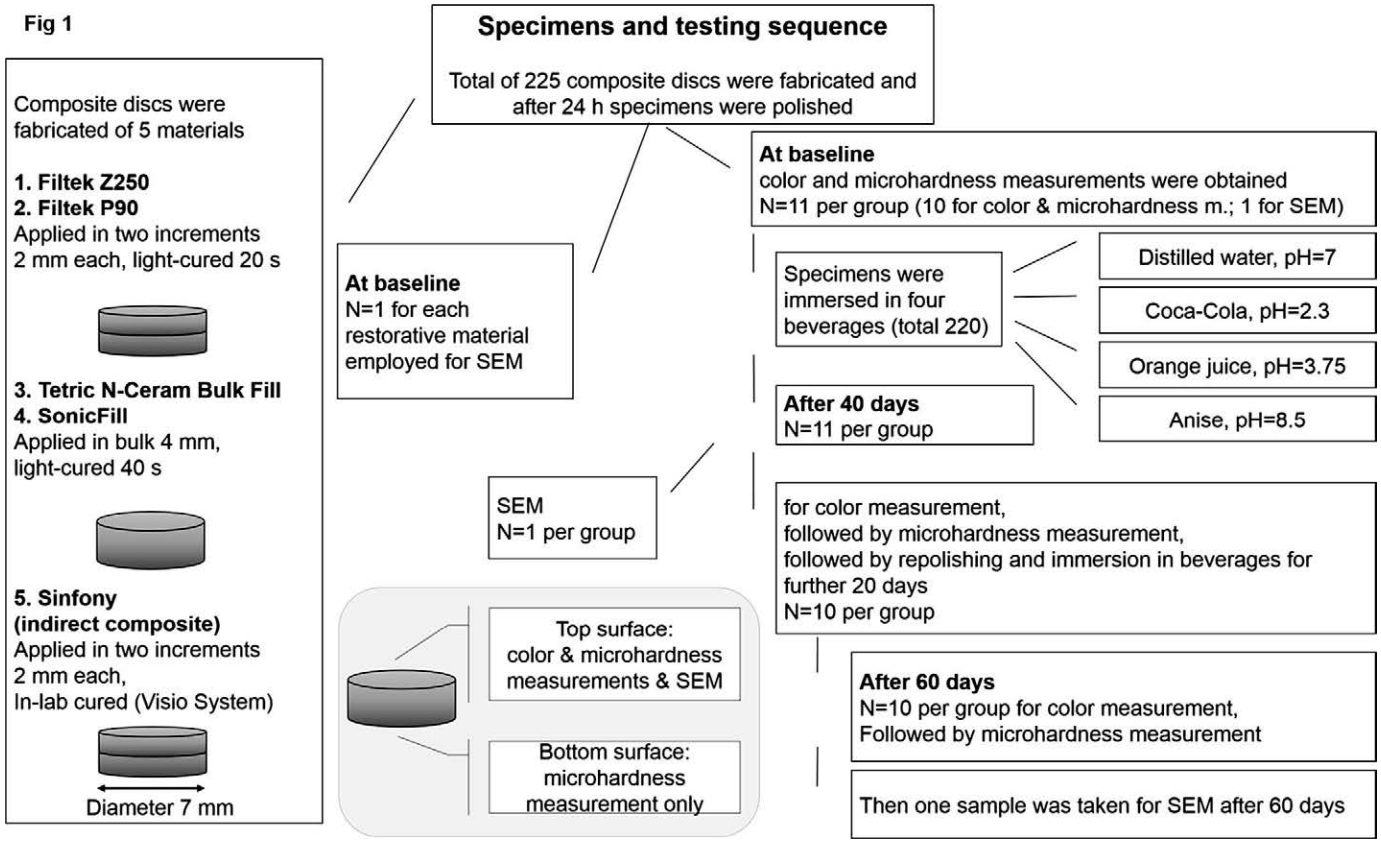


Figure 1. *Study overview.*

SEM

The specimens were examined using a scanning electron microscope (Inspect S50, FEI, Hillsboro, OR, USA) at 500× magnification to detect the surface microtopography of each material and changes due to storage in the immersion media.[27-29] At baseline, only one specimen of each restorative material was examined. After 40 days of immersion, the previously assigned specimen for SEM per each study group was examined and discarded and not used for microhardness or color-stability analysis. After 60 days of immersion, after the color and microhardness assessments, one specimen per group was randomly selected for SEM. Before scanning, the specimens were gold sputtered using the Q150R Rotary-Pumped Sputter Coater (Quorum Technologies, Ashford, UK).

Statistical Analysis

The ΔE color-stability results and the microhardness measurements were presented as mean and standard deviation values. Data were analyzed using the multivariate repeated measures analysis of variance (ANOVA) with the *post hoc* Bonferroni test for paired comparison of the materials and solutions in addition to the comparison of materials within each solution.[30-32] Moreover, the univariate ANOVA was used to compare the color-stability results ΔE of each material at 40 vs 60 days and for the comparison of top vs bottom microhardness values at each study interval.[11,32] The level of significance was 0.05 for all statistical analyses. The Pearson correlation test was performed to detect any correlation between color changes (ΔE) and microhardness changes (ΔD). Statistical data analysis was performed in IBM SPSS Statistics 22.0.

RESULTS

Color Stability

The mean values and standard deviations of the ΔE values and statistically significant differences between the materials within one beverage category and paired comparison of materials at 40 and 60 days are presented in Table 2. After 40 days of immersion in distilled water, Z250 was the only material to undergo clinically unacceptable changes

Table 2: *Color-Stability Results ΔE Mean Values and Standard Deviation (SD) of All Groups and Statistically Significant Differences Between Materials Within One Beverage Category and Paired Comparison*[a]

Solution	Material	Mean ± SD ΔE at 40 Days	Mean ± SD ΔE at 60 Days
Distilled water	Z250	3.9 ± 0.8 [a,d,1]	3.2 ± 0.8 [a,b,d,1]
	P90	2.6 ± 0.6 [b,c,d,1]	2.4 ± 0.9 [a,b,c,1]
	TetricBF	2.2 ± 0.6 [b,c,1]	1.7 ± 1.1 [b,c,1]
	SonicFill	3.1 ± 0.4 [a,b,d,1]	3.7 ± 0.2 [a,d,2]
	Sinfony	2.9 ± 0.7 [b,c,d,1]	2.6 ± 0.7 [a,b,c,1]
Coca-Cola	Z250	4.2 ± 2.6 [a,b,1]	4.5 ± 2.8 [a,b,1]
	P90	3.0 ± 0.6 [a,1]	2.6 ± 0.8 [a,1]
	TetricBF	6.0 ± 1.6 [b,1]	5.4 ± 1.9 [b,1]
	SonicFill	3.1 ± 0.7 [a,1]	3.4 ± 0.8 [a,b,1]
	Sinfony	5.3 ± 0.6 [b,1]	3.9 ± 0.9 [a,b,2]
Orange juice	Z250	3.8 ± 0.5 [a,b,1]	4.2 ± 0.5 [a,b,1]
	P90	4.1 ± 0.9 [a,b,c,1]	3.4 ± 1.6 [a,1]
	TetricBF	5.7 ± 1.6 [b,c,d,1]	5.0 ± 1.4 [a,b,1]
	SonicFill	4.2 ± 1.5 [a,b,c,1]	4.4 ± 1.2 [a,b,1]
	Sinfony	6.4 ± 1.3 [c,d,1]	5.9 ± 1.2 [b,1]
Anise	Z250	6.3 ± 2.3 [a,1]	9.9 ± 2.8 [a,2]
	P90	4.7 ± 1.1 [a,b,1]	4.3 ± 1.0 [b,d,1]
	TetricBF	4.8 ± 0.9 [a,b,1]	3.5 ± 1.4 [b,d,2]
	SonicFill	3.7 ± 1.2 [b,1]	7.0 ± 2.6 [c,d,2]
	Sinfony	5.5 ± 1.7 [a,b,1]	5.6 ± 1.2 [b,c,d,1]
Paired comparison of materials	Z250	4.5 ± 2.0 [a,1]	5.5 ± 3.3 [a,c,2]
	P90	3.6 ± 1.1 [b,1]	3.2 ± 1.3 [b,1]
	TetricBF	4.7 ± 2.0 [a,1]	3.9 ± 2.0 [b,c,2]
	SonicFill	3.5 ± 1.1 [b,1]	4.6 ± 2.0 [a,c,2]
	Sinfony	5.0 ± 1.7 [a,1]	4.5 ± 1.7 [a,c,2]

[a] *Significant differences between materials within one beverage category in each column are marked by different letters for the Bonferroni test at* $p < 0.05$*; significant differences between each material at 40 vs 60 days within one row are displayed by different numbers at* $p < 0.05$.

in color ($\Delta E > 3.3$). Immersion in Coca-Cola showed clinically unacceptable color changes for all composites except for P90 and SonicFill. When the test materials were stored in orange juice or anise, all showed clinically unacceptable color changes. After 60 days of immersion in distilled water, only SonicFill had an unacceptable color change, while storage in Coca-Cola resulted in acceptable color changes for P90 only. On storage in orange juice or anise, all materials showed clinically unacceptable color changes.

Paired comparisons showed variation of color changes in response to polishing and further immersion between materials with significant deterioration of Z250 and SonicFill and significant improvement of TetricBF and Sinfony. P90 was the only material with insignificant differences in color changes between 40 and 60 days and a clinically acceptable value of $\Delta E < 3.3$ after 60 days. The paired comparison of ΔE displayed that P90 had significantly less color change than TetricBF after 40 days and showed less color change than SonicFill after 60 days.

The results of color-stability paired comparisons based on beverages at 40 and 60 days are displayed in Table 3. Distilled water differed significantly from the other beverages ($p<0.05$), whereas the effect was insignificant between Coca-Cola, orange juice, and anise after 40 days of immersion. After 60 days, immersion in Coca-Cola and orange juice led to significantly greater color deterioration than water but significantly less than anise. Distilled water had the least deteriorating effect, while anise had the worst. For the color-stability results, there was a statistically significant interaction between the material and the solution ($p<0.001$) according to the repeated measures ANOVA.

Vickers Microhardness

Mean microhardness values and standard deviations of the top and bottom surfaces as well as paired

Table 3: *Results of Color-Stability Paired Comparisons Based on Beverages*[a]

Time Period	Beverage
At 40 days	Anise, orange juice, Coca-Cola > distilled water
At 60 days	Anise > orange juice, Coca-Cola > distilled water

[a] *> indicates clinical significance with the* post hoc *Bonferroni test at* $p<0.05$.

comparison at baseline and 40 and 60 days are presented in Table 4 in addition to the comparison of the top vs the bottom surface of each material at each study interval. There was a statistically significant deterioration in the top and bottom microhardness of all materials on aging regardless of the immersion medium. This was clear at 40 days with a significantly greater deterioration at 60 days. At baseline, the top surfaces showed a statistically significant higher microhardness than the bottom surfaces of all materials. This significant difference continued after 40 and 60 days in many groups, while in other groups it was only insignificant.

Comparisons of the top vs the bottom surfaces reveal that at baseline, the bottom surfaces of all specimens had statistically significant lower microhardness than the top surfaces ($p<0.05$). After 40 days of immersion, there was a deterioration in all of the top and bottom microhardness results. Significantly lower microhardness values of the bottom surfaces compared to the top surfaces were obtained in all materials except for Z250 in distilled water, P90 in orange juice, and Sinfony in all beverages. After repolishing and immersion for 20 more days, all materials demonstrated further microhardness deterioration of both surfaces. The microhardness of the bottom surfaces was significantly lower than that of the top surfaces in all groups except for Sinfony in all beverages.

The results of the microhardness paired comparisons based on materials are displayed in Table 5. Paired comparisons showed that at baseline, Z250 had the best statistically significant results compared to the other materials, while Sinfony had the worst. After 40 days, Z250 and P90 were not significantly different in microhardness, but both showed significantly greater microhardness compared to other materials. At 60 days, P90 showed significantly higher microhardness than other materials with least deterioration. According to the repeated measures ANOVA, there was a statistically significant interaction between the material and the solution ($p<0.001$) for the microhardness results.

SEM

The topographical analysis of specimens using the SEM images shows areas of degradation, material dislodgment, and erosion that varied in amount and extension between the materials. These degradation patterns were obvious after 40 days and progressed at 60 days of immersion in the different beverages, as shown in Figure 2. P90 underwent the least degradation.

Correlation Between (ΔE) and (ΔD)

After immersion in the beverages for 40 days, there was a strong inverse correlation between color change (ΔE) and microhardness change (ΔD) ($p=0.003$). However, after 60 days of immersion, there was a weak inverse correlation between ΔE and ΔD ($p=0.936$), as shown in Figure 3.

DISCUSSION

In vitro comparisons of the color stability and microhardness of materials have been used as a tool for predicting their degradability, especially when supported by microtopographic analysis at different levels of assessment.[1-4,11] Despite the ample amount of research indicating the clear effect of immersion in common beverages on the color-stability and surface properties of resin composite, few studies have compared the effect of variation in these properties relative to different composition and application techniques, especially for the new group of bulk-fill composites.[1-3,14,18]

Our study protocol lacked simulation of other factors, such as thermal cycling and the role of saliva and bacteria. The effect of repolishing and toothbrushing after immersion in different media on color stability was previously studied and was found to reduce staining and improve discoloration, as it removes the surface layer of pigments or absorbed stains of about 40 microns.[14,18,20,33] The effectiveness of repolishing in reducing stains and improving discoloration is related to the staining depth of the respective composite, its composition, degradability, and absorbability of water and stains.[33] Exposure of composites to beverages following repolishing is a normal consequence and addresses the reliability and longevity of improvement and can lead to a better understanding of composite degradation potential relative to the individual material composition.[24] Although immediate testing after repolishing is informative and might confirm previous findings on the effect of repolishing, it was felt that testing after further

Table 4: *Mean Values and Standard Deviation (SD) of the Vickers Microhardness of the Different Groups*[a]

Solution	Material	Mean ± SD Microhardness at Baseline			Mean ± SD Microhardness at 40 Days		
		Top	Bottom	Top vs Bottom	Top	Bottom	Top vs Bottom
Distilled water	Z250	109.8 ± 1.8[a]	89.9 ± 1.3[a]	+	81.4 ± 3.3[a]	78.3 ± 5.7[a]	–
	P90	97.2 ± 1.9[b]	83.8 ± 3.6[b]	+	89.3 ± 4.1[b]	83.1 ± 3.0[b]	+
	TetricBF	89.4 ± 2.5[c]	73.3 ± 3.7[c]	+	79.0 ± 3.3[a]	69.1 ± 2.2[c]	+
	SonicFill	96.1 ± 3.9[b]	84.3 ± 4.2[b]	+	88.7 ± 4.3[b]	81.6 ± 3.3[a,b]	+
	Sinfony	43.2 ± 5.1[d]	36.6 ± 4.9[d]	+	28.4 ± 3.9[c]	27.5 ± 4.7[d]	–
Coca-Cola	Z250	105.8 ± 2.5[f]	96.6 ± 1.9[f]	+	82.9 ± 2.2[f,h]	78.3 ± 4.7[f]	+
	P90	98.6 ± 3.0[g]	85.2 ± 5.2[g]	+	89.4 ± 4.6[g]	71.8 ± 3.6[g]	+
	TetricBF	87.1 ± 3.4[h]	74.2 ± 3.1[h]	+	83.0 ± 3.8[f,h]	71.8 ± 4.2[g]	+
	SonicFill	97.9 ± 4.5[g]	84.8 ± 3.9[g]	+	87.7 ± 3.4[f,g]	69.8 ± 5.0[g]	+
	Sinfony	44.8 ± 5.2[i]	38.8 ± 3.6[i]	+	27.6 ± 4.1[i]	24.6 ± 3.9[h]	–
Orange juice	Z250	112.1 ± 5.1[k]	89.6 ± 4.9[k]	+	94.3 ± 4.3[k]	75.6 ± 4.5[k]	+
	P90	95.1 ± 4.3[m]	88.9 ± 4.8[k]	+	85.7 ± 3.9[m]	85.9 ± 4.1[m]	–
	TetricBF	88.9 ± 2.7[n]	78.2 ± 3.3[m]	+	77.2 ± 3.5[n]	73.5 ± 2.3[k,n]	+
	SonicFill	94.8 ± 3.8[m]	81.5 ± 2.6[m]	+	89.3 ± 4.3[k,m]	69.8 ± 4.6[n]	+
	Sinfony	48.1 ± 3.6[p]	39.9 ± 2.9[n]	+	28.2 ± 4.0[p]	25.9 ± 4.0[p]	–
Anise	Z250	98.7 ± 4.2[q]	84.1 ± 4.4[q]	+	93.1 ± 4.1[q]	82.3 ± 4.9[q]	+
	P90	91.2 ± 5.2[r]	83.8 ± 4.7[q]	+	90.9 ± 5.2[q]	80.9 ± 3.7[q]	+
	TetricBF	87.9 ± 4.7[r]	74.3 ± 3.8[r]	+	82.1 ± 4.0[r]	71.3 ± 3.6[r]	+
	SonicFill	93.1 ± 3.4[q,r]	81.1 ± 4.4[q]	+	83.0 ± 3.5[r]	71.9 ± 4.2[r]	+
	Sinfony	47.9 ± 3.2[s]	39.7 ± 4.6[s]	+	26.1 ± 3.0[s]	24.7 ± 4.4[s]	–

[a] *Significant differences between materials within one beverage category in each column are marked by different letters for the Bonferroni test at* $p < 0.05$*; "+" denotes a statistically significant difference, while "–" stands for no difference at* $p < 0.05$ *in each material in one row at each study interval; significant differences between the study intervals within one material in one row of the paired comparison are displayed by different numbers for the Bonferroni test at* $p < 0.05$.

immersion following repolishing would provide a more descriptive image predicting the clinical reliability and material behavior.

Immersion times of 40 days followed by 20 more days were chosen according to Domingos and others,[34] who found that the most significant color changes take place at 60 days followed by 30 days of immersion. Moreover, the microhardness of materials immersed in coffee and Coca-Cola remained stable up to the seven-day measurement but decreased at the 30-day record and dropped more at the 60-day evaluation.[19]

The polishing technique employed in our study using Sof-Lex discs was found to provide the smoothest surface compared to other testing techniques.[20,35] Our light-curing procedure for bulk-fill composites for 40 seconds at high light intensity (1600 mW/cm^2) was chosen to obtain maximum color stability and greater microhardness.[10,36,37]

Table 5: *Results of Microhardness Paired Comparisons Based on Materials*[a]

Time Period	Material
At baseline	Z250 > P90, SonicFill > TetricBF > Sinfony
At 40 days	P90, Z250 > SonicFill > TetricBF > Sinfony
At 60 days	P90 > SonicFill, Z250 > TetricBF > Sinfony

[a] *> indicates clinical significance with the* post hoc *Bonferroni test at* ($p<0.05$).

Although restorations in the oral cavity are exposed to a variety of dietary colorants, as well as to a dynamic rather than a static exposure to beverages, most studies comprise exposing discs of the restorative material to one single coloring medium.[1] Our results showed variations in color stability among the test composites with varying levels of significance. Similar to our findings, Ren and others[1] observed that composites may have different affinities for diverse dietary colorants, possibly due to the difference in polarity between resin polymers and colorants. Composites of a resin matrix with similar polarity to yellow colorants may facilitate the absorption of the water, colored fluids, and yellow colorants into the organic phase of the material. Thus, modifying resin polymer polarities can improve resistance to staining.

Table 4: Extended.

Solution	Mean ± SD Microhardness at 60 Days			Mean ± SD Microhardness Paired Comparison (Top and Bottom)		
	Top	Bottom	Top vs Bottom	At Baseline	At 40 Days	At 60 Days
Distilled water	71.6 ± 4.5[a]	65.4 ± 3.4[a]	+	99.8 ± 10.3[1]	79.8 ± 4.8[2]	68.5 ± 5.0[3]
	84.5 ± 3.4[b]	78.9 ± 3.2[b]	+	90.5 ± 7.4[1]	86.6 ± 4.5[1]	81.7 ± 4.3[2]
	73.5 ± 3.7[a]	62.8 ± 4.2[a]	+	81.4 ± 8.8[1]	74.0 ± 5.7[2]	68.1 ± 6.7[3]
	81.5 ± 4.3[b]	74.7 ± 3.9[b]	+	90.2 ± 7.5[1]	85.1 ± 5.2[2]	78.1 ± 5.3[3]
	26.9 ± 4.0[c]	24.8 ± 2.9[c]	–	39.9 ± 5.9[1]	26.6 ± 4.1[2]	25.8 ± 3.6[2]
Coca-Cola	73.5 ± 4.8[f,g,i]	68.4 ± 5.5[f]	+	101.2 ± 5.2[1]	80.6 ± 4.3[2]	71.0 ± 5.7[3]
	79.3 ± 5.5[f,g]	67.1 ± 5.7[f,g]	+	91.9 ± 8.0[1]	80.6 ± 9.8[2]	73.2 ± 8.3[3]
	65.8 ± 5.3[h,i]	58.5 ± 5.2[h]	+	80.6 ± 7.3[1]	77.4 ± 6.9[1]	62.1 ± 6.3[2]
	67.2 ± 4.3[f,h,i]	61.3 ± 3.0[g,h]	+	91.4 ± 7.8[1]	78.8 ± 10.0[2]	64.3 ± 4.7[3]
	24.4 ± 4.2[j]	22.1 ± 3.4[i]	–	41.8 ± 4.8[1]	26.6 ± 4.2[2]	23.3 ± 3.9[2]
Orange juice	71.4 ± 3.6[k]	65.9 ± 3.9[k]	+	100.9 ± 12.5[1]	84.9 ± 10.5[2]	68.6 ± 4.6[3]
	83.2 ± 2.1[m]	74.4 ± 2.5[m]	+	92.0 ± 5.5[1]	85.8 ± 3.9[2]	78.8 ± 5.0[3]
	72.8 ± 2.9[k]	68.1 ± 3.3[k]	+	83.6 ± 6.2[1]	75.4 ± 3.4[2]	70.5 ± 3.9[3]
	80.8 ± 4.5[m]	66.8 ± 4.0[k]	+	88.2 ± 7.5[1]	79.6 ± 10.9[2]	73.8 ± 8.3[2]
	23.1 ± 3.3[n]	21.8 ± 3.1[n]	–	44.0 ± 5.3[1]	27.0 ± 4.1[2]	22.4 ± 3.2[3]
Anise	82.5 ± 5.0[q]	73.6 ± 5.4[q]	+	91.4 ± 8.5[1]	87.7 ± 7.0[1]	78.1 ± 6.8[2]
	84.1 ± 3.1[q]	70.7 ± 4.8[q,r]	+	87.5 ± 6.2[1]	85.9 ± 6.8[1]	77.4 ± 7.9[2]
	79.1 ± 4.2[q]	60.1 ± 3.9[s]	+	81.1 ± 8.1[1]	76.7 ± 6.6[1]	69.6 ± 10.5[2]
	79.6 ± 3.6[q]	67.1 ± 3.8[r]	+	87.1 ± 7.2[1]	77.5 ± 6.8[2]	73.4 ± 7.3[2]
	23.5 ± 4.9[r]	21.3 ± 5.1[t]	–	43.8 ± 5.7[1]	25.4 ± 3.7[2]	22.4 ± 5.0[2]

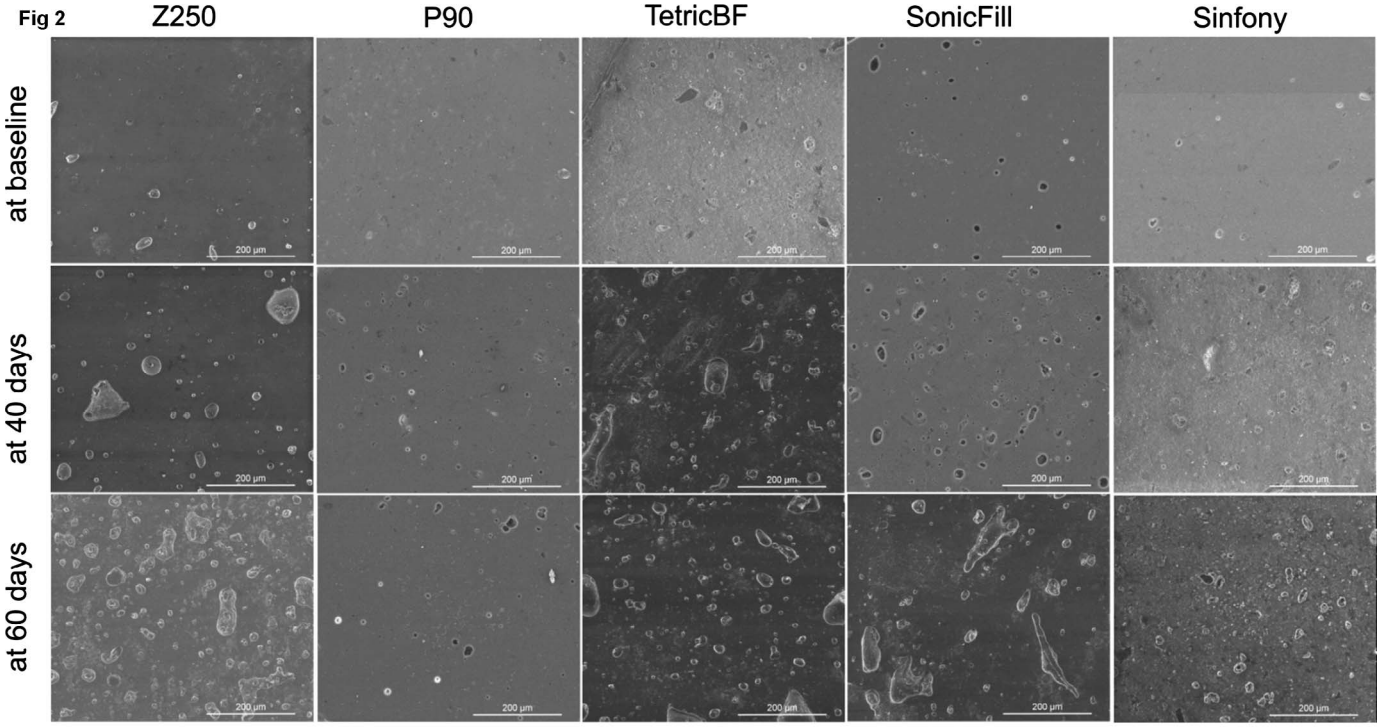
On immersion in water, most materials had clinically acceptable color changes ($\Delta E < 3.3$), which is similar to Schneider and others,[4] where 30 days of immersion in distilled water yielded clinically acceptable color changes and silorane- and ormocer-based materials did not produce higher color stability than the dimethacrylates. Additionally, silorane-based materials exhibited lower water solubility and lower hardness reductions than dimethacrylate-based composites, which is consistent with our microhardness results.

In this study, bulk-fill composites had similar color stability to Z250 after 40 days, which is similar to Tiba and others.[38] The better color stability of silorane-based P90 over TetricBF after 40 days and SonicFill after 60 days may confirm previous observations by Kang and others,[39] which may be related to the reduced hydrophilicity of silorane-based composites.[4]

Previous studies indicated that repolishing after staining reduces the ΔE value due to the removal of extrinsic stains.[18] However, our results indicate that repolishing followed by immersion had a varying effect on the color, possibly due to the variations in the organic matrix, polymer network density, filler loading, photoinitiator chemistry, and degree of conversion, which have a deciding influence on material properties, such as hydrophilicity, water sorption, adsorption, and dissolution potential and hence on the development of intrinsic or extrinsic staining.[1,4,36,39-43]

We analyzed the overall color change ΔE as an indication of degradation potential[44,45] since bulk-fill composites are more translucent and esthetically inferior than other composites.[43] However, analyzing the respective L*a*b values, which indicate the individual changes in lightness/darkness (L), red/green (a), and yellow/blue (b) scales, would have possibly provided more elaborative information about each composite. It might be more important to focus on the changes of surface reflectance of the material after staining than analyzing the color coordinate spaces to explain a range of staining effects on restorative materials.[46]

The effect of different immersion media compared to distilled water on color stability found in this study is similar to the findings of Kang and others[39] and Lepri and Palma-Dibb.[14] The significant deterioration of microhardness on the top and bottom surfaces after aging may be related to a gradual



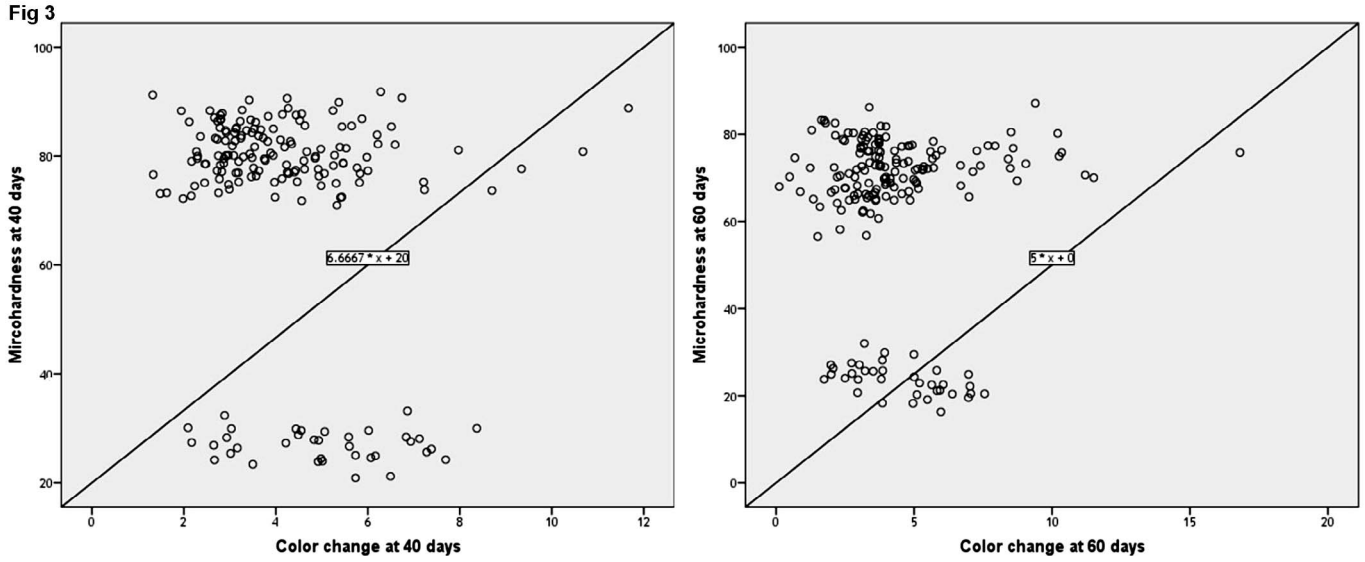


Figure 2. *SEM images (500×) of the various composites at baseline and 40 and 60 days of immersion in various beverages. The most prominent areas of degradation in the selected samples are presented.*
Figure 3. *The correlation between the ΔE and ΔD. After 40 days of immersion, there was a strong inverse correlation between color change (ΔE) and microhardness change (ΔD), while after 60 days of immersion, there was a weak inverse correlation between ΔE and ΔD.*

time-dependent degradation of the resin composite structure, which can be attributed to the hydrolysis of the ester groups in the resin matrix.[47] Further water sorption may decrease the durability of composites by expanding and plasticizing the organic matrix as well as by hydrolyzing the silane bonds between the fillers and the organic matrix.[18]

The presence of metallic ions such as zinc, barium, and strontium in fillers and radiopacifiers that are electropositive changes the silica network as they react with water, leading to charge balance changes. Therefore, hydroxyl ions increase and break the siloxane bonds inside the silica network, resulting in autocatalytic surface degradation, which might

explain the progression of surface softening with aging.[7,27,47,48]

The current study showed that at baseline, the bottom surface Vickers microhardness measures were significantly lower than those of the tops in all materials, similar to the findings of Jang and others[49] Our baseline results showed that the bottom microhardness exceeded 80% of that of the top surfaces in the SonicFill and TetricBF, which might be related to the use of the high-intensity LED curing unit. This is in line with the findings of Alshali and others.[50]

Bulk-fill materials incorporate modifications in the organic matrix and filler content, leading to higher translucency and reduced opacity with greater curing light transmission. Furthermore, they incorporate photoinitiators, such as Ivocerin in TetricBF, that yield more initiating radicals per molecule initiator than camphorquinone, leading to adequate cure depths at increased thicknesses.[37,45]

After 40 and 60 days, many groups maintained a significant difference between top and bottom microhardness values, indicating a consistent equivalent softening of the two surfaces. Others showed more deterioration in the top than the bottom surfaces. Our specimens were dropped to the floor of the beverage containers; thus, the top surface was more directly exposed to the beverage than the bottom. Nevertheless, the softening potential of a beverage is related to the compositional variations of the materials regarding the nature of the resin matrix and hydrophilicity, cross-linking density and the porosity of the network, the nature of the filler system, and the quality of the matrix/filler interface as well as the photoinitiators.[30,44,51,52] Furthermore, after light curing, dark polymerization, unpolymerized monomer conversion, and/or additional cross-linking reactions continue up to one week.[30,51,53] All of these factors vary between materials and influence the final respective material and specific surface degradation.[11,30,51,53] Therefore, it is recommended to suspend specimens rather than dropping them to the floor of the beverage container.[54]

Our results indicate that the initial proximity of the top surface to the curing light constitutes only one factor in the behavior of the material regarding surface microhardness on aging. Our findings support the multifactorial and complex nature of the degradation process.[4552] Although repolishing has removed a softened surface layer, yet further immersion produced a more detrimental effect on microhardness denoting the progressive nature of the degradation process extending deeper in the structure.[33,51,55]

SEM shows that the progressive deterioration in microhardness is related to the structural degradation of the material after immersion in different beverages, which progresses with aging.[27] This is similar to the findings of Martos and others,[48] who identified marked surface porosity and roughness in composite specimens following wet storage. The relatively superior degradation resistance pattern of P90 is similar to the findings of Schneider and others[4] and could be due to the lower hydrophilicity of the silorane-based resin matrix with a likely better resistance to hydrolytic degradation.[1,40] P90 seems to be more resistant to degradation, as observed by SEM and the microhardness as well as the color change results. Therefore, it is recommended to cover bulk-fill composites with silorane-based composite in order to delay surface degradation, which is in line with previous recommendations by Leprince and others[43] and Sunbul and others.[52]

The change in the Pearson correlation test between the ΔE and ΔD from strongly correlated at 40 days to weakly correlated at 60 days may indicate that repolishing followed by further immersion influences this correlation. This is in partial harmony with the findings of Soares-Geraldo and others,[24] who reported a significant positive correlation between the ΔE and ΔD and showed that not all color alterations were associated with surface degradation.

The findings of this study partially confirmed the null hypothesis that bulk-fill composites behave similarly to incremental and indirect composites regarding the effect of immersion in common beverages and repolishing followed by further immersion on color stability as well as microhardness. Future investigations should be directed toward combining oral environmental factors in addition to considering the depth of the individual influences of organic matrix, pigments, photoinitiator systems, and fillers in studying the degradability of this group of materials and the adverse consequences on their esthetic, physical, and mechanical qualities.[36]

CONCLUSIONS

Under the circumstances of the current investigation, it can be concluded that beverages of different pH values have a deteriorating effect on the color stability and microhardness of various formulations of resin composites. Immersion in beverages for 40 days produced variable deteriorations in color in

bulk-fill as well as incremental and indirect composites. Repolishing followed by immersion improved discoloration of TetricBF and Sinfony composites but deteriorated discoloration of Z250 and SonicFill composites. The effect of repolishing and further immersion is material dependent. Immersion in beverages produced progressive but variable patterns of deteriorations in the top and bottom microhardness of bulk-fill, incremental, and indirect resin composites. Degradability in terms of discoloration, deterioration in microhardness, and surface micromorphology is a complex multifactorial process influenced by material composition and application technique. Although color changes have a strong correlation with microhardness changes on immersion in beverages, repolishing followed by further immersion weakens such a correlation. P90 is more resistant to surface degradation than bulk-fill composites.

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 April 2016)

REFERENCES

1. Ren YF, Feng L, Serban D, & Malmstrom HS (2012) Effects of common beverage colorants on color stability of dental composite resins: The utility of a thermocycling stain challenge model in vitro *Journal of Dentistry* **40(Supplement 1)** e48-e56, 10.1016/j.jdent.2012.04.017.
2. Festuccia MS, Garcia Lda F, Cruvinel DR, & Pires-De-Souza Fde C (2012) Color stability, surface roughness and microhardness of composites submitted to mouthrinsing action *Journal of Applied Oral Science* **20(2)** 200-205.
3. Jain V, Platt JA, Moore K, Spohr AM & Borges GA (2013) Color stability, gloss, and surface roughness of indirect composite resins *Journal of Oral Science* **55(1)** 9-15.
4. Schneider LF, Cavalcante LM, Silikas N & Watts DC (2011) Degradation resistance of silorane, experimental ormocer and dimethacrylate resin-based dental composites *Journal of Oral Science* **53(4)** 413-419.
5. Shawkat ES (2009) *The Effect of the Oxygen Inhibition Layer on Interfacial Bond Strengths and Stain Resistance of Dental Resin Composites* University of Birmingham UK 109.
6. Leprince JG, Palin WM, Hadis MA, Devaux J, & Leloup G (2013) Progress in dimethacrylate-based dental composite technology and curing efficiency *Dental Materials* **29(2)** 139-156, 10.1016/j.dental.2012.11.005.
7. Pallesen U, van Dijken JW, Halken J, Hallonsten AL, & Hoigaard R (2013) Longevity of posterior resin composite restorations in permanent teeth in Public Dental Health Service: A prospective 8 years follow up *Journal of Dentistry* **41(4)** 297-306, 10.1016/j.jdent.2012.11.021.
8. Jackson RD, & Morgan M (2000) The new posterior resins and a simplified placement technique *Journal of the American Dental Association* **131(3)** 375-383.
9. Lynch CD, McConnell RJ, Hannigan A, & Wilson NH (2006) Teaching the use of resin composites in Canadian dental schools: How do current educational practices compare with North American trends? *Journal of the Canadian Dental Association* **72(4)** 321.
10. Bourbia M (2013) *Biodegradation of Dental Resin Composites and Adhesives by Streptococcus mutans: An In Vitro Study* University of Toronto 14.
11. Yanikoglu N, Duymus ZY, & Yilmaz B (2009) Effects of different solutions on the surface hardness of composite resin materials *Dental Materials* **28(3)** 344-351.
12. Cramer NB, Stansbury JW, & Bowman CN (2011) Recent advances and developments in composite dental restorative materials *Journal of Dental Research* **90(4)** 402-416, 10.1177/0022034510381263.
13. Hickel R, Peschke A, Tyas M, Mjor 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 Investigations* **14(4)** 349-366, 10.1007/s00784-010-0432-8.
14. Lepri CP, & Palma-Dibb RG (2012) Surface roughness and color change of a composite: Influence of beverages and brushing *Dental Materials* **31(4)** 689-696.
15. Nakazawa M (2009) Color stability of indirect composite materials polymerized with different polymerization systems *Journal of Oral Science* **51(2)** 267-273.
16. Cengiz S, Sarac S, & Ozcan M (2014) Effects of simulated gastric juice on color stability, surface roughness and microhardness of laboratory-processed composites *Dental Materials* **33(3)** 343-348.
17. Ilie N, Bucuta S & Draenert M (2013) Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance *Operative Dentistry* **38(6)** 618-625, 10.2341/12-395-l.
18. Mundim FM, Garcia Lda F, & Pires-de-Souza Fde C (2010) Effect of staining solutions and repolishing on color stability of direct composites *Journal of Applied Oral Science* **18(3)** 249-254.
19. Badra VV, Faraoni JJ, Ramos RP & Palma-Dibb RG (2005) Influence of different beverages on the microhardness and surface roughness of resin composites *Operative Dentistry* **30(2)** 213-219.
20. Gönülol N & Yılmaz F (2012) The effects of finishing and polishing techniques on surface roughness and color stability of nanocomposites *Journal of Dentistry* **40** e64-e70, 10.1016/j.jdent.2012.07.005.
21. Garoushi S, Sailynoja E, Vallittu PK & Lassila L (2013) Physical properties and depth of cure of a new short fiber reinforced composite *Dental Materials* **29(8)** 835-841, 10.1016/j.dental.2013.04.016.
22. Murakami M (2009) Surface properties of an indirect composite polymerized with five laboratory light poly-

merization systems *Journal of Oral Science* **51(2)** 215-221.

23. Güler AU, Duran I, Yücel AÇ, & Özkan P (2011) Effects of air-polishing powders on color stability of composite resins *Journal of Applied Oral Science* **19(5)** 505-510, 10.1590/S1678-77572011005000021.
24. Soares-Geraldo D, Scaramucci T, Steagall W Jr, Braga SR, & Sobral MA (2011) Interaction between staining and degradation of a composite resin in contact with colored foods *Brazilian Oral Research* **25(4)** 369-375.
25. Thomé T, Steagall Jr. W, Tachibana A, Braga SRM, & Turbino ML (2007) Influence of the distance of the curing light source and composite shade on hardness of two composites *Journal of Applied Oral Science* **15** 486-491.
26. Lodhi TA (2006) *Surface Hardness of Different Shades and Types of Resin Composite Cured With a High Power LED Light Curing Unit* University of the Western Cape 73.
27. Svizero Nda R, de Goes AR, Bueno Tde L, Di Hipolito V, Wang L, & D'Alpino PH (2014) Micro-sized erosions in a nanofilled composite after repeated acidic beverage exposures: Consequences of clusters dislodgments *Journal of Applied Oral Science* **22(5)** 373-381.
28. De Oliveira AL, Botta AC, Campos JA, & Garcia PP (2014) Influence of light curing units and fluoride mouthrinse on morphological surface and color stability of a nanofilled composite resin *Microscopy Research and Technique* **77(11)** 941-946, 10.1002/jemt.22421.
29. Hasanain F (2012) *An Experimental Fibre-Reinforced Dental Resin Composite* Newcastle University 262.
30. Erdemir U, Yildiz E, Eren MM, & Ozel S (2013) Surface hardness evaluation of different composite resin materials: Influence of sports and energy drinks immersion after a short-term period *Journal of Applied Oral Science* **21(2)** 124-131.
31. Ergucu Z, Turkun LS, & Aladag A (2008) Color stability of nanocomposites polished with one-step systems *Operative Dentistry* **33(4)** 413-420, 10.2341/07-107.
32. Yu H, Pan X, Lin Y, Li Q, Hussain M, & Wang Y (2009) Effects of carbamide peroxide on the staining susceptibility of tooth-colored restorative materials *Operative Dentistry* **34(1)** 72-82, 10.2341/08-42.
33. de Oliveira AL, Botta AC, Campos JA, & Garcia PP (2014) Effects of immersion media and repolishing on color stability and superficial morphology of nanofilled composite resin *Microscopy and Microanalysis* **20(4)** 1234-1239, 10.1017/s1431927614001299.
34. Domingos PA, Garcia PP, Oliveira AL, & Palma-Dibb RG (2011) Composite resin color stability: Influence of light sources and immersion media *Journal of Applied Oral Science* **19(3)** 204-211.
35. Barbosa SH, Zanata RL, Navarro MFdL, & Nunes OB (2005) Effect of different finishing and polishing techniques on the surface roughness of microfilled, hybrid and packable composite resins *Brazilian Dental Journal* **16(1)** 39-44.
36. Diamantopoulou S, Papazoglou E, Margaritis V, Lynch CD, & Kakaboura A (2013) Change of optical properties of contemporary resin composites after one week and one month water ageing *Journal of Dentistry* **41(Supplement 5)** e62-e69, 10.1016/j.jdent.2013.04.001.
37. Zorzin J, Maier E, Harre S, Fey T, Belli R, Lohbauer U, Petschelt A, & Taschner M (2015) Bulk-fill resin composites: Polymerization properties and extended light curing *Dental Materials* **31(3)** 293-301, 10.1016/j.dental.2014.12.010.
38. Tiba A, Zeller GG, Estrich CG, & Hong A (2013) A laboratory evaluation of bulk-fill versus traditional multiincrement–fill resin-based composites *Journal of the American Dental Association* **144(10)** 1182-1183, 10.14219/jada.archive.2013.0040.
39. Kang A, Son SA, Hur B, Kwon YH, Ro JH, & Park JK (2012) The color stability of silorane- and methacrylate-based resin composites *Dental Materials* **31(5)** 879-884.
40. Albuquerque PP, Moreira AD, Moraes RR, Cavalcante LM, & Schneider LF (2013) Color stability, conversion, water sorption and solubility of dental composites formulated with different photoinitiator systems *Journal of Dentistry* **41(Supplement 3)** e67-e72, 10.1016/j.jdent.2012.11.020.
41. Ardu S, Duc O, Bella E, & Krejci I (2016) Color stability of recent composite resins *Odontology* 1-7, 10.1007/s10266-016-0234-9.
42. Gregor L, Krejci I, Di Bella E, Feilzer AJ, & Ardu S (2015) Silorane, ormocer, methacrylate and compomer long-term staining susceptibility using DeltaE and DeltaE colour-difference formulas *Odontology* 1-5, 10.1007/s10266-015-0212-7.
43. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, & Leloup G (2014) Physico-mechanical characteristics of commercially available bulk-fill composites *Journal of Dentistry* **42(8)** 993-1000, 10.1016/j.jdent.2014.05.009.
44. Sabatini C (2015) Color stability behavior of methacrylate-based resin composites polymerized with light-emitting diodes and quartz-tungsten-halogen *Operative Dentistry* **40(3)** 271-281, 10.2341/14-080-l.
45. Bucuta S, & Ilie N (2014) Light transmittance and micromechanical properties of bulk fill vs. conventional resin based composites *Clinical Oral Investigations* **18(8)** 1991-2000, 10.1007/s00784-013-1177-y.
46. Manojlovic D, Lenhardt L, Milicevic B, Antonov M, Miletic V, & Dramicanin MD (2015) Evaluation of staining-dependent colour changes in resin composites using principal component analysis *Scientific Reports* **5** 14638, 10.1038/srep14638.
47. Soderholm KJ, Zigan M, Ragan M, Fischlschweiger W, & Bergman M (1984) Hydrolytic degradation of dental composites *Journal of Dental Research* **63(10)** 1248-1254.
48. Martos J, Osinaga PWR, Oliveira Ed, & Castro LASd (2003) Hydrolytic degradation of composite resins: Effects on the microhardness *Materials Research* **6(4)** 599-604.
49. Jang JH, Park SH, & Hwang IN (2015) Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin *Operative Dentistry* **40(2)** 172-180.

50. Alshali RZ, Salim NA, Satterthwaite JD, & Silikas N (2015) Post-irradiation hardness development, chemical softening, and thermal stability of bulk-fill and conventional resin-composites *Journal of Dentistry* **43(2)** 209-218, 10.1016/j.jdent.2014.12.004.

51. Moraes RR, Marimon JLM, Schneider LF, Brandt WC, Correr Sobrinho L, & Bueno M (2007) Hydrolytic stability of dental composites: One-year aging effect on surface roughness and surfce/subsurface hardness *Revista de Odontologia da UNESP* **36(4)** 383-387.

52. Sunbul HA, Silikas N, & Watts DC (2015) Resin-based composites show similar kinetic profiles for dimensional change and recovery with solvent storage *Dental Materials* **31(10)** e201-e217, 10.1016/j.dental.2015.06.003.

53. Watts DC, McNaughton V, & Grant AA (1986) The development of surface hardness in visible light-cured posterior composites *Journal of Dentistry* **14(4)** 169-174.

54. Abd Elhamid M, & Mosallam R (2010) Effect of bleaching versus repolishing on colour and surface topography of stained resin composite *Australian Dental Journal* **55(4)** 390-398, 10.1111/j.1834-7819.2010.01259.x.

55. Ozcan S, Yikilgan I, Uctasli MB, Bala O, & Kurklu ZG (2013) Comparison of time-dependent changes in the surface hardness of different composite resins *European Journal of Dentistry* **7(Supplement 1)** S20-S25, 10.4103/1305-7456.119059.

Vol 41/No 6 Nov/Dec 2016 Pages 567–682 OPERATIVE DENTISTRY ISSN 0361-7734