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113–224

Clinical Technique/Case Report

Clinical Research

Laboratory Research

Departments

Online Only Articles

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Clinical Technique/Case Report

Using Cross-Polarized Photography as a Guide for Selecting Resin Composite Shade

CA Villavicencio-Espinoza • MH Narimatsu • AY Furuse

Clinical Relevance

Cross-polarized photography is a simple technique that may improve the shade selection for the stratification of resin composites, especially when veneering discolored teeth.

SUMMARY

The restoration of single discolored maxillary anterior teeth is still a difficult task, as not only shape and surface characterization play an important role in the success of the treatment, but the propagation of light throughout the restorative material does as well. In some cases, small changes in morphology, color, and brightness will be noticeable. These factors are sometimes very tricky, and shade guides alone are difficult to use for color selection. This article proposes a protocol of employing cross-polarization imaging for improving the accuracy of the shade selection of resin composites. The step-by-step technique is presented for the restoration of a single discolored tooth.

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INTRODUCTION

Discolored teeth are classic problems that impair the esthetics of anterior teeth. When affecting multiple teeth, systemic etiologies are usually associated, and ceramic veneers are good treatment options.[1] On the other hand, a single discolored tooth is often related to root canal treatment,[2] and severely discolored teeth require the combination of walking bleach and external whitening treatments for the bleaching to be more effective.[3] However, a major problem is the recurrence of discoloration. In this case, the esthetic solution is frequently to cover the discolored tooth with dental crowns or veneers. While crowns should be precisely indicated to avoid unnecessary dental preparation,[4] more conservative treatments, such as indirect and direct veneers, have become alternatives for patients with esthetic problems with their anterior teeth.[5-7]

Direct veneer resin composites associated with adhesive systems have shown good mechanical properties and color stability as well as excellent esthetic results.[8] The current assortments of resin composites with different hues, chromas, and values make it possible to use the layering technique to

create restorations that are indistinguishable from the natural dentition.[9] However, one of the greatest challenges when employing resin composites for veneering single discolored teeth is the shade selection of natural teeth. Although some methods have been suggested in the literature, the success is considered subjective and highly dependent on the clinical experience of the operator.[10,11]

Different alternatives have been developed to facilitate the color selection, such as the use of spectrophotometers or colorimeters (devices for color measurement). However, these devices have some disadvantages related to high cost. Moreover, sometimes the device does not properly indicate the correct color, or the selected shade may not be compatible with the resin composite to be used.[12,13]

An interesting approach that can be very helpful for selecting the shade of resin composites is the use of polarizing filters associated with digital photography, known as cross-polarized photography.[14] This technique uses two linear polarizing filters: one in front of the lens and the other in front of the flash (light source). If the two filters are placed in the same plane of polarization, they are parallel and do not eliminate all reflections. However, when one of the filters is rotated 90 degrees to the other, providing crossed planes of polarization, near-to-zero light interference is produced, and the clinician can then observe the teeth in a new way, without reflections.[14-16] The advantage of this technique is that it allows a better understanding of the depth, details, characteristics, and transparencies of the dental structure. Additionally, the characteristics of the underlying dentin can be evaluated. In other words, this technique enables easier and straightforward appreciation of color.

The purpose of this case report is to describe a step-by-step clinical case in which the patient had a discolored maxillary anterior tooth restored with a resin composite veneer. The use of cross-polarized photography for choosing dentin, enamel, and incisal shade is described.

CLINICAL REPORT

A 30-year-old female patient was referred for treatment, complaining about the esthetic appearance of her maxillary right central incisor. The clinical evaluation showed a single discolored tooth and unsatisfactory old resin composite restorations (Figure 1). The main cause of the discoloration was attributed to endodontic treatment. No sensitivities to percussion were detected horizontally or vertically. However, radiographic evaluation revealed inadequate endodontic treatment with an alteration in the periapical area (Figure 2).

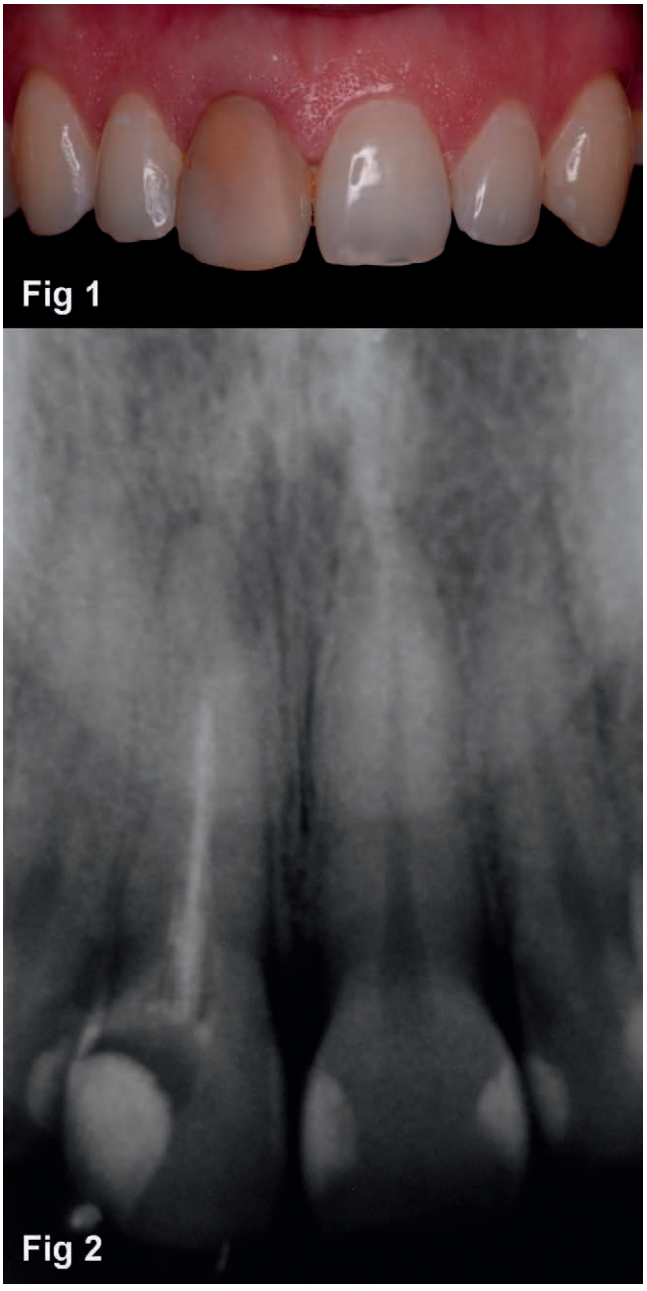

Figure 1. *Preoperative frontal view showing a discoloration on maxillary right central incisor.*
Figure 2. *Radiographic evaluation revealed an inadequate endodontic treatment with an alteration in the periapical area.*

The treatment plan included the following phases: 1) root canal retreatment, a nonvital tooth bleaching using a combination of walking bleach and in-office bleaching techniques; 2) cementation of a fiber-reinforced post and a resin composite buildup on the palatal surface; and 3) direct resin composite veneer.

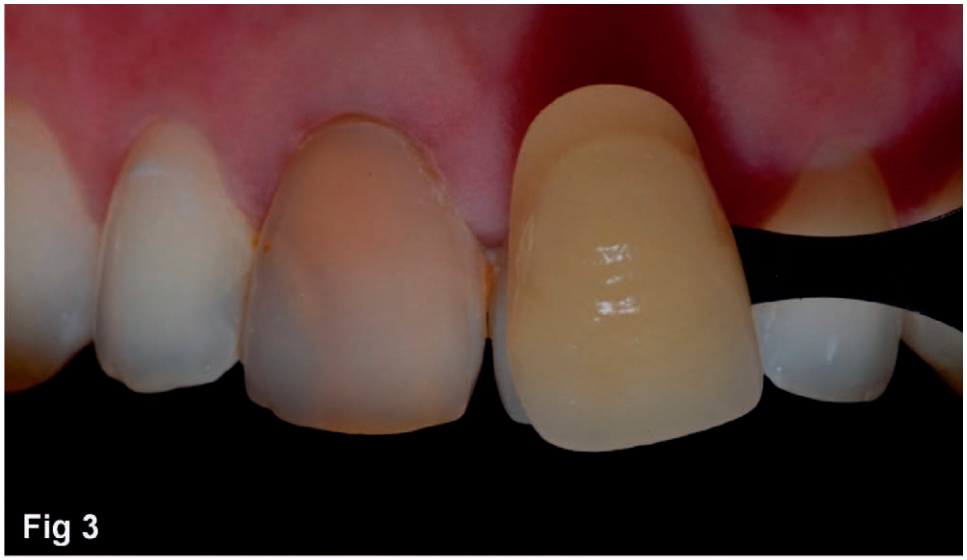

Figure 3. *Initial color of the discolored tooth evaluated with cross-polarization imaging. An A4 shade was observed with a Vita shade guide.*

Figure 4. *Different setups for obtaining cross-polarized photography. Left: a single flash with a polarized filter on the camera; right: a twin flash using two polarizing filters. Both options always had a polarized filter adapted in front of the lens.*

Phase 1

After root canal retreatment, the initial shade of the discolored tooth was selected using cross-polarized photography and a Vita Classic shade guide (Vita, Vita Zahnfabrik, Bad Säckingen, Germany). An A4 darkened tooth shade was observed (Figure 3). In contrast, the natural shade of the patient's teeth was B1. For cross-polarized photography, polarizing filters were adapted to both a Sigma Macro 105-mm lens attached to an SLR Nikon D5300 camera and the built-in flash in a way that the filters were rotated 90 degrees to one another to provide crossed planes of polarization. The camera was adjusted in manual mode to f/11, 1/100, and ISO 400. Figure 4 shows different setups for obtaining cross-polarized photography.

After rubber dam isolation, removal of coronal endodontic sealer until 1 mm apical to the cement–enamel junction, and resealing of the obturated canal with a glass ionomer cement (Vidrion R, SSWhite, Rio de Janeiro, RJ, Brazil), the walking bleach agent was prepared with a mixture of 100% carbamide peroxide and glycerin (Endoperox, Septodont, Saint-Maur-des-Fossés Cedex, France). This bleaching agent was applied to the pulp chamber, and a temporary restorative material was used (Coltosol F, Coltene, Vigodent SA, Rio de Janeiro, RJ, Brazil). The walking bleach product was replaced every seven days for three weeks. In the third week, external/internal in-office bleaching using 35% hydrogen peroxide (Lase Peroxide Flex, DMC, São Carlos, SP, Brazil) was applied only on the affected tooth.

Phase 2

Two weeks after bleaching, a post space was prepared, a fiber-reinforced post (White Post DCE,

Figure 5. *Final appearances after bleaching treatment.*
Figure 6. *Evaluation of the shade of the sound central incisor with cross-polarized photography. It is possible to note an A1 shade for the dentin.*
Figure 7. *Grayscale photography used for evaluation of value of the resin composites to be used in the stratification.*
Figure 8. *Cross-polarized photography used for evaluation of the shade of the resin composites to be used in the stratification.*

FGM, Joinville, SC, Brazil) was cemented using a self-adhesive resin cement (U200, 3M ESPE, St. Paul, MN, EUA), and a resin composite reconstruction of the palatal surface was made with B1 dentin and enamel shades (Opallis, FGM). This selection of shades was based on the color of the sound teeth and was made to avoid any possible decrease on brightness due to the thickness of the remaining facial dentin.

Phase 3

The final postbleaching shade of the maxillary right central incisor was evaluated, and A1 was selected. Cross-polarized photography and a Vita Classic shade guide were used as previously described. The conventional shade selection of the sound maxillary left central incisor using a Vita Classic shade guide showed a B1 shade (Figure 5). Wave-shaped white bands could be detected in the cervical and middle thirds, while an opalescence effect was observed at the incisal third. With cross-polarized photography, it was possible to observe that the dentin of the sound maxillary left central incisor had an A1 shade (Figure 6). The shade selection for the resin composite was made on the sound central incisor. Small portions of resin composites were applied and light activated on the tooth's facial surface. During the shade selection, grayscale and cross-polarized pictures were taken (Figures 7 and 8). The grayscale picture was taken to evaluate the value of the resin composite to be used. These photographs were also used as a reference in the stratification of the final restoration.

Afterward, modified rubber dam isolation and the silhouette technique were used for tooth preparation. A round #1014 diamond bur (KG Sorensen, Cotia, SP, Brazil) was used at high speed under water cooling to create cervical and proximal grooves. A tapered, round-ended #2135 diamond bur (KG Sorensen) was used for preparing vertical grooves and reducing the incisal edge. These grooves were used as depth cuts to facilitate the facial and incisal reduction of the veneer preparation. In order to facilitate the cervical finishing, a #000 retraction cord (Ultradent, South Jordan, UT, USA) was used for gingival displacement (Figure 9). The tooth was etched with 35% phosphoric acid for 15 seconds. After rinsing with water, an adhesive (Ambar, FGM) was applied and light activated according to the manufacturer's guidelines.

Initially, an opaque white flowable resin composite (Kolor Plus, Kerr Corp, Orange, CA, USA) was applied over the discolored dentin substrate to match the value of the patient's natural tooth (Figure 10). After this step, a thin layer of an opaque resin composite (OP, Opallis) was applied. Afterward, a small layer of dentin shade (A3, Opallis) was applied to the cervical and middle thirds, and the dentin shade (A2, Opallis) was placed in the middle and incisal areas (Figure 11). These two procedures were performed separately, and both increments were light activated for 40 seconds.

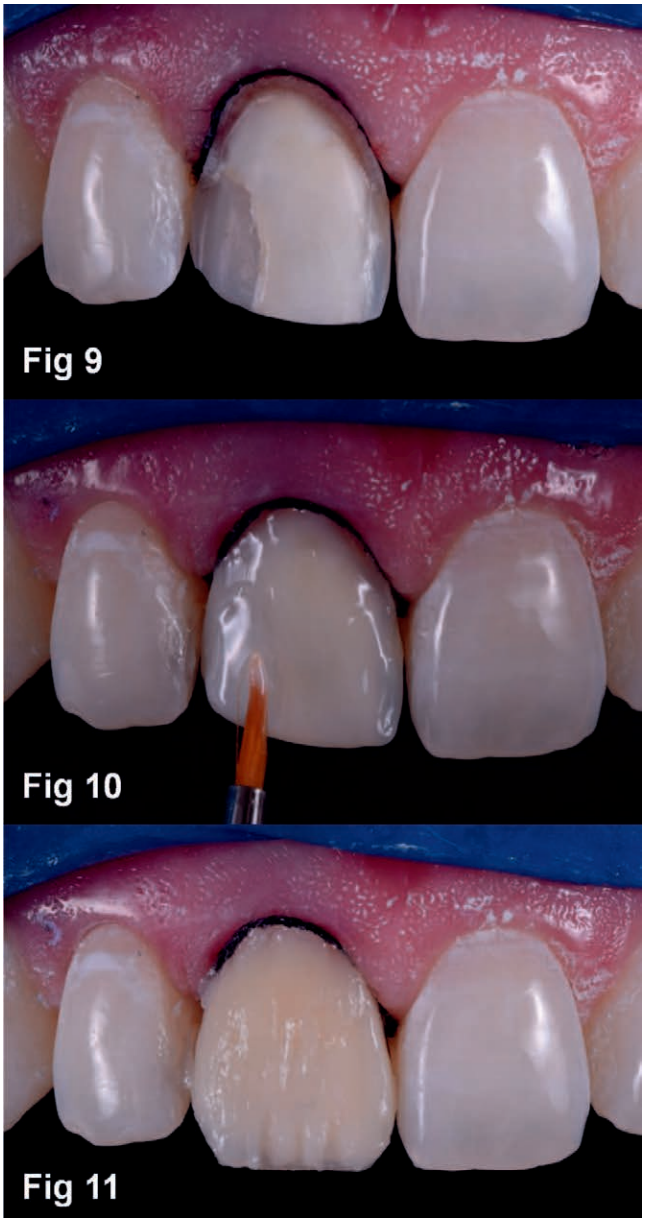

Figure 9. *Dental preparation finished.*
Figure 10. *An opaque flowable resin composite was applied over the discolored dentin substrate to match the value of the natural tooth.*
Figure 11. *A3 dentin shade applied in cervical and middle thirds and A2 dentin shade applied in middle and incisal areas.*

Before adding the enamel layer, the intrinsic characteristics and high-intensity hue of the adjacent teeth were accomplished with the aid of tints (Kolor Plus). In this stage, a white stain was used to reproduce wave-shaped bands in the cervical and middle thirds, and a blue stain was applied between dentin lobes to create an opalescence-like bluish effect at the incisal third. All tints were applied with a thin brush (Figure 12). The last layer of artificial enamel was made with B1 enamel shade (EB1, Opallis) for the cervical third and bleached enamel shade (E-Bleach, Opallis) for the middle and incisal thirds. The two portions of resin composite were applied from the cervical to incisal area with a flat spatula to completely cover the underlying resin layers in one step (Figure 13). The facial surface was light activated for 40 seconds.

After the restoration was completed, finishing was accomplished with a #12 surgical blade (Lamedid, Barueri, SP, Brazil) to remove resin composite excess in the cervical area. A pencil was used to highlight line angles of both central incisors, and a caliper was used to analyze light-reflecting areas (Figure 14). Sharp transitions between line angles were softened with a coarse finishing disc (TDV, Pomerode, SC, Brazil).

The restoration was polished with a sequence of polishing discs from coarse to superfine (TDV) and a silicone disc (TDV). The final polishing was accomplished with a felt disk and a 40-μm diamond-based paste (TDV). The final restoration can be observed in Figure 15.

DISCUSSION

The presented clinical situation reports an approach to a common problem when restoring maxillary anterior teeth and how to evaluate the inner characteristics of the dentin. While dentin has a role in the reflection and absorption of light, being responsible primarily for the shades of natural teeth and saturation, enamel is rich in minerals and behaves like a translucent glass-like object that allows light to pass through it.[17] This phenomenon results in light dispersion and scattering. Other determining factors of a successful restoration include the tooth shape, surface characterization, and propagation of light inside the restoration.[18] As demonstrated in the present case report, these last factors can be more easily observed and corrected using a pencil to highlight line angles of both central incisors and a caliper to analyze light-reflecting areas.

Shade selection of natural teeth is a complex process because it involves subjective factors that depend directly on the observer, light source, and reflection of light by the object. Moreover, the time employed for color observation, observer's experience, and type of shade guide, as well as the material's composition, are other subjective factors that critically influence the shade selection. In general, shade guides for use in dentistry follow Munsell's color parameters,[19] in which three dimen-

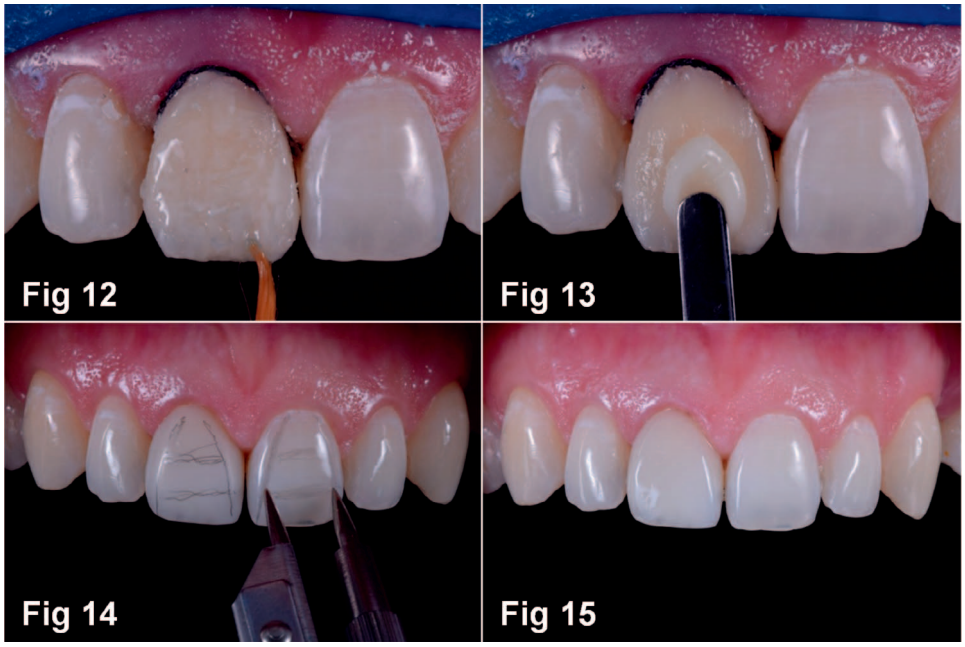

Figure 12. *Color characteristics were established using white stain in cervical and middle areas to reproduce wave-shaped bands. A blue stain was applied between dentin lobes to obtain an opalescence effect at the incisal third.*
Figure 13. *Application of the enamel layer.*
Figure 14. *A pencil was used to highlight the line angles, and a caliper was used to check the symmetry between central incisors.*
Figure 15. *Two-month follow-up showing the final result of the restored tooth.*

sions are defined: hue (basic color), chroma (saturation), and value (brightness). In the case of direct restorations, employing resin composite shade guides, although recommended for the initial evaluation of color, may be confusing and, despite several attempts to define a protocol for use, still rely on the clinical experience of the observer and the in-office illumination. Moreover, due to the optical phenomena occurring on natural teeth, it is necessary to select colors for dentin, enamel, and incisal translucent areas to obtain a better layering technique.

Over the past decade, there has been an increasing interest in illumination techniques for use in dental photography. Digital pictures can help minimize errors in clinical practice, especially during the shade matching of natural teeth. The protocol based on cross polarization, eliminating the superficial enamel light reflection, allows unobstructed visualization of surface and subsurface enamel characteristics. This technique enables easy and more accurate selection of the hue and chroma of the dentin.[20] Moreover, as demonstrated, the use of polarizing filters associated with digital photography is a simple and straightforward method for better understanding the color of natural anterior teeth.[21] For a better use of this resource, the authors recommend three pictures: the maxillary anterior teeth with a black background, the maxillary anterior teeth with a black background and the shade guide, and the maxillary anterior teeth with a black background and small portions of cured resin composites. The first picture of this protocol enables the evaluation of the characteristics of both enamel and dentin, providing an understanding and estimation of how opaque each structure is. The second picture is taken after the usual color selection, employing shade guides, and serves to compare the general color selected for the entire tooth with the estimated color of the dentin. The third picture is taken after selection of the specific resin composites for each layer and serves to check if the selection is appropriate. It should be noted that not every resin composite system is equal, and, despite the shade specification made by each manufacturer, the color itself may vary. This means that the A1 dentin shade specified by one manufacturer may not correspond to the A1 dentin shade specified by another. Moreover, the shade selection using only shade guides may be misleading if custom-made shade guides produced with the actual resin composite are not used. For this reason, this third additional step will help the selection of the correct shade of a specific system. In the present case report, in the evaluation of the color of the sound central incisor with cross-polarized photography (Figure 6), the A1 shade was selected with a shade guide originally designed for ceramics, which is not specific for the resin composite system used in the restoration. For the specific resin composite system employed, after the application of the two opaquers (ie, a first layer of a white opaque flowable resin composite and a second layer of a conventional opaque OP resin composite), the A1 dentin shade would have an artificially bright effect instead of reaching the desired color. Thus, A3 and

A2 dentin shades were used to reproduce the color variation of the dentin from cervical to incisal thirds.

Another interesting use of digital photography to help the clinician improve predictability when restoring discolored teeth is the provisional application of a thin layer of an opaque resin composite after dental preparation followed by the evaluation of the change in brightness using grayscale photographs.[22] This technique can help reproduce the brightness (value) of the adjacent teeth and could be added to the photographic protocol proposed in the present case report. However, one should take care not to overdehydrate dental tissues during restorative procedures, as brightness is elevated.

When taking digital photographs, the reflection of the flash on the enamel of the adjacent teeth may increase the brightness, especially when ring flashes are used. For this reason, twin flashes are better for taking pictures of anterior teeth. Bouncers adapted to twin flashes are other interesting accessories that can modify tooth chromaticity[21] as well as perceivable changes of brightness on the tooth. For this reason, their use is recommended, except for in cross-polarized photography.

As suggested in the present case report, the use of cross-polarized photography to assess the chromaticity and brightness (value) should be done as soon as possible (ie, in a short time) during the restorative procedure due to the natural dehydration of teeth. In addition, photographs with polarization modalities can improve the evaluation of the level of brightness of resin composites and teeth when taken in gray scale because they remove the interferences of both environmental light and photographic equipment.

CONCLUSIONS

The evaluation of hue, chroma, value, and color characteristics exhibited by natural dentition using cross polarization may help achieve more predictable outcomes during the stratification technique with resin composites.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Bauru School of Dentistry, University of São Paulo, Brazil.

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.

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Conservative Approach for Treatment of Maxillary Lateral Incisor Agenesis With the Deciduous Tooth Retained: 18-Month Follow-Up

CECM Lameira • SR Mestrener • NIP Pini • FM Salomão
AA Pesqueira • TC Fagundes

Clinical Relevance

The treatment of younger patients with maxillary lateral incisor agenesis and contralateral "peg lateral" often requires a multidisciplinary approach to achieve functional and esthetic outcomes.

SUMMARY

This case describes a female patient with agenesis of the maxillary right lateral incisor, with her permanent canine in its position and the deciduous canine retained. Additionally, she presented with a maxillary left peg lateral incisor. To solve her functional and esthetic complaints, a multidisciplinary approach involving perio-restorative procedures was proposed. Periodontal surgeries were performed to align the gingival contour, and the restorative approach utilized ceramic veneers. At the 18-month clinical and radiographic follow-up, the treatment outcome was stable, with maintenance of the clinical results achieved and without any sign of deciduous tooth resorption.

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INTRODUCTION

Agenesis in anterior teeth can negatively compromise the smile.[1,2] Maxillary lateral incisor agenesis (MLIA) is the third most common type of dental agenesis,[3] and it is more prevalent in females and bilaterally.[4] However, when MLIA is unilateral, the contralateral incisor is usually peg-shaped.[5] Both conditions may prejudice and influence the smile and facial attractiveness.[6,7]

This clinical situation often requires a multidisciplinary approach to provide a functional and esthetic resolution.[8] Orthodontics, periodontics, prosthodontics, and restorative dentistry are common fields that may be involved in all phases of treatment.[1,2,8] The main choice for MLIA patients is between orthodontic repositioning of the canine and recontouring it as a lateral incisor or opening/maintaining the space to insert an implant or prostheses.[1,8-10] According to a systematic review, there is no consensus on the best option for treating MLIA patients.[9] Regardless, the ideal treatment should be the most conservative option that satisfies the patient's functional and esthetic requirements,[8,10] taking into account aspects such as the patient's age, type of malocclusion, and tooth morphology.[8]

This case report presents and discusses an interdisciplinary management to reestablish function and esthetics in a younger patient with unilateral maxillary lateral incisor agenesis, a retained deciduous tooth, and a contralateral peg-lateral incisor. There is a lack of literature presenting similar treatment approaches for MLIA patients.

CASE REPORT

A 14-year-old female presented to the Undergraduate Clinic of Restorative Dentistry complaining about the appearance of her smile. The first clinical examination revealed that she had unilateral maxillary lateral incisor (No. 7) agenesis, with the permanent canine (6) in its position and the deciduous canine (C) in place of the permanent canine with satisfactory bone support and without mobility. Additionally, the maxillary left lateral incisor (10) was peg-shaped. However, her tooth alignment and occlusion were good. Radiographic and photographic documentation (Figure 1) were performed, and impressions of both arches were made using polyvinyl siloxane (Express XT putty light body; 3M ESPE, St Paul, MN, USA). Then, digital smile design was planned (Figure 2).

The possibility of solving the MLIA by using orthodontics was discussed with the patient and her guardian as the most conservative option, but they were resistant to it, wanting a more immediate treatment. The second option considered was maintenance of the deciduous teeth followed by periodontal procedures and indirect restorative treatment. Ceramic veneers were planned for the central incisors, the peg-lateral incisor, and the right permanent and deciduous canines. The decision for including the central incisors in the treatment was determined by the digital smile design, which clearly demonstrated that this would be necessary for a harmonic smile. The second alternative was chosen by the girl and her mother, who signed the consent form.

A diagnostic wax-up and mock-up were done, achieving the patient's expectations (Figure 3), followed by the periodontal surgeries. First, esthetic crown lengthening of the central incisors and

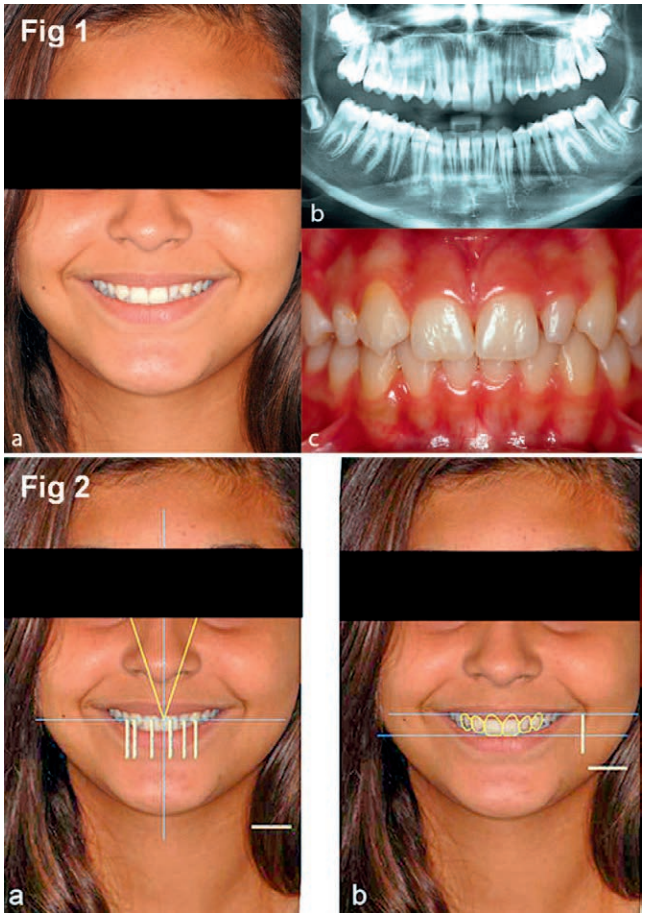

Figure 1. *Pretreatment aspect of the patient. (a) Facial, (b) radiographic, and (c) intraoral view showing the right permanent canine in the absent lateral incisor's position, the retained deciduous canine, and the maxillary left peg-lateral incisor.*
Figure 2. *Digital smile design protocol. (a) Determining the ideal horizontal plane and midline and transferring the measurements of the dental casts to the maxillary incisors and canines; (b) planning the final teeth outline and gingival contour.*

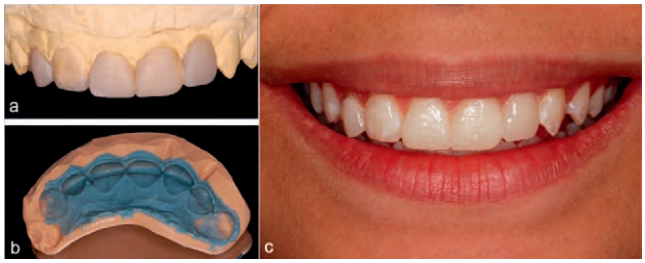

Figure 3. *Mock-up technique. (a) Diagnostic wax-up; (b) silicone matrix used to transfer the restorations previewed in the wax-up to the mouth; (c) mock-up allowing a preview of the result.*

deciduous canine and labial frenectomy were performed (Figure 4a,b). Second, the crowns of both permanent canines were repositioned coronally using connective tissue harvested from the palate to improve the gingival level. Last, a graft was placed in the recipient area and sutured to stabilize the external flap and maintain the graft position.

After 90 days (Figure 4c), the five teeth were prepared for ceramic veneers. The color selected for these veneers was B1 (Vita Classical Scale, Vita Zahnfabrik, Bad Säckingen, Germany). The preparation consisted of fine chamfering of the enamel on the facial surface, which was executed using a tapered-cylinder diamond tip (No. 4138; KG Sorensen, São Paulo, SP, Brazil), without preparing the proximal or incisal regions of any teeth. The cervical margin was at the level of the gingiva. An extra-fine diamond tip (No. 4138; KG Sorensen) and abrasive disks (Soft-Lex Pop-On, 3M ESPE) were used to refine the preparation. The right permanent canine required the most tooth preparation to reshape it into a lateral incisor. However, all preparations were considered conservative, with most of them restricted to the enamel.

The final impression of the teeth was obtained using retraction cord (Ultrapack, Ultradent Products, Indaiatuba, SP, Brazil) (Figure 5a), using a double-viscosity polyvinyl siloxane material (Express XT, 3M ESPE) (Figure 5b). Provisional restorations were fabricated for the right canine and left lateral incisor. Porcelain veneers were made of glass-infiltrated, pressed ceramics (Lithium-disilicate; IPS e.max Press, Ivoclar Vivadent, São Paulo, SP, Brazil).

The veneers were carefully verified using a try-in paste (Rely X Veneer; Ivoclar Vivadent) to verify marginal adaptation, alignment, shape, and color. Before beginning the luting procedures, we cleaned the teeth using pumice and a rubber cup. The veneers had their internal surfaces treated with 9.5% hydrofluoric acid (Condac Porcelana; FGM Products, Joinvile, SC, Brazil) for 20 seconds followed by application of a silane coupling agent (Monobond; Ivoclar Vivadent).

Then the anterior teeth were isolated using a rubber dam (Figure 6a). After conditioning with 37% phosphoric acid (Condac 37; FGM Products) for 30 seconds on the enamel and 15 seconds on the dentin (in the case of the right canine), rinsing and drying, applying a one-bottle bonding system (Single Bond; 3M ESPE), gently air-drying, and light-polymerizing for 20 seconds, each restoration was cemented individually (Figure 6b). A luting agent (Rely X Veneer shade B1/2; Ivoclar Vivadent) was applied to the internal surface of the veneer, which was positioned on the tooth. A slight polymerization of 5 seconds to stabilize the veneer was done, and excess luting material removed. Subsequently, the veneers were light cured from the facial and palatal sides for 40 seconds.

After placement of all veneers, the cervical margins were carefully checked and any excess cement was removed. Occlusion was also verified and some adjustment was performed to the decidu-

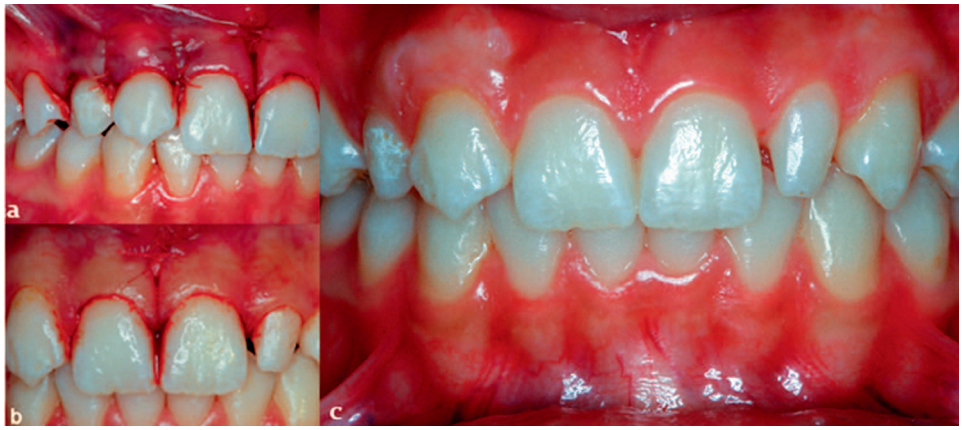

Figure 4. *Periodontal surgical procedure. (a) After correcting the gingival level with crown lengthening of the deciduous canine and repositioning it using palatal connective tissue harvested from the palate (the same procedure was performed on the left side); (b) after esthetic crown lengthening of the central incisors and labial frenectomy; (c) postoperative aspect 90 days postsurgery.*

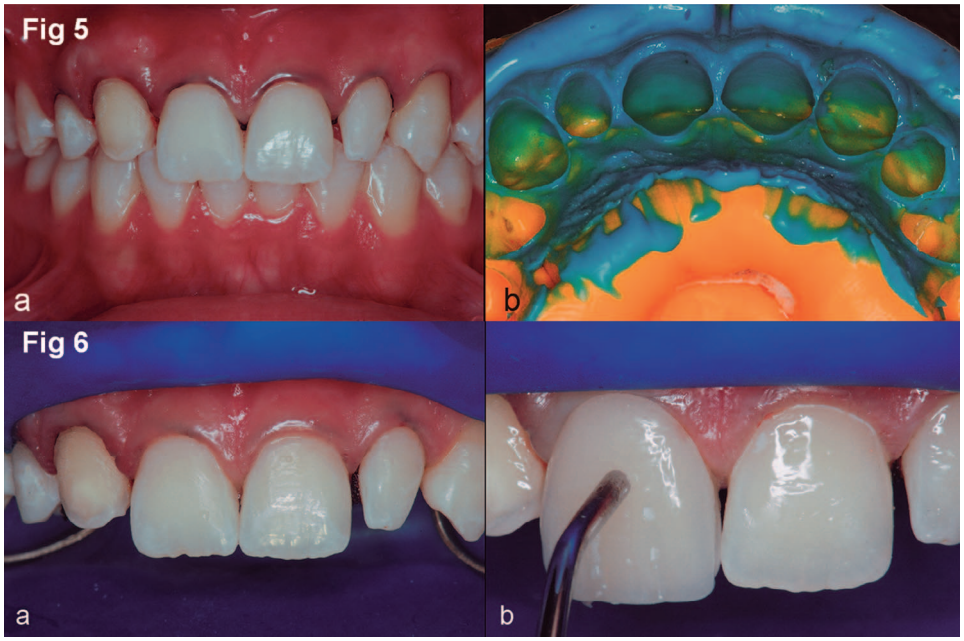

Figure 5. *Aspect of tooth preparation. (a) Placement of retraction cords for impression; (b) impression obtained using a double viscosity polyvinyl siloxane material.*
Figure 6. *Luting phase. (a) Isolation of the teeth using rubber dam and retraction cords; (b) placement of each veneer individually.*

ous canine to relive occlusal loading and to establish a group lateral disclusion on the right side. The final result can be seen in Figure 7a.

At the 6-month check-up (Figure 7b,c), maintenance of the immediate result—both biological and esthetic—were verified. At the 18-month follow-up, the same results and impressions were verified, but a new condition was noted: a slight staining on the veneer of the left central incisor. The patient admitted that she had been putting the pendant of her necklace in her mouth, precisely against this tooth, as can be seen in Figure 8. The surface of the veneer was polished with ceramic-specific rubber cups, and the patient was instructed to avoid this deleterious habit. Radiographic follow-up indicated stability of the bone around the root of the deciduous canine (Figure 9).

DISCUSSION

This case report presents a multidisciplinary approach employed to achieve the functional and esthetic rehabilitation of a young patient with MLIA. In her situation, the ideal conservative treatment would have been extraction of the deciduous canine, management of the area of agenesis with orthodontics, and placement of a provisional prosthesis for a future implant.[8,9,11] To minimize the need for provisional restorations, this treatment could be

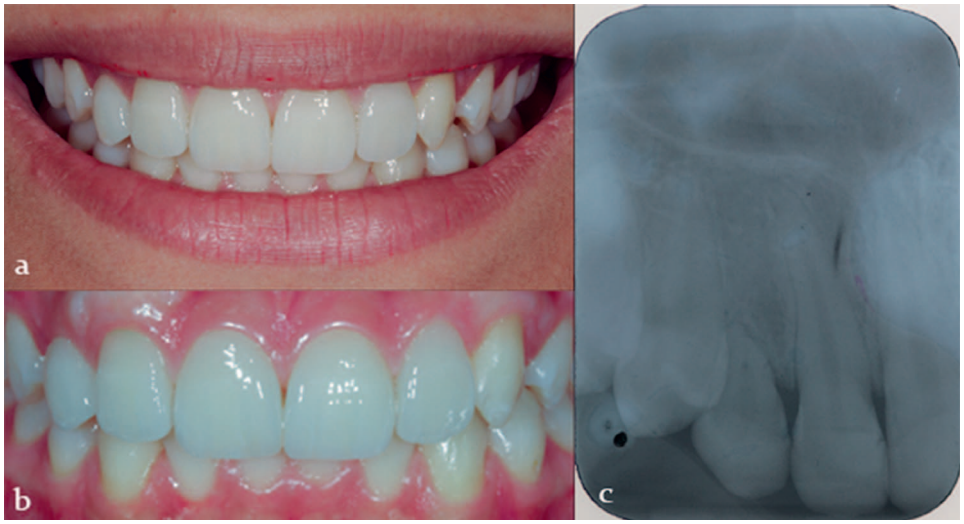

Figure 7. *(a) Posttreatment aspect of the patient. Clinical and radiographic follow-up after 6 months; (b) intraoral view; (c) periapical radiographic view.*

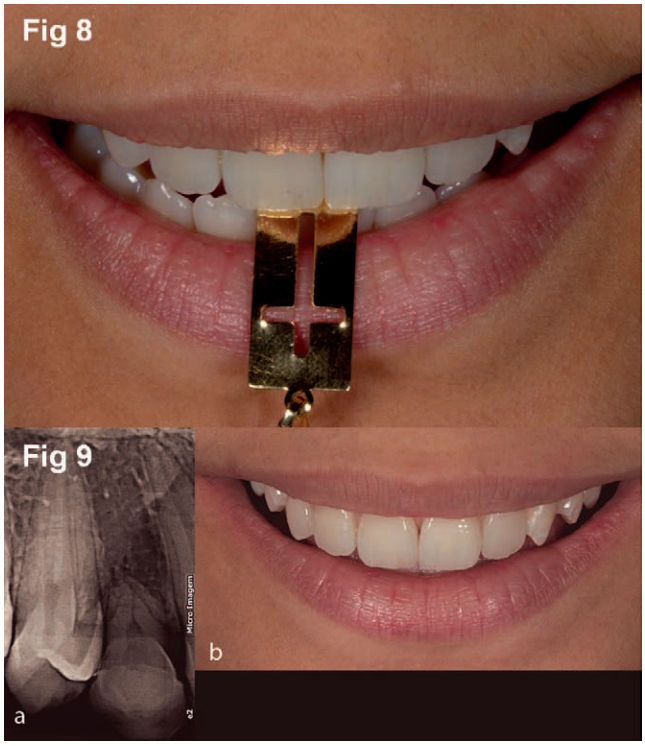

Figure 8. *Clinical follow-up after 18 months showing the veneer of the central incisors stained due to a deleterious habit of the patient.*
Figure 9. *Clinical follow-up after 18 months. (a) Periapical radiographic view showing the stability of the bone around the root of the deciduous canine; (b) clinical aspect after polishing the veneers.*

delayed until the patient reaches the optimum age for a tooth implant, which is 16-17 years old for females.[11] However, the patient was looking for a more immediate solution and complaining about her appearance, which was impairing her interpersonal relationships. Since she presented with good tooth alignment and occlusion and an adequate space for a future implant in the region of the deciduous canine, the treatment with ceramic veneers was suggested to maintain the quality of the patient's bone and to allay her esthetic dissatisfaction.

When faced with an esthetic rehabilitation, the dentist should make a dentofacial analysis by gathering the diagnostic data using esthetic checklists.[6,7,12] Currently, dental casts, photographs, and digital tools help the clinician achieve more reliable planning,[12,13] which aids in communicating with both the patient and other clinicians.[14] After planning, the mock-up procedure shifts the restorations previewed in the wax-up to the clinical situation, allowing the patient to have a tridimensional and realistic view of the treatment outcome so that anatomical changes and adjustments can be made before the definitive restorations are created.[15,16]

As previewed in the digital protocol, the ceramic veneers without the gingivoplasty to align the gingival contour would result in the centrals being shorter than the future right lateral incisor, which, in turn, would be higher (shorter) than the right deciduous canine. This condition would achieve an unbalanced smile without proportion or harmony. These periodontal procedures were thus intended to correct the gingival level and ensure proper width-to-length tooth ratios. The gingivoplasty was done to respect the biological space, and the tissue grafts were aimed to augment the thickness of keratinized tissue and improve the stability of the perio-restorative interfaces.[17] If the deciduous tooth were to exfoliate in the future and require replacement with an implant, the proshesis would have adequate space and an appropriate gingival contour.

In the restorative phase, the central incisors were included to reestablish tooth proportionality in the smile, based on the digital plan. The decision to include these teeth in the restorative phase was made because of the minimal amount of enamel preparation needed and to better fulfill the patient's high esthetic demands. The mock-up indicated no need to extend the preparation depth since the space necessary for the veneers already existed, with the exception of the right canine, which was reshaped into a lateral incisor. Recountouring canines into laterals, with direct resin or ceramics, often requires more tooth preparation due to the anatomical differences between these teeth.[8,10] Thus the canine preparation was deeper, with exposure of dentin, which was properly protected using the provisional restoration and was treated to receive the luting of the ceramic veneer.

When considering an unesthetic situation involving the restoration of anterior teeth in younger patients, one must decide between composite resin and ceramics. The option to use ceramics in this case was determined by the extensive volume of restorative material that would be necessary to reshape the deciduous tooth, the permanent canine, and the peg-shaped lateral incisor with minimal tooth preparation. Glass-infiltrated based ceramics, as used in this report, present high esthetic potential and can be used in different tonalities, in small thicknesses,[18] and are considered a safe treatment option that preserves tooth structure.[19] Clinical studies show that ceramic veneers have high survival rates,[18,19] (up to 10 years).[20] Also, their bonding efficacy in deciduous teeth is comparable to that of permanent teeth.[21]

The main concern for the present clinical situation was the stability of a ceramic veneer on a retained deciduous tooth. However, at the 18-month follow-up, radiographic evaluation indicated stability of the treatment, since the deciduous canine displayed no sign of root resorption. Clinical follow-ups should always be considered part of the treatment, since they may determine the longevity of treatment. After 18 months, the patient returned with black staining on the incisal third of the veneer of the right central incisor as a result of her deleterious habit. This clinical situation should be controlled because it can have an undesirable effect on the veneer, including fracture. The 18-month follow-up demonstrates the stability of the treatment but it cannot assess its durability. However, it is most important that the treatment done in this case provide for possible future interventions such as a single-tooth implant in the right canine site for this patient.

CONCLUSION

The results achieved in this case, as shown by the 18-month follow-up, in terms of the patient's clinical, radiographic, and psychological aspects, lead to the conclusion that our approach was safe and conservative.

Acknowledgement

The authors are grateful to Eduardo Hairashi, the prosthetics specialist who fabricated the ceramic restorations.

Regulatory Statement

This study was conducted in conformity with the local committee guidelines and policies for human subjects of the Araçatuba Dental School, State University of São Paulo. An informed consent was obtained.

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.

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Clinical Research

A Multicenter Randomized Double-blind Controlled Clinical Trial of Fiber Post Cementation Strategies

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T Pereira-Cenci • LF Valandro

Clinical Relevance

Both self-adhesive and regular resin cements obtained good survival rates and could be used for fiber post cementation.

SUMMARY

Objectives: The aim of this prospective randomized multicenter clinical trial was to evaluate the survival rate of glass fiber–reinforced posts cemented with self-adhesive or regular resin cements.

Methods: The sample was comprised of 152 teeth randomized within two centers and in accordance with the adhesive strategies for RelyX U100/U200 (3M ESPE) or Single Bond and RelyX ARC (3M ESPE). The cementation procedures were standardized and performed by previously trained operators. The primary outcome evaluated was post debonding. A trained evaluator, one for each center, assessed all subjects at intervals of 12 months for up to 6 years. Statistical analysis was performed using the Kaplan-Meier method.

Results: There was no statistically significant difference in survival rates between the two strategies assessed (p=0.991), with a 92.7% survival rate for the self-adhesive cement and 93.8% for the regular cement.

Conclusion: Both the self-adhesive and the regular resin cements are good alternatives for glass fiber post cementation.

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INTRODUCTION

Coronal reconstruction of endodontically treated teeth is frequently required before crown placement, especially when the remaining coronal tooth structure is not enough to provide adequate retention and resistance for the final restoration. Characteristics such as mechanical properties similar to that of dentin,[1] esthetics,[2,3] elimination of the laboratory step,[4] and high survival rates presented by clinical studies[4,5] make fiber posts a feasible alternative for the reconstruction of endodontically treated teeth. They also explain the increasing use of fiber posts in clinical practice.

The use of a resin cement, either with or without an adhesive system, is essential for the cementation of prefabricated glass fiber posts. A self-adhesive or conventional resin cement associated with a photoactivated adhesive system are two feasible alternatives. They present acceptable results in *in vitro* studies[6,7] and show desirable mechanical properties.[8]

Although many *in vitro* studies compared different techniques for fiber post cementation[9,10] and observed different results, the literature is inconclusive about a recommendation for the best clinical strategy for fiber post cementation. In addition, most clinical studies are focused on the type of post used for the restoration of endodontically treated teeth[5,11,12] and not on the cementation strategy for these restorations. The lack of high-level evidence to support clinical decisions on fiber post cementation strategies in randomized clinical trials is noteworthy.

The total etch-and-rinse adhesive systems associated with dual cure cements remove the smear layer and generate a better dentin hybridization.[13,14] However, this strategy needs to be executed using a wet dentin substrate, which is very difficult to control inside the canal space. In addition, it presents the limitation of light transmission decreasing through the root.[15] The self-adhesive resin cements appear to be an interesting alternative, because they do not require any dentin pretreatment before cementation and have presented good bond strength results.[9]

Thus, the aim of this randomized multicenter clinical trial was to compare the clinical performance (survival rates) of two cementing strategies (a two-step total etch adhesive system associated with a conventional resin cement vs a self-adhesive resin cement) with fiber posts. The null hypothesis tested was that there would be no difference between the two strategies.

METHODS AND MATERIALS

Experimental Design and Ethical Aspects

This study was a prospective, double-blinded (patient and evaluator), parallel-group randomized multicenter controlled trial (RCT) registered at ClinicalTrials.gov (NCT01461239). The study was developed at two dental schools and approved by both of their ethical committees (protocols 099/2009 and 0170.1.243.000-09). The report of the results followed the Consolidated Standards of Reporting Trials (CONSORT) guidelines.

Inclusion and Exclusion Criteria

The sample was composed of patients who needed post placement and single crowns in any tooth and looked for treatment at either dental school. The inclusion criteria were teeth with a clinically acceptable endodontic treatment and at least 3 mm of apical sealing, missing coronal surface indicating the need of a crown, and simultaneous bilateral occlusal contacts. The exclusion criteria were teeth with a degree of mobility higher than 1 or abutting of a removable or fixed partial denture, any systemic disease that interferes with bone quality, an apical lesion impossible to eliminate with proper endodontic treatment, as well as patients with advanced and untreated periodontal disease.

Sample Size Calculation

Sample size calculation estimated a failure rate of 10% in the experimental group (self-adhesive cement) and 0% in the control group, with 80% power and the significance level set at 5%. Thus, the sample necessary for each group was 73 teeth. However, to counter possible dropouts during the study, a sample of 76 teeth (N=152) was used.

Randomization and Allocation Process

The randomization of the experimental procedures was performed by an independent researcher, using a table of random numbers generated by a computer program and stratified considering tooth position (anterior or posterior, the latter was also divided into molars and premolars) and the two centers in which the experiment was conducted. The randomization sequence was allocated into individual consecutively numbered plain brown envelopes. The envelope was only taken and opened after the root canal prepara-

tion to prevent the choice of cement affecting this procedure.

Operator's Clinical Training

Prior to the experimental procedures, the researchers responsible for the project trained the dentists who performed the experimental procedures. One-month hands-on training and lectures were used so that all procedures were standardized in the two centers. All procedures were carried out by senior year undergraduate students under the main researchers' supervision.

Clinical Procedures

Previous to the single crown manufacturing, all patients who met the inclusion criteria and were accepted to be part of the study received a complete dental evaluation and had reestablished a healthy oral condition, if applicable. The first patients were included in 2009, and the last patients were included in 2013/2014. Patients were submitted to the procedures described below.

Diagnostic radiographs were performed to determine the working length and selection of the glass fiber post from the White Post DC system (FGM, Joinvile, Brazil). The tooth was isolated with a rubber dam, and the root canal was prepared with the system drill until two-thirds of its length was left, keeping at least 3 mm of apical sealing. If the specimen had more than one canal, the larger canal was chosen to be prepared. The post was tested, and a 4-mm coronal length was left.

For cementation procedures, the surface of all posts was cleaned with 70% alcohol, air dried, silanized (ProSil, FGM), and allowed to sit for one minute for complete evaporation of the solvent. The treatment for the root canal dentin for the post cementation followed the manufacturer's recommendations and changed according to the cementation strategy.

Regarding the first strategy, cementation with the self-adhesive resin cement RelyX U100/U200 (RelyX U100/U200 because RelyX U100 was discontinued in 2014; 3M/ESPE, St. Paul, MN, USA), the cement pastes were mixed and taken to the root canal using a Centrix syringe with an Acudose tip and with the aid of the post. Then the post was inserted into the canal, and the excess paste was removed. The post was kept in position for five minutes, and then the cement was light-cured for 40 seconds through the coronal portion of the post.

For the second strategy, cementation with two-step total etch adhesive and conventional resin cement, Single Bond and RelyX ARC (3M/ESPE), the prepared root canal was conditioned with 37% phosphoric acid for 15 seconds. This was followed by extensive washing with water and drying with light air jets and absorbent number 80 paper cones. The Single Bond adhesive system was applied in the canal with the proper microbrush, and the excess was removed with paper cones. Afterward, the adhesive system was light cured for 30 seconds, and the cement pastes were mixed and taken to the root canal using a Centrix syringe with an Acudose tip and with the aid of the post. Then the post was inserted into the canal, and the excess paste was removed. The post was kept in position for five minutes and then, at last, the cement was light-cured for 40 seconds through the coronal portion of the post.

After cementation, a diagnostic radiograph was taken for the baseline. The coronal reconstruction was made using Scotch Bond + Z 250 composite resin (3M ESPE). The coronal preparation was made according to the literature recommendation for metal-ceramic crowns, on the level of gingiva or, at most, 0.5 subgingival, and using a chamfer marginal design. The prepared teeth were impressed with a polyether material (3M ESPE) using an acrylic unitary tray along with a full arch alginate impression. After fabrication, the metal frameworks (CrCo) were verified clinically, a transfer casting was made, and the ceramic color was selected. The Final crowns were verified by cervical fit, occlusal adjustments were made when necessary, and all crowns were cemented with RelyX U100/U200 (3M ESPE) resin cement.

Evaluation Parameters

Participants were recalled annually for clinical and radiographic examinations. The main outcome evaluated was fiber post debonding. If the fiber post was in place at the moment of evaluation, it was considered a survival. All fiber post debonding was considered as failure. Root fractures were also considered a failure, because studies showed that fiber post decementation could lead to root fracture.[16] When a patient returned for an examination with a tooth lacking a post, the time of failure (post debonding) was based on his/her self-report. Periapical radiographs were taken to evaluate any endodontic problem. If any apical alteration was observed, this was not considered failure but was considered unsuccessful. Failures of metal-ceramic

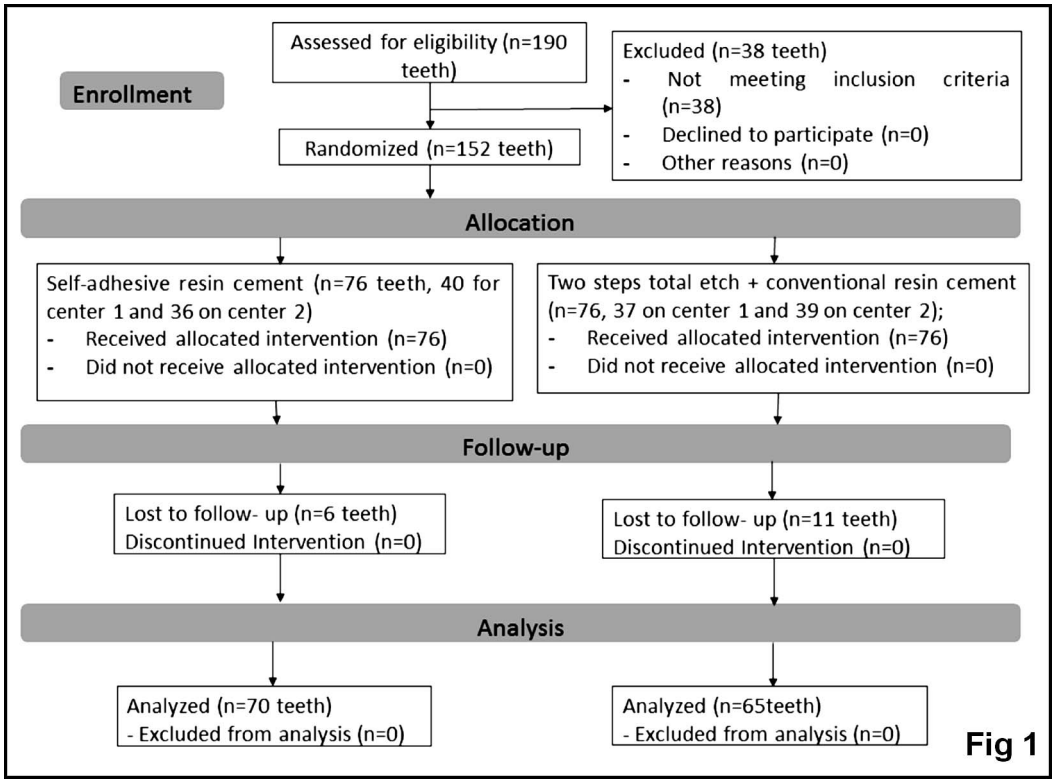

Figure 1. *Flowchart of trial phases.*

crowns were not considered, because they were not related to the bonding potential of resin cements. The outcome evaluated was the presence or absence of decementation, so the calibration of the evaluators was not necessary. All clinical problems observed or related by patients were treated by the researchers. The latest recalls were performed in the first months of 2016.

Statistical Analysis

Statistical analysis was performed using the SPSS 22 for Mac software (SPSS Inc, Chicago, IL, USA). Descriptive analysis was used to describe those patients included in the study and the reasons of failures. The longevity of the posts and teeth was assessed using the Kaplan-Meier model and the long-rank test (α=0.05).

RESULTS

Participants

A total of 129 patients with an average age of 47.7 years, received 152 glass fiber posts. After six years, 15 patients (n=17 teeth) were lost to follow-up (12 patients were unable to be contacted; three patients changed address) resulting in a recall rate of 91.4 % for the six-year period of the study (Figure 1). A total of 114 patients and 135 teeth were evaluated at the final count. Ninety-five of the patients were women, with a mean age of 47.4 years, and 19 were men, with a mean age of 49.25 years.

In total, 70 fiber posts were cemented with self-adhesive resin cement, and 65 fiber posts were cemented with two-step total etch adhesive + conventional resin cement. From the 135 evaluated fiber posts, 60 were cemented in the anterior teeth and 75 were cemented in the posterior teeth (52 premolars and 23 molars), as shown in Table 1. The mean observation time was 37 months (3.1 years).

Failures

After six years, nine failures were observed, with five failures for RelyX U100/U200 and four failures for RelyX ARC. RelyX U100/U200 showed a survival rate of 92.7%, and RelyX ARC showed a survival rate of 93.8%, with no statistical difference (p=0.991) to each other (Figure 2). Three failures were root fractures (two in the RelyX ARC group and one in the RelyX U100/U200 group), four were fiber post decementations (three in the RelyX ARC group and one in the RelyX U100/U200 group), one was a core fracture (in the RelyX ARC group), and one was a

Table 1: *Distribution of restoration evaluated according to patient sex, patient age group, tooth, and class type*

Sex	RelyX ARC				RelyX U200				Grand Total
	Ant	PM	Mol	Total	Ant	PM	Mol	Total	
Female (age group in years)	23	20	11	55	25	25	8	57	112
17-40	3	5	3	11	2	4	1	7	18
41-50	10	8	1	19	13	12	4	29	48
51-60	6	4	3	14	8	8	—	16	30
>60	4	3	4	11	2	1	3	6	17
Male (age group in years)	3	4	2	10	9	3	2	14	23
17-40	—	1	1	2	3	—	—	3	5
41-50	2	1	—	3	4	—	2	6	9
51-60	—	—	—	—	—	1	—	1	1
>60	1	2	1	5	2	2	—	4	9
Grand total	26	24	13	65	34	28	10	70	135

Abbreviations: Ant, anterior teeth, Mol, molars; PM, premolars.

post fracture (in the RelyX U100/U200 group). The nine failures occurred in seven patients (two patients presented two failures each), and all the failures occurred in teeth that had few remaining coronal structures (0 or 1 remaining wall).

Considering the location of failure, six failures occurred in the posterior region and three occurred in the anterior region (Figure 3). Considering the tooth type, six failures occurred in the premolars, three occurred in the incisors (two in the lateral incisors and one in a central incisor), and no failures were observed in the molars. The statistical analysis showed no statistically significant difference between them ($p=0.210$).

Regarding the treatment centers (Figure 4), five failures occurred in center 1: three root fractures and two decementations. Four failures occurred in center 2: three decementations and one post fracture. There was no statistical difference found when comparing survival curves between the centers ($p=0.339$).

Periodontal problems and endodontic alterations were not observed during the evaluations at both centers.

DISCUSSION

The results presented in this study show that the survival rate of glass reinforced fiber posts is not influenced by the type of tested approach to cement fiber posts. Therefore, the null hypothesis was not rejected.

The evaluated cementation strategies present distinctive clinical approaches. Although one requires multiple steps, involving acid etching and dentin moisture control, the other does not need any steps prior to post cementation. One would expect that those differences could influence the results, but the findings of this study may be related to the fact that all procedures were standardized, all operators were previously trained, and the procedures followed the technique recommended by the manufacturers. For example, the technique used in this study to insert the resin cement into the root canal already generated less voids in the resin cement layer and presented high bond strength values than other techniques.[17]

The survival similarities could be explained by the fact that high bond strength values along all root spaces are not as important to the clinical behavior. It is possible that high bond strength values at the cervical region could be enough to generate the same clinical outcome.[18] However, if this study had a different clinical design (retrospective), the results could be different as most retrospective studies do not have standardized procedures and generally present a higher sample.

Considering the region of the failures, more failures occurred in the anterior teeth and premolars in comparison to molars, as no failures occurred in the latter. It is likely that, different from molars, premolars and anterior teeth receive oblique occlusal forces, which are more dangerous to the restored teeth than vertical forces.[19] Also, molars have a larger structure that may dissipate occlusal strength more evenly. However, important factors such as remaining coronal structure and ferrule height, which were recorded to be used in future statistical analysis, showed no influence on the results. This could be related to the fact that most of the teeth presented no coronal structure. In addition, this characteristic appears to have more influence on

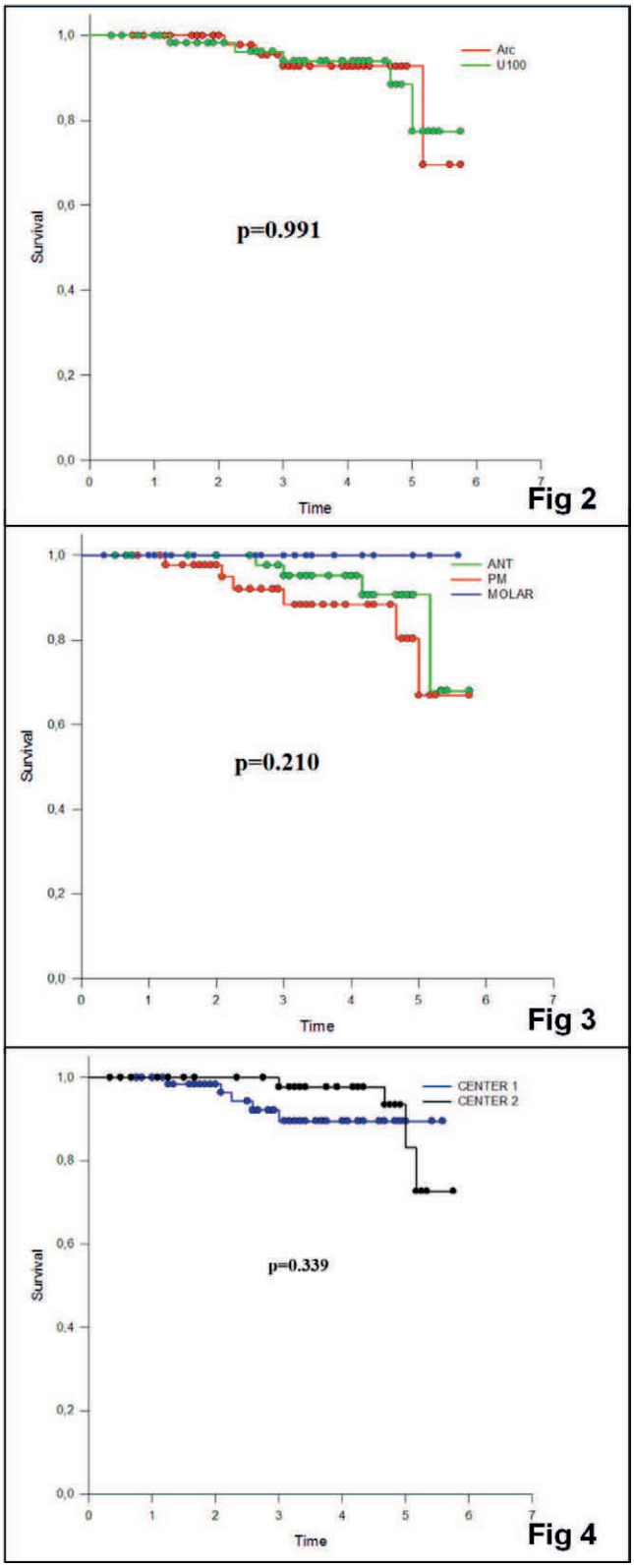

Figure 2. *Kaplan-Meier survival curves for the comparison between cementation strategies.*
Figure 3. *Kaplan-Meier survival curves for the comparison between region.*
Figure 4. *Kaplan-Meier survival curves for the comparison between the centers.*

fracture resistance than on decementation properties.

Another important issue to discuss is that all patients presented a perfect occlusal condition and had no current parafunctional habits. It is a perfect scenario for the dentist to make a single crown; however, it is not always the reality of clinical practice. In the future, it may be important to include all types of patients, so that the influence of these variables on outcomes can be evaluated.

An interesting point is the fact that the patient may influence the results to the same degree or more than the material used for the restoration, as the hygiene habits and the occlusal characteristics may affect the survival of the restoration. In this study, there were nine failures in all, and four of these failures occurred in two patients. Each patient had two failures and that received different cementation strategies, showing that the patient's characteristics are an important issue to be considered.

Although the Kaplan-Meier method estimates survival until the sixth year of observation, the mean time of observation in this study was 3.1 years. Longer observation periods are, of course, always better, and this could be a limitation of this study. However, the survival rates presented in this study are satisfactory compared with other clinical trials with longer (five years) follow-up survival rates, such as Schmitter and others.[20] They found survival rates near 70% for teeth restored with glass fiber posts.

Randomized multicenter clinical trials are important tools to obtain relevant data with a high level of evidence, but they also present some drawbacks. These include achieving the size of the sample, the observation time, the high rates of withdrawal, and the need to follow strict criteria such as the CONSORT statement.

It is also important to highlight the fact that the characteristics of the cities where the study was developed may affect the results. Although no statistically significant differences were found between the survival rates of the centers, in one center there was a higher patient dropout because it is a city with a low fixed permanent residence, with a student and military population that often moves. A more fixed population facilitated the lower dropout in the other city. This is very relevant, as the only difference observed regarding the centers was the follow-up of the patients.

Another important issue is that all the teeth received a metal-ceramic crown as the final restora-

tion, which was performed using the same technique and the same resin cement. The control of these factors also helped to avoid possible bias and allowed for the assessment of the real effects of the resin cement on fiber posts. It is also important to point out that, as the first randomized multicenter clinical trial to evaluate the influence of cementation strategies on the survival of teeth restored with glass fiber posts, the results should be interpreted carefully. From this study, it was found that both resin cements performed adequately, and restorations had adequate survival rates. However, more clinical studies considering critical clinical factors are essential to generate more evidence to help clinicians decide the best clinical protocol when planning restorations with glass fiber posts.

CONCLUSION

Self-adhesive and regular resin cements are feasible options to cement glass fiber posts, with an adequate survival of the restorations.

Acknowledgments

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Santa Maria, Brazil. The approval codes for this study are 099/2009 and 0170.1.243.000-09.

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.

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Laboratory Research

Bulk-Fill Composites: Effectiveness of Cure With Poly- and Monowave Curing Lights and Modes

JK Gan • AU Yap • JW Cheong • N Arista • CBK Tan

Clinical Relevance

For camphorquinone-based bulk-fill composites, photopolymerization with monowave light-emitting diode lights may be more efficient than polywave ones. Despite manufacturers' claims, not all bulk-fill composites can be effectively cured to depths of 4 mm.

SUMMARY

This study compared the effectiveness of cure of bulk-fill composites using polywave light-emitting diode (LED; with various curing modes), monowave LED, and conventional halogen curing lights. The bulk-fill composites evaluated were Tetric N-Ceram bulk-fill (TNC), which contained a novel germanium photoinitiator (Ivocerin), and Smart Dentin Replacement (SDR). The composites were placed into black polyvinyl molds with cylindrical recesses of 4-mm height and 3-mm diameter and photopolymerized as follows: Bluephase N Polywave High (NH), 1200 mW/cm^2 (10 seconds); Bluephase N Polywave Low (NL), 650 mW/cm^2 (18.5 seconds); Bluephase N Polywave soft-start (NS), 0-650 mW/cm^2 (5 seconds) → 1200 mW/cm^2 (10 seconds); Bluephase N Monowave (NM), 800 mW/cm^2 (15 seconds); QHL75 (QH), 550 mW/cm^2 (21.8 seconds). Total energy output was fixed at 12,000 mJ/cm^2 for all lights/modes, with the exception of NS. The cured specimens were stored in a light-proof container at 37°C for 24 hours, and hardness (Knoop Hardness Number) of the top and bottom surfaces of the specimens was determined using a Knoop microhardness tester (n=6). Hardness data and bottom-to-top hardness ratios were subjected to statistical analysis using one-way analysis of variance/Scheffe's post hoc test at a significance level of 0.05. Hardness ratios ranged from 38.43% ± 5.19% to 49.25% ± 6.38% for TNC and 50.67% ± 1.54% to 67.62% ± 6.96% for SDR. For both bulk-fill composites, the highest hardness ratios were obtained with NM and lowest hardness ratios with NL. While no significant difference in hardness ratios was observed between curing lights/modes for TNC, the hardness ratio obtained with NM was significantly higher than the hardness ratio obtained for NL for SDR.

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INTRODUCTION

Bulk-fill composites are not a new notion, and many products have come and gone over the past three decades. Potential advantages of bulk-fill composites include reduction of voids in material mass as well as faster and easier placement or curing, leading to improved chairside efficiency. Polymerization shrinkage may, however, be more pronounced, and cure in deep preparations might be inadequate.[1] Contemporary bulk-fill composites differ considerably from their predecessors. Through the use of novel photoinitiators, proprietary resins, special modulators, unique fillers, and filler distribution, current bulk-fill composites are claimed to have lower polymerization shrinkage and depth of cure of 4 mm or more. Studies pertaining to the depth of cure of bulk-fill composites have, however, been equivocal. While some authors have reported adequate cure at 4-mm depths,[2-5] others have reported otherwise.[6-8] The apparent discrepancies can be attributed to differences in testing methodologies, viscosity and translucency of the bulk-fill composites evaluated, and light-curing conditions.[9]

Light-cured composites set via radical photopolymerization. Light photons are absorbed by photoinitiators, and free radicals are formed in the presence of activators. The free radicals subsequently trigger the polymerization reaction, resulting in the conversion of monomers into polymers.[10] Camphorquinone (CQ) is the most widely used photoinitiator and has a sensitivity peak near 470 nm in the blue range of the visible light spectrum. Because of its intense yellow color, alternative lighter-colored initiators that completely bleach out after photopolymerization have been recently promoted. These include phenyl propanedione (PPD), acyl phosphine oxide (APO), and Ivocerin. While the absorption spectrum of PPD extends from the ultraviolet (UV) wavelength range to approximately 490 nm, APO such as Lucirin TPO mainly absorbs light in the UV range. The sensitivity peak of APO is approximately 370 nm, which is considerably lower than that of CQ. Ivocerin is a newly developed germanium photoinitiator that absorbs light at a higher wavelength range than APO and has a sensitivity peak of about 420 nm.

Until the 1990s, halogen or quartz tungsten halogen curing lights were the standard curing option. Because of their wide spectral output, halogen lights required band-pass filters to limit wavelengths of light between 370 nm and 550 nm for the absorption of CQ. Their wide range of wavelengths also allows for the curing of composites employing PPD and APO as photoinitiators. However, the curing efficacy of halogen lights is low, and the high temperatures generated require external cooling and limit the lifetime of bulbs, reflectors, and filters.[11] As such, the use of light-emitting diode (LED) curing lights, which emit blue light, began in the 2000s to address the problems associated with halogen curing lights. Instead of hot filaments, LED curing lights use a combination of two different doped semiconductors for light production. They consume less energy, do not require cooling fans, and have extended lifetimes without significant loss of light intensity.[11] The first-generation LED curing lights had low light intensities of approximately 400 mW/cm^2, while the second generation lights were able to achieve intensities of up to 1000 mW/cm^2. Both first- and second-generation LED lights used only one type of LED (monowave [single-peak] technology) and were unable to cure composites with PPD and APO initiator systems. Current third-generation LED curing lights feature higher light intensities, multiple curing modes (high, low, and soft start), and avoid wavelength-compatibility issues by deploying polywave (dual/multipeak) technology.

Research pertaining to the use of polywave LED curing lights on the effectiveness of cure of bulk-fill composites is still limited.[3,12,13] The curing efficacy in these studies was assessed using different methodologies, including micro-Raman spectroscopy, microhardness testing, and the ISO 4049 scraping test. Comparison of polywave and monowave LED curing lights was addressed in only one study.[12] Using the ISO scraping test, no significant difference in depth of cure was found between the two LED technologies for the bulk-fill composites evaluated (Tetric Evoceram and Filtek Bulk Fill). More studies are therefore required to address the current gaps in knowledge, especially with the increased use of PPD and APO as photoinitiators, particularly in bleached shade composites. Therefore, the objective of this study was to compare the effectiveness of cure of two bulk-fill composites with polywave LED, monowave LED, and conventional halogen curing lights using microhardness testing. Curing efficacy of the high-intensity, low-intensity, and soft-start modes of the polywave LED was also appraised. It was hypothesized that no difference in the effectiveness of cure existed between the polywave LED (and its various curing modes), monowave LED, and halogen curing lights if the total light energy was kept constant.

Table 1: *Technical Profiles of Bulk-Fill Composites Evaluated*

Material	Abbreviation	Shade/Batch Number	Composition	Filler % by Weight (Volume) and Filler Size	Recommended Thickness, mm	Recommended Curing Time and Light Intensity
Tetric N-Ceram Bulk Fill	TNC	Universal (IVA)/LOT S21119, Exp 2017-06	Resin: dimethacrylates Filler: barium glass, ytterbium trifluoride, mixed oxide and copolymers	80%-81% (55%-57%) 0.04-3 μm	4	20 s for ≥ 500 mW/cm^2 or 10 s for ≥ 1000 mW/cm^2
SDR Posterior Bulk Fill Flowable Base	SDR	Universal/ 1405000811, Exp 2016-04	Resin: modified UDMA, EBPADMA, TEGDMA Filler: barium-alumino-fluoro-borosilicate glass, strontium alumino-fluoro-silicate glass	68% (45%) Mean 4.2 μm	4	20 s, for ≥ 550 mW/cm^2

Abbreviations: Bis-MPEPP, 2,2-bis[(4-methacryloxy polyethoxy)phenyl)propane; EBPADMA, ethoxylated bisphenol A dimethacrylate; SDR, Smart Dentin Replacement; S-PRG, surface pre-reacted glass ionomer; TEGDMA, triethylene glycol dimethacrylate; TNC, Tetric N-Ceram bulk-fill; UDMA, urethane dimethacrylate.

METHODS AND MATERIALS

The technical profiles and composition of the bulk-fill composites evaluated are shown in Table 1. Tetric N-Ceram Bulk Fill (TNC; Ivoclar Vivadent, Schaan, Liechtenstein) uses CQ and Ivocerin as photoinitiators, giving it a wide absorption range of 370 nm to 460 nm.[14] According to the manufacturer's instructions, TNC can be cured reliably in 4-mm increments by LED lights of 1000 mW/cm^2 over 10 seconds. While TNC is a "sculptable" bulk-fill restorative, Smart Dentin Replacement (SDR; Dentsply-Caulk, Milford, DE, USA) is a "flowable" bulk-fill base material. The photoinitiator in SDR is CQ, and 4-mm increments of SDR can be cured by halogen lights of 550 mW/cm^2 over 20 seconds.

The composites were placed in a single increment into black polyvinyl molds with cylindrical recesses of 4-mm height and 3-mm diameter. Excess material was removed by compressing the molds between two glass slides (1-mm thick). The composites were then irradiated through the top glass slide using either a polywave LED with different curing modes (Bluephase N Polywave, Ivoclar Vivadent, Schaan, Liechtenstein), a monowave LED (Bluephase N Monowave, Ivoclar Vivadent), or a halogen (QHL75, Dentsply-Caulk) curing light. The polywave LED light offered several different curing modes including high-power, low-power, and soft-start polymerization, whereas the monowave LED light presented with only high-power curing. The exit windows of the light-curing tips were 8 mm in diameter for the three curing lights, and light intensity was verified with a radiometer (Demetron LED radiometer; Kerr Corporation, Middleton, WI, USA) prior to use to ensure consistency of energy output. The five curing light/mode combinations are detailed in Table 2. These were Bluephase N Polywave high power (NH), Bluephase N Polywave low power (NL), Bluephase N polywave soft start (NS), Bluephase N Monowave (NM), and QHL75 (QH). The total energy output (intensity × time) was standardized at 12,000 mJ/cm^2 for all curing lights/modes, with the exception of NS. Because of the preset soft-start curing profile, the closest total energy achievable for NS was 13,625 mJ/cm^2. Six specimens were fabricated for each composite for the various curing light/mode combinations. Immediately after light polymerization, the composite specimens were removed from their molds and stored in a light-proof container at 37°C in a humidified atmosphere for 24 hours. They were then subjected to microhardness testing with a Knoop hardness tester (FM-7, Future-Tech, Tokyo, Japan). A 10 g load was applied with a dwell time of 15 seconds to obtain the Knoop hardness number

Table 2: *Technical Profile of Curing Lights and Modes Evaluated*

Curing Light	Curing Mode	Recommended Curing Profile	Study Curing Profile
Bluephase N Polywave	High (NH)	1200 mW/cm^2 (10 s)	1200 mW/cm^2 (10 s)
Bluephase N Polywave	Low (NL)	650 mW/cm^2 (10 s)	650 mW/cm^2 (18.5 s)
Bluephase N Polywave	Soft start (NS)	0-650 mW/cm^2 (5 s) → 1200 (10 s)	0-650 mW/cm^2 (5 s) → 1200 (10 s)
Bluephase N Monowave	High (NM)	800 mW/cm^2 (15 s)	800 mW/cm^2 (15 s)
QHL-75	Normal (QH)	550 mW/cm^2 (20 s)	550 mW/ cm^2 (21.8 s)

Table 3: *Mean Top KHN, Bottom KHN, and Hardness Ratio (%) With the Different Lights/Modes for TNC and SDR*

Material	Curing Light	Curing Mode	Top KHN	Bottom KHN	Hardness Ratio, %
TNC	Bluephase N Polywave	NH	25.78 (3.56)	10.32 (1.04)	40.95 (8.44)
	Bluephase N Polywave	NL	30.10 (4.13)	11.42 (1.03)	38.43 (5.19)
	Bluephase N Polywave	NS	29.09 (4.74)	11.67 (1.01)	41.10 (8.14)
	Bluephase N Monowave	NM	22.82 (1.91)	11.24 (1.75)	49.25 (6.38)
	QHL-75	QH	30.31 (1.82)	13.43 (1.52)	44.27 (4.08)
SDR	Bluephase N Polywave	NH	15.21 (2.42)	8.74 (0.83)	58.37 (8.67)
	Bluephase N Polywave	NL	16.39 (1.37)	8.30 (0.69)	50.67 (1.54)
	Bluephase N Polywave	NS	17.16 (1.65)	9.98 (0.72)	58.44 (5.13)
	Bluephase N Monowave	NM	16.19 (1.49)	10.95 (1.43)	67.62 (6.96)
	QHL-75	QH	18.35 (1.41)	10.51 (0.82)	57.64 (6.97)

Abbreviations: KHN, Knoop hardness number; SDR, Smart Dentin Replacement; TNC, Tetric N-Ceram Bulk Fill.

(KHN) for both top and bottom surfaces of each specimen. The KHN corresponding to each indentation was determined by measuring the dimensions of the indentations using the following formula:

$$\mathrm{KHN} = 14.2 \times (\mathrm{F}/\mathrm{d2})$$

where F is the test load in kilograms and d is the longer diagonal length of an indentation in millimeters. For each surface, three readings were taken, and the mean KHN value was calculated. The KHN of the bottom was divided by the KHN of the top surface to establish the hardness ratios, which were subsequently converted to a percentage. KHN data and hardness ratios were subjected to statistical analysis using one-way Analysis of Variance and Scheffe's post hoc test at a significance level of 0.05.

RESULTS

The mean KHN and hardness ratios (%) of the various curing light/mode combinations for TNC are shown in Table 3. The mean KHN of the top surface ranged from 22.82 ± 1.91 to 30.31 ± 4.13 when irradiated using NM and QH, respectively. The mean KHN of the bottom surface ranged from 10.32 ± 1.04 to 13.43 ± 1.52 for NH and QH, respectively. The hardness ratio for TNC ranged from 38.43% ± 5.19% to 49.25% ± 6.38% for NL and NM, respectively. Results of statistical analysis for TNC are reflected in Table 4. At the top surface, the KHNs of QH and NL were significantly higher than NM, while for the bottom surface, the KHN of QH was significantly higher than that of NH. However, no significant difference in hardness ratio was observed.

The mean KHN and hardness ratios (%) of the various curing light/mode combinations for SDR are also shown in Table 3. The mean top KHN ranged from 15.21 ± 2.42 to 18.35 ± 1.41 for NH and QH, while the mean bottom KHN ranged from 8.30 ± 0.69 to 10.95 ± 1.43 for NL and NM, respectively. The hardness ratio for SDR ranged from 50.67% ± 1.54% to 67.62% ± 6.96% for NL and NM, respectively. Results of the statistical analysis for SDR are reflected in Table 4. At the top surface, the KHN of QH was significantly higher than that of NH. At the bottom surface, the KHNs for NM and QH were significantly higher than that of NL. The bottom KHN of NM was also significantly higher than that of NH. Unlike TNC, significant differences in hardness ratio for SDR were observed. The hardness ratio obtained with NM was significantly higher than with NL.

DISCUSSION

The effectiveness of cure of bulk-fill composites with polywave LED, monowave LED, and conventional

Table 4: *Statistical Comparison of Top KHN, Bottom KHN, and Hardness Ratio (%) for TNC and SDR*

Material	Variable	Result[a]
TNC	Top KHN	QH, NL > NM
	Bottom KHN	QH > NH
	Hardness ratio, %	NS
SDR	Top KHN	QH > NH
	Bottom KHN	NM > NH, NL
		QH > NL
	Hardness ratio, %	NM > NL

Abbreviations: KHN, Knoop hardness number; NS, no statistical significance; SDR, Smart Dentin Replacement; TNC, Tetric N-Ceram Bulk Fill.
[a] > denotes statistically significant differences. Results of one-way Analysis of Variance/Scheffe's post hoc test ($p<0.05$).

halogen curing lights was evaluated. The null hypothesis was rejected as significant differences in effectiveness of cure existed between the different lights and curing modes despite regulating the total light energy. Curing efficacy can be assessed by direct and indirect methods. Direct methods, such as infrared and Raman spectroscopy, are not routinely used as they are complex, expensive, and time-consuming to perform.[15] Indirect methods include visual appraisal, scraping (ISO 4049), and hardness testing. While visual appraisal does not offer scientific objectivity, the ISO scraping test generally results in greater depths of cure when compared with hardness testing.[6] Hardness is an indicator of the degree of polymerization, and a good correlation between Knoop hardness and infrared spectroscopy has been reported.[16,17] In view of its relative efficiency and popularity, Knoop hardness testing was selected to determine the effectiveness of composite cure. Hardness testing was done 24 hours after photopolymerization to allow for composite postcure.[18]

Adequate photopolymerization is essential for optimization of physicomechanical properties and clinical longevity of composite restorations.[19] In addition, inadequately cured composites are also cytotoxic because of residual monomers and other reactive components.[20,21] Composite restorations should ideally be equally cured throughout. The bottom-to-top hardness ratio of the 4-mm-thick bulk-fill specimens should approximate or equal 1 (100%). Many studies have, however, used a hardness ratio of 0.8 or 80% as the standard for satisfactory cure due to material and light-curing constraints.[22,23] Material factors affecting photopolymerization involve thickness, shade, opacity, and composition, while those related to curing lights include light intensity, wavelength, exposure time, size, location, and orientation of the light probes.[19] As thickness, materials, total light energy (intensity $\times$ time), and light probe variables including curing distance were controlled during the experiment, the results can be largely attributed to light type, spectral wavelengths, and light intensity modification during curing (ie, continuous versus soft-start curing).

At both top and bottom surfaces, the KHN of TNC was higher than SDR regardless of curing light/mode. Results corroborated those of similar studies concerning these materials and can be attributed to TNC's higher filler content when compared with SDR.[6,7] While TNC can be placed up to the surface of the overall composite resin restoration and functionally loaded, SDR requires a "capping layer" of conventional composite to sustain functional loads because of its lower filler loading. At the top surface, significant differences in KHN were observed between curing lights/modes. For TNC, curing with the halogen light and polywave LED (NL in particular) resulted in harder top surfaces than with the monowave LED light (NM). This finding may be attributed to the narrower spectral output of the NM. For SDR, significant differences in the top KHN was observed between the halogen (QH) and polywave LED in high-power mode (NH). The harder surface associated with QH could be contributed in part to a thermal effect and longer exposure time as emission spectrum and total energy output were similar.[24] Exposure time for QH was approximately double that of NH (21.8 vs 10 seconds), and heating of composites from halogen lights has been shown to increase hardness.[25]

As light passes through the bulk-fill composites, intensity is clearly reduced because of light absorption and scattering by the materials, attenuating the potential for photopolymerization.[26] At the bottom surface, photopolymerization of TNC with the halogen light (QH) resulted in significantly higher bottom KHN than with the polywave LED at high power (NH). Thermal effects are negligible at the bottom surface of restorations as composites are poor conductors of heat.[27] As emission spectrum and total energy output were comparable, differences may well be due to variations in light attenuation, light exposure time, and the ensuing polymeric network type. Light attenuation with NH may be greater, especially considering its short curing time. Previous studies have reported the need for longer curing times with LED lights when compared with halogen lights for achieving a similar depth of cure and mechanical properties.[28,29] Hardness of composites is dependent not only on the degree of conversion but also on the nature of and bonding between monomers.[16] Polymers with more cross-linked chains are harder than those with linear chains.[30] Theoretically, the use of high light intensity in the initial phase of curing should result in a greater number of growth centers and higher cross-link density.[31] The aforementioned may, however, be mitigated by the short curing time associated with NH. As such, the cross-link density of bulk-fill composites associated with the various lights/modes requires further investigation.

For SDR, the bottom KHN with the halogen light (QH) was significantly higher than the polywave LED in low-power mode (NL). Significant differences in bottom hardness were also observed between the

LED lights. Photopolymerization with the monowave light (NM) resulted in significantly higher bottom KHN than curing with the polywave LED in both high- and low-power modes (NH and NL). The incongruence in findings when compared with TNC may be attributed to variances in composite composition. In addition to photoinitiator and resin differences, SDR has a lower filler loading and larger filler particle sizes than TNC, resulting in a more translucent material. Monomer conversion has been found to be inversely proportional to filler loading owing to decreased light transmission.[32] Light scattering from smaller filler particles has been found to reduce depth of cure, especially when filler sizes are similar to the wavelength of the emitted light.[33] Despite comparable curing times, photopolymerization with the halogen light still resulted in harder bottom surfaces than with the polywave LED light, reinforcing the necessity for cross-link density studies. Photopolymerization with the monowave LED light resulted in significantly higher bottom surface KHN than the polywave light, with the exception of the soft-start mode. The better performance of the polywave soft-start mode could be attributed to its slightly higher total energy (13,625 mJ/cm^2). The total energy of the soft-start mode could not be harmonized to 12,000 mJ/cm^2 because of the manufacturer's programmed settings. The polywave LED curing offered no advantage over its monowave counterpart as SDR uses primarily CQ as its photoinitiator.

For both TNC and SDR, the hardness ratio was lower than 80% for all curing lights/modes. This can be attributed to attenuated irradiance reaching the bottom surfaces.[26] The highest hardness ratio achieved was 49.25% ± 6.38% and 67.62% ± 6.96% for TNC and SDR, respectively. Results corroborated a recent independent study by Yap and others involving the same materials.[6] To achieve a hardness ratio of 80%, TNC and SDR need to be limited to increments of 2.5 mm and 3 mm correspondingly.[6] Other authors have, however, reported a bottom-to-top hardness ratio of 80% or more.[34,35] The divergence in outcomes can be attributed to differences in bulk-fill materials evaluated, curing light, or parameters and methodologies employed. For both bulk-fill composites, the highest hardness ratio was obtained with NM and lowest with NL despite their identical total energy output. Ranking of hardness ratios differed slightly between TNC and SDR and were as follows: TNC – NM > QH > NS > NH > NL; SDR – NM > NS > NH > QH > NL. When only the LED curing lights were considered, the ranking of hardness ratio was similar for both bulk-fill composites. Photopolymerization with monowave LED gave the highest hardness ratio followed by the polywave LED light in soft-start, high-power, and low-power modes. The monowave LED light thus appears to be somewhat more effective than the polywave LED at 4-mm depths.

For TNC, no significant difference in hardness ratio was observed between the various curing lights/modes. Results supported those of Meenes and others[12] using the ISO 4049 scraping test and customized tooth molds. They found the influence of curing lights to be insignificant, but there was a relatively significant interaction between materials and mold types on composite depth of cure. The use of stainless steel molds led to a deeper depth of cure for Tetric Evoceram bulk-fill, rationalizing the use of unreflective black polyvinyl molds in the present study. For SDR, photopolymerization with the monowave LED resulted in a significantly higher hardness ratio than with the polywave LED at low-power mode despite standardization of the total energy (67.62% vs 50.67%). The irradiance of the polywave LED light even with a lower power mode was already 650 mW/cm^2, well above the traditionally recommended minimum light intensity of 400 mW/cm^2.[36] The 17% variance in hardness ratio between the two LED lights is of concern and warrants further investigation. It may be attributed in part to the lower percentage of light transmitted through composites offered by polywave LED lights when compared with monowave LED lights.[37] Clinically, the reduction in hardness ratio may be even higher as the curing light probe may be 8 mm or more from the composite surface. The latter has been shown to significantly reduce the degree of conversion at the bottom surface of restorations.[38] The effect of light probe distance on curing efficacy of poly- and monowave LED curing lights should also be explored.

CONCLUSION

Within the limitations of this *in vitro* study, the following conclusions can be made:

1. Top and bottom surface hardness achieved with the halogen curing light was usually higher than that obtained with the LED lights when total energy was controlled.
2. The ideal bottom-to-top hardness ratio of 0.8 (80%) was not achieved by either bulk-fill composite evaluated regardless of curing lights/modes.

3. For both bulk-fill composites, the highest hardness ratios were obtained with the monowave LED curing light and lowest with the polywave LED curing light in low-power mode.
4. Photopolymerization with monowave LED lights may be more effective than with polywave LED lights for camphorquinone-based bulk-fill composites.

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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.

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Effect of Sonic Resin Composite Delivery on Void Formation Assessed by Micro-computed Tomography

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Clinical Relevance

Voids in resin composite restorations may be generated during restorative procedures, leading to a reduction in mechanical, physical, and biological properties. A sonic delivery method may lead to a higher incorporation of air within the restoration and, consequently, higher void formation.

SUMMARY

Objectives: The aim of this study was to quantify the internal void volume formation in commercially available, resin composites inserted using conventional or sonic insertion methods, and analyzed using three-dimensional (3D) micro-computed tomography (μCT).

Methods and Materials: Four resin composites were evaluated: one conventional (Herculite, Ultra, Kerr Corporation, Orange, CA, USA), one flowable bulk fill (SureFil SDR Flow, Dentsply International, York, PA, USA), and two packable bulk fill (SonicFill, Kerr Corporation, and Tetric EvoCeram Bulk Fill, Ivoclar Vivadent Inc, Schaan, Liechtenstein). Eight groups were evaluated according to each resin composite type and insertion method (conventional or sonic; n=5). Forty ABS 3D-printed cylindrical molds, 5.0 mm in diameter and 4.0 mm in depth, were fabricated. For the conven-

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tional resin composite, the mold was filled incrementally (two layers), while for bulk-fill resin composites, insertion was performed in a single increment. The sonic insertion method was performed using a specific handpiece (SonicFill Handpiece, Kerr Corporation). Resin composites were light cured using a multipeak light-emitting diode light-curing unit (VALO, Ultradent Products Inc, South Jordan, UT, USA) in its regular mode. Samples were evaluated by μCT, and data were imported into software (Amira, version 5.5.2, VSG, Burlington, MA, USA) for 3D reconstruction, from which the percentage of void volume was calculated. Data were analyzed using two-way analysis of variance and Tukey post hoc test at a preset alpha of 0.05.

Results: The conventional insertion method resulted in reduced porosity, compared with sonic insertion, for SureFil SDR Flow and Tetric EvoCeram bulk fill. The sonic insertion method did not demonstrate any influence on void formation for Herculite Ultra or SonicFill.

Conclusion: Results suggest that the sonic insertion method might increase void formation during resin composite delivery, depending on restorative material brand.

INTRODUCTION

The number of resin composite posterior restoration procedures has greatly increased, due to higher esthetic demands from patients and legal constraints of amalgam use within many countries.[1] A multitude of factors affect the clinical outcome of resin composite restorations, such as quality of the polymerization process,[2] mechanical properties,[3] and insertion technique.[4]

For decades, the incremental (or layering) technique has been the most common insertion method for resin composite, in an attempt to reduce the effects of polymerization shrinkage and stress.[5] The polymerization reaction results in a volumetric reduction caused by formation of covalent bonds established between monomers, which may lead to stress at the tooth/restoration interface, resulting in inadequate adaptation, micro-cracking, postoperative sensitivity, marginal staining, and secondary caries.[5] By placing small resin composite increment thickness (up to 2.0 mm), a reduction in gap formation between the restoration and preparation walls has been observed.[6] In addition, improved optical properties and esthetic outcomes may be achieved by using different composite shades, opacities, translucencies, and a variety of layering techniques.[7]

Bulk-fill resin composites have recently been introduced for insertion into a preparation using one single increment, followed by one light-curing exposure.[8] The main advantages of these products are stated to be lower volumetric shrinkage and shrinkage stress when compared with conventional, methacrylate-based materials[9] and also less chair-side time. A specific sonic handpiece has been developed, in combination with a bulk-fill resin composite (high inorganic content, regular consistency), to improve material adaptation into a preparation. It is thought that the sonication method imparts energy into the material, changing its rheological properties, decreasing viscosity, and leading to enhanced adaptation between resin composite and preparation surfaces, while maintaining mechanical properties similar to those of a hybrid resin composite.[10]

Incorporation of air bubbles and voids in resin composite restorations is an area of concern to all clinicians. The presence of voids may be influenced by material manipulation failures during product manufacturing and packaging, filler size and content, and high-viscosity monomer use, among others.[11-16] An increased amount of voids could result in increased water sorption and, consequently, increased staining.[17] Also, these defects are potential loci of stress concentration and may act as initiation points for fracture and crack propagation, reducing mechanical strength and wear resistance.[17] Voids at the adhesive interface reduce bond strength between composite and an adhesive system, by reducing the total bonded surface area.[18] In addition, higher bacteria retention and biofilm formation could result from voids on the restoration surface.[19]

Void formation has been evaluated by sectioning samples, followed by observation and quantification using a light microscope.[15,20] This process is destructive, tedious, and time-consuming and does not provide quantification of the entire restoration mass. Micro-computed tomography (μCT) analysis has been used for bulk volumetric evaluations (using x-ray scanning), generating 3D rendering and spatial quantification of materials.[21] Thus, this method is proven to be a suitable tool for porosity assessment inside of resin composite restorations.[13,22,23]

The purpose of this study was to quantify porosity within resin composites (regular and bulk-fill) that were placed using either conventional or sonic

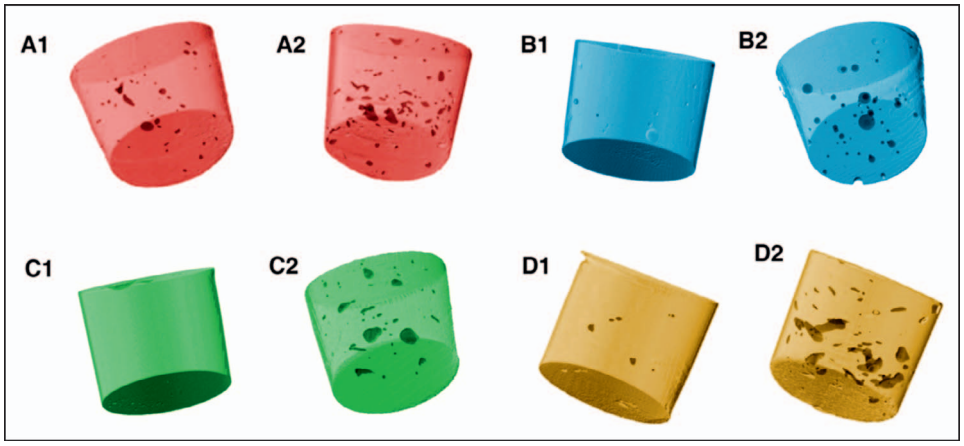

Figure 1. *3D rendering of one representative sample of each composite showing presence of inner voids. A1- Herculite Ultra using conventional insertion method; A2- Herculite Ultra using sonic insertion method; B1- SureFil SDR Flow using conventional insertion method; A2- SureFil SDR Flow using sonic insertion method; C1- Tetric EvoCeram Bulk Fill using conventional insertion method; C2- Tetric EvoCeram Bulk Fill using sonic insertion method; D1- SonicFill using conventional insertion method; D2- SonicFill using sonic insertion method.*

insertion methods and analyzed using μCT technology. The hypothesis tested was that the sonication insertion method would reduce resin composite void formation compared with a conventional placement method.

METHODS AND MATERIALS

Specimen Preparation

Cylindrical molds were three-dimensionally (3D) printed using a white, thermoplastic polymer (acrylonitrile butadiene styrene [ABS], Octave Systems Inc, Santa Clara, CA, USA) having a 4.5-mm internal diameter and a height of 4.0 mm (Figure 1). The internal walls were standardized by using a standard 5-mm drill bit, resulting in parallel, uniform surfaces. A polyester strip (Mylar Type D, 0.08 in., DuPont, Wilmington, DE, USA) was placed under the mold, prior to resin composite placement. Four different resin composites were used (Table 1). Each material was inserted into the molds using two different methods: conventional (according to the manufacturer's instructions) and by extrusion through a sonic delivery instrument (SonicFill Handpiece, speed level 3, Kerr Corporation, Orange, CA, USA).

To fabricate each specimen, one resin composite compule was used. For the sonic insertion method, resin composite material was transferred from each original compule into a SonicFill compule (compatible with the SonicFill Handpiece). The SonicFill compule is unique in that it can be dismantled and reassembled. A SonicFill compule was dismantled, the content was emptied, and the compule form was rinsed and immersed in acetone followed by ultrasonic agitation, to remove any composite residue. Afterward, the curved tip of each evaluated composite compule was removed by using a heavy-duty cutter, and the resin composite content was extruded into one of the previously prepared, empty SonicFill compules, followed by replacement of the piston. All procedures were performed in a light- and temperature-controlled room. Conventional placement consisted of directly extruding the material into the mold from the supplied resin composite delivery device. The plastic mold was filled from bottom to top, followed by vertical pressure, using a metallic spatula (AECRJ2XP, American Eagle, Missoula, MT, USA). A piece of polyester strip was then placed

Table 1: *Restorative Materials Used*

Material	Manufacturer	Shade	Organic Composition[a]	Inorganic Composition[a]	Lot No.
Herculite Ultra	Kerr Corporation	A2	Bis-GMA, TEGDMA, Bis-EMA	SiO_2 glass, oxide (78%wt)	5123035
SureFil SDR Flow	Dentsply International	U	Modified UDMA, TEGDMA, EBPDMA	Ba-Al-F-B-Si glass and Sr-Al-F-Si glass (68%wt)	0511
Tetric EvoCeram Bulk Fill	Ivoclar Vivadent, Inc	IVA	Bis-GMA, Bis-EMA, UDMA	Ba-Al-Si glass, prepolymer filler (monomer, glass filler, and ytterbium trifluoride), spherical mixed oxide (79-81%wt)	S38401
SonicFill	Kerr Corporation	A2	Bis-GMA, TEGDMA, EBPDMA	SiO_2 glass, oxide (83.5%wt)	5135439

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; Bis-EMA, ethoxylated bisphenol A dimethacrylate; UDMA, urethane dimethacrylate; EBPDMA, ethoxylated bisphenol A dimethacrylate.
[a] *Composition information obtained from the manufacturer.*

on the top surface and slightly pressed flat, using a microscope glass slide (Gold Seal, Becton Dickinson and Company, Franklin Lakes, NJ, USA). For the sonic insertion method, sonication was generated by a handpiece attached to a high-speed multiflex connection,[24] and all resin composites were inserted in a single increment ("bulk filled"), with the exception of Herculite Ultra (Kerr Corporation), which was inserted using two separate, 2.0-mm-thick increments, as recommended by the manufacturer. Samples were light cured, using a characterized multiwave (400 nm, 440 nm, and 460 nm) light-emitting diode (LED) light-curing unit (VALO, Ultradent Products Inc, South Jordan, UT, USA), for 40 seconds (n=5), in its regular output mode (1521.2 ± 9.2 mW/cm^2). The distal end of the curing tip was in contact with the polyester sheet during photopolymerization.

μCT Analysis

Each polymerized specimen was scanned using a μCT instrument (μCT40, Scanco Medical, AG, Basserdorf, Switzerland), calibrated using a phantom standard, at 70 KVp/BH 200 mgHA/ccm. Five specimens at a time were placed in the sample holder. The instrument was operated at medium resolution (16 μm/slice) using 70 kVp (114 μA) resulting in approximately 380 slices per sample. Data were analyzed using software (Amira, version 5.5.2, VSG, Burlington, MA, USA). A similar, virtual cylindrical shape was then designed, having the same specimen measurements (5.0 mm in diameter and 4.0 mm height), and its cylinder volume was calculated using the "Material Statistics" software function. Images obtained from samples were subjected to the "Boolean" function, to subtract the mold from the analysis. Using the calculated ideal cylinder volume, image data were cropped to obtain similar cylindrical volumes for analysis. The inner resin composite void volume was calculated using the "Threshold" function, to separate voids from resin composite, and the "Material Statistics" software function determined porosity for each specimen. Using these values, the volume of voids was calculated as a percentage of the total volume of resin composite. Data were analyzed using a two-way analysis of variance (ANOVA) and Tukey post hoc test (IBM SPSS, 22, Armonk, NY, and Chicago, IL), at a preset alpha of 0.05.

RESULTS

Table 2 presents the means and standard deviations of void volumes (in percentages) for each material, as a function of insertion method (regular and sonic). Preliminary analysis of data showed heterogeneous variances between groups. Analyses that homogenized the variances led to similar conclusions to the unadjusted analysis that is presented. The two-way ANOVA indicated that the factors "material" ($p=0.002$) and "placement technique" ($p<0.001$) significantly influenced results. The ANOVA also detected a significant interaction between the major factors ($p=0.002$). The analysis indicated sufficient power for the interaction term (94%).

Table 2: *Mean (SD) Void Volume (%) for Each Composite and Insertion Method*[a]

Material	Insertion Method	
	Conventional	Sonic
Herculite Ultra	1.93 (1.14) Aa	2.53 (0.52) Ba
SureFil SDR Flow	1.64 (0.75) Ab	7.08 (3.72) Aa
Tetric EvoCeram Bulk Fill	0.71 (0.50) Bb	2.55 (0.68) Ba
SonicFill	2.43 (0.36) Aa	2.76 (0.95) Ba

[a] *n=5 specimens per experimental group. Means followed by similar letters (lowercase letters compare insertion method for each material and uppercase letters compare material for each insertion method) are not statistically different ($p>0.05$).*

SonicFill and Herculite ultra resin composites demonstrated significantly higher void percentage compared with Tetric EvoCeram Bulk Fill and Surefil SDR Flow, when using the conventional insertion method ($p=0.0217$). However, when using the sonic device, SureFil SDR Flow showed significantly increased void volume compared with that of Herculite Ultra, SonicFill, and Tetric EvoCeram bulk fill ($p=0.0065$).

Sonication had no significant effect on void volume percentage for Herculite Ultra and SonicFill resin composites, but significantly increased void percentages were observed for SureFil SDR Flow and Tetric EvoCeram bulk fill. Qualitative 3D image analysis of internal void formation showed an increase in void volume for all materials sonically inserted (Figure 1).

DISCUSSION

The sonic insertion method is an alternative to manual extrusion and placement of resin composite. In theory, resin composite sonication would facilitate insertion and increase marginal and internal adaptation, because of a decrease in the material's viscosity, while maintaining original composition and physicochemical properties. The research hypothesis was rejected, because the sonic insertion method resulted in significantly higher void volume for some resin composites, compared with the use of

a conventional insertion method for the same material. An increased void formation may be associated with changes in resin composite rheological properties (reduced viscosity under sonication) from that originally provided by the manufacturers. Sonication tended to increase void formation for some materials evaluated, but not for others, as observed in quantitative and qualitative analyses (Table 2; Figure 1).

Such increased void formation was more evident for the two bulk-fill resin composites evaluated, which were specifically formulated to be applied as a single increment. Bulk-fill resin composites are relatively new materials that usually demonstrate lower volumetric shrinkage,[9] even when applied in 4.0- to 5.0-mm increment thicknesses.[8] The use of these resin composites tends to simplify restorative procedures, reducing chairside time and requiring less manipulation for each increment, which would lead to fewer flaws, as confirmed by this study. Currently, the bulk-fill resin composite SonicFill is the only material recommended by the manufacturer for use with the SonicFill Handpiece. For this material, the sonic insertion method did not significantly increase void volume. Even though the void volume of SonicFill was not significantly changed as a result of the different insertion methods, its void value was still fairly high. Because this material is designed specifically to be dispensed by the sonic handpiece, it seems reasonable that manufacturers could control the innate void volume of the product. In so doing, a denser and less pitted polished surface might be produced, providing enhanced potential for restoration longevity and greater esthetics.[25] Lower mechanical properties have been reported for SonicFill resin composite, when used in association with a sonic handpiece, compared with use of a conventional insertion method.[26]

Use of μCT analysis provided an accurate measurement of the total internal void volume, without the need of destructive sectioning and polishing, usually required for traditional methods.[9,21,23] The 3D rendering allowed void visualization within the intact restorative materials and also provided quantitative measurements.[13] As noted previously, the presence of internal and surface-formed voids can drastically affect restoration properties and appearance. Voids located at tooth-restorative material interfaces may lead to marginal staining and reduced bond strength.[18] In addition, porosity potentially leads to crack formation and consequently to bulk failure.

The conventional insertion method demonstrated a more homogeneous structure for SureFil SDR Flow and Tetric EvoCeram bulk fill compared with that of SonicFill and Herculite Ultra (the latter applied in two separate increments). The conventional layering technique used for the regular resin composite (Herculite Ultra) proved to increase the volume of voids as compared with a flowable, bulk-fill resin composite (SureFil SDR Flow) and high-viscosity, bulk-fill resin composite (Tetric EvoCeram Bulk Fill). Previous studies using high-resolution tomography also detected porosities inside resin composite restorations, reporting detrimental effects of layering application, in terms of porosity.[13,23] These results corroborate with another study, in which the prolonged packing time had a negative effect on the resin composite surface hardness, and conversely, increased mechanical properties were obtained by placing the material in small increments.[4]

In the present study, the lowest void formation was observed using Tetric EvoCeram bulk fill (which is a high-viscosity, bulk-fill resin composite) when applied without sonication. Similar results were observed in another study that reported less porosity with higher-viscosity resins.[13] A reduction in resin composite viscosity, in conjunction with the vibration generated by the sonic handpiece, may lead to a higher incorporation of air within the restoration, which was observed for Surefil SDR Flow, resulting in a high standard deviation. Because of the reduced viscosity of the material (the only flowable composite evaluated), void formation was increased and unpredictable. In addition, the sonication process may cause smaller, isolated bubbles (already present in the material) to coalesce and form a consolidated, large bubble, now termed a *void*. When resin composite recovers its original rheological properties, the entrapped air is more obvious and potentially has a greater effect on the bulk material properties.

The void volume measured in the current work was calculated based on an ideal cylindrical designed in the software. Thus, the void formation was limited to the cylinder volume and did not take into consideration the void formation on the walls of a preparation. Also, only SonicFill compules were used for all the materials, because the respective compule for each material was not compatible with the SonicFill handpiece. Furthermore, only one frequency of sonication was evaluated (SonicFill handpiece), and further studies should be conducted to evaluate the effect of different frequencies and different resin composite formulations on the

potential for void formation. The influence of these voids on mechanical properties of bulk-fill restorations and their clinical implications should be evaluated as well.

CONCLUSIONS

Within the restrictions imposed by the experimental methods used, the following conclusions may be made:

1. There was no significant difference in void volume of SonicFill restorative material when using either the sonic or conventional, manual method of placement.
2. The void volumes of other bulk-fill resin composites (a flowable and a high-viscosity paste) were significantly increased when using the sonic insertion method.
3. Sonic insertion of a 2.0-mm-thick increment of conventional resin composite did not significantly change void volume compared with placement using conventional, manual methods.

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Conflict of Interest

The authors 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.

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Marginal Fit and Retention Strength of Zirconia Crowns Cemented by Self-adhesive Resin Cements

R Pilo • M Folkman • A Arieli • S Levartovsky

Clinical Relevance

The marginal fit of zirconia crowns cemented by self-adhesive resin cements is clinically acceptable, but the retention after severe aging processes is unsatisfactory.

SUMMARY

The absolute marginal gap (AMG) precementation and postcementation and the retention of zirconia crowns cemented to standardized molar preparations (4×10) by self-adhesive resin cements (SARCs) were evaluated. The following SARCs were used: RelyX U-200 (RXU200; 3M ESPE, Seefeld, Germany), SmartCem 2 (SC2; Dentsply, Milford, DE, USA), and G-Cem Automix (GCA; GC, Alsip, IL, USA). The control adhesive resin cement was Panavia 21 (PAN; Kuraray Dental Co Ltd, Osaka, Japan). Twenty measuring locations at a constant interval along the margins were marked, and the AMG was measured by an image analysis system connected to a stereomicroscope (20×). The cemented copings were aged 270 days at 100% humidity and 37°C and then underwent 10,000 thermal cycles, 5°C-55°C. After aging, the crowns were tested for retention, and the debonded surfaces were examined at 3× magnification. The mean marginal gaps precementation and postcementation were 34.8 ± 17.4 μm and 72.1 ± 31 μm, respectively, with no statistically significant differences between the cements. A significant difference ($p \leq 0.001$) in retention between the cements was found. The highest values were obtained for SC2 and GCA (1385 Pa and 1229 Pa, respectively), but these presented no statistically significant differences. The lowest values were found for PAN and RXU200 (738 Pa and 489 Pa, respectively), but these showed no statistically significant differences. The predominant mode of failure in all of the groups was mixed, and no correlations were found between marginal gap and retention.

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INTRODUCTION

All-ceramic indirect restorations, especially those of high-strength ceramic materials such as zirconia, offer esthetically pleasing restorations, even for long-span, fixed partial dentures (FPDs)[1]; however,

their use has made luting procedures more challenging. The aim of cementation is to integrate the restorations with the dental hard tissues, especially with the dentin, supplying retention, marginal sealing, and esthetics.

Zirconia FPDs can be luted with conventional cements (zinc phosphate cement and glass ionomer) only if the appropriate tooth preparation provides sufficient retention. However, the introduction of cements with adhesive capabilities to dental practice has given practitioners the opportunity to increase retention in many other clinical situations (eg, short or tapered tooth preparation) and achieve the desired optimal esthetic results. As a result, particle-reinforced resin composite materials are presently the luting materials of choice. Adhesive bonding systems and resin cements demonstrate superior mechanical properties and stability,[2] reduced solubility,[3] high adhesive strength to restorative materials and dental hard tissues, and improved esthetics.[4] However, the complexity involved in multi-step systems requires simplification to reduce their technical sensitivity and chairside time.[5] Thus, self-adhesive resin composite cements (SARC), which do not require dentin pretreatment, have been developed.[6]

The main components of SARCs are as follows: 1) aromatic and aliphatic dimethacrylate monomers to form a cross-linked network, 2) acidic methacrylate monomers to adhere with enamel and dentin and copolymerize with the cross-linking monomers, 3) glass filler particles or basic compounds to neutralize residual acidic monomers, 4) conventional silanated filler particles to provide strength by an inert reinforcing effect, 5) appropriate catalysts and stabilizers to comply with the dual-cure characteristic and shelf-life requirements, and 6) pigments and opacifiers to fulfill the esthetic requirements.[7,8] The retention strength and marginal fit of zirconia-based ceramic restorations luted by SARCs were extensively investigated as independent variables. Palacios and others[9] reported no statistically significant differences in crown retention values luted by composite resin cement with adhesive agent (Panavia F 2.0 and ED Primer A & B), a resin-modified glass ionomer cement (Rely X Luting), and a SARC (RelyX Unicem). A nonsignificant difference between Panavia F 2.0 and RelyX Unicem was also reported in other studies,[10,11] irrespective of the internal surface treatment of the crowns.[11] The retention was higher only when the RelyX Unicem Aplicap was used[10] and lower when total-etch adhesive resin cement (Duo-Link) was used without crown surface treatment.[10] Ernst and others[12] tested the retention strengths of four resin-cement systems as well. They also found no significant differences in the retention strength values between the compomer, the resin-modified glass ionomer, the SARC, and the adhesive resin luting cements Superbond C&B and Panavia F 2.0.

Three different parameters of marginal fit have been traditionally defined in the literature: *vertical marginal discrepancy* measured parallel to the path of draw of the casting and *horizontal marginal discrepancy* measured perpendicular to the path of draw of the casting. The angular combination of the vertical and horizontal marginal discrepancies has been classified as the *absolute marginal discrepancy*.[13] These terms are difficult to define in underextended and overextended cases without sectioning. For simplicity, most current clinical studies divide these parameters into the following: *marginal gap* (vertical marginal discrepancy), which is the perpendicular distance from the internal surface of the coping to the margin of the preparation, and *absolute marginal gap* (AMG; absolute marginal discrepancy), which is the distance from the external edge of the coping margin to the cavosurface of the preparation finish line.[14-16] The latter definition was adopted in the current study.

The marginal fit is still one of the most important criteria for the clinical success of all-ceramic FPDs. A large marginal discrepancy can compromise the longevity of the restoration, not only because of caries but also because of periodontal disease.

Copings of zirconia-based ceramic restorations are routinely produced by computer-aided design and computer-assisted manufacturing (CAD-CAM). These CAD-CAM restorations are popular because they provide reproducible results with high esthetic value, short fabrication time, and reduced technical errors.[15] Numerous studies have reported an absolute marginal discrepancy of <90 μm with various CAD-CAM systems,[16-20] all within the acceptable clinical limit of 120 μm;[17,21] however, few have tested this variable precementation and postcementation.[17,22]

The marginal seal capability of cemented all-ceramic crowns with self-adhesive resin cement in comparison with other cements is still under study. Yuksel and others[23] reported a lower level of microleakage with the SARC compared with glass ionomer luting cement. Similarly, Behr and others[24] have reported that the SARC had significantly lower dye penetration in comparison with resin cement with a

smear layer–removing adhesive system and compomer cement with a smear layer–dissolving adhesive system. Ghazy and others[25] have demonstrated that the resin cement with separate primer/bonding agent results in significantly lower microleakage scores, irrespective of crown material. Rosentritt and others[26] have found no significant difference in marginal integrity between the SARC and conventional resin cements after total etching, priming, and bonding.

Although the marginal seal is a cement material–dependent property, it is strongly linked to the marginal gap precementation and postcementation because the higher the marginal gap, the less probability of perfect sealing. The marginal gap of cemented CAD-CAM zirconia-based FPDs depends on the design of the finishing line,[19] the impression technique,[20] the type of system used,[16,17] and the cement space.[27] Given that the aim of cementation is to concomitantly enhance the retention and marginal fit, these variables should be investigated in the same study. However, no study has addressed both of these variables in CAD-CAM zirconia-based FPDs cemented by SARCs.

Therefore, the aims of the current study were 1) to evaluate the marginal fit of CAD-CAM zirconia crown copings before and after cementation by three types of SARCs and a control adhesive resin cement; 2) to test the retention strength of these crowns postcementation; and 3) to correlate the marginal fit with the retention values.

The null hypotheses tested were as follows: 1) there is no difference in AMG before or after cementation or in retention between the different cements; and 2) no relationship between retention and marginal fit exists.

METHODS AND MATERIALS

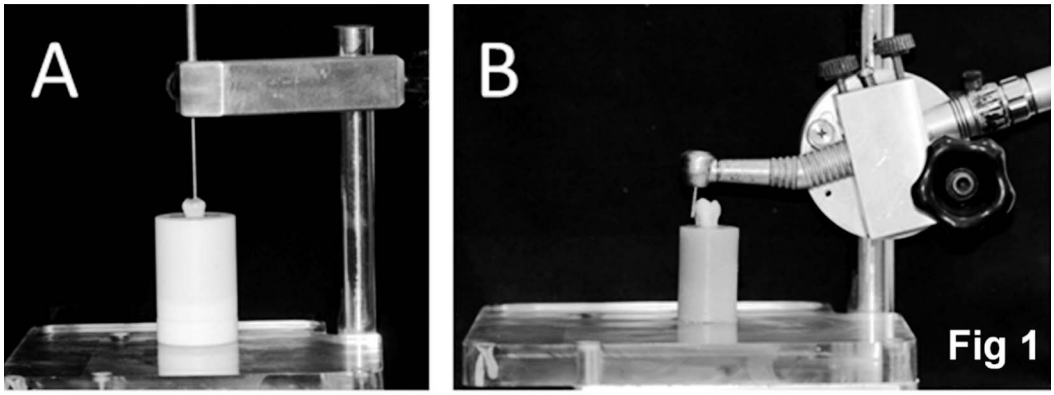
A total of 40 freshly extracted, caries-free, intact molars were collected for the study. The study protocols were approved by the Tel Aviv University Ethics Committee. The patients were informed about this study and consented to the use of their teeth. All external debris was removed by curettes, and the teeth were stored in a germ-free 0.1% thymol solution at room temperature for three days. They were then switched to tap water for no longer than three months until experimentation. The roots of the teeth were notched for retention purposes by a diamond bur (C1, Strauss, Ra'anana, Israel) and embedded parallel to the long axis of the tooth with a custom-designed alignment apparatus. Each tooth was suspended in the middle of a polytetrafluoroethylene (Teflon) ring and mounted 2 mm apical to the cementoenamel junction (CEJ) in a polymethyl methacrylate resin (Quick Resin, Ivoclar Vivadent, Schaan, Liechtenstein) (Figure 1a). After mounting, the teeth were stored in tap water at room temperature.

All teeth were prepared according to the following standardized protocol. The occlusal surface was sectioned perpendicular to the long axis, 6 mm above the CEJ, with a water-cooled precision saw (Isomet Plus, Buehler, Lake Bluff, IL, USA). A 0.4-mm, 360° chamfer finish line located 1 mm above the CEJ with a 10° taper was prepared with a rigidly secured high-speed handpiece equipped with a diamond bur (C1, Strauss) mounted on a custom-designed surveyor-like apparatus (Figure 1b). Final preparation took place exclusively in dentin, and a new bur was used for each tooth.

Impressions of the prepared teeth were made using a two-step technique. The preliminary impression was made with a copper ring filled with an acrylic material (Unifast, GC, Alsip, IL, USA). In the second step, an acrylic layer with a thickness of 2 mm was removed from the inner aspect of the ring, and an occlusal vent hole was created. A condensation silicone wash impression material (Xantopren, Heraeus Kultzer, Hanau, Germany) was dispensed with an automatic mixing syringe. To compensate for the difference between room and mouth temperatures, the setting time was doubled. The impressions were poured with a type 4 dental stone (Silky Rock, Whip Mix, Louisville, KY, USA) mixed with a Multivac (Degussa, Essen, Germany) on a vibrating machine (Sun, Barst, Las Vegas, NV, USA).

A total of 40 crown copings were produced using CAD-CAM technology with Lava frame zirconia blocks (3M ESPE, Seefeld, Germany). The Lava CAD-CAM system includes an optical scanner (Lava Scan), a CAM machine (Lava Form), and a sintering oven (Lava Therm). CAD-CAM zirconia copings of 0.5-mm axial and 1.0-mm occlusal thickness were milled at a commercial dental laboratory (Lava Milling Centre, Dental Centre, Tel Aviv, Israel). A virtual spacer layer of 50-μm thickness 0.5 mm short of the margins was planned. The Lava zirconia coping was designed with a loop (4-mm outer diameter and 2-mm inner diameter) extending coronally from the occlusal surface (Figure 2a).

Prior to cementation, the areas of the axial and occlusal surfaces of each prepared tooth were measured as previously described by Pilo and



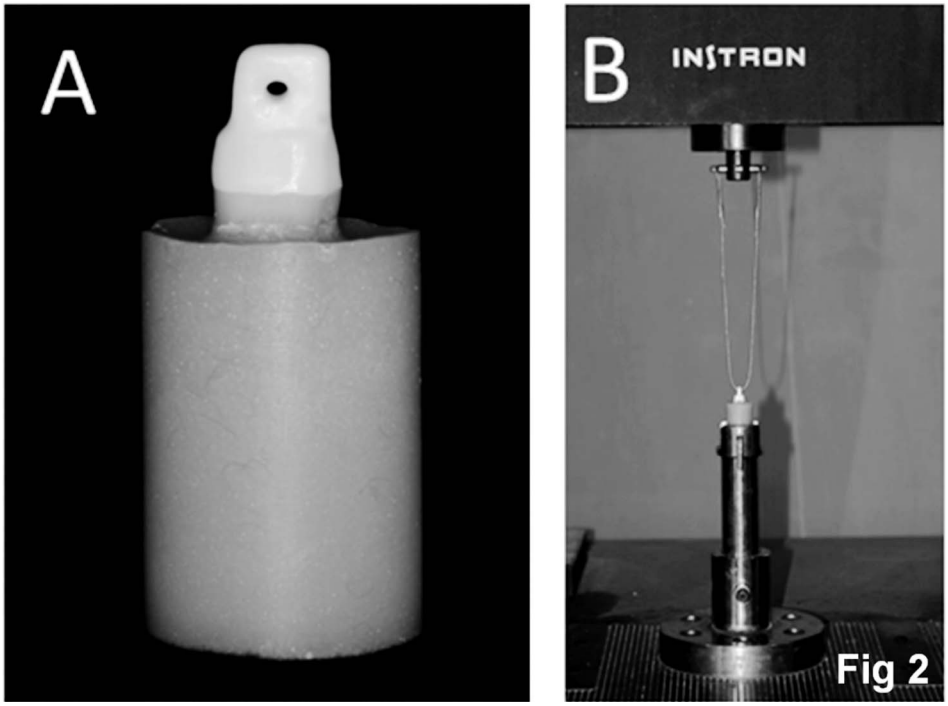


Figure 1. *Sample preparation. (A): Teeth were suspended in the middle of a polytetrafluoroethylene (Teflon) ring and mounted 2 mm apical to the cementoenamel junction in a polymethyl methacrylate resin. (B): Surveyor-like apparatus for the standardized preparation of mounted extracted teeth.*
Figure 2. *Retention strength experiment. (A): Zirconium oxide coping showing the design of the occlusal surface. (B): Metal cable connecting the coping with the universal testing machine.*

others[28] The AMG precementation was measured with a Bioquant Osteo image-analysis system using digital photographs acquired with a stereomicroscope (20×, M8, Wild, Heerbrugg, Switzerland). For all measurements, the specimens (copings on respective teeth) were placed on a metal device to secure the copings while applying a uniform load of 10 N. The marginal gap was measured at 20 measuring locations at an approximately constant interval along the margins of the tooth. The mean marginal gap width for each tooth was calculated. The locations were marked by an indelible marking pen (Lumocolor Permanent, Staedtler Mars GmbH, Nürnberg, Germany).

The 40 prepared teeth were randomly assigned to four groups (4×10: 3 SARCs and a control), according to the cement type used: RelyX U-200 (RU200; 3M ESPE, Seefeld, Germany), SmartCem 2 (SC2; Dentsply, Milford, DE, USA), G-Cem Automix (GCA; GC, Alsip, IL, USA), and Panavia 21 (PAN; Kuraray Dental Co Ltd, Osaka, Japan) (Table 1). The intaglio surfaces of the crowns were not sandblasted but were cleaned with Ivoclean (Ivoclar Vivadent). All cements were mixed following the manufacturers' instructions. The cement was placed on half of the axial surfaces of the copings, simulating the clinical procedure. Each coping was aligned and seated on the respective tooth with firm

Table 1: *Resin Cements, Manufacturers, and Chemical Composition*

Resin Cement	Manufacturer	Composition
RelyX U200	3M ESPE, Seefeld, Germany	Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheologic additives, alkaline fillers, pigments
SmartCem 2	Dentsply, Milford, DE, USA	Urethane dimethacrylate, phosphoric acid-modified acrylate resin, barium boron fluoroaluminosilicate glass, organic peroxide initiator, camphorquinone, phosphene oxide, accelerators, butylated hydroxytoluene, UV stabilizer, titanium dioxide, iron oxide, hydrophobic amorphous silicon dioxide
G-Cem Automix	GC, Leuven, Belgium	Fluoroaluminosilicate glass, initiator, pigments, 4-META, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer
Panavia 21 + ED Primer A&B	Kuraray Dental Co Ltd, Osaka, Japan	HEMA, MDP, 5-NMSA, water, accelerator, ethanol, MPTS, initiator
		MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, fillers, BPO, hydrophilic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfonate

Abbreviations: BPO: benzoyl peroxide, *HEMA:* hydroxylethyl methacrylate, , *MDP*: methacryloyloxydecyl dihydrogen phosphate, *4-META*: 4-mathacryloylethyl trimellitate anhydride, *MPTS*: methacryloyloxypropyltrimethoxysilane, *5-NMSA*: 5-N-methacryloyl 5-aminosalicylic acid, *UDMA*: urethane dimethacrylate,

finger pressure according to the previously made markings and mounted under a constant load of 50 N (force gauge FG 20, Lutron, Taipei, Taiwan) for 10 minutes. The excess cement was removed with an explorer.

Marginal gaps were measured at the same locations with the same methodology 24 hours postcementation.

The cemented copings were stored in 100% humidity at 37°C for 270 days and subsequently underwent thermal cycling between temperatures of 5°C and 55°C for 10,000 cycles with a 10-second dwell time (Y. Manes, TA, Israel). The crown copings were subjected to dislodgment forces through a 1.2-mm diameter metal cable entangled through the coronal loop along the apico-occlusal axis until failure using a universal testing machine (Model 4502, Instron Corp, Buckinghamshire, UK) at a crosshead speed of 1 mm/min (Figure 2b). The force at dislodgment was recorded and divided by the total surface area of each preparation to yield the retention values (Pa).

Examination of the debonded surfaces of the teeth and crowns was performed under 3× magnification (Orascoptic HiRes 3, Middleton, Wi, USA). Each matched surface of the dentin-crown was analyzed separately (five surfaces per tooth).

Failure was classified according to the criteria presented in Table 2.

The data for the AMG precementation and post-cementation were analyzed using a two-way analysis of variance (ANOVA) with repeated measures. The dependent variable was the mean marginal gap. The type of cement (n=4) and precementation/postcementation (n=2) served as the independent variables.

The data for the retention strength were analyzed using a one-way ANOVA with repeated measures. The Scheffé test for multiple comparisons was used; the dependent variable was the mean retention strength, and the independent variable was the type of cement used. The significance level for both tests was set to α=0.01.

Premarginal and postmarginal gap values were correlated to the respective retention values with the Pearson correlation coefficient. The significance level was set at α=0.05.

RESULTS

Table 3 presents the mean AMG (±SD) before and after cementation of all cementation groups, which for all groups combined were 34.83 ± 17.4 μm and 72.00 ± 31.22 μm, respectively. Analysis of variance

Table 2: *Classification of Failure Criteria*

Criteria	Description
Mixed mode	Adhesive and cohesive cement equally
Mixed (crown dominant)	Mixed but mainly adhesive cement-crown
Mixed (dentin dominant)	Mixed but mainly adhesive cement-dentin
Adhesive cement-dentin	Cement principally on crown surface
Adhesive cement-crown	Cement principally on dentin surface
Cohesive cement	Cement equally distributed on dentin and crown

Table 3: *Mean and Standard Deviation (SD) for Absolute Marginal Gap Before and After Cementation of All Cementation Groups*

Cement	Precementation	SD	Postcementation	SD
RelyX U-200	31.5	15.03	69.9	30.06
SmartCem 2	31.9	10.74	74.1	20.66
G-Cem Automix	43.3	20.16	76.1	32.17
Panavia 21	32.4	21.36	68.0	42.57
Total	34.8	17.40	72.0	31.22

with repeated measures revealed that this difference was statistically significant ($p<0.001$).

However, within each of the four cementation groups, the difference in AMG precementation (ie, as fabricated) and postcementation (ie, influence of the cements) was not statistically significant ($p=0.454$), and no interaction was found between the times (pre/post) and cements (0.379).

Figure 3 presents the mean retentive strength (±SD) for the different cementation groups. There were significant differences ($p\leq0.001$) in mean coping removal stress among the different groups. The highest mean dislodgment stresses were observed for SC2 and GCA (1385±615 Pa and 1229±491 Pa, respectively), which were not significantly different from each other, followed by 738±395 Pa for PAN and 489±265 Pa for RXU200, which were not significantly different from each other.

The failure modes of the different cementation groups are presented in Figure 4. Examination of the failure mode under magnification revealed that the main mode of failure for all cementation groups was mixed mode or mixed (crown dominant), implying that the failure was between the cement and crown in most surfaces, and thus most of the cement was trapped on the dentin. However, the cement group with the lowest retentive values, RXU200, was the only group to exhibit the pure adhesive cement-crown mode of failure (50%), whereas the groups with the highest retentive values, GCA and SC2, were the only ones to exhibit the pure adhesive cement dentin mode of failure, in 50% and 25% of the surfaces, respectively. PAN exhibited equal failure in mixed mode and mixed (crown dominant). SC2 was the only cement exhibiting the mixed (dentin dominant) mode of failure (approximately 30%).

The Pearson correlation coefficient test yielded nonsignificant low correlations between the before ($r=-0.14$, $p=0.37$) and after ($r=-0.33$, $p=0.17$) AMG values and their respective retentive values. The

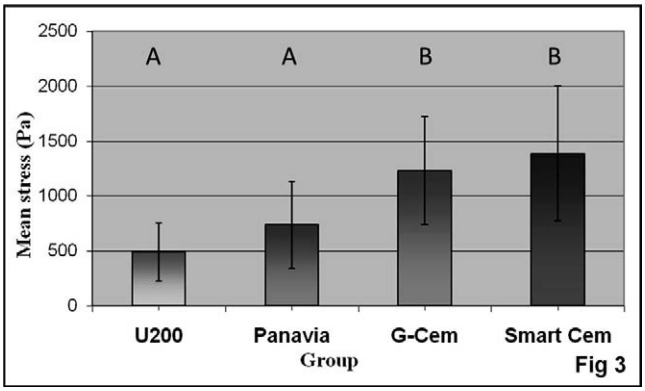


Figure 3. *The mean retention strength (Pa) (±SD) for the different cementation groups. Groups with identical letters presented no statistically significant differences.*

differences between the after and before AMG values and the respective retentive values also gave low, nonsignificant coefficients ($r=-0.34$, $p=0.11$).

DISCUSSION

The AMG of the zirconia crown copings (CAD-CAM) examined before and after cementation did not differ significantly among the four cements examined. The results thus support our first null hypothesis. The mean AMG values of 35 μm precementation and 72 μm postcementation are all within the limits of clinical acceptability. A limit of up to 120 μm was first set for cast gold copings[29,30] but is also widely accepted for all-ceramic crowns.[17,21]

The AMG depends on the type of system used.[16,17] Our results with Lava correspond well to previous reports of 24-87 μm for this system.[15,19,31-33] Karatasli and others[15] compared the marginal adaptation of Lava copings (CAD-CAM) with that of Celay and Zirkonzahn copings (MAD/CAM), with casted metal copings as the control. The lowest marginal gap was observed in the Lava copings (24.6±14.0 μm), whereas the highest marginal gaps were observed in the Zirkonzahn (112.11±22.6 μm) and metal (120.1±33.1 μm) groups. Another study[34] that examined three different all-ceramic systems manufactured by CAD/CAM showed that the Lava system produced gap measurements (approximately 46 μm) that were statistically smaller than those obtained using the Everest (65 μm) and Procera systems (approximately 62 μm). Reich and others[35] compared the marginal gap of Lava three-unit FPDs (65 μm) with those of conventional FPDs (54 μm) and concluded that the clinical fit of all CAD/CAM systems tested competed well with that of conventional systems. The small differences in marginal

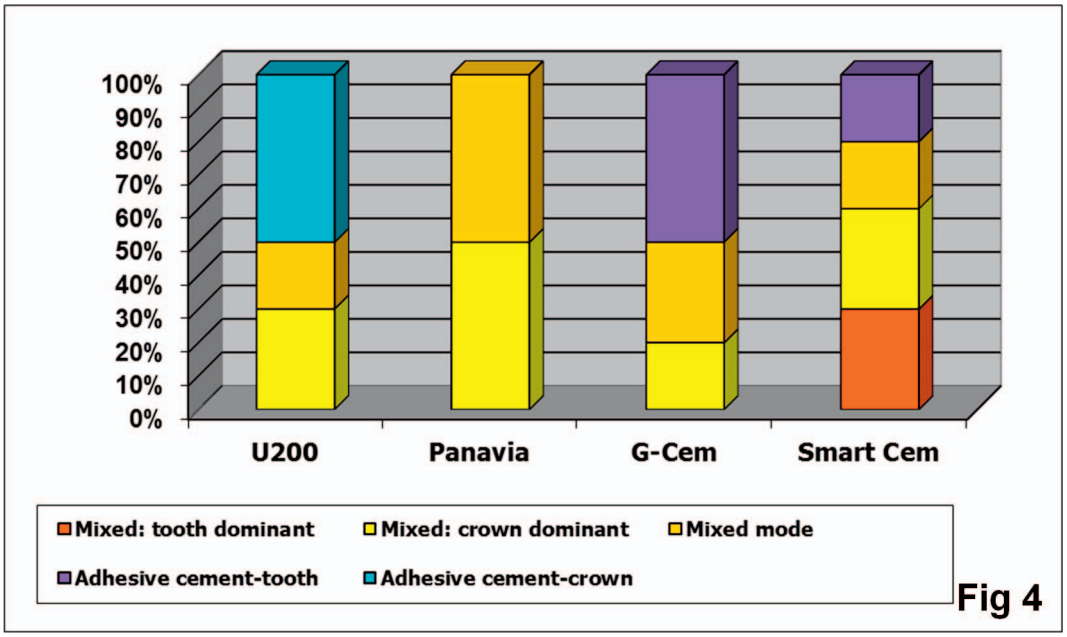


Figure 4. *The failure modes of the different cementation groups.*

gaps among the aforementioned studies are attributable to either variations in fabrication or differences in the virtual cement space,[27] which was not reported in most research. In our study, a virtual spacer width of 50 μm was set 0.5 mm short of the margins.

The AMG should be examined before and after cementation because elevation of the crown might occur during cementation, resulting in open margins and exposure of a large amount of cement to oral fluids.[36] Incomplete seating of crowns postcementation has been extensively studied and reviewed[37] for cast metal–based crowns. The complications caused by incomplete seating include the creation of premature contacts, alteration of contact areas, reduction of retention, and dissolution of extra cement with secondary caries.[37] However, few studies have examined the marginal gaps of all-ceramic crowns precementation and postcementation.[17,22]

Gonzalo and others[17] reported that cementation of zirconia crowns with glass ionomer cement did not cause a significant increase in the vertical marginal discrepancy (3-8 μm) for three different systems. In contrast, in the current work, there was a statistically significant difference in the mean marginal gaps (37 μm) between precementation and postcementation for all cements. However, the two studies differ in methodology: Gonzalo and others used standardized stainless steel abutments, whereas the current study used natural teeth.

Martínez-Rus and others[22] studied the effect of two luting cements, resin and glass ionomer cement, on marginal openings among four all-ceramic systems. The AMD of the crowns was measured before and after cementation with scanning electronic microscopy. In accordance with the current study, they observed statistically significant differences in the mean marginal openings before and after cementation. The resin cement resulted in larger marginal discrepancies compared with those of the glass ionomer cement. Zirconia copings (Y-TZP) produced a more favorable marginal fit than CAD-CAM and pressed lithium disilicate copings cemented with resin cement on implant abutments.[38] Similar to the current study, the difference in the marginal discrepancy of the Y-TZP copings between precementation and postcementation was 46 μm.[38] In summary, based on the findings of this *in vitro* study, self-adhesive resin cements with 50-μm virtual cement space cause only slight elevation of the crown within the limits of clinical acceptability.

However, marginal leakage postcementation depends not only on the amount of cement exposed but also on the solubility and adhesive properties of the cement. Accordingly, Gu and others [39] demonstrated that the adhesive composite resin luting system resulted in the least leakage when zinc-phosphate cement, compomer cement, and adhesive composite resin cement were evaluated for marginal discrepancies and leakage of all-ceramic crowns.

Zirconia crown copings can be luted with conventional cements (zinc phosphate cement and glass ionomer) but attain retention only in favorable tooth preparations by mechanical interlocking of the luting agent in the irregularities present on the surfaces of both the restoration and the tooth. However, the lack of adhesion and the absence of a chemical bond to the tooth structure result in poor retention compared with the SARCs.[40,41] The second part of our study aimed to evaluate the retentive strength of the Lava zirconia crown copings after cementation with three SARCs and resin cement with self-etch primer as a control after nine months of aging in 100% humidity. Our null hypothesis for this study was rejected because the mean coping removal stress was significantly different among the four groups. The highest mean dislodgment stresses were observed for SC2 and GCA (approximately 1.4 MPa and 1.2 MPa, respectively), and the lowest were observed for PAN and RXU200 (approximately 0.7 MPa and 0.5 MPa, respectively). The relatively high SD reflects the variability between the teeth and is common with the results of experiments performed with natural teeth,[9,11,12] but a significant difference could be detected. These removal stress values were appreciably lower than those reported in other studies evaluating the retentive strength of identical cements[9,12,42,43] for luting zirconia crowns, which reported values of 4.8-9.7 MPa. These differences might be attributable to the surface treatment method and aging. The manufacturers of PAN and RXU200 recommend sandblasting the inner surface of the crown before cementation, whereas the manufacturers of SC2 and GCA do not require this step. For the purpose of standardization, we can either include or omit that step for all of the cements. The issue of sandblasting the intaglio of the crown is controversial because it has the potential to create surface microcracks that can decrease the strength of the zirconia.[41,44] We therefore decided to omit this step for all of the cements for the purpose of standardization. However, previous studies that have reported much higher retentive values treated the copings by either airborne-particle abrasion with 50- μm aluminum oxide (Al_2O_3),[9,43] sandblasting with 100-μm alumina,[42] or tribochemical coating with silica.[12,42] The value of grit-blasting the zirconia surface with 50-μm Al_2O_3 as a simple and effective treatment for improving retention compared with no treatment was recently reported by Şanlı and others.[44] Moreover, Inokoshi and others[45] conducted a meta-analysis of bonding effectiveness to zirconia and reported a trend of higher bonding effectiveness when zirconia was pretreated by either Al_2O_3 sandblasting or tribochemical silica coating, particularly compared with studies in which zirconia underwent no mechanical pretreatment. However, when deciding to improve mechanical retention by grit-blasting, the particle size must be limited to minimize subsurface damage and the risk of cracking the zirconia substrate.[44]

In our study, the cemented copings underwent 100% humidity at 37°C for 270 days and were subsequently thermal cycled between water temperatures of 5°C and 55°C for 10,000 cycles. In studies reporting much higher retentive values, the aging processes involved much shorter times of 24 hours,[9] one week,[12] or 14 days.[42,43]

A few recent studies have reported results similar to our results after prolonged aging. De Sá Barbosa and others[46] evaluated the microshear bond strength of four SARCs to yttria-stabilized tetragonal zirconia surfaces after water storage for 24 hours or one year. The latter period significantly decreased the microshear bond strengths to zirconia for all cements by 53%-91%. Da Silva and others[47] compared the bond stability of RelyX adhesive resin cement (conventional) and RelyX Unicem (self-adhesive) resin cement with a yttria-stabilized tetragonal polycrystalline zirconia (ceramic submitted to two surface treatments after six months of aging in water). Irrespective of surface treatments, the self-adhesive resin cement was not able to maintain the bond to Y-TZP ceramic after six months of aging in water, with a decrease in retentive values of 43%-93%.[47] Of note, the RelyX SARC family exhibited the highest extent of degradation, >90%.[46,47] Degradation of RelyX Unicem after aging was also reported by Perdigão and others[48] and de Melo and others.[49] Thus, it is quite reasonable that the severe aging processes in the current study influenced the SARCs as well as the control and caused their degradation and hydrolysis. This conclusion was reinforced by a recent study by our group in which the retentive strength of zirconia crowns was tested using an identical methodology.[50] The crowns were cemented with RXU200 without sandblasting and tested after only 30 days. The retentive values obtained were higher, 2.28 MPa, because no aging over a prolonged period was performed.[50] Examination of the failure mode revealed that the main mode of failure for all cementation groups was mixed mode. However, the groups with the lowest retentive values (RXU200 and PAN) exhibited dominance of surfaces with failures between the cement and crown, whereas the groups with the highest retentive values (GCA and SC2) exhibited dominance of surfaces with failures

between the cement and dentin. These observations suggest that, as per the manufacturers' recommendations for RXU200 and PAN, it is crucial to sandblast the intaglio surfaces of the Y-TZP crowns to avoid adhesive cement-crown failures. An appreciable improvement in the adhesion of RXU200 to zirconia by surface grit blasting was recently reported,[44,47] whereas adhesive cement-crown failure is mainly anticipated without grit-blasting.[50] An alternative option to increase the retention between RXU200 and the zirconia substrate is the use of zirconia primers.[51]

It is anticipated that greater incomplete seating postcementation will reduce retention, based on studies of gold castings cemented by zinc phosphate cement showing a reduction of 19%-32% in crown retention due to incomplete seating.[52] The results of the current study support our second null hypothesis, given that no relationship was observed between retention and AMG. The before and after AMG values, as well as the differences between them, gave low negative nonsignificant Pearson correlation coefficients with retention. Although the negative signs indicate that an inverse relationship exists, the low and nonsignificant correlations suggest a weak relationship. With the current CAD-CAM crowns, the predictable preplanned 50-μm virtual spacing provided sufficient cementation space for the excess cement. By contrast, the manually provided 20- to 40-μm die spacer for cast gold crowns was unpredictable.[37]

CONCLUSIONS

We have shown that AMGs of LAVA Y-TZP crowns as fabricated (35 μm) and after cementation by self-adhesive resin cement (72 μm) were within the limits of clinical acceptability of 120 μm. This limit was traditionally set for cast gold copings but is also widely accepted for all-ceramic crowns. Self-adhesive resin cements and resin cement with self-etch primer are anticipated to cause minimal crown elevation postcementation provided that a virtual spacer of 50-μm thickness 0.5 mm short of the margins is set. In contrast, the retention strength of these Lava Y-TZP crowns after severe aging conditions yielded unsatisfactory values (<1.4 MPa), which greatly differed between the cements. The lowest values (<0.74 MPa) were found for RXU200 and PAN, which exhibited mainly adhesive cement-crown failures. These values could have been improved by sandblasting the intaglio surfaces of the Y-TZP crowns. No relationships were found between retention and AMG.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Tel Aviv University. The approval code for this study is 13.11.2015.

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.

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Influence of Different Restoration Techniques on Fracture Resistance of Root-filled Teeth: *In Vitro* Investigation

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Clinical Relevance

Endodontically treated teeth with large mesio-occluso-distal cavities may be restored with different techniques to enhance structural integrity.

SUMMARY

Objective: The purpose of this study was to determine the fracture strength of endodontically treated mandibular premolar teeth restored with composites and different reinforcement techniques.

Methods and Materials: Forty-eight freshly extracted human mandibular premolar teeth were randomly divided into four groups: group IN, group CR, group FRC, and group PRF. Group IN consisted of teeth with intact crowns and served as the control group. In the other three groups, endodontic treatment was performed and standard mesio-occluso-distal (MOD) cavities were prepared. Then cavities were restored with hybrid resin composite only, flowable composite and hybrid resin composite, and Ribbond, flowable composite and hybrid resin composite in groups CR, FRC and PRF, respectively. All of the teeth were subjected to fracture by means of a universal testing machine, and compressive force was applied with a modified stainless-steel ball at a crosshead speed at 0.5 mm/min.

Results: The highest values were observed in group IN, while the lowest values were determined in group CR. There was not any statistically significant difference between group CR and group FCR ($p>0.05$). When groups CR, FCR, and PRF were compared, group PRF showed significantly better fracture strength than did groups CR and FCR ($p<0.05$). It was determined that there was not any significant

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difference between group IN and group PRF ($p>0.05$).

Conclusions: Polyethylene ribbon fiber considerably increases the fracture strength of mandibular premolar teeth with MOD cavities restored with composite.

INTRODUCTION

Endodontically treated teeth (ETT) are more prone to fractures compared to teeth with healthy pulps because of dentin dehydration and hard tissue loss.[1,2] There is a significant decrease in strength and fracture resistance of ETT, especially with the preparation of wide mesio-occluso-distal (MOD) cavities, so root canal treatment should not be considered complete until the teeth are restored permanently.[1-3] Previous studies[4,5] proposed that restorations improve structural integrity and increase the prognosis of ETT when subjected to considerable masticatory occlusal loads. Although there is no agreement on the desired type of restoration for ETT, it should reproduce anatomical form, esthetics, and function in addition to stopping bacterial microleakage, ensuring periodontal health, and protecting the remaining tooth structure against occlusal loads and antagonistic teeth.[6-8] To avoid failure of the root canal treatment, an easy, high-strength, fast, direct, and cost-effective restorative method may be appropriate.

Adhesive technology is progressing every day, making it possible to create conservative and highly esthetic restorations with direct bonding to the teeth. Flowable composites are preferred by clinicians because of the better adaptation they offer; they act as an extendable and flexible intermediate layer with a low modulus of elasticity. They involve less filler (60%-70% by weight and 46%-70% by volume) and a larger percentage of resin matrix than hybrid resins. Flowable resin composite applied before the placement of restorative material may form an elastic liner.[9,10]

Recent improvements in resin-bonded composite properties have encouraged clinicians performing adhesive restorations of ETT. Ribbond (Ribbond, Seattle, WA, USA) is a polyethylene fiber that has an ultrahigh elastic modulus. These fibers increase the flexural properties of composite resin, have similar elastic modulus to dentin, provide better levels of fatigue resistance, and permit effective force transmission.[11] In addition, Ribbond's easy manipulation and adaptation to the contours of the teeth make it a material of choice. In addition, it was reported that Ribbond behaves in such a manner that it distributes stresses and absorbs energy. It reduces the stress concentrations via distributing forces over a larger area, which in turn stops crack formation and propagation. In addition, it absorbs the energy from repeated occlusal effects.

The purpose of this study was to determine the fracture strength of endodontically treated mandibular premolars restored with composites and different reinforcement techniques.

The null hypothesis of the study was that the fracture resistance of MOD composite restorations in root-filled teeth is not affected by different reinforcement techniques, such as use of flowable composites or Ribbond.

METHODS AND MATERIALS

This study was approved by the Istanbul University Faculty of Dentistry Ethical Committee. Forty-eight single-rooted human mandibular premolar teeth with similar dimensions (mesiodistal: 5.08±0.5 mm; buccolingual: 7.15±0.7 mm) extracted for orthodontic reasons were used. The completely formed teeth were extracted from donors aged 18-30 years. The teeth were cleaned of debris and soft tissue remnants immediately after extraction and kept in saline solution for 24 hours. The samples were divided randomly into four groups (n=12 each).

Group IN consisted of intact teeth without any cavity preparations or endodontic treatment. In the other three groups, all of the teeth had endodontic treatment first. Endodontic access cavities were prepared with diamond burs at high speed, and the pulp tissues were extirpated. The working length of each tooth was determined using #15 K-files (Kendo, VDW, Munich, Germany), and all the teeth were instrumented until an apical size of #35 with K-files. The step-back technique was used to give a taper with H-files #40, #45, and #50 (Kendo). During the preparation, the root canals were irrigated with 2 mL of 5.25% NaOCL before each file was introduced into the canal. After the instrumentation and irrigation, root canals were dried with absorbent paper points (META BIOMED Co, Ltd, Chungbuk, Korea) and obturated with gutta-percha (GP; META BIOMED Co, Ltd) and AH Plus sealer (Dentsply De Trey, Konstanz, Germany) using a cold lateral condensation technique. Excessive coronal root canal filling materials were removed with heated instruments and then GP was removed 1 mm apically from the canal orifices and covered with light-cured,

Table 1: *Types of Materials Used in Coronal Restorations and their Compositions*

Type of Materials	Lot No.	Manufacturers	Compositions
Clearfil SE Bond	000165	Kurary Co, Ltd, Japan	Primer: MDP, HEMA, hydrophilic dimethacrylate, *N,N*-diethanol-*p*-toluidine, water Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, CQ, *N,N*-diethanol-*p*-toluidine, silanated colloidal silica
Clearfil Liner 2V Bond	000002	Kurary Co, Ltd, Japan	Bond liquid A: Bis-GMA, HEMA, MDP, hydrophobic dimethacrylate, CQ, accelerators
Clearfil Majesty Flowable Composite	3K0009	Kurary Co, Ltd, Japan	Silanated barium glass filler, silanated colloidal silica, TEGDMA, hydrophobic aromatic dimethacrylate, DL-CQ. The total amount of inorganic filler is approximately 62 vol%, particle size of inorganic filler ranges from 0.02 μm to 19 μm
Clearfil AP_X Composite	630062	Kurary Co, Ltd, Japan	Bis-GMA, TEGDMA, silanated barium glass filler, silanated silica filler, silanated colloidal silica, DL-CQ. The total amount of inorganic filler is approximately 71 vol%, particle size of inorganic filler ranges from 0.02 μm to 17 μm
Ribbond	9592	Ribbond Inc, Seattle, WA, USA	Ultra–high molecular weight polyethylene, Homopolymer H-$(CH_2\text{-}CH_2)n$-H

Abbreviations: Bis-GMA: bisphenol A glycidyl dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, HEMA: Hydroxyethyl methacrylate

resin-modified glass ionomer cement (GIC; Fusion I seal, PREVEST Denpro, Kasmir, India) 1.5 mm coronal to the cemento-enamel junction (CEJ). After all of these steps were completed, standard MOD cavities were prepared. The thicknesses of the remaining buccal and lingual walls were standardized to 2.5 ± 0.2 mm, and the height from base of the fissure to the GP was standardized to 3 mm. The height of the axial walls from the proximal sides was approximately 1.5 mm. Following MOD preparation, the teeth were divided randomly into three experimental groups.

The materials used in these experimental groups and their compositions are described in Table 1.

Group Composite Resin (CR) (n=12 Samples)

The cavities were washed and dried with air water sprays. After priming for 20 seconds (SE Primer; Kuraray, Tokyo, Japan) the cavity surfaces were gently dried. SE Bond (Kuraray) was applied to the cavity surfaces and cured for 10 seconds. The cavities were then restored with resin composite (Clearfil AP-X, Kuraray) using an incremental technique and each layer was cured for 20 seconds (Figure 1).

Group Flowable Composite Resin (FCR) (n=12 Samples)

After completion of priming and bonding procedures with Clearfil SE Bond, as for group CR, the pulpal floors of the cavities were coated with a layer of low-

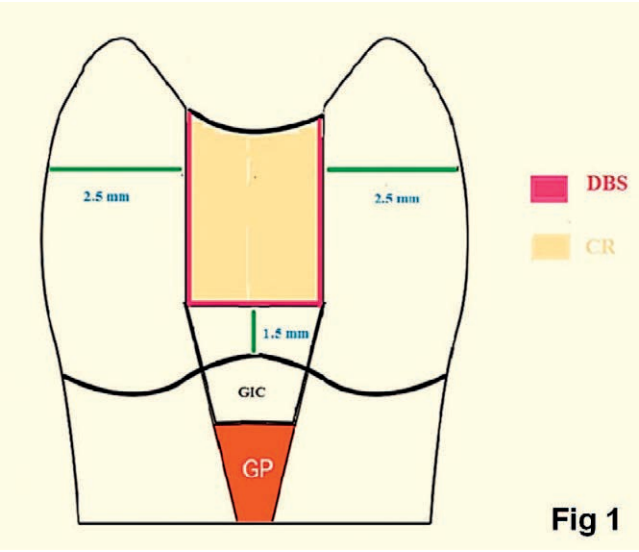

Figure 1. *The restoration of teeth in group CR with DBS (dentin bonding system) and CR.*

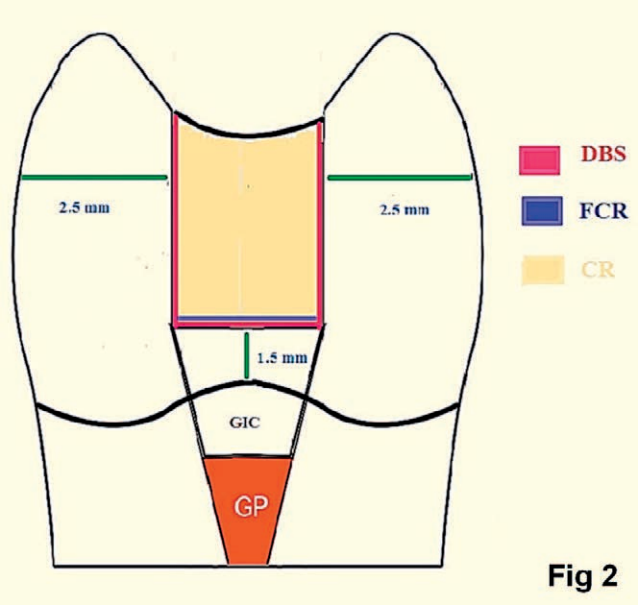

Figure 2. *The restoration of teeth in group FRC with DBS, FCR, and CR.*

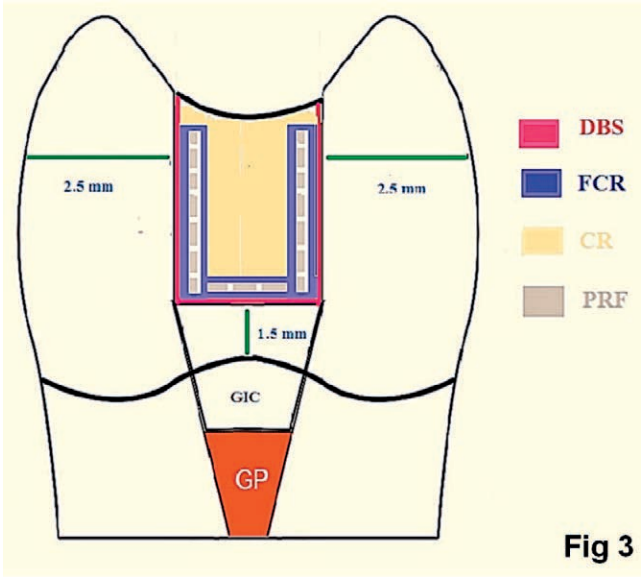

Figure 3. *The restoration of teeth in group PRF with DBS, FCR, and PRF.*

viscosity resin composite (Clearfil Majesty Flowable Universal Composite, Kuraray) and cured for 20 seconds. Then the cavities were restored with Clearfil AP-X resin composite using an incremental technique and each layer cured for 20 seconds (Figure 2).

Group Polyethylene Ribbon Fiber (PRF) (n=12 Samples)

After priming and bonding procedures with Clearfil SE Bond, as for group CR, the cavity surfaces (buccal walls, lingual walls, and pulpal floors) were covered with a layer of universal flowable, low-viscosity resin. Prior to curing, three pieces of polyethylene fiber (3-mm length, 2 mm wide) (Ribbond THM; Ribbond Inc, Seattle, WA, USA) were cut and wetted with Clearfil Liner Bond 2V Bond Liquid A (Kuraray). The Ribbond THM was kept in a dark container before the restoration process. Each polyethylene fiber was embedded inside the flowable composite on buccal, lingual walls and pulpal floors as one layer and was then cured for 20 seconds. Once cured, the cavities were restored with composite (Figure 3).

After the restoration was complete, finishing and polishing procedures with finishing burs, polishing tips, and discs (TOR VM Ltd, Moscow, Russia) were performed on all of the samples.

All of the samples (control and experimental groups) were mounted into self-curing polymethyl methacrylate resin at a level 1-1.5 mm below the CEJ with a cylinder metal mold (30-mm length, 20-mm width). During these procedures, care was taken to keep the long axis of the tooth parallel to that of the mold. Following the mounting, the samples were subjected to fracture using a universal testing machine (No. 3345J7324, Instron, Norwood, MA, US). A compressive force was applied with a modified stainless-steel ball (6 mm in diameter) parallel to the long axis and centered over the teeth until the ball contacted the internal surface of buccal functional cusps and the small part of the restoration. Compressive loading of teeth was performed at a crosshead speed at 0.5 mm/min. The mean loads required to fracture the samples were recorded in Newtons (N).

According to the failure modes, fractures were divided into two groups: 1) favorable fractures at the CEJ level and above and 2) unfavorable fractures below the level of the CEJ.

Statistical Analysis

Statistical calculations were performed with the NCSS 2007 program for Windows. In addition to standard descriptive statistical calculations (mean, standard deviation), a Kruskal-Wallis test was used in the comparison of groups, post hoc Dunn. The statistical significance level was established at $\alpha = 0.05$.

RESULTS

The fracture strength and statistical comparisons for each group are shown in Table 2. According to the

Table 2: *Mean Fracture Resistance in N, Standard Deviation (SD), Standard Error (SE), and Statistical Comparison of the Groups*[a]

Groups	n	Mean ± SD	SE	Minimum	Maximum
IN	12	2156.79 ± 628.04 A	181.30	1475.99	3284.23
CR	12	1315.83 ± 352.38 B	101.72	749.63	1777.05
FCR	12	1445.35 ± 506.18 B	146.12	906.16	2272.00
PRF	12	1951.64 ± 330.94 A	95.53	1316.02	2345.91

[a] *Groups with different letters show a statistically significant difference ($p<0.005$).*

results, the highest values were seen in group IN, and the lowest values were seen in group CR. The fracture strength of the restored teeth exhibited no statistically significant difference between group CR and group FCR ($p>0.05$). Group PRF showed the highest fracture strength compared with group CR and group FCR ($p<0.05$). No statistically significant difference was observed between group IN and group PRF ($p>0.05$).

The failure modes for each group and statistical comparisons are displayed in Table 3. Regarding the failure mode, the highest percentage of favorable fractures was observed in group PRF, numerically, followed by group IN. When failure modes were compared, group PRF showed significantly more favorable fractures than did group CR ($p<0.05$).

DISCUSSION

Endodontically treated teeth are at greater risk of fracture, mostly as a result of treatment techniques that result in hard tissue loss and large cavity preparations.[1,12-14] An extensive MOD cavity preparation in a root-filled tooth may lead to cuspal fracture of the tooth if it is not restored appropriately.[15,16] Fennis and others[17] used data from over 46,000 patients in 28 dental practices and reported that of the 20.5 cusp fracture incidents per 1000 person-year, 21% involved premolar teeth.

In the oral cavity, posterior teeth are exposed to more masticatory occlusal loads and have a greater risk of fracture than is associated with anterior teeth.[18] In our study, we used premolar teeth to evaluate the strengthening and reinforcing effect of different restoration techniques. Cavity size may be another factor that altered the strengthening of the teeth. In the present study, the floor of the MOD cavity was measured to be approximately 2.15 mm in width, as was the case in a previous study.[12] We applied axial forces on the functional cusps, parallel to the long axis of the teeth, to mimic the forces of centric occlusion. Several studies[19,24] have used a universal testing machine to produce a compressive load to the specimen by means of several metallic load devices, such as steel spheres, steel cylinders, and wedge-shaped devices with a straight and cast-metal antagonist tooth. Burke and others[25] determined that the best method with which to evaluate the resistance of premolars to fracture involves using a ball of a defined diameter. In addition, in our study, we subjected the teeth to vertical compressive loading with a 6-mm-diameter, stainless-steel sphere. All of the teeth were stored in saline solution for a day before the tests. We avoided longer times of storage in solution to eliminate the postmortal changes of the tooth structure. Moreover, we used only intact teeth as a control group to compare the biomechanical properties of healthy teeth to ETT restored with different techniques.

Recently, a variety of materials have been developed to improve the mechanical properties of structurally weakened teeth. Direct composite restorations offer a good option that can strengthen teeth while maintaining esthetics.[1,26,27] Current studies[26-28] have revealed that adhesive restorations improve resistance and have superior properties for transmitting and distributing functional stress. In our study, the samples from groups CR and FRC showed the lowest fracture resistance. Although the fracture strength of the specimens can be increased by using flowable composite under the restorations, no significant difference was observed between these groups. In accordance with our results, it was reported that MOD composite restorations of maxillary premolar teeth with or without cavity liners underneath showed similar resistance to fracture.[29]

Table 3: *The Fracture Modes, Percentages, and Statistical Comparisons of the Groups*[a]

Groups	Favorable Fracture		Unfavorable Fracture	
	No.	%	No.	%
IN	8 AB	77.78	4	22.22
CR	5 A	61.11	7	38.89
FCR	6 AB	66.67	6	33.33
PRF	11 B	94.44	1	5.56

[a] *Groups with different letters show a statistically significant difference ($p<0.005$).*

Ribbond is a leno-woven, ultra–high-molecular-weight polyethylene fiber with an ultrahigh elastic modulus. Its intrinsic fabric architecture, which has fibers oriented in various directions forming an interwoven structure, permits for forces to be dispersed over a broader area, diminishing stress altitudes.[11,30] Ribbond needs to be impregnated with wetting resin before being placed in the flowable resin composite. The leno design enhances impregnation of the wetting resin and thereby enhances the chemical bonding of the fiber with flowable resin, creating a unique united structure. In a previous study, Belli and others[31] claimed that the lock-stitch property delivered the forces through the weave without stress propagation into the resin. As a result of the interwoven nature of the fabric, it was expected that polyethylene fiber had a stress-altering effect.[30] Cobankara and others[14] reported that inserting Ribbond below the composite did not reduce the fracture strength of endodontic molar teeth with MOD cavity preparations. On the other hand, Sengun and others[32] described that inserting a ribbon fiber on the occlusal surface of endodontically treated premolar teeth with MOD cavity preparations improved fracture strength. Belli and others[33] evaluated the influence of using low-viscosity flowable composite with or without Ribbond fiber on the fracture resistance of endodontically treated mandibular molar teeth with MOD cavity preparations, and they concluded that inserting Ribbond significantly improved fracture resistance. Similarly, in our study, inserting Ribbond to pulpal, buccal, and lingual walls of the cavity improved the fracture resistance of endodontically treated premolar teeth with MOD adhesive restorations.

In our study, fiber insertion procedures on cavity walls (pulpal, buccal, and lingual walls) showed a positive influence on distributing stress along the teeth with significantly similar fracture resistance to that observed for group IN. In accordance with our results, Costa and others[34] also reported that the fracture strength of endodontically treated premolars restored with polyethylene ribbon fiber was similar to that of intact teeth. Belli and others[33] determined that the use of polyethylene ribbon fiber below composite restorations in root-filled molar teeth with MOD preparations produces a significant increase of fracture resistance compared to that associated with intact teeth. The discrepancy between these studies may involve the lack of standardized preparation and/or standardized test design. In the present study, we used premolar teeth with smaller cavity sizes than molar teeth. Inserting more than one strip of ribbon fiber in premolar teeth with smaller cavities results in a bridging effect between the cusps of the teeth and improvement of strength.

In the current study, the failure modes of each test group were also analyzed. Our results indicated that teeth restored with only composite and flowable composite were more prone to unfavorable fractures with the fracture line lower than 1 mm below the CEJ. However, group PRF revealed favorable fractures that can be restored more simply, and the teeth may be preserved in clinical service without any extra treatment. Sengun and others[32] examined the influence of a new fiber-reinforced composite restoration method on the fracture resistance of endodontically treated premolars, and they determined that most of the failure modes of the reinforced teeth were restricted to the level of the enamel, whereas the other groups revealed fractures generally at the level of the CEJ or below it. The possible explanation for this can be the leno-wave pattern of polyethylene fibers, which has crack-stopping or crack-deflecting mechanisms.

The results of this study are only introductory and comparative. The study has some limitations and does not completely simulate the clinical situation. Although fracture resistance was considered, the biomechanical properties of the periodontium were not included. The forces applied in this study were at a constant direction and speed, while forces produced intraorally differ in magnitude, speed of application, and direction. Additional research is necessary to determine the influence of mechanical, thermal, and chemical stress on the durability of the restorations.

CONCLUSIONS

Within the limitations of the study, the null hypothesis is rejected, and it is concluded that

- Intact teeth and polyethylene ribbon fiber groups revealed significantly similar fracture resistance.
- Use of flowable composite resin below composite restorations did not significantly increase the fracture strength of root-filled premolar teeth with MOD cavity preparations.
- Use of reinforced polyethylene ribbon fiber beneath composite restorations in root-filled premolar teeth with MOD preparations considerably increased the fracture strength.
- Most of the failure modes of group PRF were restricted to the level of the enamel, whereas the

other three groups revealed fractures mostly at the level of lower than 1 mm below the CEJ.
- Polyethylene ribbon fiber–reinforced composite resin restorations appeared to represent a more reliable restorative technique than did composite restoration for wide cavities.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Istanbul University Faculty of Dentistry Ethical Committee. The approval code for this study is 2016/23.

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.

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Performance of Monolithic and Veneered Zirconia Crowns After Endodontic Treatment and Different Repair Strategies

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Clinical Relevance

Two-step repair fillings with silica coating, silanization, and bonding provide improved fracture loads in monolithic and better marginal integrity in veneered zirconia restorations than one-step fillings. Monolithic restorations provide higher fracture loads but require more time for access preparation.

SUMMARY

Objectives: To investigate failure loads of monolithic and veneered all-ceramic crowns after root canal treatment and to analyze marginal integrity of repair fillings.

Methods and Materials: Seventy-two human molars were restored with monolithic (Zr-All) or veneered (Zr-Ven) zirconia crowns. Molars were assigned to six groups (n=12 per group) depending on restoration material, access type (no access cavity [control] or endodontic treatment [test]), and type of filling (one-step [1-st] or two-step [2-st]). For type of filling, molars were treated using a self-etch universal adhesive and cavities were either filled with layered composite (1-st) or filled until the crown material was reached, which was additionally conditioned and then filled (2-st). Scanning electron microscopic analysis of the restoration margins was performed before and after thermomechanical loading (TML), and the percentage of continuous margins was assessed. Crowns were then loaded to failure.

Results: Preparation of the access cavity required more time in monolithic (445 s) than in veneered crowns (342 s). Loads to failure were higher in control groups (Zr-All: 5814 N; Zr-Ven: 2133 N) and higher in monolithic test (2985 N) than in veneered test crowns (889 N). In monolithic crowns, 1-st had lower fracture

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loads than 2-st fillings (2149 N vs 3821 N). Continuous margins of 66% to 71% were achieved, which deteriorated after TML by 39% to 40% in Zr-All, by 34% in Zr-Ven-1-st, and by 24% in Zr-Ven-2-st.

Conclusions: Endodontic access and adhesive restorations resulted in reduced fracture load in monolithic and veneered zirconia crowns. Two-step fillings provided higher fracture loads in Zr-All and better marginal quality in Zr-Ven crowns.

INTRODUCTION

The need for root canal treatment (RCT) is one of the most common biological complications following reconstructive treatment and may lead to subsequent technical complications with veneering fractures after preparation of the endodontic access cavity.[1] Among abutment teeth initially positive for sensitivity testing, approximately 11% required RCT during an observation period of 10 years.[2] For single-crown abutments, the pulp survival rate reached 84.4% after 10 years and 81.2% after 15 years, while abutments in fixed dental prostheses (FDP) had a lower pulp survival rate of 70.8% after 10 years and 66.2% after 15 years.[3] The increased risk of pulpal necrosis following abutment preparation is related to an additive effect of several noxious agents, such as caries, repeated fillings, periodontal disease, and physical or restorative trauma.[4,5] Extensive reduction of the tooth structure to facilitate a similar path of insertion in long-span FDP or to provide sufficient space for all-ceramic restorations is another potential cofactor.[3,6]

In a recent review, factors influencing damage around endodontic access cavities and fracture resistance of all-ceramic crowns after access repair were investigated.[7] Although the authors were not able to provide a "best practice" clinical protocol, decisive factors were identified, such as the crown material and its adhesive potential, the cement material used, damage along the access cavity, and the ratio between cavity and crown size.[1,8-12] Further, the grit size of the diamond bur used for access preparation[9] and the filling technique to restore the access cavity[13] have been documented to influence fracture resistance of all-ceramic crowns. While glass ceramic crowns had lower fracture resistance than zirconia restorations,[14] veneered (bilayered) zirconia was not as resistant as monolithic zirconia.[8,15] In lithium disilicate glass ceramics, the use of larger-grit rotary diamonds (180 μm) reduced the failure load of bonded restorations to 2354 N as compared to small-grit diamond burs (126 μm, 3464 N).[9] In zirconia restorations, external stress from grinding induces a phase transformation from tetragonal to monoclinic and is associated with a 4% increase in volume. This volume increase results in compressive stresses and increased fracture toughness. Crack propagation occurs only when these clamping constraints are surpassed and stress factors exceed arresting factors.[16,17] Following RCT, an adhesive composite filling restoration should provide successful endodontic outcome, re-establish crown stability, and ensure crown retention.[13,18-21] To meet these requirements, a variety of techniques for pretreatment of the restoration have been examined, including etching or sandblasting of the surface as well as the application of special bonding agents.[20,22] While surface sandblasting with an aluminum oxide powder created microretention,[20,22] corundum particles coated with silicium oxide (CoJet, 3M ESPE, Rüschlikon, Switzerland) led to a retentive silicated surface and, in combination with a silane, enabled a chemical connection to the filling material.[23-25]

The aim of this experimental study was to investigate the fracture load of monolithic and veneered all-ceramic crowns following RCT and access cavity filling and to analyze the marginal integrity of one- or two-step repair fillings before and after thermomechanical loading (TML).

METHODS AND MATERIALS

Ethical approval was obtained for the use of extracted teeth for material testing of dental restorations. Seventy-two extracted human mandibular molars that had no caries, fractures, fillings, or restorations were selected. Teeth were kept in 0.1% thymol suspension for disinfection and to prevent dehydration.[26] The specimens were divided into six groups (n=12 per group) and labeled according to the intended crown restoration type and the filling procedure (group 1: Zr-All; 2: Zr-All-1-st; 3: Zr-All-2-st; 4: Zr-Ven; 5: Zr-Ven-1-st; 6: Zr-Ven-2-st; Figure 1).

Preparation of Specimens, Crown Fabrication, and Cementation

To simulate the periodontal ligament, a gum resin (Anti-Rutsch-Lack, Wenko-Wenselaar, Hilden, Germany) was applied in a thin layer on the root surface. To fix the specimens in the loading device, the roots were embedded in acrylic resin (Dermotec 20, Dermotec Siegfried Demel, Nidderau, Germany). Teeth were prepared for crown restoration by two

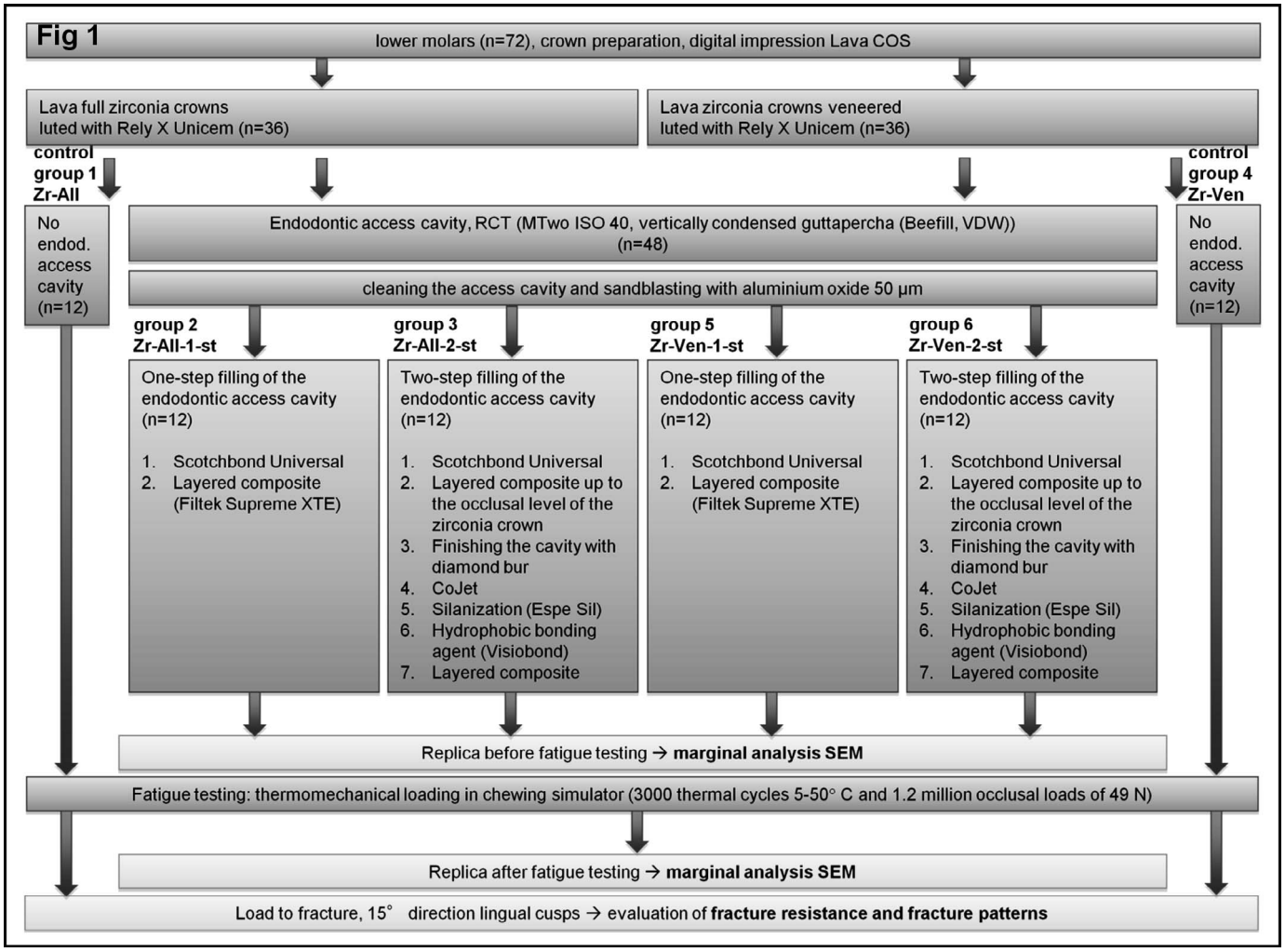

Figure 1. *Schematic overview of the study design and group treatment.*

operators (AS and AH) according to the guidelines for Lava All-Zirconia restoration (3M ESPE). A circular shoulder preparation of 1 mm (horizontally) was performed along the cemento-enamel junction using cylindrical burs (no. 307 [106-µm grit] and no. 4315 [40 µm-grit]; Intensiv, Grancia, Switzerland). Football-shaped diamonds were used for the preparation of the occlusal relief (no. 4250 [40-µm grit], no. 5250 [15-µm grit]; Intensiv). While the no. 307 bur was used only twice, the nos. 4315, 4250, and 5250 burs were used a maximum of four times. Standardization of a similar preparation was ensured using a silicon key (Affinis putty, Coltène, Whaldent, Altstätten, Switzerland) made from the original crown contour, which was sectioned and applied during substance reduction to illustrate sufficient clearance.

Digital impressions of all specimens were taken with the Lava scanner (3M ESPE). In groups 1 to 3, the monolithic zirconia crowns representing the anatomic contour of a mandibular molar were designed and computerized, ensuring a minimal crown thickness of 1 mm. Crowns were milled from ceramic blocks (Lava, 3M ESPE), and the surfaces were finally glazed. For groups 4 to 6, 3M zirconia copings of 0.5-mm thickness were manufactured (Lava, 3M ESPE). A professional dental technician (Diethard Schwarz, Dental Laboratory, Velden/Vils, Germany) conducted the feldspathic porcelain veneering at a minimal thickness of 0.5 mm with final glazing. Before cementation, the crown thickness of each specimen was measured in the area of the endodontic access with a thickness gauge (M&W Dental, Illnau, Switzerland). The prepared abutment surface was cleaned with pumice mixed with Ringer's solution (Ringer Ecotrainer Plus, B. Braun, Maria Enzersdorf, Austria), rinsed with water, and air-dried. The restorations were degreased with trichloroethylene (Merck KGaA, Darmstadt, Ger-

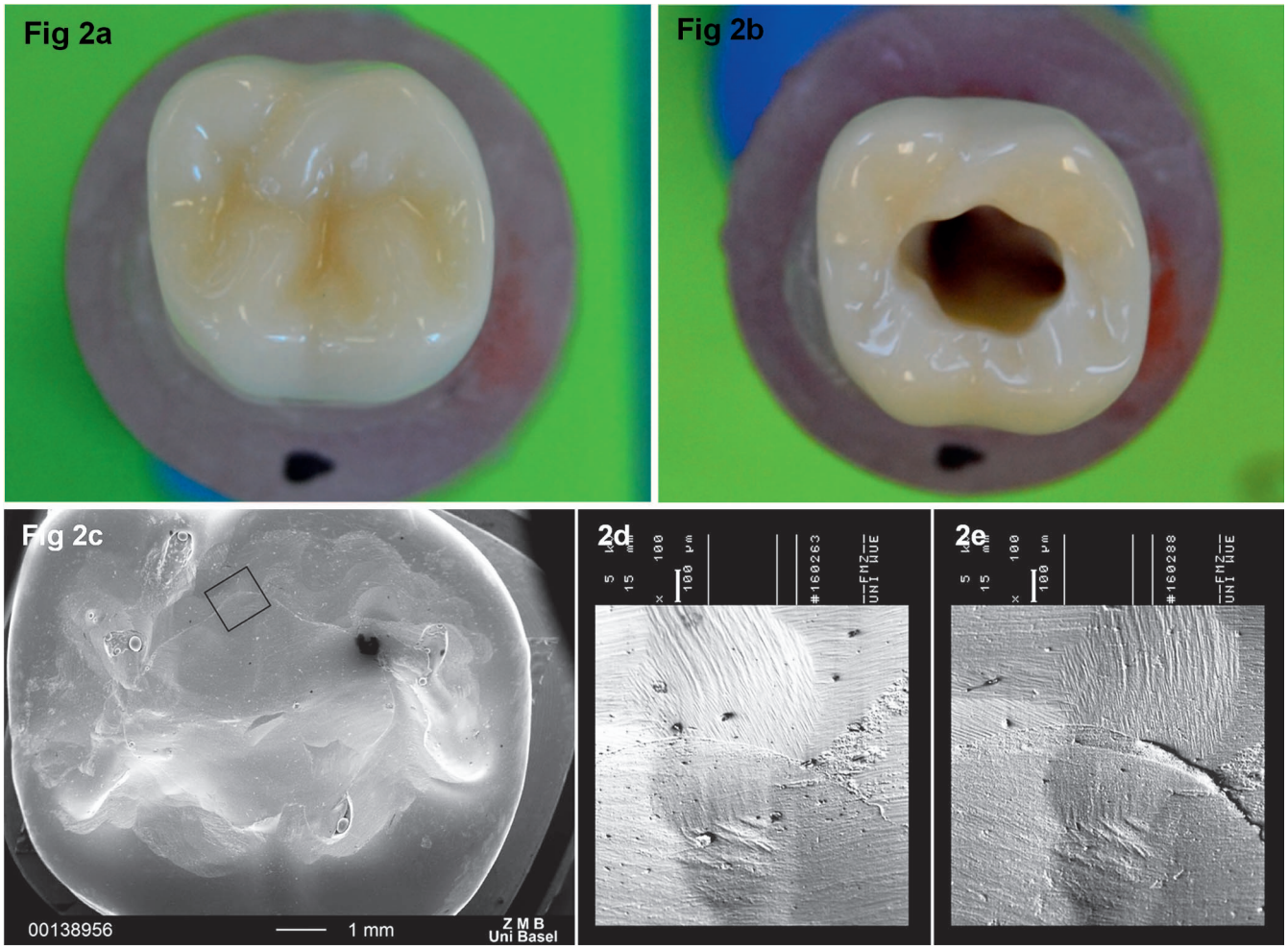

Figure 2. *(a): Sample (no. 3) of group 2 Zr-All-1-st crown after cementation. (b): Endodontic access and cavity preparation. (c): Overview with scanning electron microscope; detail indicated by square. (d): Detail of the restoration margin with 100% continuous margin before thermomechanical loading. (e): Same area after thermomechanical loading with proportion of continuous margin reduced to 53%.*

many) and cemented with a self-adhesive resin cement (RelyX Unicem, 3M ESPE).[1] After initial light curing for two seconds (Bluephase C8, Ivoclar Vivadent, Schaan, Liechtenstein), excess cement was removed, and final light curing was performed with 800-mW/cm^2 light intensity from four directions for 20 seconds each. Photographs and periapical radiographs (Insight Dental film, Kodak, Rochester, NY, USA) were taken, and after 10 minutes, the specimens were stored again in the 0.1% thymol suspension.

Endodontic Access, Root Canal Treatment, and Restoration of the Endodontic Access Cavity

The endodontic access preparation was performed by two operators (AS and AH) applying a standardized trapezoidal access shape for mandibular molars. A new cylindrical diamond (no. 307 [106-μm grit], Intensiv) was used for each specimen in a high-speed hand piece (40,000 min^{-1}) under water cooling (Figure 2a,b). The time required to penetrate the crown and complete the access cavity with all root canal entrances was recorded in seconds. Root length was defined based on the radiograph. Root canals were prepared using rotary instruments (Mtwo, VDW, Munich, Germany) up to a master apical file 40/04. Between the different instrument sizes the canals were flushed with 10 mL sodium hypochlorite (1%, Caelo, Hilden, Germany). The root canals were dried with paper points, coated with sealer (AH Plus, Dentsply De Trey GmbH, Konstanz, Germany), and filled with BeeFill 2 in 1 gutta-percha (BeeFill Gutta-percha cartridge, VDW) by the warm vertical compaction technique. The gutta-percha filling was then reduced with a round bur (0.9-1.4-mm diameter, Komet, Lemgo, Germany) up to 1 mm underneath the root canal orifice to increase the adhesive

surface for the filling material. The access cavity was finished with cylindrical burs (no. 307 [106-μm grit] and no. 4315 [40-μm grit], Intensiv) and sandblasted with 50 μm Al_2O_3 particles (Dento-Prep, Ronvig, Daugaard, Denmark) to remove any remnant of sealer or gutta-percha and to increase adhesion. To generate adhesion to both dentin and the ceramic surface, a novel self-etch universal adhesive containing the functional monomer 10-methacryloyloxydecyl dihydrogen phosphate (10-MPD) and silane (Scotchbond Universal, 3M ESPE) was applied for 20 seconds, gently air-dried for five seconds, and light cured with Elipar S10 (3M ESPE) for 10 seconds. The endodontic access cavities were then filled with a one- or two-step filling procedure, applying 2-mm layers of a universal composite (Filtek Supreme XTE, 3M ESPE). With the one-step procedure, the cavity was filled with several layered increments. For the two-step procedure, composite layers were applied until the level of the ceramic material was reached. The remaining cavity was then conditioned with silica coating (CoJet Sand, 3M ESPE), water sprayed and air-dried, silanated (ESPE Sil, 3M ESPE), and air-dried after one minute. A light-cured adhesive (Bonding Visio Bond, 3M ESPE) was applied for 20 seconds and light cured for 20 seconds, and the filling was finished with two oblique layers. For both techniques, the surface was finished with a fine diamond bur (no. 4250 [40-μm grit], Intensiv) and polished with Occlubrush (Kerr, Bioggio, Switzerland) and a one-step diamond paste (Unigloss, Intensiv).

Replica Before and After Fatigue Testing, Load to Fracture

An impression of the sealed endodontic access cavity was taken with a polyvinyl siloxane impression material (Affinis Regular Body). Replicas were manufactured with epoxy resin (Stycast 1266 A & B 2 Part clear epoxy, Emerson & Cuming, Westerlo, Belgium). These replicas were controlled with an optical microscope (Wild M3B, Heerbrugg, Switzerland) to ensure that the entire filling margins were visible. All specimens underwent TML in a chewing simulator (CoCoM 2, OOK, Zürich, Switzerland). Stressing comprised 1.2 million occlusal loads of 49 N at 1.7 Hz and simultaneous thermal cycling (3000 thermal cycles between 5°C and 50°C) using antagonistic natural teeth. Following TML, the specimens were visually inspected, and the presence of any ceramic chipping or fracture was recorded. An additional set of replicas was manufactured as described above. All samples were then loaded to fracture in a universal testing machine (Allround-Line, Zwick GmbH& Co., Ulm, Germany) and a 20-kN-load cell. Specimens were fixed in a metal holder with the long axis of the roots at an angle of 15° to the direction of the load. A linear load (crosshead speed of 0.5 mm/min) was applied in the direction of the lingual cusp until fracture.

SEM Analysis of Marginal Quality

All replicas taken before and after TML were sputter coated with gold (EMITECH K550 Emitech, Taunus Stein, Germany) and numbered to facilitate a blinded analysis by one of the authors (SS) not involved in the restoration procedure. The restoration margins of the endodontic access cavities were examined with a scanning electron microscope (DSM 940, Zeiss, Oberkochen, Germany) at 100× to 1000× magnification and analyzed using dedicated measurement software (RaEm, programmer Peter Müller, Würzburg, Germany). Prior to all measurements, a measuring grid (copper mesh, item no. S150, Plano, Wetzlar, Germany) was scanned, and the measurement software was normalized at 100× magnification. Images of the restoration margins were saved and measured at 100× magnification. To investigate the margin quality of the restorations, criteria modified from the classification introduced by Blunck and Zaslansky[27] were applied to distinguish between 1) continuous margin (without any signs of gap formation), 2) noncontinuous margin (hairline crack or gap), and 3) not judgeable margin (due to excess composite material or fracture).[27] Finally, the proportion of continuous margin in each specimen was calculated and presented as a percentage of the individual judgeable margin (Figure 2c through e).

Statistical Analysis

The prediction variable fracture load was log transformed as verified by preliminary analysis, including a quantile comparison plot. Descriptive statistics included means (standard deviation) for metric variables and median (interquartile range) for fracture load. Linear models were performed to predict either thickness, time for access, or fracture load; these models provided estimates of slope values (for continuous variables) or difference of means as well as ratios (for categorical variables). For the percentage change of continuous margin (after vs before TML), linear regression models were performed to compare crown (Zr-All vs Zr-Ven) and access restoration (2-st vs 1-st). The corre-

Table 1. *Results of Different Parameters in the Six Groups With Crown Thickness, Time for Trepanation, Fracture Load, and Changes in Continuous Margin Before and After Loading, Mean (Standard Deviation), and Median (Interquartile Range) for Fracture Load*

Material	Zr-All (Monolithic)				Zr-Ven (Veneered)				*p*-Value (All Groups)
	Control	Test			Control	Test			
Parameter	1 Zr-All	2 Zr-All-1-st	3 Zr-All-2-st	2 and 3	4 Zr-Ven	5 Zr-Ven-1-st	6 Zr-Ven-2-st	5 and 6	
Crown thickness (μm)	1.38 (0.35)	1.46 (0.26)	1.5 (0.33)	1.48 (0.29)	1.34 (0.19)	1.44 (0.29)	1.55 (0.23)	1.50 (0.26)	0.474
Time for trepanation (s)	—	517 (80)	374 (73)	445 (105)	—	389 (131)	295 (89)	342 (120)	0.003
Fracture load (N)	5955 (5105-6603)	1975 (1480-2335)[b]	3265 (2718-4540)[b]	2705 (1983-3698)[a]	2200 (1855-2400)	923 (601-1274)	844 (568-1008)	844 (577-1141)[a]	<0.001
Continuous margin before load (%)	—	67 (13)	71 (21)	69 (17)	—	66 (10)	67 (12)	66 (11)	0.838
Continuous margin after load (%)	—	28 (19)	31 (22)	30 (20)	—	33 (10)[c]	43 (14)[c]	38 (13)	0.148
Change in continuous margin (%)	—	39 (16)	40 (29)	40 (23)[a]	—	34 (10)[c]	24 (12)[c]	29 (12)[a]	0.121

p-*values derived from analysis of variance (*F*-test) except for the following:*
[a] *Statistically significant differences between groups 2 3 and 5 and 6 (fracture load* p*<0.001; rank sum test; change in continuous margin* p*=0.042;* t*-test).*
[b] *Statistically significant differences between groups 2 and 3 (*p*<0.001; linear regression predicting log transformed fracture load values).*
[c] *Statistically significant difference between groups 5 and 6 after load (*p*=0.038) and changes (*p*=0.043;* t*-test).*

sponding 95% confidence intervals and *p*-values were calculated for all regressions. Nested model designs were performed to separately analyze selected study groups. The level of significance was set at $\alpha = 0.05$. Adjustment of significance level for multiple comparisons was omitted because of the descriptive nature of the study. All analyses were performed with the statistical program R version 3.1.2 (R Core Team 2014).[28]

RESULTS

The occlusal crown thickness in the area of the access varied between 1.34 and 1.55 mm without significant differences among the groups (Table 1). The time to complete the access cavity was significantly longer for Zr-All crowns (groups 2 and 3) with 445.3 ± 104.5 seconds than for Zr-Ven (groups 5 and 6) with 342.3 ± 119.5 seconds ($p=0.003$). Visual inspection following chewing simulation revealed that ceramic chippings had occurred in the Zr-Ven groups (two crowns in Zr-Ven-1-st, four crowns in Zr-Ven-2-st), while no ceramic chipping or fracture was observed in preserved Zr-Ven crowns (control group 4) or in any Zr-All specimens (groups 1 to 3).

Load to fracture varied between 5814 ± 1084 N for Zr-All and 806 ± 273 N for Zr-Ven-2-st ($p<0.001$). For both materials, fracture loads were significantly higher for preserved control groups than for the test groups (Table 1; Figure 3). Fracture loads were higher for Zr-All test specimens (groups 2 and 3; 2985 N) than for Zr-Ven specimens (groups 5 and 6; 889 N; $p<0.001$). The comparison of one-step and two-step filling restorations within the different crown materials revealed significantly higher fracture loads for Zr-All-2-st than for Zr-All-1-st, while no difference existed between the two procedures in the Zr-Ven groups 5 and 6 (Table 1).

The relative proportion of continuous margin along the endodontic access restoration varied between 66% and 71% before TML and was reduced to 28% to 43% after TML (Table 1; Figure 4). The deterioration of the marginal quality was greater in the Zr-All groups with 40% change compared with the Zr-Ven groups with 29% change of continuous margin ($p=0.042$; Table 1). The reduction in the proportion of continuous margin was significantly greater with the one-step procedure (Zr-Ven-1-st 34%) than with the two-step procedure (Zr-Ven-2-st 24%; $p=0.043$).

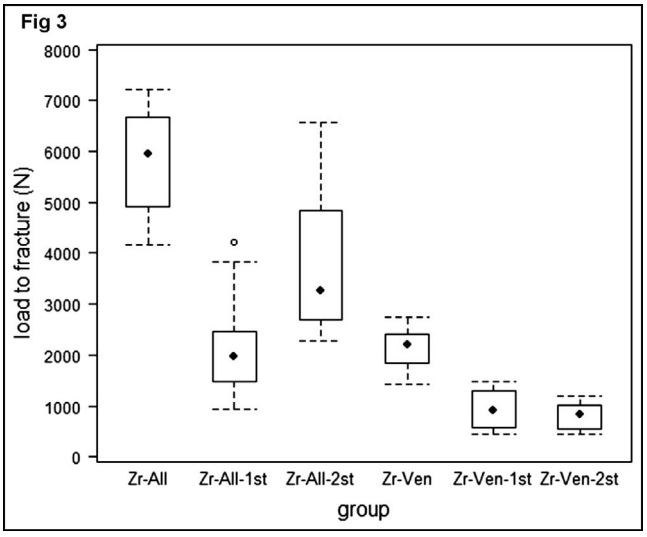

Figure 3. *Box plots of loads to fracture in groups 1 to 6.*

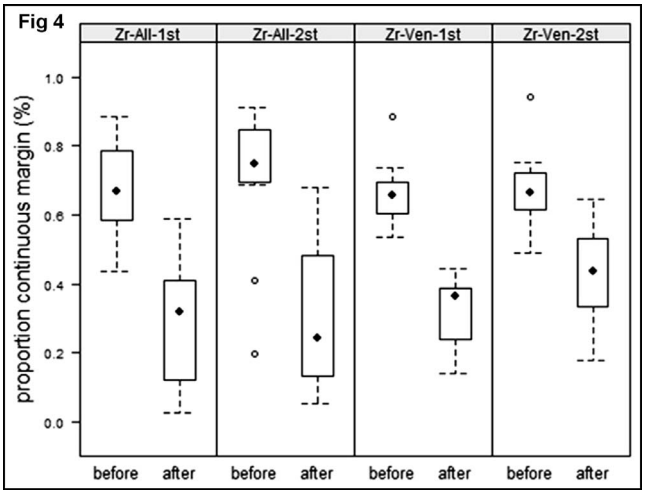

Figure 4. *Box plots of proportions of continuous margin before and after thermomechanical loading in groups with repair fillings (groups 2, 3, 5, and 6).*

DISCUSSION

The aim of this *in vitro* experiment was to compare monolithic and veneered zirconia crowns after access cavity preparation, RCT, and repair fillings. It was observed that preparation of the access cavity required more time in monolithic than in veneered crowns. Furthermore, loads to failure were significantly higher in monolithic crowns than in veneered crowns, and the two-step filling technique had a positive influence on fracture resistance with monolithic crowns. While an approximately two-thirds-perfect margin of the repair filling was initially achieved with either technique, thermomechanical load resulted in a deterioration of the marginal quality, particularly in the Zr-All groups, while the two-step filling procedure provided better marginal integrity in veneered crowns.

In the present *in vitro* study, extracted human teeth were used. Each tooth was individually prepared, and crown specimens were fabricated with computer-assisted design and manufacturing in accordance with the manufacturers' guidelines. A possible limitation of the current study protocol is that a certain variation in abutment dimensions and crown thickness existed. In other experimental studies, standardized epoxy or composite resin dyes with similar crown specimens were used and RCT was simulated.[1,9,10] In the present study, RCT was performed in groups 2 to 5 to ideally reflect the clinical situation. For the preparation of the endodontic access cavity, new burs were used for each specimen to ensure comparability, while repeated use of burs is common practice in the clinic. The coarse-grit diamonds of 106 μm that were used provided adequate access without weakening the ceramic integrity but may have resulted in marginal microfractures. However, the documented preparation time of 7.25 minutes for monolithic zirconia crowns and 5.42 minutes for veneered zirconia crowns would have been even more prolonged with the use of smaller grit sizes. To avoid heat-induced crack initiation and propagation in the ceramic material, the use of a diamond bur with sufficient water cooling has been recommended.[11,29-31] Carbide burs were found to be ineffective and associated with a higher risk of fractures and craze lines, particularly in glass-ceramic materials.[7,11,12] In an experimental study using lithium-disilicate crowns (IPS e.max CAD) cemented with dual-polymerizing resin cement (Multilink Implant, Ivoclar Vivadent), the use of a 126-μm-grit-size diamond rotary instrument and subsequent composite filling restoration did not affect fracture load (3464 N) as compared to the unprepared control (3316 N), while failure loads were reduced to 2915 and 2354 N when using coarse-grit diamonds of 150 and 180 μm, respectively.[32] In the study by Qeblawi and others,[32] destructive experimental testing was applied without any artificial aging, which is known to have a considerable impact on the values generated in load-to-fracture tests.[33] In the current experimental protocol, cyclic loading within physiological limits and simultaneous thermocycling was selected, and the periodontal ligament was simulated to mimic oral cavity conditions.[34] These factors may be responsible for the observed reduction in fracture loads among repair restorations,

with none of them being able to restore the maximum load capability to the same level as was recorded for the specimens without endodontic access cavity.

In a recent review of *in vitro* studies, protocols were assessed with regard to fracture resistance of endodontically accessed and repaired all-ceramic crowns.[7] In addition to the initial baseline strength of the ceramic material, the application of adhesive cementation techniques, the size of the access cavity in relation to the crown size, and the residual tooth structure were discussed as potential key factors influencing fracture resistance.[7] In the current study, higher fracture resistance was found with monolithic compared to veneered all-ceramic crowns. Following endodontic access and cavity restoration, veneered crowns demonstrated reduced fracture values of 955 and 806 N, which is close to the maximal bite forces of 807 $\pm$ 140 N in the molar region of 20- to 24-year-old males[35] but exceeds normal chewing forces ranging from 70 to 150 N.[36] Previous studies have demonstrated favorable mechanical properties for monolithic crowns compared to veneered all-ceramic restoration.[14,15] Highest loads to fracture were documented for monolithic zirconia crowns (6517 N), while for the two veneered designs with or without a cervical collar of zirconia, average values of 4712 and 4091 N, respectively, were achieved.[15] For veneered Procera crowns cemented with Rely X Luting Plus cement (3M ESPE) on epoxy resin dies and provided with repair fillings, the endodontic access did not influence failure loads of alumina crowns (1459 N control, 1531 N with access restoration), while data for zirconia showed differences with 2514 N for the unprepared control and 2246 N for the repaired crowns.[1]

In the current study, ceramic chipping during TML occurred in six out of 24 veneered and prepared crown specimens, while no chipping fractures were observed in the unprepared control group Zr-Ven and in none of the monolithic crowns. Edge chipping around the endodontic access restoration was also observed when Procera crowns were loaded to failure.[1] In this experimental study, Procera crowns were fabricated based on alumina and zirconia copings with the veneering porcelain pressed over the copings.[1] In alumina crowns, core fractures and veneer shear were observed, while with zirconia copings, the veneering delaminated frequently from the core.[1] Analyzing the occlusal surface following endodontic access preparation in monolithic and veneered zirconia three-unit FDP also demonstrated that more microfractures and chippings occurred in veneered restorations.[8,11] Achieving durable bonding to zirconia restorations is challenging, and despite a wide variety of recommended surface conditioning methods, to date no universally accepted protocol exists.[37] In the present study, both repair methods applied for restoration of the access cavities of the monolithic zirconia crowns showed favorable results in terms of percentage of continuous margins. However, gap formation significantly increased under *in vitro* stress conditions, confirming previous studies that revealed that artificial aging affects the bonding effectiveness to zirconia.[38,39] For the one-step protocol, a universal adhesive containing the phosphate-based functional monomer 10-MDP was selected because of its proven chemical bonding capability to zirconia. However, since 10-MDP is one among many ingredients mixed into one solution, bond durability to zirconia is inferior compared to dedicated ceramic primers based on the same monomer.[38] These factors are possibly responsible for the rather low marginal quality achieved in group Zr-All-1-st after TML.

For the veneered zirconia crowns, both repair protocols used in the present study comprised silanization because there is consensus that application of a silane after mechanical conditioning of the veneering porcelain is crucial to achieve a chemical bond to the composite resin.[40] In the one-step protocol, the silane is incorporated into the formulation of the utilized adhesive Scotchbond Universal. In this group, marginal quality was inferior, though not statistically significant, compared to the two-step approach. These findings are comprehensible since there is recent evidence that the silane coupling agent in universal adhesives is less efficient compared to dedicated silanes.[41-44]

CONCLUSIONS

It can be concluded that the two-step repair filling with silica coating, silanization, and bonding of the marginal crown material led to improved fracture loads in monolithic zirconia crowns and better marginal integrity in veneered zirconia restorations as compared to the one-step filling. While monolithic restorations provided generally higher fracture loads and no chipping fractures, more time was required for preparing the endodontic access cavity than in veneered zirconia crowns.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethik beider Basel. The approval code for this study is EK 221/12.

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.

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Marginal Gap Formation in Approximal "Bulk Fill" Resin Composite Restorations After Artificial Ageing

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Clinical Relevance

With regard to marginal gap formation, certain flowable "bulk fill" resin composites may be viable alternatives to packable "regular" resin composites—especially in deep cavities with extensive dentin margins.

SUMMARY

The aim of this *in vitro* study was to investigate the marginal gap formation of a packable "regular" resin composite (Filtek Supreme XTE [3M ESPE]) and two flowable "bulk fill" resin composites (Filtek Bulk Fill [3M ESPE] and SDR [DENTSPLY DeTrey]) along the approximal margins of Class II restorations. In each of 39 extracted human molars (n=13 per resin composite), mesial and distal Class II cavities were prepared, placing the gingival margins below the cemento-enamel junction. The cavities were restored with the adhesive system OptiBond FL (Kerr) and one of the three resin composites. After restoration, each molar was cut in half in the oro-vestibular direction between the two restorations, resulting in two specimens per molar. Polyvinylsiloxane impressions were taken and "baseline" replicas were produced. The specimens were then divided into two groups: At the beginning of each month over the course of six months' tap water storage (37°C), one specimen per molar was subjected to mechanical toothbrushing, whereas the other was subjected to thermocycling. After artificial ageing, "final" replicas were produced. Baseline and final replicas were examined under the scanning electron microscope (SEM), and the SEM micrographs were used to determine the percentage of marginal gap formation in enamel or dentin. Paramarginal gaps were registered. The percentages of marginal gap formation were statistically analyzed with a nonparametric analysis of variance followed by Wilcoxon-

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Mann-Whitney tests and Wilcoxon signed rank tests, and all *p*-values were corrected with the Bonferroni-Holm adjustment for multiple testing (significance level: α=0.05). Paramarginal gaps were analyzed descriptively. In enamel, significantly lower marginal gap formation was found for Filtek Supreme XTE compared to Filtek Bulk Fill (p=0.0052) and SDR (p=0.0289), with no significant difference between Filtek Bulk Fill and SDR (p=0.4072). In dentin, significantly lower marginal gap formation was found for SDR compared to Filtek Supreme XTE (p<0.0001) and Filtek Bulk Fill (p=0.0015), with no significant difference between Filtek Supreme XTE and Filtek Bulk Fill (p=0.4919). Marginal gap formation in dentin was significantly lower than in enamel (p<0.0001). The percentage of restorations with paramarginal gaps varied between 0% and 85%, and for all three resin composites the percentages were markedly higher after artificial ageing. The results from this study suggest that in terms of marginal gap formation in enamel, packable resin composites may be superior to flowable "bulk fill" resin composites, while in dentin some flowable "bulk fill" resin composites may be superior to packable ones.

INTRODUCTION

Today, placement of direct resin composite restorations is a routine treatment in restorative dentistry. Since the introduction of resin composites more than 50 years ago, they have undergone constant development and have proved to be clinically effective.[1] Esthetic and mechanical properties have been improved, and handling, polishability, and abrasion resistance have been optimized.[1-4] Despite these improvements, the dentist is still faced with some challenges when using resin composites. One of these challenges is shrinkage due to polymerization. Polymerization shrinkage creates stress within resin composites, at the interface between the resin composite restoration and the tooth substance as well as within the tooth structure. Polymerization shrinkage can lead to marginal or paramarginal gap formation and, thus, to marginal discoloration, nanoleakage, or secondary caries.[1-3,5,6] One clinical means of minimizing these negative effects of polymerization shrinkage is the incremental technique.

According to this technique, regular resin composites are applied in increments of a maximum thickness of 2 mm, with each increment being light-cured separately.[1,2] Generally, the incremental technique is time consuming—particularly in posterior teeth with deep cavities. In response to the demand for simplified application and reduced application time, so-called "bulk fill" resin composites have been developed for restoration of Class I and Class II cavities. "Bulk fill" resin composites can be applied in a single increment of 4-5 mm thickness, depending on the product,[1,7-9] which obviously simplifies application and reduces the application time.[1] "Bulk fill" resin composites can be divided into two categories: 1) packable "bulk fill" resin composites and 2) flowable "bulk fill" resin composites. Packable "bulk fill" resin composites can be used for restoration of the entire cavity, including the occlusal surface. Flowable "bulk fill" resin composites, however, are to be used as a "base" restoration and are intended to be covered by a final, occlusal layer of a packable resin composite. This final layer of packable resin composite is necessary because flowable "bulk fill" resin composites have lower surface hardness, elastic modulus, and abrasion resistance due to their reduced filler content.[10]

Previous studies[11-14] have shown that mechanical properties such as surface hardness and abrasion resistance of flowable "regular" resin composites are inferior to those of their packable counterparts. This might also apply to flowable "bulk fill" resin composites, and whereas the occlusal surface of a Class II flowable "bulk fill" resin composite restoration is covered by a final layer of packable resin composite, the flowable "bulk fill base" in the gingival part of the approximal surface is not. Because of inferior mechanical properties, flowable "bulk fill" resin composites used as "base" restorations in Class II cavities are likely to undergo degradation when exposed to the various incidents that commonly occur in the oral cavity (eg, toothbrushing and interdental hygiene procedures as well as thermal variations). Thus, the aim of the present *in vitro* study was to investigate the marginal gap formation along the approximal margins of Class II flowable "bulk fill" resin composite restorations and to compare it with the marginal gap formation of packable "regular" resin composite restorations after artificial ageing. The null hypothesis was that there would be no difference between the two flowable "bulk fill" resin composites investigated and the packable "regular" resin composite in terms of marginal gap formation before and after artificial ageing.

METHODS AND MATERIALS

Cavity Preparation and Restoration

A total of 39 extracted human permanent molars were used (n=13 molars/group; three groups: one

Table 1: *Resin Composites and Restorative Procedures*

Resin Composite	Restorative Procedure
Filtek Supreme XTE (3M ESPE, St Paul, MN, USA) Lot No. N628811	1. Increment Filtek Supreme XTE 2 mm; light-curing 20 s 2. Increment Filtek Supreme XTE 2 mm; light-curing 20 s 3. Increment Filtek Supreme XTE 2 mm; light-curing 20 s
Filtek Bulk Fill (3M ESPE, St Paul, MN, USA) Lot No. N421893	1. Base restoration Filtek Bulk Fill 4 mm; light-curing 10 s 2. Occlusal layer Filtek Supreme XTE 2 mm; light-curing 20 s
SDR (DENTSPLY DeTrey, Konstanz, Germany) Lot No. 1408000235	1. Base restoration SDR 4 mm; light-curing 10 s 2. Occlusal layer Filtek Supreme XTE 2 mm; light-curing 20 s

packable "regular" resin composite [Filtek Supreme XTE (3M ESPE, St Paul, MN, USA)] and two flowable "bulk fill" resin composites [Filtek Bulk Fill (3M ESPE) and SDR (DENTSPLY DeTrey, Konstanz, Germany)]). Before extraction, patients had been informed about the use of the teeth for research purposes, and verbal consent had been obtained. After extraction, the teeth were pooled. The local ethics committee categorizes pooled teeth as an "irreversibly anonymized bio-bank" and, thus, no ethical approval was needed. The molars to be used were cleaned under tap water with a scaler to remove debris and then stored in 2% chloramine solution in the refrigerator (4°C) until needed. Before preparation of the cavities, the roots of the molars were shortened under water-cooling with a grinding machine (Struers LaboPol-21; Struers, Ballerup, Denmark) and silicon carbide abrasive papers of #220 grit size (Struers). After grinding and to facilitate handling, the molars were embedded in cylindrical stainless-steel molds with self-curing acrylic resin (Paladur; Heraeus Kulzer, Hanau, Germany). After curing of the acrylic resin, the stainless-steel molds were removed. In each molar, a standardized mesial and distal Class II cavity was prepared through use of a coarse-grained preparation and a fine-grained finishing diamond bur (Intensiv 8113NR and FG 223B; Intensiv AG, Montagnola, Switzerland). The dimensions of the standardized cavity were 4 mm in oro-vestibular width, 6 mm in occluso-cervical height (including a margin below the cemento-enamel junction), and 2 mm in mesio-distal depth. The margins of the cavity were not beveled. Then, a circular curved transparent matrix (Lucifix Molar; KerrHawe, Bioggio, Switzerland) was placed, on which the thickness of the future increments of resin composite (depending on the group, as listed in Table 1) was marked with a water-resistant felt pen. The cavities were pretreated with the three-step etch-and-rinse adhesive system OptiBond FL (Kerr, Scafati, Italy) according to the manufacturer's instructions (etching of the cavity for 15 seconds with 37.5% phosphoric acid [Kerr Gel Etchant, Lot No. 5329366; Kerr], 15-second water spray, three-second air-dry, 15-second application of OptiBond Prime [Lot No. 48574776; Kerr], five-second air-dry, 15-second application of OptiBond Adhesive [Lot No. 4851978; Kerr], three-second air-dry, and 10-second light-cure). Subsequently, the restorations of both cavities were carried out as specified in Table 1 (n=13 molars/group, resulting in n=26 cavities per resin composite; three resin composites). All light-curing was conducted with an LED curing unit (Demi LED; Kerr; irradiance 1500 mW/cm^2; validation of the light efficiency by a radiometer [MARC PS; Blue-Light Analytics Inc, Halifax, NS, Canada]). After removal of the matrix, the restorations were finished and polished with Sof-Lex XT Discs (Sof-Lex XT Discs coarse, medium, fine, and superfine; 3M ESPE). The discs were changed after polishing of each restoration. The restored molars were then kept in tap water for 24 hours at 37°C.

Specimen Preparation and Production of "Baseline" Replicas

After tap water storage for 24 hours, each molar was cut in half in the oro-vestibular direction between the two restorations with a water-cooled diamond saw (IsoMet Low Speed Saw; Buehler, Lake Bluff, IL, USA), resulting in two specimens per molar. These specimens were also embedded in cylindrical stainless-steel molds with self-curing acrylic resin (Paladur; Heraeus Kulzer), letting the restorations protrude from the acrylic resin. After curing of the acrylic resin, the specimens were cleaned with deionized water in an ultrasonic bath (TUC-150; Telsonic AG, Bronschhofen, Switzerland) for three minutes and then thoroughly air-dried. From each specimen, polyvinylsiloxane impressions were taken (addition-type silicone, surface-activated PRESIDENT heavy body [Lot No. F93948] and regular body [Lot No. F83175]; Coltène/Whaledent, Altstät-

ten, Switzerland). The impressions were poured with epoxy resin (EpoFix; Struers) to produce "baseline" replicas.

Artificial Ageing of the Specimens

The specimens were divided into two groups of artificial ageing: At the beginning of each month during six months' storage, one specimen per molar was subjected to mechanical toothbrushing (syndicad LR1; syndicad Dental Research, Munich, Germany) for 500 cycles (~8.5 minutes) using a toothpaste slurry (50 g with a ratio of 1:1 deionized water and toothpaste [M-Budget toothpaste; Migros Genossenschaftsbund, Zurich, Switzerland; RDA ~70]), while the other specimen was subjected to thermocycling (1000 cycles; 5°C/55°C, 30-second exposure time). During the six months' storage, all specimens were kept in tap water at 37°C.

Production of "Final" Replicas and Measurement of Marginal Gap Formation

After storage, all specimens were again cleaned with deionized water in an ultrasonic bath (TCU-150; Telsonic AG) for three minutes, polyvinylsiloxane impressions were taken from each specimen, and new impressions were poured with epoxy resin, as previously described, resulting in "final" replicas.

The baseline and the final replicas were mounted on aluminum stubs and sputter-coated (100 seconds, 50 mA) with gold/palladium by use of a sputter-coating device (Balzers SCD 050; Balzers, Liechtenstein). Baseline and final replicas were then examined under a scanning electron microscope ([SEM] JEOL JSM6010PLUS/LV; JEOL, Tokyo, Japan), and SEM micrographs were produced. Since the restorations were located partly in enamel and partly in dentin (ie, below the cemento-enamel junction), marginal gap formation of the restorations was assessed separately at the "margin located in enamel" and at the "margin located in dentin." For the "bulk fill" resin composite restorations and in analogy to the clinical situation, the restorations were each regarded as one unit, and no distinction was made between the layer of flowable "bulk fill" resin composite and the top layer of packable "regular" resin composite.

First, the length of the entire enamel margin of each restoration was measured (in micrometers). In the case of gaps along the margin, the length of each gap was measured (in micrometers) and the individual gap lengths were added. The percentage of the total gap length was then calculated relative to the entire enamel margin. This procedure was repeated for the restorative margin located in dentin. All measurements of marginal gap formation were performed with the SEM software InTouch Scope Version 2.01 (JEOL) by one operator (SM) and were performed twice in order to calculate the intra-operator reliability.

The paramarginal gap formation was registered as being either present or absent, and the percentage of paramarginal gaps was calculated for each group.

Statistical Analysis

The intraoperator reliability of the two measurements of marginal gap formation was calculated using the Kendall Tau. The percentages of marginal gaps in enamel as well as in dentin were statistically analyzed with a nonparametric analysis of variance (ANOVA) to test for an effect of the three factors "resin composite" (ie, Filtek Supreme XTE, Filtek Bulk Fill, or SDR), "artificial ageing" (ie, baseline or final), and "type of artificial ageing" (ie, mechanical toothbrushing or thermocycling) and of their interactions. In case of a significant effect of one of the factors, post hoc analysis was performed using the Wilcoxon-Mann-Whitney test. The comparison between gap formation in enamel and dentin was done using the Wilcoxon signed rank test.

All p-values were corrected with Bonferroni-Holm adjustment for multiple testing. The statistical analysis was performed with R, version 3.3.1 (The R Project for Statistical Computing, Vienna, Austria) using the packages "irr" and "nparLD" after the level of significance had been set at $\alpha=0.05$. The distribution of paramarginal gaps was analyzed descriptively.

RESULTS

Representative SEM micrographs of restorative margins in enamel and dentin are shown in Figure 1 for margins without gap formation and in Figure 2 for margins with gap formation.

The Kendall Tau value regarding the intraoperator reliability between the two measurements of marginal gap formation was 0.96 for enamel and 0.97 for dentin. As a result of the high Kendall Tau for both tooth substances, the percentages of marginal gap formation in enamel as well as in dentin from the first measurement were pooled with the percentages of the second measurement for each of the 13 restorations in each group. These results are shown in Table 2 as mean values and standard deviations whereby differences in dentin gap forma-

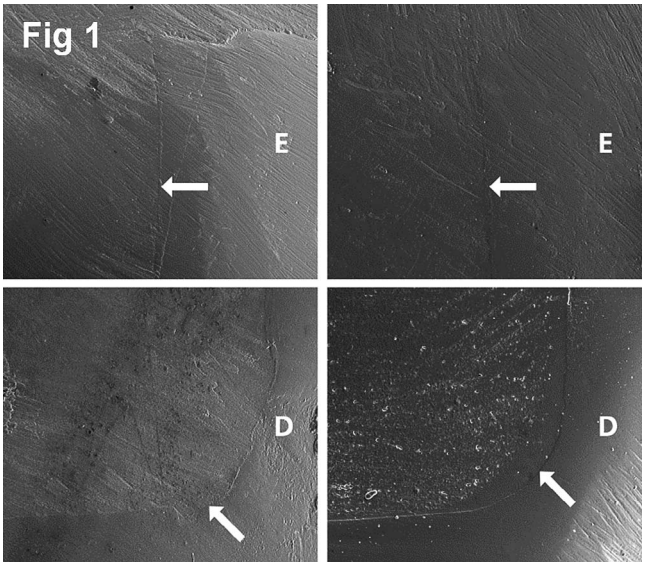

Figure 1. *Representative scanning electron microscope micrographs of restorative margins in enamel (E) and dentin (D) without gap formation.*

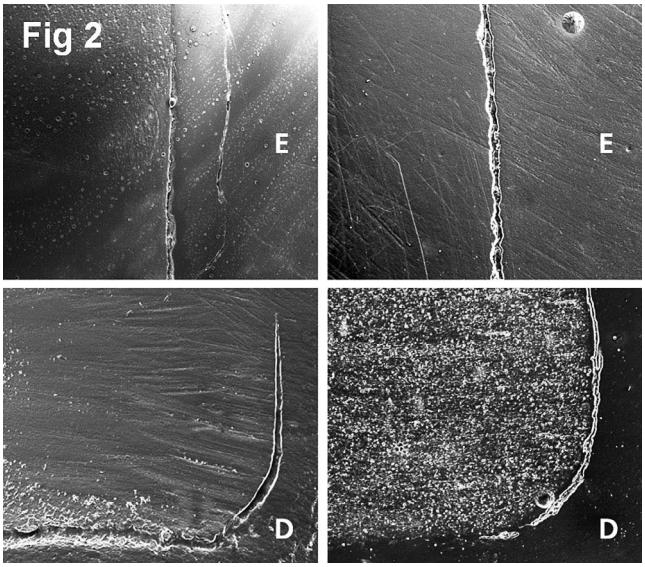

Figure 2. *Representative scanning electron microscope micrographs of restorative margins in enamel (E) and dentin (D) with gap formation.*

tion, undisclosed by median values, become apparent.

For enamel, the nonparametric ANOVA showed a significant effect of the factors "resin composite" ($p=0.0068$) and "artificial ageing" ($p<0.0001$) but no significant effect of the factor "type of artificial ageing." There were no significant interactions between the three factors. With regard to the effect of artificial ageing, gap formation increased significantly for all three resin composites after mechanical toothbrushing or thermocycling. With regard to the effect of resin composite, this factor did not interact significantly with the factor "artificial ageing" or with the factor "type of artificial ageing." Consequently, the percentages of marginal gap formation in enamel from the two points in time (baseline and final) and from the two types of artificial ageing were pooled for each of the three resin composites (Figure 3). Subsequently, Wilcoxon-Mann-Whitney tests showed significantly lower marginal gap formation in enamel for Filtek Supreme XTE compared to Filtek Bulk Fill ($p=0.0052$) and SDR ($p=0.0289$) and no significant difference between Filtek Bulk Fill and SDR ($p=0.4072$).

For dentin, the nonparametric ANOVA showed a significant effect of the factor "resin composite" ($p=0.0317$) but no significant effect of the factors "artificial ageing" or "type of artificial ageing." There were no significant interactions between the three factors, and consequently the percentages of marginal gap formation in dentin from the two points in time (baseline and final) and two types of artificial ageing were pooled for each of the three resin composites (Figure 3). Subsequently, Wilcoxon-Mann-Whitney tests showed significantly lower marginal gap formation in dentin for SDR compared to Filtek Supreme XTE ($p<0.0001$) and Filtek Bulk Fill ($p=0.0015$) and no significant difference between Filtek Supreme XTE and Filtek Bulk Fill ($p=0.4919$).

Furthermore, a Wilcoxon signed rank test found significantly lower marginal gap formation in dentin than in enamel ($p<0.0001$).

The distribution of paramarginal gaps is presented in Table 3. The percentage of restorations with paramarginal gaps varied between 0% and 85%, and the percentages were markedly higher after artificial ageing for all three resin composites, regardless of type of ageing. There were no characteristic or systematic differences between the three resin composites.

DISCUSSION

The present study investigated marginal gap formation along the approximal margins of Class II restorations and showed significant differences between the three resin composites compared. In enamel, the packable "regular" resin composite Filtek Supreme XTE showed less gap formation than did the two flowable "bulk fill" resin composites, Filtek Bulk Fill and SDR, before as well as after artificial ageing. In dentin, on the other hand, one of the "bulk fill" resin composites (SDR) showed less

Table 2: *Marginal Gap Formation (%) in Enamel and Dentin Before ("Baseline") and After the Two Types of Artificial Ageing ("Final") (Mean Values and Standard Deviations)*

Type of Artificial Ageing	Resin Composite		
	Filtek Supreme XTE (n=13)	Filtek Bulk Fill (n=13)	SDR (n=13)
Mechanical toothbrushing			
Enamel			
Baseline	29.7 ± 21.8	49.6 ± 21.2	46.9 ± 28.5
Final	68.6 ± 24.2	81.6 ± 11.6	72.1 ± 19.6
Dentin			
Baseline	2.9 ± 4.8	7.9 ± 27.7	0 ± 0
Final	4.1 ± 6.2	9.1 ± 26.9	7.0 ± 25.3
Thermocycling			
Enamel			
Baseline	29.0 ± 14.0	48.5 ± 28.5	47.0 ± 21.2
Final	66.6 ± 21.5	84.1 ± 13.9	81.0 ± 15.4
Dentin			
Baseline	4.6 ± 8.5	7.9 ± 27.5	0 ± 0
Final	4.6 ± 8.9	19.1 ± 28.6	0 ± 0

Table 3: *Distribution of Paramarginal Gaps (%) Before ("Baseline") and After the Two Types of Artificial Ageing ("Final")*

Type of Artificial Ageing	Resin Composite		
	Filtek Supreme XTE (n=13)	Filtek Bulk Fill (n=13)	SDR (n=13)
Mechanical toothbrushing			
Baseline	23.1	30.8	23.1
Final	84.6	46.2	65.4
Thermocycling			
Baseline	7.7	0	38.5
Final	53.9	61.5	50.0

gap formation than the other two resin composites. These results lead to rejection of the null hypothesis.

Whether or not marginal gaps are formed and the extent to which gaps are formed depend on an interplay between multiple factors,[15-23] some related to the resin composite, others related to the specific cavity and restorative procedure. In this study, we sought to keep constant the factors related to the cavity and the restorative procedure. Thus, care was taken during cavity preparation to obtain standardized cavities and, consequently, a constant C-factor for all restorations. Furthermore, a gold standard adhesive system was chosen[24] and applied strictly according to the instructions for use to ensure best possible adhesion, and light-curing was performed with a high-performance LED curing unit, the power intensity being continuously monitored.

Two factors related to the resin composite are of key importance to gap formation: polymerization shrinkage[16,17,25,26] and elastic modulus.[11,27,28] In adhesively bonded resin composite restorations, polymerization shrinkage generates stress at the tooth-restoration interface,[16,29,30] which has undesirable consequences, such as marginal gap formation, tooth deflection, and paramarginal enamel fractures.[30-34] According to Hooke's Law, polymeri-

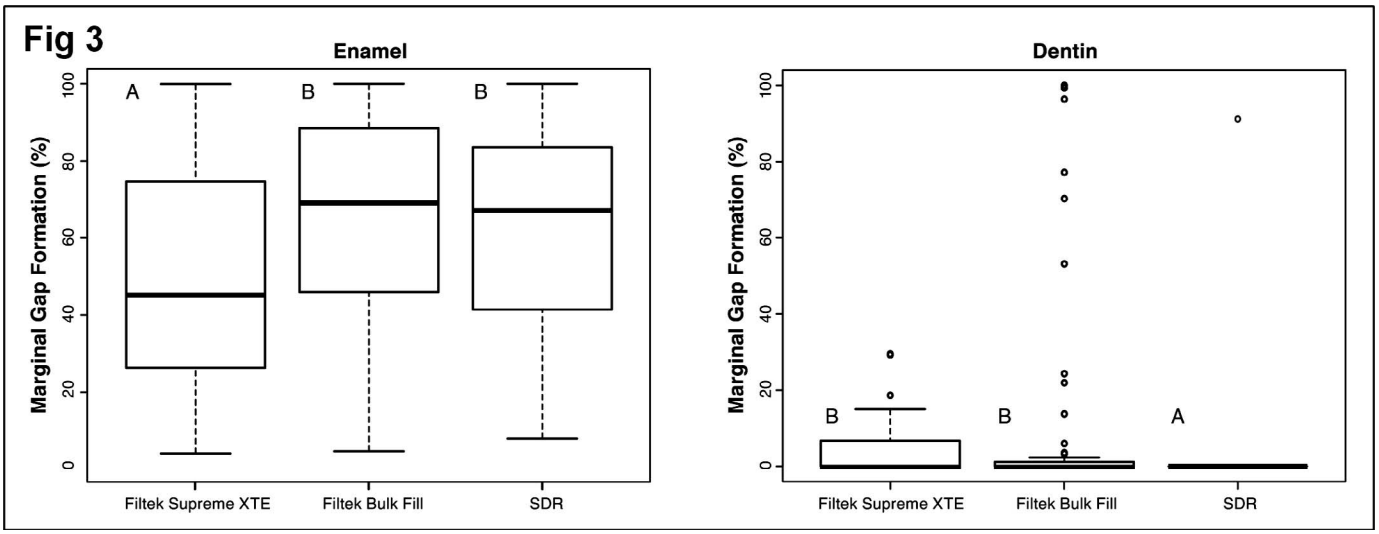

Figure 3. *Percentages of marginal gap formation in enamel and dentin (n=52 per group; for each of the resin composites, the percentages were pooled because of the absence of significant interactions between the three factors "resin composite" [ie, Filtek Supreme XTE, Filtek Bulk Fill, or SDR], "artificial ageing" [ie, baseline or final], and "type of artificial ageing" [ie, mechanical toothbrushing or thermocycling]; different letters indicate significant differences between the three resin composites within enamel or dentin).*

zation stress is the product of elastic modulus and strain.[30] This implies that resin composites with a combination of high polymerization shrinkage and high elastic modulus are expected to result in the highest polymerization stresses. Polymerization shrinkage and elastic modulus both depend highly on the filler content of the resin composite: the higher the filler content, the lower the polymerization shrinkage[16,35] and the higher the elastic modulus.[11,28,29] To allow for a higher increment thickness while maintaining an acceptable degree of monomer-polymer conversion, "bulk fill" resin composites need to be more translucent than "regular" resin composites. This is mainly obtained through a reduction in filler content.[36] Indeed, whereas the "regular" resin composite Filtek Supreme XTE had a filler content of 63.3%vol, that of Filtek Bulk Fill was 42.5%vol and that of SDR 45%vol.[8,9,36] As anticipated, the latter two materials have been reported to show higher polymerization shrinkage (3-4%vol vs 2%vol)[8,9,11,37,38] and lower elastic modulus (4-5 GPa vs 10-11 GPa).[11,26,36,37,39] While higher polymerization shrinkage would be expected to increase gap formation, lower elastic modulus would be expected to have the opposite effect.

The present study found higher gap formation in enamel of the two flowable "bulk fill" resin composites Filtek Bulk Fill and SDR compared to the packable "regular" resin composite Filtek Supreme XTE. Apparently, the lower elastic modulus of the "bulk fill" resin composites did not compensate for the higher polymerization shrinkage. In their study of four "bulk fill" and two "regular" resin composites, Kim and others[37] reported lower polymerization shrinkage stress of SDR compared with Filtek Z250, whereas Filtek Bulk Fill showed similar polymerization shrinkage stress as the "regular" resin composite. Despite an overall, strong positive correlation between polymerization shrinkage stress and tooth-composite interfacial debonding in Class I cavities for all six resin composites tested, no significant difference in interfacial debonding was found between Filtek Bulk Fill, SDR, and Filtek Z250, and the study[37] does not corroborate our findings. This may be explained by the numerous differences in study design between the two studies, such as cavity type, adhesive system, and methodology used to determine marginal integrity. However, a favorable effect of high elastic modulus of resin composites on enamel gap formation in Class II cavities has been reported by Benetti and others.[27] Of particular relevance for the present results is the finding that the use of a resin composite with low elastic modulus and high polymerization shrinkage (Charisma, Heraeus Kulzer) resulted in more severe gap formation in enamel than did the use of a resin composite with higher elastic modulus and lower polymerization shrinkage (Grandio, VOCO, Cuxhaven, Germany), and this result is in agreement with our findings. Unfortunately, after thermocyclic and mechanical loading the lower gap formation of Grandio was accompanied by a higher frequency of paramarginal enamel fractures. In the current study, a tendency was indeed observed toward more paramarginal gaps for Filtek Supreme XTE than for the two "bulk fill" resin composites after the artificial ageing involving mechanical toothbrushing but not after the artificial ageing involving thermocycling.

In dentin, one of the "bulk fill" resin composites (SDR) showed significantly less marginal gap formation than did Filtek Supreme XTE and the other "bulk fill" resin composite (Filtek Bulk Fill). This positive result for SDR may be explained by SDR having generated less polymerization shrinkage stress than Filtek Supreme XTE and Filtek Bulk Fill,[14,37] possibly as a result of containing a "polymerization modulator" embedded in the backbone of the polymerizable resin,[40] which supposedly counteracts polymerization stress through lower polymerization rate.[14] The positive results for SDR also corroborate with findings of previous studies[26,41,42] in which SDR was used as a base filling in Class II cavities and which found the marginal integrity to be as good as that of a conventionally layered "regular" resin composite. Moreover, the result indicates that the elastic modulus is not as important for gap formation in dentin as it is for gap formation in enamel. This result is in harmony with that of Benetti and others,[27] who found no difference in dentin gap formation among three "regular" resin composites of highly varying elastic modulus.

Clearly, gap formation was more severe at enamel margins than at dentin margins before as well as after artificial ageing. First, this indicates that the adhesive system is able to create a durable bond to dentin. Second, this may imply that the situation at the enamel margins was more challenging than at the dentin margins. One important factor could be the elastic modulus. It has often been claimed that the elastic modulus should be as similar as possible to the tooth structure so that the resin composite is able to flex with the tooth structure under mechanical load.[16,43] The elastic moduli of the three resin composites investigated in the present study (4-5 GPa to 10-11 GPa)[11,26,36,37,39] are much closer to that of dentin (13-19.0 GPa)[43-45] than to that of enamel

(80-94 GPa),[43] the much bigger difference for enamel resulting in higher stress formation. In their study of the effect of a 4-mm SDR base in Class II MOD cavities, Roggendorf and others[41] found more severe gap formation in dentin than in enamel after thermomechanical loading. This result is in contrast with our findings and cannot easily be explained. One reason could be the difference in cavity type between the two studies (Class II MO/OD cavities in the present study vs Class II MOD cavities in the other study). Another reason could be the more rigorous thermomechanical loading procedure endured by the MOD restorations, both factors exposing the MOD restorations to much higher levels of stress.

The current study subjected the Class II restorations to two artificial ageing protocols intended to simulate not only long-term exposure to the high humidity of the oral cavity but also some of the mechanical and thermal challenges that these restorations endure during normal function. Such challenges may give rise to stress formation due to cyclic, subcatastrophic mechanical loading as well as a mismatch between the coefficient of thermal expansion of the resin composite and the tooth substance. Both artificial ageing protocols led to aggravated gap formation in enamel and an increase in paramarginal gaps, but they had no detrimental effect on gap formation in dentin. The aggravation of gap formation in enamel as a consequence of the artificial ageing protocols is in agreement with previous findings,[27,41] but these studies also reported an aggravation of gap formation in dentin. The fact that we found no difference between the two artificial ageing protocols may imply that the aggravation in gap formation observed was caused primarily by the long-term water storage *per se* and that the mechanical toothbrushing and thermocycling protocols were inadequate to provoke a significant effect. Indeed, with regard to thermocycling, it has been reported[46] that simulation of one-year clinical function requires a total of 10,000 cycles, which is almost double the 6000 cycles applied in the present study.

Obviously, randomized clinical trials are the ultimate tool for evaluating the performance of dental treatments. However, clinical trials are not only extremely resource demanding and time consuming to perform, but once the results of clinical trials with a meaningful follow-up time are published, the information tends to be obsolete as the materials and techniques employed are no longer on the market. Furthermore, the large number of materials available within practically every material category gives an almost infinite and unrealistic number of combinations of materials and techniques to be investigated. For these reasons, researchers conduct preclinical screenings in the form of laboratory studies of properties and performances that are deemed clinically relevant. *In vitro* models allow for testing hypotheses in a controlled laboratory set-up that would be unviable *in vivo*. In the present study, we intended to get an impression of the middle-term performance of Class II restorations with two "bulk fill" resin composites investigated through six months' water storage combined with either mechanical toothbrushing or thermocycling. The lack of effect on dentin gap formation indicates that the protocols were not sufficiently harsh or long-lasting to challenge the materials. Furthermore, the apparent difference in paramarginal gap formation at baseline between the restorations later to be subjected to toothbrushing and those to be subjected to thermocycling implies that the registration of paramarginal gaps (absent vs present) was too crude. Although marginal integrity is not the only factor to influence clinical success, in view of the scarce amount of data on marginal gap formation of "bulk fill" resin composites, more studies applying clinically relevant thermal and mechanical simulation protocols are warranted.

CONCLUSIONS

Marginal gap formation was higher in enamel than in dentin. In enamel, the "regular" resin composite Filtek Supreme XTE showed less marginal gap formation than did the two "bulk fill" resin composites. In dentin, however, one of the "bulk fill" resin composites, SDR, showed less marginal gap formation than did Filtek Supreme XTE and the other "bulk fill" resin composite, Filtek Bulk Fill.

These results suggest that in deep Class II cavities, flowable "bulk fill" resin composites can be an alternative to packable "regular" resin composites.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Kantonale Ethikkommission KEK, Bern, Switzerland. The approval code for this study is Req-2016-00332.

Conflict of Interest

The authors declare no conflicts of interest, real or perceived, financial or nonfinancial.

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Effect of Toothpaste Use Against Mineral Loss Promoted by Dental Bleaching

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Clinical Relevance

Dental bleaching promotes slight mineral loss associated with the surface changes of enamel, which might be prevented by toothpaste use prior to treatment.

SUMMARY

Aim: To investigate the effect of different toothpaste formulations used prior to dental bleaching with 35% hydrogen peroxide (HP) on the mineral content and surface morphology of enamel. Methods: Seventy bovine enamel blocks (4×4×2 mm) were submitted to *in vitro* treatment protocols using a toothbrushing machine prior to dental bleaching or a placebo procedure (n=10) as proposed in the following groups: unbleached control (PLA), bleached control (HP), and brushing with differing toothpastes prior to HP bleaching, including: potassium nitrate toothpaste containing sodium fluoride (PN), sodium monofluorophosphate/MFP toothpaste (FT), arginine-carbonate (8% arginine) (PA) or arginine-carbonate (1.5% arginine) toothpaste (SAN), and toothpaste containing bioactive glass (NM). Phosphorus concentration in gel ([P]) was evaluated (μg of P/mg of gel), and the elemental levels (wt%) of Ca, P, and Na as well as the proportion between Ca and P and spectra graphics were determined using an energy-dispersive X-ray spectrometer (EDS). The surface morphology was assessed using scanning electron microscopy (SEM). The data were subjected to analysis of variance and the Tukey test (α=0.05).

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Results: HP demonstrated the greatest [P] values in gel, being statistically different from PLA. The [P] of NM was statistically similar to PLA. HP showed a significant decrease in the Ca% and Ca/P values when compared to PLA in EDS analysis. PA showed Ca/P values statistically different from HP. In accordance with SEM analysis, the PA, SAN, and NM groups presented a smooth and uniform enamel surface, while HP and FT demonstrated some alterations in morphology. Conclusion: The toothpastes containing bioactive glass or arginine carbonate used prior to dental bleaching were effective in protecting enamel against mineral loss promoted by the whitening procedure.

INTRODUCTION

Tooth whitening has become a popular procedure for patients who seek to improve the color appearance of their teeth. Currently, dental bleaching procedures utilize hydrogen peroxide (HP) as an oxidizing agent.[1] Although the bleaching mechanism is not totally understood, it has been suggested that, when in contact with dental tissues, there is an oxidation reaction, and HP releases free radicals, hydroxyl anions, and reactive oxygen molecules,[2] all of which break up the organic pigmented molecules,[3,4] resulting in smaller and less heavily pigmented molecules that absorb less light in dentin.[1,4] This change in pigmentation causes teeth to appear lighter. With careful diagnosis and appropriate attention to the bleaching procedure, dental bleaching is considered a relatively conservative and safe approach for the treatment of discolored teeth.[5]

Dental bleaching may cause structural changes to the tooth surfaces, including changes to surface morphology and physical-chemical properties. Some studies have demonstrated enamel surface alterations following exposure to HP-based gels, suggesting that the procedure results in mineral loss that may be related to changes in surface microhardness, roughness, and the mineral content of enamel.[6-10] Calcium and phosphate ions are the main constituents of the hydroxyapatite crystal found in dental enamel, and their concentration is largely affected by the dissolution process, as occurs during a demineralization/erosive event.[11] Nevertheless, few studies have chemically assessed the concentration of these ions after tooth bleaching.[9,10] It is unknown how the bleaching procedure can change the concentration of phosphorus in the hydroxyapatite crystals of enamel. Additionally, tooth dissolution may result in defects and porosities on the enamel surface visualized under microscopic evaluation.[7,12]

Saliva and other active agents play an essential role in maintaining or creating an active environment to reduce demineralization or to promote remineralization in tooth surfaces exposed to bleaching treatment.[13,14] Currently, a commercially available bioactive glass-based toothpaste (calcium sodium phosphosilicate , NovaMin, SmithKline Beecham Consumer Healthcare, Berkshire, United Kingdom) has been promoting enamel remineralization[15] and is indicated for dentin hypersensitivity treatment.[16] Bioactive glass was originally developed as a bone regeneration material that demonstrated beneficial effects when incorporated into the bleaching gel or when used before or after dental bleaching.[17,18] Additionally, an arginine-carbonate–based toothpaste has been commercially available for caries control[19] and hypersensitivity treatment.[20] However, few studies exist in the literature that discuss the effects of arginine-carbonate toothpaste on preventing erosive surface loss[21] and its effect prior to, during, or after bleaching.[18,22] Therefore, the effect of toothpastes on the enamel surface when used prior to dental bleaching has been rarely assessed.

The aim of this study was to evaluate the potential effect of different toothpaste formulations used prior to dental bleaching on the surface morphology and mineral content of enamel. The null hypotheses tested were 1) that the dental bleaching would not affect the calcium or phosphorus content of the enamel, 2) that the toothpaste would not affect the mineral content of the enamel when used prior to treatment, and 3) that there would be no difference between the different toothpastes tested in protecting enamel against calcium or phosphorus loss promoted by dental bleaching.

METHODS AND MATERIALS

Sample Preparation

Freshly extracted and intact young bovine incisors were selected and stored in a 0.1% thymol solution for one month. Seventy enamel-dentin blocks, with dimensions of 16 mm^2 and 2 mm in height (1 mm of enamel and 1 mm of dentin), were prepared from the buccal surfaces of the teeth. The sectioning of teeth was performed using a high-concentration diamond disc (Buehler, Lake Bluff, IL, USA) coupled with a precision cutting machine (Isomet 1000, Buehler). Sectioned blocks were serially planed and polished using a water-cooled mechanical grinder and 600-, 1000-, and 2000-grit SiC papers (Buehler), and at the

end, they were polished using cloths and diamond spray (1, 0.5, and 0.25 μm, Buehler). The specimens were immersed in deionized water and placed in an ultrasonic bath for 10 minutes between and after the polishing procedures to obtain a standardized enamel surface. All block surfaces, excluding the enamel surface, were protected using an acid-resistant varnish (Risqué, Taboão da Serra, Brazil). Twenty-four hours before and during the study, all prepared samples were stored in artificial saliva, which was renewed every day, in a 37°C incubator. The artificial saliva contained a known composition of 1.5 mM Ca, 0.9 mM P, 150 mM KCl, 0.05 μg F/mL, and 0.1 M Tris buffer with a pH of 7.0.[23]

Toothpaste Treatment

Samples were submitted to linear brushing movements using toothbrush heads (Oral-B Indicator 40 Soft, Gillette do Brazil Ltda, Manaus, Brazil) attached to an automatic toothbrushing machine (Equilabor, Piracicaba, Brazil), using a static axial load of 200*g* and a speed of 5 movements/s at 37°C.[24] The samples were randomly assigned to the groups. In control groups (unbleached or bleached), the brushing was performed only with distilled water, while in the toothpaste groups, toothpaste/distilled water slurries (1:3) were used. The composition of each toothpaste and bleaching agent used, as well as the description of the experimental groups, is listed in Table 1. Toothbrushing was simulated using 825 cycles, which corresponded to one month, considering that approximately 10 to 15 strokes are performed in each toothbrushing event.[25] The blocks were washed with distilled water for 10 seconds and stored in artificial saliva for 24 hours prior to the dental bleaching procedures.

Bleaching Treatment and Gel Collection

A 35% HP gel (Whiteness HP, FGM, Joinville, Brazil) was applied to the exposed enamel area following the manufacturer's instructions. The composition of the gels is described in Table 1, with the initial and final pH of the bleaching agent measured in triplicate, thus presenting three readings for the bleaching gel (means: initial pH=5.64; after 15 minutes=4.87) and for the placebo gel (buffered to pH 6.0) using a pH meter (Procyon, São Paulo, Brazil). The bleaching or placebo gel was applied to the enamel surfaces three times for 15 minutes per application, without intervals between applications. Each application was performed using a precise amount of gel measured (0.010±0.002 g) using an analytical balance (AUW 220 d, Shimadzu, Kyoto, Japan). The weight values of the gel were used to calculate the phosphorus concentration per gram of gel. After exposure of the bleaching gel to the tooth, the specimens were placed in 0.5 mL of deionized water (gel rising water) in closed bottles and submerged in an ultrasonic bath for 30 seconds (Marconi, Piracicaba, São Paulo, Brazil). Immediately after, the specimens were submitted to 1 minute of vigorous stirring. This process was repeated three times for each sample, considering that the application of gel was performed following the manufacturer's instructions. The mixture of gel and distilled water used to rinse off the sample in each application was analyzed to assess the phosphorus concentration.

Phosphorus Measurement in Gel

Phosphorus concentration was evaluated relative to the weight of the bleaching or placebo gel used in each specimen. The phosphorus quantification for each sample proposed in this study was calculated by adding the three applications (μg of P/mg of gel), using the method described by Fiske and Subbarow.[26] In this method, the reaction consisted of the mixture of bleaching gel and distilled water, ultrapurified water, and molybdic acid solution (ammonium molybdate at 2.5% in sulfuric acid), which were vigorously vortexed. After 10 minutes, the reducing agent (1-amino-2-naphthol-4-sulfonic acid, sodium sulfite, and bisulfate) was added, and the mixture was vortexed again. After 20 minutes, the blue color intensity was measured using a spectrophotometer (DU 800, Beckman Coulter, Brea, CA, USA) at 660 nm and calibrated with standards from 0 to 24 μg P/mL. In a previous pilot study, the addition of a phosphorous standard (6 μg of P) in the mixture and the use of a control (6 μg of P+distilled water) for evaluation of the minimal variation of phosphorus concentration between the specimens of the same group were planned. In order to evaluate the variation of phosphorus concentration during bleaching procedures, the baseline values of phosphorus in gel not exposed to enamel were evaluated. The phosphorus concentration of gels at baseline was obtained by diluting the fresh gel in distilled water, thus repeating the process described for the collection methods of gel.

Energy-Dispersive X-Ray Spectrometry

Five samples per group were randomly selected for analysis by energy-dispersive X-ray spectrometry (EDS). The selected samples were submitted to sputtering under vacuum (Desk II, Denton Vacuum, Moorestown, NJ, USA) for the application of a fine

Table 1: *Groups, Products, and Toothpastes Used in This Study*[a]

Groups	Toothpaste	Treatment/Bleaching[b]	Manufacturer	Composition[b]
Unbleached control (PLA)	*Without toothpaste (distilled water)*	*Placebo gel (three times for 15 min each application)*	*Proderma, Piracicaba, Brazil*	*Water, neutralized carbopol, glycerin, triethanolamine, buffered in pH 6.0*
Bleached control (HP)	*Without toothpaste (distilled water)*	*35% HP (three times for 15 min each application)*	*Whiteness HP, FGM, Joinville, Brazil*	*35% hydrogen peroxide, thickener (carbopol), glycol, water*
Potassium nitrate toothpaste containing NaF (PN)	Sensodyne Fresh Impact	35% HP	GlaxoSmithKline Brasil Ltda, Rio de Janeiro, Brazil	5% potassium nitrate, dodium fluoride (NaF) 1426 ppm, water, hydrated silica, sorbitol, glycerin, cocamidopropyl betaine, xanthan gum, titanium dioxide, sodium sacharin, sucralose, mentha piperita, d-limonene
MFP fluoride toothpaste (FT)	Colgate Maximum Cavity Protection	35% HP	Colgate-Palmolive, São Bernardo do Campo, Brazil	Sodium monofluorophosphate (MFP) 1450 ppm, water, calcium carbonate, glycerin, sodium lauryl sulfate, cellulose gum, tetrasodium pyrophosphate, sodium bicarbonate, benzyl alcohol, sodium saccharin, sodium hydroxide
Arginine- carbonate–based toothpaste (8% arginine) (PA)	Colgate Sensitive Pro relief Pro-Argin	35% HP	Colgate-Palmolive, São Bernardo do Campo, Brazil	8% arginine, MFP 1450 ppm, water, calcium carbonate, sorbitol, arginine bicarbonate, sodium lauryl sulfate, cellulose gum, titanium dioxide, tetrasodium pyrophosphate, sodium bicarbonate, benzyl alcohol, sodium saccharin, xanthan gum, limonene
Arginine- carbonate–based toothpaste (1.5% arginine) (SAN)	Colgate Maximum Cavity Protection PLUS Sugar Acid Neutralizer	35% HP	Colgate-Palmolive, São Bernardo do Campo, Brazil	1.5% arginine, MFP 1450 ppm, water, calcium carbonate, glycerin, arginine bicarbonate, sodium lauryl sulfate, cellulose gum, titanium dioxide, tetrasodium pyrophosphate, sodium bicarbonate, benzyl alcohol, sodium saccharin, sodium hydroxide
Toothpaste containing bioactive glass (NM)	Sensodyne Repair & Protect Novamin	35% HP	SmithKline Beecham Consumer Healthcare, Berkshire, United Kingdom	5% calcium sodium phosphosilicate, MFP 1426 ppm, glycerin, silica, PEG-8, titanium dioxide, carbomer, cocamidopropyl betaine, sodium methyl cocoyl taurate, sodium saccharin, d-limonene

Abbreviation: HP, hydrogen peroxide.
[a] *Italic cells are related to the gel composition, and roman cells show the composition of toothpastes.*
[b] *According to the manufacturer's instructions.*

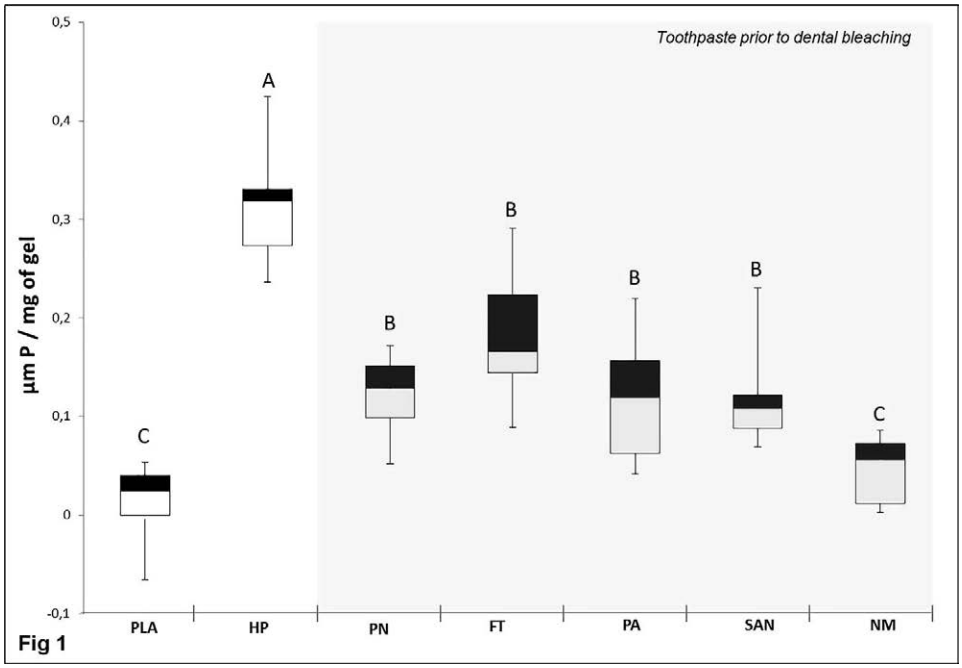

Figure 1. *Box plot of phosphorus concentration in the gel. The higher box plot means that values represent phosphorus loss of enamel with gel. PLA, placebo; HP, 35% hydrogen peroxide; PN, potassium nitrate (with NaF); FT, fluoride as MFP; PA, 8% arginine; SAN, 1.5% arginine; NM, bioactive glass.*

carbon layer. The EDS point analysis (Vantage, Acquisition Engine Co, Tokyo, Japan) was performed on the enamel surface to determine the elemental presence of Na, Mg, Al, Si, P, and Ca. The elemental levels (wt%) of Ca, Na, and P and the proportion between Ca and P were determined. For each sample, five points were randomly selected (300 μm^2 for each point), and the mean values were calculated.

Scanning Electron Microscopy

For analysis by scanning electron microscopy (SEM), another five specimens from each group were randomly selected and subjected to vacuum in a sputter coater (SCD 050, Balzers Union Aktiengesellschaft, Balzers, Liechtenstein) to deposit a thin layer of gold, equivalent to 10^{-6} mm, in order to increase the surface reflectance. Then images (4000×) of representative areas of the specimens were obtained using SEM (JSM-5600LV, JEOL, Tokyo, Japan).

Statistical Analysis

The data of phosphorus concentration in the gel and elemental levels (wt%) of Ca, Na, and P and the proportion between Ca and P were statistically analyzed using SAS software (SAS Institute Inc, Cary, NC, USA). The normal distribution of the values was verified, and a parametric analysis was performed. The data were subjected to a one-way analysis of variance and the Tukey test for multiple comparisons (α=0.05).

RESULTS

Phosphorus Concentration in Gel

Based on the phosphorus concentration [P] analysis for the gel (Figure 1), the results demonstrated that dental bleaching (HP, bleached control) resulted in greater phosphorus loss ($p<0.001$) when compared to the unbleached control (PLA) group. Furthermore, all toothpaste applications prior to dental bleaching decreased the [P] values, which differed statistically from the bleached control (HP) group; however, the toothpaste containing bioactive glass (NM) group was the only one that did not show significant difference in relation to PLA ($p>0.05$).

Elemental Levels (wt%) and EDS

The values of the relative percentage weight of calcium (Ca%) are presented in Table 2. The Ca% values of the HP group were significantly less than the PLA group ($p<0.001$). The application of NM and arginine-carbonate (1.5% arginine) toothpaste (SAN) did not significantly affect the relative Ca% compared to the PLA group ($p>0.05$) or the HP group ($p>0.05$).The Ca% values of the arginine-carbonate (8% arginine) (PA) group were statistically similar to the HP control group ($p>0.05$). The smallest values of Ca% were found in the potassium nitrate (PN)

Table 2: *Mean (Standard Deviation) of Elemental Levels (wt%) for EDS Analysis of Enamel Surface According to the Treatment Group*[a]

	Calcium	Phosphorus	Sodium	Ca/P
PLA	65.52 (0.15) A	33.68 (0.11) C		1.95 (0.01) A
HP	65.03 (0.07) B	34.10 (0.11) AB	—	1.91 (0.01) BC
PN	64.61 (0.14) C	34.31 (0.10) A	—	1.88 (0.01) B
FT	65.07 (0.19) B	34.06 (0.23) AB	—	1.91 (0.01) BC
PA	65.09 (0.26) B	33.59 (0.27) C	0.54 (0.12) A	1.94 (0.01) A
SAN	65.19 (0.22) AB	34.17 (0.21) A	—	1.91 (0.02) BC
NM	65.13 (0.25) AB	33.73 (0.15) BC	0.64 (0.03) A	1.93 (0.01) AB

[a] *Identical letters indicate no significant difference (*p*>0.05) among different groups (vertical). Italics indicate the control groups. — represents % wt < 0.01. Abbreviations: PLA, placebo; HP, 35% hydrogen peroxide; PN, potassium nitrate (with NaF); FT, fluoride as MFP; PA, 8% arginine; SAN, 1.5% arginine; NM, bioactive glass.*

group, which significantly differed from all other groups ($p<0.01$).

The relative percentage weight values of phosphorus (P%) are presented in Table 2. The relative P% was significantly higher in the HP group when compared to PLA group ($p<0.05$). The PA and NM groups did not significantly differ from the unbleached control; therefore, only PA differed statistically from the HP group ($p<0.05$), presenting lower values of P%. The P% values observed in the PN and SAN groups were statistically higher than the PLA, PA, and NM groups ($p<0.01$), which were statistically similar between them ($p>0.05$) and between the sodium monofluorophosphate/MFP toothpaste (FT) and HP groups ($p>0.05$).

The EDS results showed absence (wt%) of Mg, Al, or Si quantifiable by specific software. Considering the representative spectra graphics presented in Figure 2, the PA, SAN, and NM groups demonstrated the presence of sodium (Na; note the sets); therefore, the sodium (Na%) was quantified (Table 2) only in the PA and NM groups, and it was not significantly different between them ($p>0.05$). The proportion between the Ca% and P% values of the HP group was significantly less than the PLA group ($p<0.001$). PLA obtained 1.95 (Ca/P), which was statistically similar ($p>0.05$) to the PA and NM groups; in addition, the PA was the only one statistically different from the HP group ($p>0.05$).

SEM

The SEM images collected (Figures 3 and 4) present a smooth and uniform enamel surface in the groups submitted to toothpaste application prior to the whitening procedure. Superficial alterations were found in the HP (Figure 3B) and FT groups (Figure 3D), demonstrating different levels of enamel demineralization with a loss of interprismatic substance and an increase in porosity. Figures 4C,D show retained mineral particles on enamel surfaces treated with NM, presenting a surface covered with a precipitate, which was not solubilized by the whitening procedures.

DISCUSSION

Null hypotheses 1 and 2 were rejected because the dental bleaching affected the calcium and phosphorus content of enamel and the toothpastes acted in the mineral content of the bleached enamel when used prior to the whitening procedure. Null hypothesis 3 was rejected because there was a difference in the effects found in the toothpastes used for protecting enamel against slight mineral loss promoted by dental bleaching. Dental enamel is the hardest mineralized biological tissue, containing approximately 96% mineral, 3% water, and 1% organic matter by weight.[27] The principal inorganic constituent of enamel is hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$, which tends to incorporate a number of elements (Na, K, Cl, F, Mg, Zn, Pb, Cu, Sn, and Al).[27,28] In this study, bovine teeth were used because the chemical and physical morphology of bovine enamel resembles that of human enamel.[29] Within this context, the mineral loss is associated with the release of calcium and phosphorus ions due to hydroxyapatite crystal dissolution, resulting in a decrease of enamel properties during dental bleaching.[6-10]

The effect of toothpaste on dental bleaching related to the mineral content of enamel is still unclear in that few studies[18,22,30] have evaluated the effect of the use of toothpastes in tooth whitening on enamel. Toothpastes are the most widespread products used in oral hygiene, and their interaction with dental treatment, as tooth bleaching, should be known. Potassium nitrate toothpaste, conventional fluoride toothpaste, arginine-based toothpaste, and

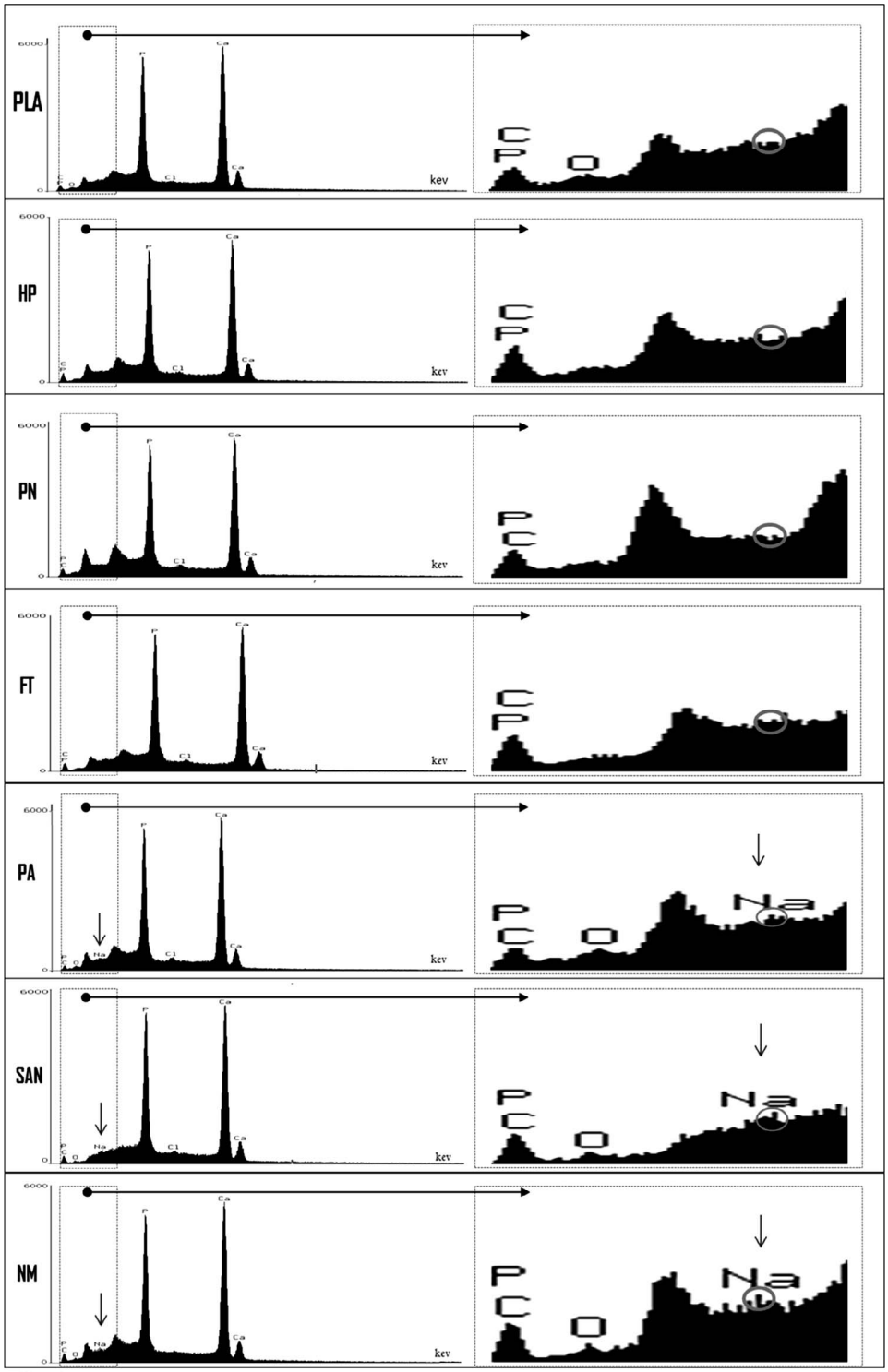

Fig 2

Figure 2. *Representative spectra graphics for EDS analysis. In image enlargement, a slight increase in the sodium peak (Na) is indicated by sets in the PA, SAN, and NM groups. PLA, placebo; HP, 35% hydrogen peroxide; PN, potassium nitrate (with NaF); FT, fluoride as MFP; PA, 8% arginine; SAN, 1.5% arginine; NM, bioactive glass.*

toothpaste containing bioactive glass assessed in the present study do not affect the whitening effectiveness of the bleaching procedure.[18] In the present study, the focus was to evaluate the potential effects of toothpaste application prior to dental bleaching by analyzing the enamel surface morphology and mineral content, especially by the new approach of quantifying the phosphorus content. The use of MFP toothpaste (FT) has been suggested as a nonactive control because MFP requires enzymatic hydrolysis

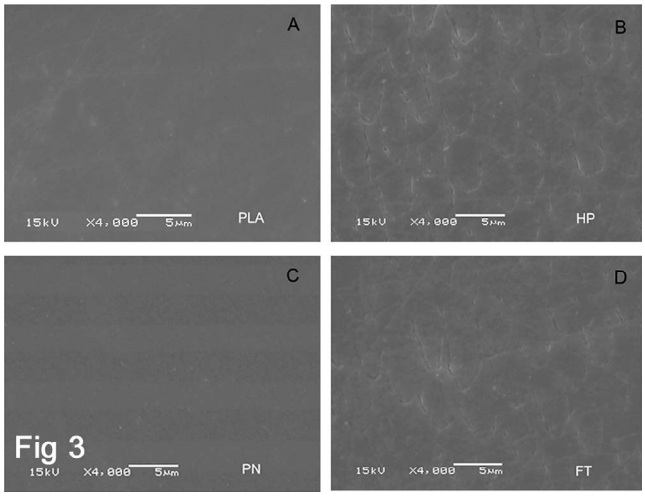

Figure 3. *Representative SEM images (4000×) of the specimens from the treatment groups. (A): Placebo (PLA). (B): Only bleached (HP). (C): Potassium nitrate toothpaste containing NaF (PN). (D): MFP fluoridated toothpaste (FT). PLA, placebo; HP, 35% hydrogen peroxide; PN, potassium nitrate (with NaF); FT, fluoride as MFP.*

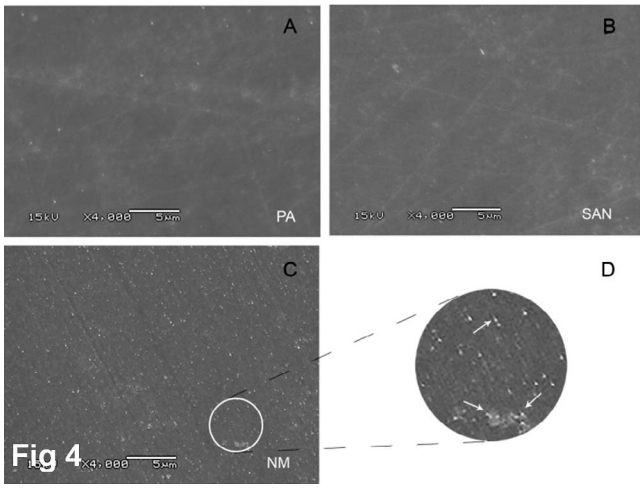

Figure 4. *Representative SEM images (4000×) of the specimens from the treatment groups. (A): 8% arginine-carbonate–based toothpaste (PA). (B): 1.5% arginine-carbonate–based toothpaste (SAN). (C): Toothpaste containing bioactive glass (NM). (D): Presence of mineral precipitates found in the NM group. PA, 8% arginine; SAN, 1.5% arginine; NM, bioactive glass.*

in human saliva and dental biofilm to release free fluoride.[31] Thus, as artificial saliva was used, the MFP had a low effect in *in vitro* models to prevent the demineralization of enamel. In another way, a NaF-based toothpaste was tested (PN group) and was considered to be an active group once this compound was ionized in aqueous solution, with fluoride ions available and active. NaF has been proven with *in vitro* results,[32] which is important because fluoride is currently used as an agent that promotes remineralization of dental hard tissues and that decreases the effects of demineralization.[32] The low effect of MFP on decreasing demineralization in *in vitro* studies was validated in SEM analysis. Figure 3 shows alterations in the enamel topography of the FT group, indicating mineral loss in the interprismatic areas and surface enamel similar to the HP group.

In dental bleaching, a mineral dissolution with loss of calcium and phosphorus occurs.[33,34] This adverse effect can be attributed to the action of free radicals resulting from the oxidation of the organic and inorganic elements on teeth[3] and also to the acidic pH of the bleaching agent applied.[35] EDS analysis showed a decrease in Ca% values for the bleached control group (HP); however, the P% values increased in the HP group. These percentage values are relative to the area analyzed in weight. The mineral gain and loss is possibly due to the crystal structure of $Ca_{10}(PO_4)_6(OH)_2$.[36] Due to the characteristics of the EDS methodology and once the amounts of all elements were presented as a mass percentage (%), a greater loss of calcium can increase the relative values of phosphorus, which exists in a small proportion in the hydroxyapatite crystal, and it also influences the analysis of other elements. Thus, an increase in calcium loss would lead to higher proportions for the P% values, but this does not mean that the bleached enamel surface incorporated phosphorus ions when compared to the placebo group. This was clearly demonstrated in the chemical analysis of gel, in which there was an increase in phosphorus loss of enamel with the gel (Figure 1).

The formation of mineral precipitates on enamel surface when using the bioactive glass toothpaste (NM) occurred due to the interaction of bioactive glass particles, Ca, P, and Na,[15] as shown by the representative images (Figure 4D) and spectra graphic (Figure 2). EDS analysis in the NM group suggested the relative percentage weight of Na (mean=0.64%) and Na presence in spectra graphics, indicating that it could be possible for the NM group to promote the formation of a hydroxycarbonate apatite layer[15] on the tooth surface. When bioactive glass is brought into contact with body fluids, there is a rapid capitation of Na, Ca, and P, thus creating precipitated minerals that are able to achieve remineralization of the damaged enamel surfaces[15,17,30,37] or reduce the effects of the demineralization process.[17,18] The results of SEM analysis (Figure 4D) show that the NM group presented uniform mineral precipitates covering the enamel surface

that remained intact. The NM toothpaste did not allow a morphological change on enamel in SEM images; this finding is corroborated by the roughness results described by Vieira-Junior and others[18] that reported similarity between the unbleached enamel and the bleached enamel previously exposed to bioactive glass toothpaste. Based on SEM images, EDS analysis, and the low concentration of phosphorus in the gels, it is possible to suggest that NM decreased the mineral loss of enamel, likely due to the fact that the hydroxycarbonate apatite precipitate was not lost by dissolution with the gel application. These results indicate a beneficial effect of bioactive glass toothpaste use prior to dental bleaching therapy and are in agreement with previous studies,[17,18] suggesting that the combination of the nonreacted bioactive glass particles and the newly formed hydroxycarbonate apatite layer results in preventing further demineralization.

In relation to arginine-containing toothpastes (PA and SAN), the effects on microhardness, roughness, or the mineral content of enamel in literature are rarely assessed.[18,38] It is thought that arginine is a source of calcium since it is a positively charged amino acid[39] that binds to negatively charged dental tissues and calcium carbonate.[40] Moreover, the alkaline environment promoted by toothpaste could buffer the pH of the gel, which might encourage calcium and phosphate ions to precipitate themselves under dental tissues.[40] When considering the Ca/P ratio and the P% values by EDS analysis (Table 2), the results may indicate a lower dissolution of hydroxyapatite crystal in the PA group (8% arginine) in the case that the PA group did not differ from the unbleached control (PLA). This effect can be mediated by the arginine concentration of toothpaste since these results were not replicated in the SAN group, which is composed of 1.5% arginine. However, these findings should be carefully evaluated since EDS analysis is relative (wt%) and performed in random sections of the enamel. The role of arginine in different demineralization/remineralization processes should be evaluated in future investigations.

The observed morphological changes on the enamel surface (Figures 3 and 4) suggest that the damaging effects of dental bleaching may be decreased by prior application of different toothpastes (PN, SAN, PA, and NM). Despite the values of Ca% and P% in EDS analysis, the PN group presented a polished and unchanged enamel surface in SEM analysis, indicating no morphologic alterations resulting from dental bleaching. Added to this, it is likely that there was an incorporation of fluoride ions (from NaF) on the enamel surface (calcium fluoride/ fluorhydroxyapatite), which decreased the values of the other element %s in EDS. This proposition is corroborated, since the phosphorus concentration in gel did not show an increase of phosphorus loss for the PN group. From this statement, it is clear that EDS analysis is a semiquantitative/qualitative method that should be applied in conjunction with further methodologies for chemical quantification of enamel after bleaching, as performed in this study.

In the present study, an automatic toothbrushing machine was used in order to standardize different variables. In *in vitro* models, the effect of toothpastes on dental structure may be influenced by several factors, such as the velocity of brushing, the type of movement, and the amount of load applied and temperature. Currently, considering the frequency with and time in which people perform their toothbrushing,[41] the overall recommendation for simulating the toothbrushing under *in vitro* conditions is to perform the brushing twice a day, 10 to 15 strokes per tooth.[25,42] Thus, the purpose was to simulate approximately 30 days of brushing, using 825 cycles, to create an enamel surface treated with the different active compounds studied.

In accordance with the presented results, there were differences in the potential effects among the different toothpastes used. Specifically, the toothpastes containing bioactive glass (NM) or arginine (PA) were efficient in minimizing the negative bleaching effects related to mineral loss and changes in enamel topography. Nevertheless, natural human saliva could provide an effective environment for the remineralization of dental hard tissues demineralized by dental bleaching; therefore, the pretreatment with toothpaste helps prevent the slight enamel phosphorus loss from dental in-office bleaching procedures, which may not be clinically relevant unless the patient's own postsalivary remineralization is impaired. Further research is needed to investigate the performance of bioactive glass or arginine containing toothpastes in *in situ* and *in vivo* studies regarding the combination of toothpaste and effects of bleaching agents on dental substrates.

CONCLUSIONS

The dental in-office bleaching procedure promoted slight phosphorus and calcium loss of enamel; however, the toothpastes containing bioactive glass or arginine-carbonate used prior to treatment were effective in preventing mineral loss.

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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.

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Two-year Effects of Chlorhexidine-containing Adhesives on the *In Vitro* Durability of Resin-dentin Interfaces and Modeling of Drug Release

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CAG Arrais • PV Farago • A Reis • AD Loguercio

Clinical Relevance

The addition of diacetate chlorhexidine up to 0.2% is a viable method by which to provide a drug release system in adhesive systems to maintain stable resin-dentin adhesive interfaces after two years of water storage.

SUMMARY

Objectives: To evaluate the effects of addition of diacetate chlorhexidine (CHX) at different concentrations into two etch-and-rinse adhesive systems on CHX release, as well as the immediate (IM) and two-year (2-Y) resin-dentin microtensile bond strength (μTBS) and nanoleakage (NL).

Methods: CHX was added to XP Bond (XP) and Ambar (AM) at concentrations of 0.0 wt% (control); 0.01 wt%; 0.05 wt%; and 0.1 to 0.2 wt%. To assess the cumulative CHX release,

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adhesive disks were made in a metallic matrix and after light-curing were stored in water. Ultraviolet spectrophotometric measurements of the storage solution were performed to examine the release kinetics of CHX. For μTBS and NL, the occlusal enamel of molars was removed and the adhesives were applied to the dentin surface after acid etching. After composite resin build-up, specimens were sectioned to obtain μTBS sticks. The specimens were subjected to μTBS and NL at IM and after 2-Y. In addition, specimens underwent examination for CHX using micro–Raman spectroscopy. All data were submitted to statistical analysis (α=0.05).

Results: With regard to CHX release, AM showed a slower and gradual release of CHX while XP released CHX more quickly ($p<0.05$), and CHX was still present in the hybrid layers after 2-Y. Both adhesives showed CHX release at 2-Y water storage. Both CHX-containing adhesives showed higher μTBS values than did the control group ($p<0.05$).

INTRODUCTION

New restorative techniques and materials have become more and more available, allowing increasingly esthetic and conservative procedures. As a result, "Adhesive Dentistry" has gained more attention, with the success of most restorative procedures relying mostly on adhesive systems providing optimal and durable bonding to tooth substrates.

Current adhesive systems are made up of three basic steps: etch, primer, and adhesive (bond). Briefly, an acid is used to demineralize dentin and enamel substrates and to increase their porosity, and the pores are filled with a primer, which is subsequently covered by a hydrophobic agent (bond) to ensure bonding with the resin composite material.[1-3] In the dentin substrate, mineral removal by acid etching exposes the collagen mesh and noncollagenous proteins. It is known that commercially available adhesive systems invariably fail in dentin infiltration[4-8]; thus, many sites of dentin may remain exposed. Thus, matrix metalloproteinases (MMPs) and cysteine cathepsins, which are involved in the degradation of exposed collagen, remain during the bonding procedure via an endogenous proteolytic mechanism, even in the absence of bacteria. These assumptions have been confirmed by *in vitro* and *in vivo* studies.[6,9-14]

Alternatives have been proposed for improving the bonding of adhesives to tooth, such as the use of agents that exhibit enzymatic inhibition.[15] Studies have shown potential inhibition of MMPs in compounds such as epigallocatechin-3-gallate[16,17]; galardin[18]; tetracyclines[19]; benzalkonium chloride[20]; quaternary ammonium salts, such as 12-methacryloyloxydodecylpyridinium bromide[21,22]; and ethylenediamine tetraacetic acid salt,[23,24] thus helping preserve the hybrid layer.

The most commonly and extensively tested compound is chlorhexidine (CHX),[25,26] which is an antimicrobial agent and is one of the first protease inhibitors evaluated in dentistry.[14] However, a majority of the *in vitro* studies applied CHX as an additional primer on acid-etched dentin. This procedure adds an extra step to the bonding protocol and does not fulfill the clinicians' need for simplification.

Sabatini[27] suggested that the inclusion of these protease inhibitor substances into one of the components of the bonding protocol could be an interesting alternative. Recently, the addition of CHX to the acid etchant[28,29] or to the adhesive solution[27,30-34] has been shown to effectively protect the adhesive interface against degradation over time. However, although the addition of CHX to the acid etchant showed promising results, a CHX reservoir is not produced immediately after acid etching. It has been hypothesized[33] that any approach that could provide this controlled release of CHX within the demineralized dentin could improve the durability of the resin/dentin interface by slightly transferring CHX to adjacent collagen. The CHX inclusion into primers and/or adhesives could produce a reservoir for a controlled release. However, no study to date has been found that evaluated the two-year stability of resin/dentin interfaces to CHX-containing adhesive as well as the kinetics of CHX release after two-year storage in water.

Therefore, this *in vitro* study evaluated the effects of CHX diacetate concentration added to two simplified etch-and-rinse (ER) adhesive systems on the release profile of CHX from adhesives as well as the immediate and two-year effects on the micro-tensile bond strength (μTBS) to dentin and nanoleakage at the bonding interface. In addition, micro–Raman spectroscopy was used to detect CHX within the hybrid layer. The following null hypotheses were tested: 1) There is no difference in the CHX release profile for each adhesive system evaluated; 2) The addition of CHX to adhesive composition at different concentrations does not impair immediate μTBS values to dentin and nanoleakage pattern at the bonding interface; and 3) There is no significant difference between immediate and two-year μTBS

Table 1: *Adhesive Systems: Composition and Application Mode*

Adhesive Systems	Composition	Application Mode
XP Bond (DENTSPLY)	TCB-resin, *terc*-butyl alcohol, PENTA, PPD, UDMA, TEGDMA, HEMA, modified carboxylic acid, nanofiller and dimethacrylate	1. Apply phosphoric acid to dentin for 15 s 2. Rinse for 15 s. Dry with absorbent paper. Keep dentin wet. 3. Apply the adhesive for 20 s undisturbed. 4. Gently air for 5 s to evaporate the solvent. 5. Light-cure for 20 s.
Ambar (FGM)	Methacrylate monomers (UDMA and MDP), photoinitiators, co-initiators, stabilizers, inert silica nanoparticles and ethanol	1. Apply phosphoric acid to dentin for 15 s. 2. Rinse for 15 s. Dry with absorbent paper. Keep dentin wet. 3. Apply two coats vigorously by rubbing the adhesive for 20 s (10 s each). 4. Gently air for 10 s to evaporate the solvent. 5. Light-cure for 10 s.

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; PENTA, pentaerythritol pentacrylate monophosphate; PPD, phenylpropanoid; TCB-resin, 1,2,3,4-butane-tetracarboxylic acid ester di2-hydroxyethylmethacrylate; TEGDMA, triethylene glycol dimethacrylate; 10-MDP, 10-methacryloyl oxidecil dihydrogenphosphate; UDMA, urethane dimethacrylate or 1,6-di (methacryloyloxyethylcarbamoil)-3,30,5-trimethylhexano.

values to dentin and nanoleakage patterns at the bonding interface when adhesive systems with CHX are used, regardless of CHX concentration.

METHODS AND MATERIALS

Formulation of the Experimental Adhesives

Four experimental adhesive systems were formulated using the simplified ER adhesive systems XP Bond (XP; Dentsply, York, PA, USA) and Ambar (AM; FGM Produtos Odontológicos Ltda, Joinville, SC, Brazil), according to the addition of different concentrations of CHX (99.9% pure chlorhexidine diacetate; Sigma Chemical Company, St Louis, MO, USA) (wt%): 0.01, 0.05, 0.1, and 0.2. The CHX was added to the adhesive and mechanically mixed by a motorized mixer (stirring). The bonding agents were also tested without any CHX added to their composition (control, commercial material). Therefore, a total of five groups for each adhesive were tested in the present study. Table 1 depicts their detailed composition and application mode.

Release Profile of CHX from Adhesives

Water standard solutions containing 1, 5, 10, 15, 20, 25, 30, 35, 40, 45, and 50 μg/mL of CHX were made to obtain an analytical curve with a linear regression between absorbance values and CHX concentrations, using the Genesys 10S UV-Vis Spectrophotometer (Thermo Scientific, Madison, WI, USA). The maximum absorbance of CHX at 260 nm (data not shown) was confirmed. Afterwards, 10 resin discs of each experimental group were produced in a brass mold (5.8 mm diameter, 1.0 mm thick). The unpolymerized adhesive was dispensed to completely fill the mold, and all visible air bubbles trapped in the adhesives were carefully removed. An air stream evaporated the solvent for 40 seconds at a distance of 10 cm. Under a glass cover slip, the adhesive was light-cured for 40 seconds using an LED light source at 600 mW/cm^2 (Radii-cal, SDI, Victoria, Australia). Then, the specimens were removed from the brass mold without undergoing permanent deformation, and each one was individually stored in deionized water.

At appropriate time intervals (one, three, and 12 hours and one, two, three, four, five, six, seven, 10, 12, 14, 21, and 28 days and two years), absorbance values of these storage solutions were obtained at 260 nm and converted into the amount of CHX released based on the linear analytical curve. Thus, the ultraviolet absorbance values at 260 nm of the control groups (0% CHX discs) were subtracted from the values produced from the CHX-containing discs, considering the concomitant monomer release. The cumulative release for the two-year period was represented as the percentage of CHX released and the mass (in milligrams) of CHX release per gram of the adhesive sample.[35,36] MicroMath Scientist TM 2.01 software (Salt Lake City, UT, USA) used mathematical models[37] to build cumulative profiles of CHX released from resin discs and to evaluate the CHX release profile.

Data were fit to first-order, biexponential, zero-order, Weibull, and monolag equations (Table 2). The best fit was chosen considering the correlation coefficient (r), the model selection criteria (MSC), and graphical adjustment. The power law (Korsmeyer-Peppas model: $ft=a\times t^n$) was the semiempirical equation developed to simulate the CHX release mechanism and to describe drug release from polymeric systems.[38] In this equation, ft is the

Table 2: *Mathematical Models Related to Chlorhexidine (CHX) Release Experiments*

Model	Equation[a]
Monoexponential	$\%D = 100(1 - e^{-kt})$
Biexponential	$\%D = 100[1 - (Ae^{-\alpha t} + Be^{-"\beta t})]$
Order zero	$\%D = kt$
Weibull	$\%D = 100[1 - e^{-(t/TD)b}]$
Monolag	$\%D = 100[1 - e^{-k(t-x)}]$

[a] *Where % is the percentage of dissolved drug over time; t, k, α, and "β are the observed dissolution rate constants; A and B represent the initial concentrations of the drug contributing to the two stages of dissolution; TD indicates the time in which 63.2% of the drug is dissolved; and b is the parameter related to structural and geometric characteristics of the resin disc.*

dissolved fraction of CHX at time t; n is the release exponent, indicative of the mechanism of the substance release; and a is the constant incorporating structural and geometric features of the resin disk. After the best model that described the release of CHX from the adhesive systems was determined, the time required to allow 50% release of CHX from each material was calculated, assuming that this equation remained as the dominant release mechanism over time, using MathWorks Matlab TMR 2012a software (Natick, MA, USA).[38]

Tooth Preparation and Bonding Procedures

Twenty-five caries-free extracted human third molars were used. The teeth were collected after the patient's informed consent was obtained. The teeth were disinfected in 0.5% chloramine and stored in distilled water. A flat dentin surface was exposed on each tooth after wet-grinding the occlusal enamel with 180-grit silicon-carbide (SiC) paper. The enamel-free, exposed dentin surfaces were further polished with 600-grit SiC paper for 60 seconds to standardize the smear layer. The adhesives were applied following the manufacturers' instructions (Table 1) and were light-cured using an LED light for 10 seconds at 600 W/cm^2 (Radii-cal, SDI). Resin composite blocks (Opallis, FGM) were incrementally built up on the bonded surfaces (three 1 mm–thick increments), and each resin layer was cured using the same curing light for 40 seconds. A single operator performed all bonding procedures in an environment with controlled temperature and humidity. Five teeth were used for each experimental group (n=5). After storage of the bonded teeth in distilled water at 37°C for 24 hours, they were longitudinally sectioned in both "x" and "y" directions across the bonded interface using a diamond saw in an automated sectioning device (Labcut 1010, Extec Corp, Enfield, CT, USA) under water cooling at 300 rpm to obtain bonded sticks with a cross-sectional area of approximately 0.8 mm^2. The cross-sectional area of each stick was measured using a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) to the nearest 0.01 mm and recorded for subsequent calculation of the μTBS. The bonded sticks from the same tooth were randomly divided and assigned to be tested immediately (IM) or after two-year storage [2-Y] in distilled water at 37°C. The storage solution was not changed and its pH was monitored monthly.

μTBS Test

Seven to nine bonded sticks from each tooth at each storage period were attached to a modified device for μTBS testing using cyanoacrylate resin (Super Bonder, Loctite, São Paulo, SP, Brazil) and subjected to a tensile force in a universal testing machine (Kratos, São Paulo, SP, Brazil) at 0.5 mm/min. The failure mode was evaluated at 40× (HMV-2, Shimadzu, Tokyo, Japan) and was classified as cohesive in dentin (failure exclusive within dentin; CD); resin cohesive in resin composite (failure exclusive within resin; CR); adhesive (failure at resin/dentin interface; A), or mixed (failure at resin/dentin interface that included cohesive failure of the neighboring substrates; M).

Nanoleakage (NL) Evaluation

Two bonded sticks from each tooth at each storage period that were not tested for μTBS were subjected to NL evaluation. All sticks were placed in 50 wt% ammoniacal silver nitrate in darkness for 24 hours, thoroughly rinsed in distilled water, and immersed in photo developing solution for eight hours under a fluorescent light to reduce silver ions into metallic silver grains within voids along the bonded interface. Specimens were mounted on aluminum stubs and polished with 1000-grit SiC paper and 6-, 3-, 1-, and 0.25-μm diamond paste (Buehler Ltd, Lake Bluff, IL, USA). Afterwards, they were ultrasonically cleaned, air-dried and gold sputter-coated (MED 010, Balzers Union, Balzers, Liechtenstein) for analysis in a scanning electron microscope (SEM) operated in the backscattered mode and using energy-dispersive X-ray spectrometry (LEO 435 VP, LEO Electron Microscopy Ltd, Cambridge, UK).

Three pictures were taken of each specimen. The first picture was taken in the center of the bonded stick. The other two pictures were taken 0.3 mm to

Table 3: *Chlorhexidine (CHX) Release (in % and mg/g) for All Experimental Conditions After 28 Days and Two Years of Water Storage*[a]

Adhesive System	CHX Concentration, wt%	28 Days		2 Years	
		% of the Original Concentration,	mg/g	% of the Original Concentration,	mg/g
XP Bond	0.01	25.0 ± 1.1 A	0.025 ± 0.013 e	2.0 ± 0.2 B	0.002 ± 0.003 g
	0.05	23.0 ± 1.2 A	0.115 ± 0.002 c	1.6 ± 0.4 B	0.008 ± 0.002 f
	0.1	24.5 ± 1.4 A	0.245 ± 0.018 b	3.0 ± 0.8 B	0.030 ± 0.018 e
	0.2	22.6 ± 1.5 A	0.452 ± 0.047 a	4.2 ± 0.7 B	0.083 ± 0.047 d
Ambar	0.01	10.0 ± 0.9 C	0.010 ± 0.003 k	9.0 ± 0.7 C	0.009 ± 0.003 k
	0.05	10.4 ± 0.7 C	0.052 ± 0.003 j	10.6 ± 0.9 C	0.053 ± 0.004 j
	0.1	13.2 ± 1.1 C	0.132 ± 0.045 i	10.2 ± 0.9 C	0.103 ± 0.045 i
	0.2	12.0 ± 1.2 C	0.240 ± 0.048 h	10.5 ± 1.1 C	0.210 ± 0.048 h

[a] *Comparisons are valid only within adhesive. Analysis per column (n=10 per group). For each adhesive, same capital letters for CHX (%) and lowercase for CHX (mg/g) indicate that there is no statistically significant difference ($p>0.05$).*

the left and right of the first one. As two bonded sticks per tooth were evaluated and a total of five teeth were used for each experimental condition, a total of 30 images were evaluated per group. All images were taken by a technician who was blinded to the experimental conditions. The relative NL percentage within the adhesive and hybrid layer areas was measured in all pictures using image editing software (UTHSCSA ImageTool 3.0 software, University of Texas Health Science Center, San Antonio, TX, USA).

CHX Detection by Micro–Raman Spectroscopy

Two bonded sticks from each tooth at each storage period that were not tested for μTBS were used for CHX detection at the resin/dentin bonding interface. Micro–Raman spectroscopy was performed using Senterra spectroscopy (Bruker Optik; Ettlingen, Germany). The micro–Raman spectrometer was first calibrated for zero and then for coefficient values using a silicon sample. The bonding area was analyzed using the following micro-Raman parameters: 20-mW neon laser with 532-nm wavelength, spatial resolution of ca 3 pm, spectral resolution of ca 5 cm^{-1}, and 100× magnification (Olympus UK, London, UK) to a ca 1-pm beam diameter.[31,39]

Spectra were taken in the middle of the hybrid layer, in an arbitrary area of the intertubular dentin. Care was taken to select an area between two dentin tubules. One site per slice was examined. Accumulation time per spectrum was 30 seconds, and six co-additions were taken per point. Postprocessing of spectra was performed using the dedicated Opus Spectroscopy Software, version 6.5 (Bruker Optik) and consisted of analysis with modeling, which allowed distinguishing spectral components of the adhesive and dentin.

Statistical Analysis

For μTBS and NL, the experimental unit in the current study was the hemi-tooth, since half of the tooth was tested IM and the other half was tested after 2-Y of water storage. The μTBS and NL values of all sticks from the same hemi-tooth were averaged for statistical purposes. The μTBS (MPa) and NL (%) data of each adhesive were subjected to two-way repeated-measures analysis of variance (ANOVA; CHX concentration and storage time). No comparison was made between products. For CHX release, the data (% and mg/g) for each adhesive were analyzed by two-way ANOVA (CHX concentration vs time). A Tukey post hoc test was used for pairwise comparisons ($\alpha=0.05$) using the Statistica for Windows software (StatSoft, Tulsa, OK, USA).

RESULTS

Release Profile of CHX from Adhesive Systems

The analytical curve with linear regression between absorbance values and CHX concentrations had a correlation coefficient of $r = 0.99959$, making it suitable for determining the CHX release (data not shown). The release profiles were fitted to mathematical models and the best model was selected based on the correlation coefficient (r), the MSC (Table 2), and graphic adjustment was conducted using the biexponential equation.[33]

The CHX release values at times 28 days and two years are presented in Table 3. For both adhesives, the cross-product interaction was statistically significant ($p=0.0001$ and $p=0.001$, respectively, for the

Table 4: *Number (%) of Specimens According to Fracture Pattern Mode for All Experimental Conditions Immediately and After Two Years of Water Storage*

CHX Concentration, wt%	Ambar				XP Bond			
	Immediate		2 Years		Immediate		2 Years	
	A/M	C	A/M	C	A/M	C	A/M	C
Control (without CHX)	34 (81.0)	8 (19)	30 (83.4)	6 (16.6)	31 (81.6)	7 (18.4)	27 (77.2)	8 (22.8)
0.01	38 (79.2)	10 (20.8)	32 (84.3)	6 (15.7)	31 (75.7)	10 (24.3)	35 (77.7)	10 (23.3)
0.05	39 (81.3)	9 (18.7)	37 (88.1)	5 (11.9)	34 (81)	8 (19)	29 (78.4)	8 (21.6)
0.1	37 (80.5)	9 (19.5)	33 (86.9)	5 (13.1)	30 (69.8)	13 (30.2)	36 (94.8)	2 (5.2)
0.2	32 (72.7)	12 (27.3)	33 (76.8)	10 (23.2)	37 (74)	13 (26)	32 (82.1)	7 (17.9)

Abbreviations: A/M, adhesive/mixed fracture mode; C, cohesive fracture mode; CHX, chlorhexidine.

percentage of the original concentration and the amount in mg/g). Different CHX concentrations influenced CHX release from the tested adhesive systems. In general, a higher CHX concentration resulted in higher CHX release in both products, and AM showed slower and gradual release when compared to XP, which released CHX at an apparently higher rate. The total amount of released CHX was proportional to the initial concentration of CHX added to the adhesives.

μTBS Test

The fracture patterns of all experimental groups are shown in Table 4. No premature failures were observed in the present study. Cohesive failures were not included in the statistical analysis because of the lower number of specimens with those failure modes. The μTBS means and standard deviations for all groups are shown in Table 5. For both adhesives, the cross-product interaction was statistically significant ($p=0.002$ and $p=0.001$ for AM and XP, respectively). A μTBS drop was noted over time in all XP groups and most AM groups ($p=0.001$ and $p=0.002$, respectively). However, this decrease was much more pronounced in the control group (reduction of 40% to 53%) when compared with the CHX-containing adhesive systems (reduction ranged from 16% to 23% for AM and from 29% to 33% for XP). The only exception was observed when CHX at 0.1 wt% and 0.2 wt% was added to AM, in which case no significant differences were observed between the IM and 2-Y μTBS values.

Nanoleakage

The NL means and standard deviations are shown in Table 6. The cross-product interaction was statistically significant for both adhesives ($p=0.03$ and $p=0.0001$ for AM and XP, respectively). None of the conditions resulted in NL-free interfaces.

For AM and XP, significant differences between immediate and 2-Y groups were seen for all formulations ($p=0.03$ and $p=0.0001$, respectively). However, the highest NL was found in the control group when compared to the NL observed on the bonding interfaces created by CHX-containing adhesives. The 2-Y NL values in the experimental groups were significantly lower than the values observed in the control groups mainly when 0.1 wt% and 0.2 wt% of CHX were added to both adhesives.

Illustrative SEM images of the bonding interfaces created in the control group and experimental group

Table 5: *Means and Standard Deviations of the Microtensile Bond Strength (MPa) for All Experimental Conditions Immediately and After Two Years of Water Storage*[a]

CHX Concentration, wt%	Ambar			XP Bond		
	Immediate	2 Years	% Reduction	Immediate	2 Years	% Reduction
Control (without CHX)	50.3 ± 4.1 A	30.1 ± 4.3 C	40.2	60.2 ± 3.1 a	28.1 ± 3.9 c	53.3
0.01% CHX	53.8 ± 4.0 A	44.8 ± 2.3 B	16.7	63.3 ± 4.7 a	41.9 ± 3.9 b	33.8
0.05% CHX	50.0 ± 4.7 A	41.5 ± 4.0 B	17.0	59.4 ± 4.0 a, b	40.3 ± 3.9 b	32.1
0.1% CHX	52.7 ± 3.5 A	47.4 ± 3.4 A, B	10.0	64.1 ± 4.0 a	45.5 ± 3.9 b	29.1
0.2% CHX	55.6 ± 3.3 A	49.8 ± 3.1A	10.4	63.2 ± 3.1 a	44.7 ± 3.9 b	29.3

Abbreviations: CHX, chlorhexidine.
[a] *Comparisons are valid only within adhesives. For each adhesive, means with the same capital or lowercase letters indicate means statistically significantly different (Tukey test, p>0.05).*

Table 6: *Means and Standard Deviations of the Nanoleakage (%) for All Experimental Conditions After 24 Hours and Two Years of Water Storage*[a]

CHX Concentration, wt%	Ambar		XP Bond	
	Immediate	2 Years	Immediate	2 Years
Control (without CHX)	16.3 ± 4.5 A	24.3 ± 3.1 C	24.4 ± 3.6 a	44.1 ± 4.3 c
0.01	12.5 ± 3.8 A	22.3 ± 3.6 B, C	26.5 ± 3.9 a	36.5 ± 4.7 b
0.05	14.5 ± 4.1 A	21.3 ± 2.6 B, C	27.4 ± 4.6 a	39.7 ± 4.9 b, c
0.1	13.6 ± 4.4 A	17.2 ± 3.3 B	24.5 ± 4.1 a	33.5 ± 3.8 b
0.2	14.3 ± 3.9 A	18.4 ± 4.3 B	26.1 ± 3.3 a	34.5 ± 4.7 b

[a] *Similar uppercase letters indicate means statistically similar (p>0.05) (Tukey test, α=0.05).*

using 0.2 wt% CHX from both adhesive systems at immediate and 2-Y intervals are shown in Figure 1. In the immediate period, NL was nearly restricted to the hybrid layer for both adhesives. Although we did not compare products statistically, XP Bond presented an apparently higher amount of NL, regardless of the CHX concentration, than did Ambar. After two years, all groups showed noticeably more NL, which was present in almost the entire thickness of the hybrid layer and was more pronounced in the control group.

Identification of CHX by Micro–Raman Spectroscopy

Representative Raman spectra performed at the adhesive interface for both adhesive systems are shown in Figures 2 and 3. The relative intensities for CH_2 and CH_3 deformation (1450 cm^{-1}) as well as C-O-C phenyl groups (1113 cm^{-1}) and CH-OH (1260 cm^{-1}) vibrations are associated with methacrylate monomers in the hybrid layer. All these peaks are typically observed when resin/dentin interfaces are evaluated.[39-41]

The peaks associated with CHX digluconate are around 1585 cm^{-1}.[42] This peak was not observed in the control group (Figures 2 and 3). However, it was clearly identified in all CHX-containing adhesives (0.01 wt% and 0.2 wt% CHX groups, Figures 2 and 3). These figures were very similar for the immediate observation and after two years of water storage, indicating that CHX is still present within the hybrid layer even after two years of water storage.

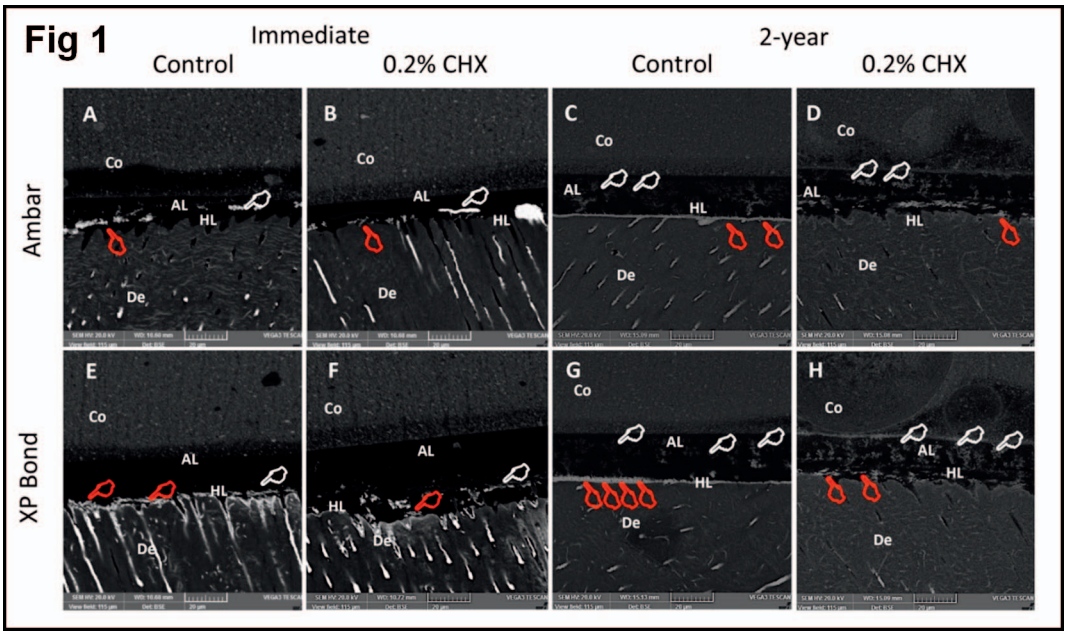

Figure 1. *Scanning electron micrographs of the adhesive interfaces of the experimental groups. Silver nitrate deposits in all groups are mainly within the hybrid layer (red hands), although some deposits were also found within the adhesive layer (white hands). However, after two years of water storage, this deposition was more pronounced in the control group (C and G) when compared to the CHX groups (D and H). As the CHX-containing adhesive showed similar amount and pattern of silver nitrate deposition, we decided to show only one of the groups (Co = composite resin; AL = adhesive layer; HL= hybrid layer; De = dentin).*

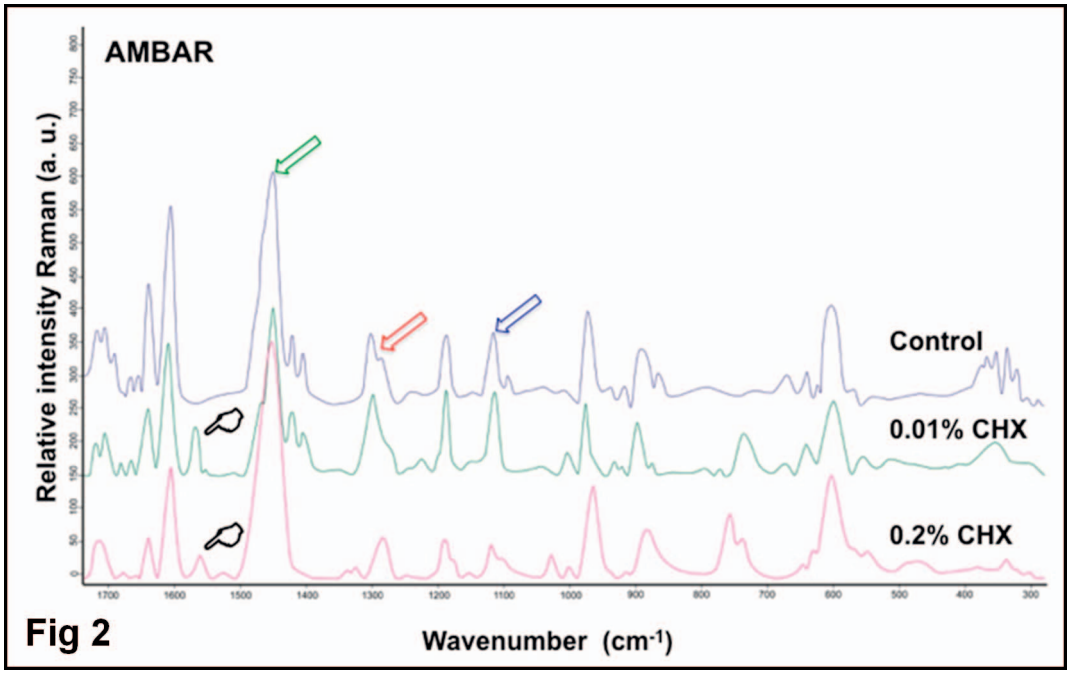

Figure 2. *Raman line-spectra acquired at the adhesive/dentin interface created by Ambar (without CHX [control] and with a 0.01 wt% CHX and 0.2 wt% CHX). The relative intensities for CH2, CH3 deformation (1450 cm^{-1}; green arrow), C-O-C phenyl groups (1113 cm^{-1}; blue arrow), and CH-OH (1260 cm^{-1}; red arrow) vibrations are associated with methacrylate monomers in the hybrid layer; this was observed in all groups. The representative peak of CHX diacetate (1585 cm^{-1}) was only evident in the CHX groups (black hands).*

DISCUSSION

In the current study, a higher concentration of CHX within the bonding agent resulted in a greater amount of released CHX. Moreover, AM showed slower and more gradual release in comparison to that observed in XP. Thus, the first hypothesis was rejected. Differently from the releasing mechanism observed when CHX was added to a primer or to a phosphoric acid, CHX release from bonding agents is quite a bit more complex. When the polymer structure is created after light curing, a porous polymer network composed mostly of hydrophilic monomers and some hydrophobic monomers is formed.[7,8] The amount of hydrophilic monomer within the polymer network determines water sorption and solubility of the bonding agent.[43] Some studies[33,35] have shown a direct positive correlation between these properties and the release profile of drugs added to the bonding agents. In other words, bonding agents with higher water sorption and solubility are capable of releasing more CHX at a higher rate than are bonding agents with lower water sorption and solubility. Differently from AM, XP bond has the highly hydrophilic monomers 2-hydroxyethyl methacrylate (HEMA) and dipentaerythritol pentacrylate monophosphate (PENTA), which were responsible for the higher water sorption and solubility than that observed in AM. As a consequence, faster CHX release from XP Bond (23% of the amount of CHX initially added to the bonding agent) was observed within 28 days when compared to CHX release from AM (11.4% of the amount of CHX initially added to the bonding agent).

After the initial burst release of CHX, both bonding agents showed a continuous but slow release after two years. These results corroborate previous findings[33,35] and may be explained by the water sorption mechanism. After swelling, the mass change in the resin discs due to polymer swelling attains an equilibrium level.[35] After that point, water was not capable of penetrating the polymer network as it did within 28 days[35]; therefore, a small amount of CHX was released from both bonding agents over two years. Indeed, because AM presented lower water sorption and solubility, the slower and continuous water absorption in AM resulted in the apparently higher 2-Y CHX release from AM (an average of 7.8% of the total amount of CHX added to the bonding agent) when compared to that shown by XP (an average of 2.7%).

The addition of CHX to the bonding agents did not impair the immediate μTBS to dentin, nor did it

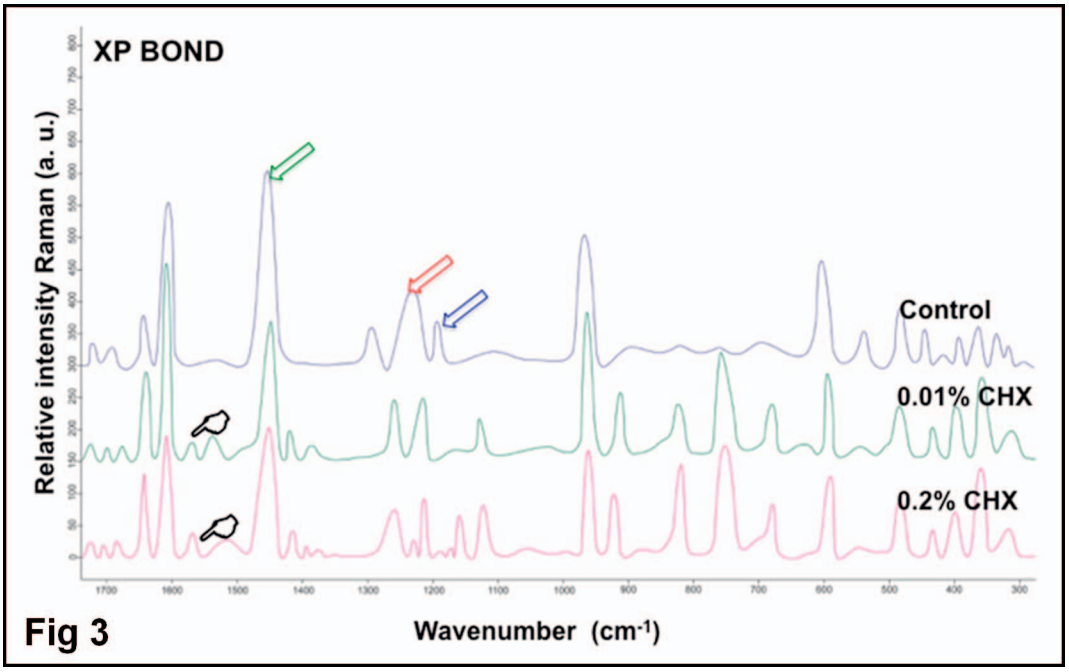

Figure 3. *Raman line-spectra acquired at the adhesive/dentin interface created by XP Bond (without CHX [control] and with a 0.01 wt% CHX and 0.2 wt% CHX). The relative intensities for CH2, CH3 deformation (1450 cm^{-1}; green arrow), C-O-C phenyl groups (1113 cm^{-1}; blue arrow), and CH-OH (1260 cm^{-1}; red arrow) vibrations are associated with methacrylate monomers in the hybrid layer; this was observed in all groups. The representative peak of CHX diacetate (1585 cm^{-1}) was only evident in the CHX groups (black hands).*

impair the NL pattern, so the second hypothesis was accepted. These results corroborate other findings[32,33] and may be mainly attributed to the low CHX content added to the bonding agents. Conversely, previous studies demonstrated that the addition of higher concentrations of CHX (from 1% to 5%) into the bonding agent caused increased resin solubility[35] and decreased modulus of elasticity and degree of conversion of the formed polymer.[44] In addition, CHX diacetate was evaluated in the current study, while other studies[34,45] have incorporated CHX digluconate. The main difference between these compounds is that CHX diacetate is available as a powder, while CHX digluconate is only available as an aqueous solution. In other words, the addition of CHX digluconate to the bonding agents would also add water to the formulation, damaging the adhesive properties through water entrapment.[46,47]

The presence of CHX diacetate did not eliminate hybrid layer degradation within two years in most tested conditions, as lower μTBS values were observed when compared to the immediate values. The only exceptions were noted when AM with CHX at 0.1 wt% and 0.2 wt% were tested, since no significant differences between the immediate and 2-Y μTBS values were recorded. Therefore, the third hypothesis was rejected. It should be noted, however, that all experimental groups showed higher 2-Y μTBS values than did the control groups. Therefore, CHX was apparently capable of attenuating the deleterious effects of MMPs on exposed collagen fibrils, even when a very small amount of CHX was released from the bonding agents. Therefore, it should be noted that CHX has a strong affinity to the dental structure, as it binds to the phosphate groups of mineralized dentin crystallites and to the carboxyl groups of the collagen matrix.[48] This interaction between CHX and dentin matrices is based on electrostatic forces between protonated $NH3^{+}$ in the CHX molecule and negative molecular electrostatic potential of $COOH^{-}$ and OH^{-} in dentin. Therefore, the initial release of CHX may have oversaturated the enzyme binding sites and remained bound to collagen fibrils for later release along with the slow continuing release overtime.[15] For this reason, it is not possible to attribute the CHX protective activity against collagen degradation solely to the small amounts of CHX released from the bonding agents over time. The presence of CHX within the bonding interface over time was confirmed by the Micro-Raman analysis, since the Raman spectra of bonding agents containing CHX still showed the peak referring to the presence of this drug even after two-year storage in water.

Despite the evidence that long-term release of CHX from the bonding agents protected exposed collagen fibrils against collagenolytic attack, most experimental groups still showed a drop in μTBS over time. One could state that most evaluated concentrations were not capable of effectively inhibiting the effects of MMPs to prevent its catalytic activation. However, it should be mentioned that the drop in mechanical strength of the bonding interface after 2-Y is not only related to collagen degradation but also to the degradation of other constituents, such as the composite resin and bonding agent.[18] In this regard, the results indicated that CHX cannot prevent polymer degradation. In other words, water swelling of the bonding agent within the hybrid layer may also result in hygrothermal degradation during aging, such as swelling stresses and the formation of microcracks,[49] which impair the mechanical properties of the plasticized polymer within the hybrid layer. This also helps explain why AM, an adhesive system with lower water sorption and solubility, showed an overall lower percentage of μTBS drop over time than did XP, a bonding agent with more hydrophilic monomer content and higher water sorption and solubility. These results were confirmed by the nanoleakage pattern and SEM analysis, as bonding interfaces created by XP clearly showed more silver deposition than those created by AM. Indeed, the use of bonding agents with lower water sorption and solubility, such as AM, having CHX added at 0.1 wt% or 0.2 wt%, resulted in the most stable bonding interface among all groups. Therefore, the current results demonstrated that the stability of the bonding interface does not rely solely on the CHX dose but also on the mechanical properties of the bonding agent.

In the current study, only two ER adhesive systems were tested, so the results cannot be extrapolated to other adhesive systems, such as self-etching or universal systems. In addition, all analyses were performed on sound, intact third molars. Therefore, different results might be expected when bonding agents containing CHX are applied to caries-affected dentin. Further studies are required to address these issues.

CONCLUSIONS

Within the limitations imposed by this *in vitro* study, the following conclusions were made:

- Although both bonding agents showed an initial CHX burst release profile followed by continuous low release, the general release profile was product-dependent. None of the CHX concentrations impaired the immediate μTBS values and NL patterns, regardless of bonding agent.
- Although most groups showed lower long-term μTBS values when compared to the immediate values, the use of CHX-containing bonding agents resulted in more stable bonding than was associated with the use of regular commercial products.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the State University of Ponta Grossa. The approval code for this study is 1693/09.

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.

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Does Classification of Composites for Network Meta-analyses Lead to Erroneous Conclusions?

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Clinical Relevance

Different classification systems of composites can be used for meta-analyses. The results are rankings of composite classes, which can be used to decide to use a specific composite. We found a high agreement of such rankings between most classification systems.

SUMMARY

Objectives: Composites can be classified differently, according to manufacturer information, filler particle size, resin-monomer base, or viscosity, for example. Using clinical trial data, network meta-analyses aim to rank different composite material classes. Dentists then use these ranks to decide whether to use specific materials. Alternatively, annual failure rates (AFRs) of materials can be assessed, not requiring any classification for synthesis. It is unclear whether different classification systems lead to different rankings of the same material (ie, erroneous conclusions). We aimed to evaluate the agreement of material rankings between different classification systems.

Methods: A systematic review was performed via MEDLINE, Cochrane Central Register of Controlled Trials, and EMBASE. Randomized controlled trials published from 2005-2015 that investigated composite restorations placed in load-bearing cavitated lesions in permanent teeth were included. Network meta-analyses were performed to rank combinations of composite classes (according to manufacturer, filler particle size, resin-monomers, viscosity) and adhesives. Material combinations were additionally ranked using AFRs.

Results: A total of 42 studies (6088 restorations, 2325 patients) were included. The ranking of most material class combinations showed significant agreement between classifications (R^2 ranged between 0.03 and 0.56). Comparing material combinations using AFRs had low precision and agreement with other systems. AFRs were significantly correlated with follow-up periods of trials.

Conclusion: There was high agreement between rankings of identical materials in different classification systems. Such rankings

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thus allow cautious deductions as to the performance of a specific material. Syntheses based on AFRs might lead to erroneous results because AFRs are determined by follow-up periods and have low precision.

INTRODUCTION

Placing restorations is still the most frequently performed and expensive dental treatment.[1-3] Nowadays, the majority of these restorations are adhesively placed composites, with a vast number of materials from numerous manufacturers being available to dentists. These composites are tested clinically by a similarly large number of controlled trials. Although dentists will not be able to evaluate the findings of each trial, they usually require some kind of synthesis of study findings that summarizes the results and gives guidance as to which materials to use under certain conditions.

In such syntheses, outcomes of trials are usually compared by either pairwise meta-analysis (comparing, for example, the number of failures in one group vs the other and pooling the resulting risk ratios across trials) or via network meta-analysis using direct (ie, evidence stemming from comparisons made within a trial) and indirect evidence (ie, evidence stemming from comparisons between different trials), thereby allowing comparison of more than two groups.[4,5] The result is a ranking, either of two comparators ("A is superior to B in condition C") or multiple groups ("A has a lower risk of failure than B, which has a lower risk than C in condition D" and so on). Given the number of materials available, such syntheses require some kind of classification of materials both for statistical evaluation and for interpretation (comparing 20 or more material brands without summarizing them in classes will likely not allow robust synthesis and will also prohibit easy interpretation of findings).

For dental composites, a number of classification systems are available. Composites might be classified according to manufacturers' information (with some products being actively separated from the "field," such as ormocers or siloranes). Alternatively, they might be classified on the basis of their filler particle size, their resin-monomer base, or their amount of filler particles, which is also correlated with viscosity. For syntheses, the same material can thus belong to different classes, and could—on the basis of these different classifications—be ranked differently. That could mean the same material (product) is found suitable by one analysis but not another, depending on the group of materials in which it is classified.[6] It is thus unclear whether deductions can be made from such class rankings with regard to the performance of a specific material/product.

An alternative to the described meta-analytic syntheses is the comparison of annual failure rates (AFRs). This avoids classification, but pools findings from possibly very different trials (follow-ups, patient groups) via AFRs, which could heavily distort the results. For example, certain materials might have been used only in short-term trials in low-risk patients, whereas others have been investigated over longer periods in high-risk patients. This is likely to lead to higher AFRs in the latter group but does not necessarily prove this material to be performing worse than the material in the former. Whereas this might also be the case in pairwise or network meta-analysis, the risk of distortion is lower here, given that comparisons are made within trials first (yielding risk ratios, as described) before pooling.

In summary, using different classification systems of dental composites might lead to different results and possibly erroneous conclusions. Our aim was to assess the agreement of materials' rankings with regard to risk of failure based on meta-analyses using different classification systems. Moreover, we aimed to evaluate whether such rankings are reflected by AFRs, and whether AFRs are precise (ie, reproducible in different trials) or depend on other factors (such as a follow-up period). Our research question was "For identical material (product) combinations, what is the agreement in rankings yielded by network meta-analyses using different composite classifications, and how do these rankings agree with rankings based on AFRs?" We hypothesized that there was no significant agreement of rankings yielded by different classification systems.

METHODS

Study Design

We built on data previously collected as part of a systematic review of randomized trials on dental restoration materials including composites.[6,7] The therein-investigated composites were classified and combinations of composite classes and adhesives submitted to network meta-analyses, yielding a ranking of each material class combination according to its risk of failure. We then compared how identical material (product) combinations were ranked using different classification systems and investigated the agreement of rankings. We further

compared these rankings with rankings based on AFRs. Our analysis exemplarily focuses on composites placed in load-bearing cavitated lesions of permanent teeth.

Systematic Review

The performed systematic review had included randomized, controlled clinical trials (RCT) published from 2005-2015 comparing the survival of two or more different composite and/or adhesive materials. RCTs were excluded if they compared different treatment techniques (eg, conventional vs atraumatic restorative treatment), not materials, or placed restorative materials or adhesives as a sealant or for orthodontic bonding, not a restoration. To ensure uniform randomization across trials, we only included trials reporting on survival of composite restorations placed in adult or adolescent (ie, children with permanent teeth) patients in need of restoration of load-bearing cavities in posterior permanent teeth.

The Cochrane Central Register of Controlled Trials, MEDLINE (via PubMed) and EMBASE (via OVID) were searched on March 2, 2015. The search strategy and screening procedure is shown in appendix Figure S1. Screening, inclusion, and data extraction had been performed independently by two reviewers. Consensus was obtained by discussion. Full details regarding the performed review can be found elsewhere.[6,7]

Classification Systems

Composite materials are placed using adhesives. Therefore, all trials essentially compared combinations of adhesives and composites. Adhesives were classified as follows: 1) 4- or 3-step etch-and-rinse adhesives (3ER); 2) 2-step etch-and-rinse adhesives (2ER); 3) 2-step self-etch adhesives (2SE); 4) one-step self-etch adhesives (1SE). For composites, four classification systems were used, based on

- Manufacturers' classification. This system had been used in previous publications on this issue[6,7] and categorized composites as 1) conventional composites (CC); 2) ormocer composites; 3) bulk-fill composites (both flowable and packable bulk fills); and 4) siloranes.
- Filler composition (ie, hybrid or single particle size) according to the manufacturer's information: 1) microhybrid, 2) nanohybrid, 3) microfilled, and 4) nanofilled.
- Resin-monomer base regarding the main component of the resin matrix: 1) conventional monomers (eg, urethane dimethacrylate, bisphenol A glycol dimethacrylate, triethylene glycol dimethacrylate), 2) ormocers, and 3) siloranes.
- Viscosity according to the manufacturer's classification: 1) composites with conventional viscosity, 2) packable composites, and 3) flowable composites. Where composites were neither classified as packable nor as flowable, we assumed they had conventional viscosity.

If two composite materials had been used in the same cavity (as for some bulk fills, with bulk material being covered by another composite), the material within the focus of the study (in this case, always the bulk fill) was used for classification. Combining adhesive and composite material allowed the classification of each placed material combination (eg, a microhybrid composite placed using a 3ER). Given the different number of classes, the classification systems were differently granular (eg, the filler-based classification included 13 class combinations, whereas the classification according to the resin component included only seven combinations). The full list of assessed materials and their classification can be found in appendix Table S1.

In addition, composites were not classified at all, and instead combinations of each composite with the used adhesive system class were constructed (eg, Tetric Ceram placed using a 3ER). This resulted in 71 unique combinations of composite product and adhesive class, which were used for analyses based on AFRs.

Data Syntheses

Our outcome parameter for syntheses was risk of failure (ie, restorations needing any restorative reintervention—that is, replacement or repair). That included retention loss, but also any United States Public Health Services (USPHS) ratings of charlie or delta, for example. Our analysis only accounted for restorations that were followed up, with risk of failure per study being derived as events (failures) per total sample followed. The unit of analysis was restorations. Note that most trials were clustered (ie, patients contributed more than one lesion), which usually calls for some adjustment because resulting confidence intervals of effect estimates (such as risk ratios) are otherwise artificially narrow. We did not perform such adjustment, because our aim was not to compare materials but to assess the agreement between rankings yielded by different classification systems. Moreover, we did not present confidence or credible intervals, because we focused on rankings.

Therefore, network meta-analyses (NMA) were used for estimating class combination ranks, with Bayesian random-effects models and a Markov chain Monte Carlo simulation being conducted via the Bayesian software package GeMTC 0.6 and WinBugs[8] implemented in R 3.0.3 (R Foundation, Vienna, Austria). For each classification system, one separate NMA was performed. Binomial likelihood was used to model the data.[9,10] To fit the model, we used a noninformative prior for the basic parameters from a normal distribution $N(0,10^4)$ and a flat prior U(0,2) for the random-effects standard deviation. The convergence was assessed on the basis of the Brooks-Gelman-Rubin criteria[11] and inspection of trace plots. The first 50,000 iterations were discarded as "burn-in" and then a further 50,000 iterations were undertaken for two chains at thinning intervals of five. Different class combinations were ranked according to their probability of having the lowest vs the highest risk of failure,[12] and the average rank was calculated. The surface under the cumulative ranking (SUCRA) line was plotted and the area under the plot (SUCRA value) calculated. SUCRA values were eventually used to rank class combinations (eg, from 1 to 13 for the filler-based classification system). Loop inconsistency (ie, the difference between direct and indirect estimates for different treatments within a loop) was evaluated by the Inconsistency Factor (IF) for the loop.[13,14] Within each loop, the IF value is defined as $IF = E_{direct} - E_{indirect}$ (E: estimate). We rejected the null hypothesis that evidence is consistent (H_0: $IF = 0$) when the IF was significantly greater or smaller than 0.[6] Note that usually, performing pairwise meta-analyses is recommended alongside network meta-analyses. No such pairwise meta-analyses were performed, given that we were not interested in determining material efficacy but to assess agreement of material combinations.

In addition to NMA, material combinations were ranked according to their AFRs. Materials with AFRs of 0 were all ranked first, resulting in 62 ranks of the 71 individual material combinations.

Assessment of Agreement

Agreement of rankings of the same composite-adhesive combination was assessed twofold: First, graphical assessment using heat maps was performed, assigning a color code to each rank, homogeneously distributing different red and green hues between the lowest and the highest ranks. This allowed us to overcome the issue of a different number of ranks in different classification systems. Similarly, statistical assessment using the Kendall correlation coefficient allowed us to evaluate agreement regardless of the absolute number of ranks. A possible association of AFRs with follow-up periods was assessed using Pearson correlation.

RESULTS

Search and Studies

In the original review, 114 studies (147 articles) were included. From these, we used 42 studies (with 103 comparator groups), solely focusing on composites being placed in load-bearing cavities of permanent teeth. In three comparator groups, a combination of two materials was used for placement of the restorations. After a mean follow-up period of 42 months (range, 12 to 120 months), 4820 restorations had been assessed (Figure S1, Table S2).

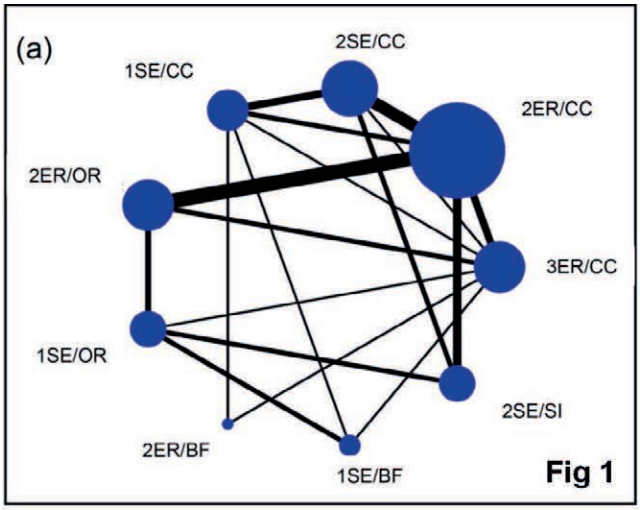
Ranking of Different Material Class Combinations

Using manufacturers' classification of composites, nine different material class combinations were assessed (Figure 1a). When ranking them according to their probability of failure (Figure 1b) and the resulting SUCRA value, CC placed using 3ER or 2SE were ranked highest, whereas siloranes placed using 2SE showed the lowest ranking (Figure 1c). There was statistical inconsistency in one assessed loop (finding 2ER/CC nonsignificantly inferior to 1SE/CC using direct comparison [ie, comparisons within a trial], and vice versa using indirect comparison [ie, comparisons between different trials]; the level of inconsistency was limited with $p=0.04$). Because these material combinations were ranked fourth and fifth (ie, very similarly), the resulting uncertainty was limited.

Classifying composites according to their filler particle size (Figure 2a) and ranking them according to risk of failure (Figure 2b), nanohybrids placed using 2SE or 1SE as well as nanofilled composites placed using 2SE were ranked highest, whereas microhybrids placed using 2SE were ranked lowest (Figure 2c). There was no statistical inconsistency.

Classification according to the main resin component (Figure 3a) and subsequent ranking (Figure 3b) found composites containing conventional resins placed with 3ER or 2SE ranked highest; silorane resin composites placed using their 2SE adhesive were ranked lowest (Figure 3c). Again, there was no statistical inconsistency.

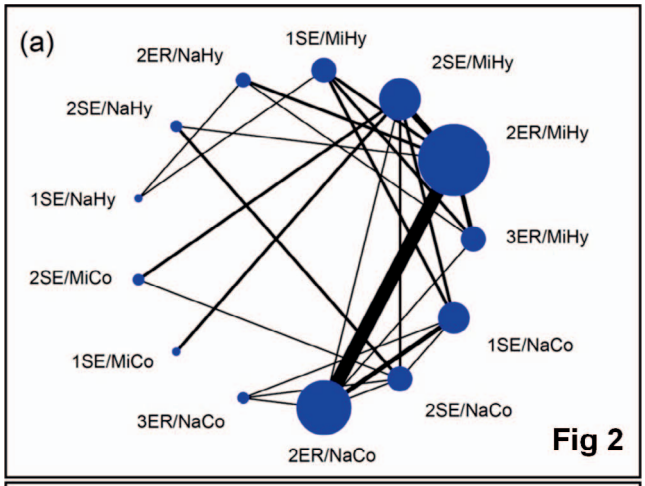
Classification according to viscosity found packable or conventionally viscous composites placed

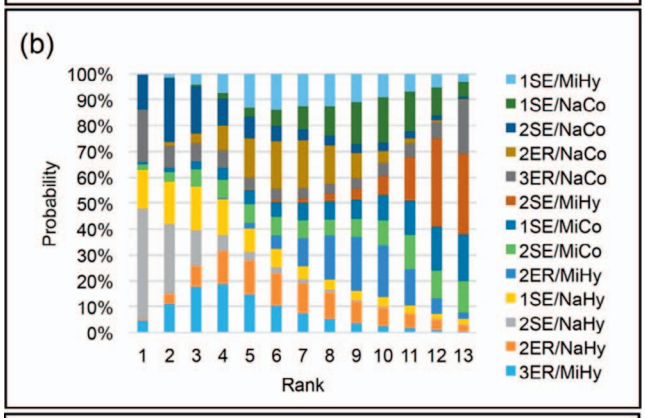


(c)

Class	Mean rank	SUCRA value	Overall rank
2SE/CC	2.9	16	1
3ER/CC	3.3	15	2
2ER/BF	4.4	13	3
1SE/CC	4.4	12	4
2ER/CC	4.8	12	5
1SE/OR	5.0	11	6
2ER/OR	5.9	9	7
1SE/BF	6.9	7	8
2SE/SI	7.4	6	9

Figure 1. *Results if composites are classified according to manufacturers as conventional composites (CC), bulk fills (BF), ormocers (OR), siloranes (SI). Adhesives were categorized as follows: 3ER, 4- or 3-step etch-and-rinse; 2ER, 2-step etch-and-rinse; 2SE or 1SE, 2- or 1-step self-etch. (a): Networks of composite adhesive class combinations. The size of the nodes is proportional to the number of placed restorations. The width of the lines is proportional to the number of trials comparing the connected treatments. (b): Ranking of composite adhesive class combinations according to their chance of restoration survival. (c): Mean rank, surface under the cumulative ranking (SUCRA) value and resulting overall rank of different class combinations.*



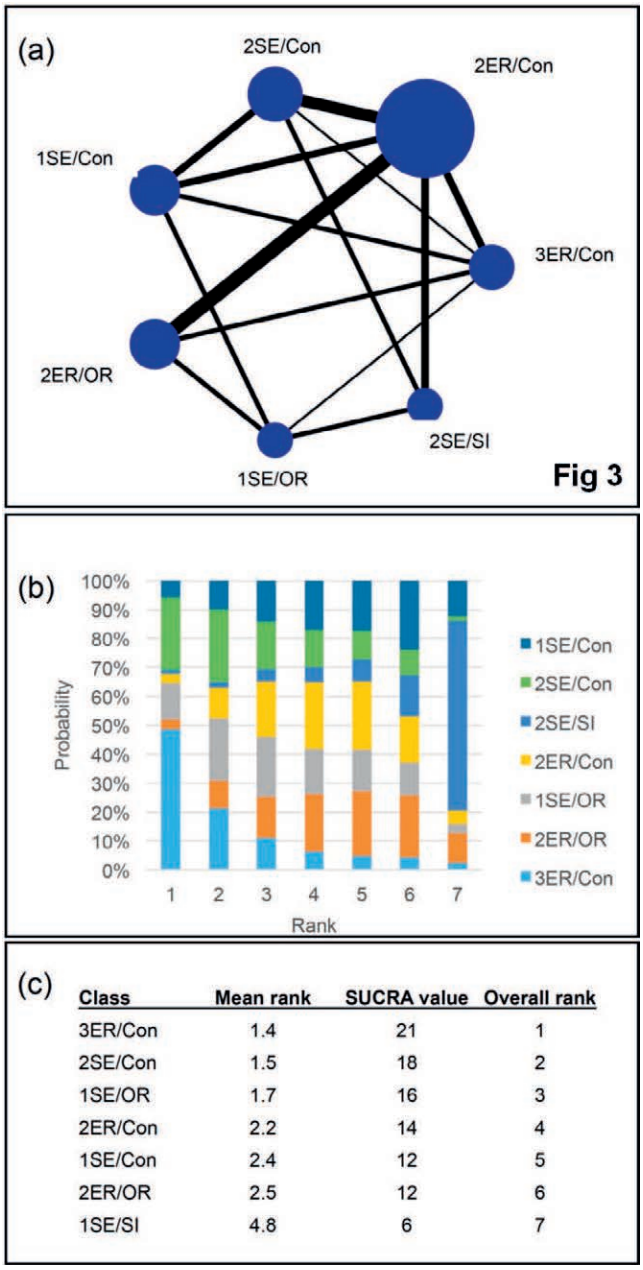


(c)

Class	Mean rank	SUCRA value	Overall rank
2SE/NaHy	2.2	13	1
2SE/NaCo	3.3	11	2
1SE/NaHy	3.6	10	3
3ER/MiHy	4.1	10	4
2ER/NaCo	5.1	8	5
2ER/NaHy	5.0	8	6
3ER/NaCo	5.4	8	7
1SE/MiHy	5.5	7	8
2SE/MiCo	6.1	6	9
2ER/MiHy	9.5	5	10
1SE/NaCo	8.7	5	11
1SE/MiCo	7.3	5	12
2SE/MiHy	9.1	2	13

Figure 2. *Results if composites are classified according to filler particle size: Microhybrids (MiHy), nanohybrids (NaHy), microfilled composites (MiCo) or nanofilled composites (NaCo). Adhesives were categorized as follows: 3ER, 4 or 3-step etch-and-rinse; 2ER, 2-step etch-and-rinse; 2- or 1SE, 2- or 1-step self-etch. (a) Networks of composite adhesive class combinations. (b) Ranking. (c) Mean rank, surface under the cumulative ranking (SUCRA) value and resulting overall rank of different class combinations.*

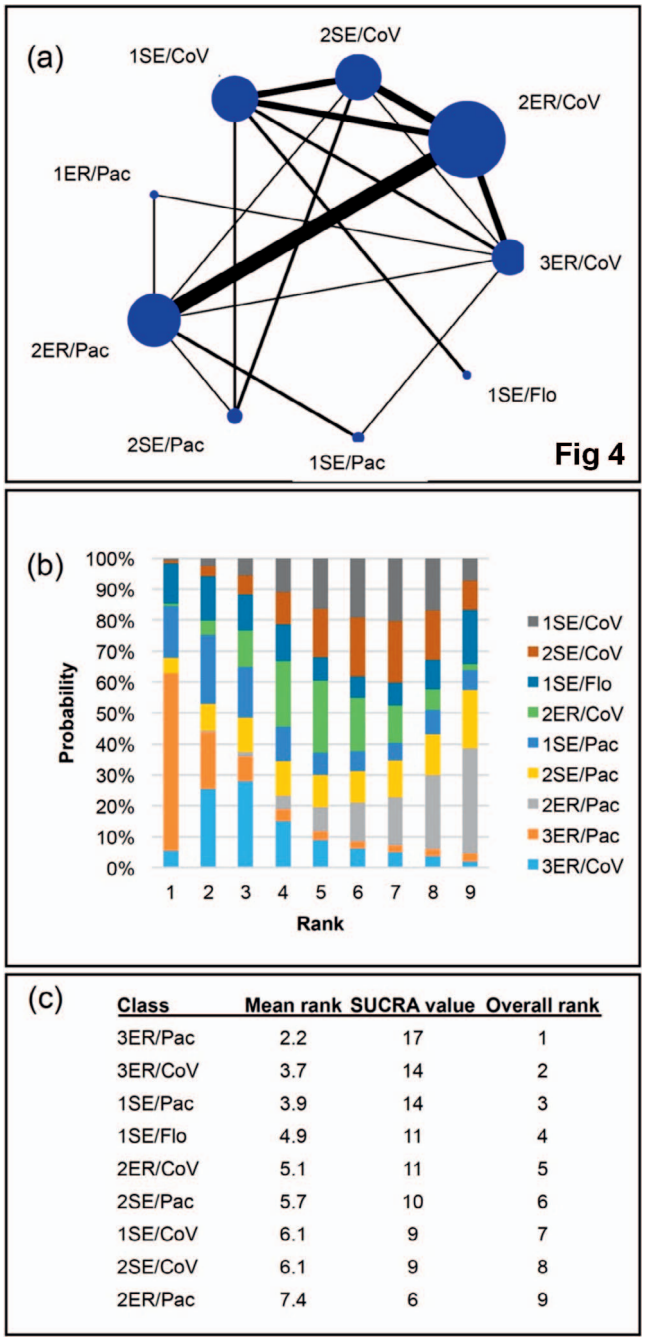
using 3ER ranked highest, and conventionally viscous or packable composites placed with 2ER on the lowest rank (Figure 4a-c). There was statistical inconsistency (p=0.01), with direct evidence finding conventionally viscous composites placed using 1SE nonsignificantly more suitable than those placed using 2SE, whereas indirect evidence found the opposite. Given that those class combinations were



(c)

Class	Mean rank	SUCRA value	Overall rank
3ER/Con	1.4	21	1
2SE/Con	1.5	18	2
1SE/OR	1.7	16	3
2ER/Con	2.2	14	4
1SE/Con	2.4	12	5
2ER/OR	2.5	12	6
1SE/SI	4.8	6	7

Figure 3. *Results if composites are classified according to resin-monomer base: Conventional monomers (Con), ormocers (OR), siloranes (SI). Adhesives were categorized as follows: 3ER, 4 or 3-step etch-and-rinse; 2ER, 2-step etch-and-rinse; 2- or 1SE, 2- or 1-step self-etch. (a) Networks of composite adhesive class combinations. (b) Ranking. (c) Mean rank, surface under the cumulative ranking (SUCRA) value and resulting overall rank of different class combinations.*



(c)

Class	Mean rank	SUCRA value	Overall rank
3ER/Pac	2.2	17	1
3ER/CoV	3.7	14	2
1SE/Pac	3.9	14	3
1SE/Flo	4.9	11	4
2ER/CoV	5.1	11	5
2SE/Pac	5.7	10	6
1SE/CoV	6.1	9	7
2SE/CoV	6.1	9	8
2ER/Pac	7.4	6	9

Figure 4. *Results if composites are classified according to their viscosity: Conventional viscosity (CoV), packable (Pac), flowable (Flo). Adhesives were categorized as follows: 3ER, 4 or 3-step etch-and-rinse; 2ER, 2-step etch-and-rinse; 2- or 1SE, 2- or 1-step self-etch. (a) Networks of composite adhesive class combinations. (b) Ranking. (c) Mean rank, surface under the cumulative ranking (SUCRA) value and resulting overall rank of different class combinations.*

ranked seventh and eighth, the impact of this inconsistency was limited.

Agreement of Rankings

Using a heat map (Figure 5), we found materials being ranked highly in one classification system to be ranked similarly in other systems in most cases (details can be found in the Appendix Table S3). One notable exception was composites classified as micro-hybrids (for example, Filtek Z250) placed using 2SE

(for example, Clearfil SE Bond); these were ranked poorly, whereas the same material combination (Z250 + Clearfil SE) was ranked highly in other classifications. The reason for this discrepancy was that microhybrids placed with 2SE included silorane-based composites, which performed rather poorly and lowered the ranking of this class combination.

The relatively high agreement was reflected in correlation coefficients as well (Table 1), which found a significant association ($p<0.05$) of ranks yielded by different classification systems with only one exception (agreement between classification according to the manufacturer and classification according to viscosity). However, R^2-values were low for most of the groups, indicating low confidence for the found agreements between classification systems.

Limited agreement was found when comparing NMA rankings with AFR rankings (Figure 5), with AFR-based ranks mostly not showing significant association to ranks yielded by other classification ($p>0.05$) except for a borderline-significant agreement between AFR ranking and NMA rankings according to filler ($p=0.04$). When evaluating AFRs, it was apparent that the same material combinations could have been ranked very differently depending on the study (for example, Surefil placed using Prime+Bond NT was ranked first as well as 55th), highlighting the limited precision of AFR-based ranking. For material combinations that had been investigated in a minimum of three studies, we assessed the association between AFR and follow-up, and found significant associations for nine of 12 material combinations (the mean correlation coefficient between follow-up and AFR was 0.45).

DISCUSSION

Dentists will use syntheses of original data or their derivatives (such as guidelines) for clinical decision-making rather than consulting all available individual trials. These syntheses usually build on some kind of classification system both for statistical pooling and ease of interpretation. Using the resulting ranking of a specific material class, dentists might decide to use or not to use a specific

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Figure 5. *Heat map of agreement of ranks. Combinations of composite and adhesive classes were used to yield ranks. Green indicates a class combination was ranked high, whereas red indicates a low rank. Four composite classification systems were assessed: manufacturer classification (M), or according to filler particle size (F), resin-monomer base (R), viscosity (V). In addition, rankings based on annual failure rates (AFR) were estimated. Details can be found in Appendix Table S3.*

Table 1: *Agreement of Ranks of Material Combinations According to Different Composite Classification Systems as Well as Annual Failure Rates (AFR). Four Classification Systems Were Assessed: Manufacturers' Classification, Classification According to Filler Particle Size, to Resin-base, to Viscosity. Kendall's Correlation Was Used (Level of Significance in Parentheses).*

	Filler	Resin	Viscosity	AFR
Manufacturer	0.24 (0.02)	0.56 (<0.01)	0.03 (0.74)	0.05 (0.49)
Filler		0.22 (0.01)	0.31 (<0.01)	0.15 (0.04)
Resin			0.18 (0.03)	0.14 (0.07)
Viscosity				0.06 (0.46)

material (product). On the basis of our findings, this is largely justified, because a material (product) combination that is ranked highly in one classification system is likely to be ranked highly in another classification system and vice versa. Given our findings, we refute our hypothesis of no significant agreement between classification systems.

There are several possible reasons underlying this agreement. First, it might be that there were too few materials in each class, leading to one or few materials dominating each class, with limited risk of distortion via pooling materials. Second, it is possible that material properties such as filler particle size or resin-base (which are reflected by classifications) are indeed the true drivers of performance: This would be encouraging, given that these properties can be technically altered and are the focus of large parts of dental materials science. If material properties are in fact determining a material's performance, the present review would allow the assessment of which combinations are better suited than others for restoring load-bearing cavities in permanent teeth. Third, it can be assumed that the used adhesive partially determined the ranking of a class combination. Because the adhesive system classes remained stable regardless of the applied composite classification, the found agreement might partially stem from there. We expect a combination of these factors to contribute to the recorded agreement. For other medical disciplines, it can be shown that variability in definitions of treatment classifications (ie, variations due to differences in dose or administration routes in trials on drugs) can affect the ranking of the resulting treatment nodes within NMA.[15,16] However, the agreement of material class rankings yielded by different tested classification systems was relatively high within our study, as long as ranking was not based on AFRs.

Ranking materials based on AFRs did not correlate significantly with most other rankings. That could mean that most other rankings do not reflect the true performance of a material, as AFRs might be expected to do. However, when closely inspecting AFRs of identical material combinations, we found these to vary widely (up to 5.5% for the same material). This highlights the limited precision of AFRs and AFR-based rankings. Moreover, we found AFRs to correlate significantly with a trial's follow-up period: Longer trials recorded significantly more failures, which increases AFRs compared with short-term trials. Although we did not assess the possible underlying reasons (eg, higher risk of failure later in a restoration's lifetime, long-term trials being from different settings or in different patients), it is clear that pooling such AFRs might not only reflect a material's performance but also the design and conduct of the trial. On the basis of these findings, we cannot recommend continuing to synthesize data on clinical restoration performance using AFRs.

This study has several limitations. First, a number of confounders (such as the adhesive system used) might affect a material's performance and could artificially increase agreement between classes. However, even if this were the case, dentists indeed place combinations of composite and adhesive; any classification that truly predicts the performance of such combination is thus clinically relevant. Moreover, this element of bias would be present in AFR-based rankings, too (where it did not seem to increase agreement). Second, given the limited differences in SUCRA values between many material class combinations, there seems to be a relatively high degree of uncertainty. Several confounders (such as differences between batches of the same material, inclusion of studies that used material combinations) might have contributed to this uncertainty. However, some materials seem to be more suitable than others. For example, and regardless of the classification systems, composites containing ormocers and siloranes were ranked lower, whereas nanohybrids were generally ranked high. It should be noted that despite the discussed uncertainty, we did not find statistical inconsistency to greatly affect

our findings. This confirms that the constructed networks were relatively robust and our findings not mere statistical artefacts. Third, we included only randomized trials. Whereas the internal validity of such trials is higher than that of observational trials (mainly due to reduced selection bias), they are usually performed under controlled and somewhat artificial conditions and suffer from limited follow-up periods. Future analyses might consider including controlled nonrandomized trials. Moreover, we included only recently published trials, with an artificial inclusion cutoff of 10 years (2005-2015). That was justified because many materials were only recently available, and pooling these with much older materials might lead to distortion. However, one should note that some included more recent trials followed up older materials after 10 years or longer. Almost half of the included trials had a follow-up period of two years or less. Given that such short-term failures are often related to retention loss, which itself is associated with the adhesive bond, it is likely that the adhesive had a significant influence on the ranking of the class combinations in this study. Long-term studies (which were rare in our review), in contrast, might show 1) a higher rate of complications and 2) different types of complications.[17] In this case, the adhesive as determinant for the material class ranking might play a more limited role. Last, the assessed outcome parameter was failure. It is unclear whether using other components of available outcome measures like the USPHS criteria (such as surface properties, or secondary caries) will affect ranking agreements.

CONCLUSIONS

On the basis of our findings, the agreement of material class rankings yielded by different classification systems was relatively high. Nevertheless, interpretation of material class performances should be made with caution, because the same material might be ranked differently depending on the classification system used. Comparing materials solely on the basis of AFRs might lead to erroneous results, given that AFRs are determined by follow-up periods rather than material performance.

Conflict of Interest

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

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Combination of Acetaminophen/Codeine Analgesics Does Not Avoid Bleaching-Induced Tooth Sensitivity: A Randomized, Triple-Blind Two-Center Clinical Trial

FM Coppla • M Rezende • E de Paula • PV Farago • AD Loguercio • S Kossatz • A Reis

Clinical Relevance: The use of an opioid analgesic drug was not capable of preventing tooth sensitivity arising from in-office dental bleaching.

doi: http://dx/doi.org/10.2341/17-092-C

Longitudinal Evaluation of Radiopacity of Resin Composites: Influence of Photoactivation and Accelerated Photoaging

AD Cruz • IAM Costa • FS Calazans • MF Aguiar • MO Barceleiro

Clinical Relevance: Radiopacity has been considered an unchangeable property with great significance for assessing the quality of a restoration by radiographic images. Any changes in this property over time, under the influence of photoactivation and photoaging, might affect radiographic diagnosis.

doi: https://doi.org/10.2341/16-240-L

Effect of Chlorhexidine Treatment Prior to Fiber Post Cementation on Long-Term Resin Cement Bond Strength

M Durski • M Metz • G Crim • S Hass • R Mazur • S Vieira

Clinical Relevance: The addition of chlorhexidine application prior to adhesive cementation appears to be effective in enhancing the long-term push-out strength with glass fiber posts in all root thirds in vitro.

doi: http://dx/doi.org/10.2341/16-241-LR2

Fatigue Failure Load of Restored Premolars: Effect of Etching the Intaglio Surface of Ceramic Inlays With Hydrofluoric Acid at Different Concentrations

T Missau • AB Venturini • GKR Pereira • C Prochnow • LF Valandro • MP Rippe

Clinical Relevance: Etching with hydrofluoric acid at different concentrations (1%, 5%, 10%) does not affect the fatigue failure load of premolars restored with feldspar inlays (milled by a computer-aided design / computer-aided manufacturing system). Thus, those acids might be used for ceramic etching.

doi: http://dx/doi.org/10.2341/16-345-L

Degradation of Multimode Adhesive System Bond Strength to Artificial Caries-Affected Dentin Due to Water Storage

AC Follak • LL Miotti • TL Lenzi • RO Rocha • FZM Soares

Clinical Relevance: When using multimode adhesives on sound dentin, clinicians can choose to use either an etch-and-rinse or a self-etch strategy in terms of bond strength; however, on caries-affected dentin, bonding degradation will be greater irrespective of etching mode.

doi: http://dx/doi.org/10.2341/17-129-L

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Clinical Research

Combination of Acetaminophen/ Codeine Analgesics Does Not Avoid Bleaching-Induced Tooth Sensitivity: A Randomized, Triple-Blind Two-Center Clinical Trial

FM Coppla • M Rezende • E de Paula • PV Farago • AD Loguercio • S Kossatz • A Reis

Clinical Relevance

The use of an opioid analgesic drug was not capable of preventing tooth sensitivity arising from in-office dental bleaching.

SUMMARY

Bleaching-induced tooth sensitivity (TS) is highly prevalent. Objective: This study aimed to determine if the combination of opioids and nonopioids analgesics (Tylex) may provide a better analgesic effect. Method: A triple-blind, parallel, randomized two-center clinical trial was conducted with 105 healthy patients who received either a placebo or a combination of acetaminophen/codeine. The first dose of Tylex 30 mg (acetaminophen 500 mg/codeine 30 mg) or placebo was administered one hour before the in-office bleaching (35% hydrogen peroxide), and extra doses were administered every six hours for 48 hours. The TS was recorded using a visual analog scale of 0 to 10 and a numeric rating scale of 0 to 4 in different periods: during bleaching, one hour up to 24 hours, and 24 hours up to 48 hours postbleaching. The color was measured before and one month after dental bleaching with a visual shade guide (Vita Classical), Vita Bleached-

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guide 3D-MASTER, and the spectrophotometer Vita Easyshade. The absolute risk of TS was evaluated using the Fisher exact test. Data of TS intensity with numeric rating scale of the two groups were compared with the Mann-Whitney *U*-test and the Friedman test, while data from the visual analog scale were evaluated by two-way repeated measures analysis of variance and the Tukey test for pairwise comparison. The color changes between groups were compared using the Student *t*-test (α=0.05). Results: No significant differences between the groups were observed in the risk and intensity of TS. The overall absolute risk of TS was approximately 96%. No significant differences between groups were observed in terms of color change ($p>0.05$) for any scale. Conclusion: The use of an acetaminophen/codeine combination prior to in-office bleaching does not reduce the risk and intensity of bleaching-induced TS.

INTRODUCTION

In-office bleaching is an alternative to the popular at-home bleaching, and a dental professional can perform it on the same day the patient walks into the dental office.[1] The in-office bleaching protocol has some advantages, as it allows close dentist control, avoids soft tissue exposure to peroxides, avoids the risk of material ingestion, and reduces total treatment time. However, in-office bleaching using 35% hydrogen peroxide (HP) has a long history of tooth sensitivity (TS),[2-4] producing TS intensity that is, on average, four times higher than that produced by at-home bleaching.[5] In recent clinical trials, authors reported an absolute risk of TS varying from 67% to 100%,[5-9] meaning that under the best conditions, six patients in every 10 will experience pain during treatment.

The high variability of pain induced by bleaching can be explained by variables such as age, gender, baseline tooth color, and bleaching technique (at-home or in-office bleaching, a single bleaching session or even shorter application times of 10 to 20 minutes, or variations in the concentrations and composition of the products). Rezende and others[10] showed an association between baseline tooth color and bleaching-induced TS: every 1-shade-guide-unit (SGU) increase in baseline color reduces the odds of TS by 17% and the intensity by 13%. Baseline color can predict TS; in other words, the darker the teeth, the lower the intensity and risk of TS.

To the extent of the authors' knowledge, the reasons for bleaching-induced TS are not clear. TS seems to result from the easy passage of HP through the enamel and dentin to the pulp,[11] causing pulp damage and a reversible inflammation process.[12] Since 2009, some authors have investigated the use of anti-inflammatory drugs to reduce this adverse effect.[5,6,13-15] In their studies, the use of ibuprofen, a nonsteroidal anti-inflammatory, reduced TS immediately up to one hour after bleaching.[14,15] The administration of other medicines, such as a selective anti-inflammatory drug (etoricoxib, 60 mg) or a steroidal anti-inflammatory drug (dexamethasone, 4 mg), was not capable of reducing this side effect.[5,14]

Another medicine that can be used per the oral route but not yet investigated could be an alternative approach. Nonopioid analgesics produce analgesia through peripheral action consisting of the inhibition of the cyclooxygenase enzyme system.[16] This class of analgesics comprises commonly used drugs for pain control due to their low toxicity, with rare side effects in cardiovascular and respiratory systems.[16] However, compared to opioid analgesics, they have a limited analgesic effect.[16] Opioid analgesics act on specific sites in the central nervous system. At the cellular level, they promote the closing of voltage-gated calcium channels, a reduction in the production of cyclic adenosine monophosphate, and the stimulation of potassium efflux resulting in cell hyperpolarization. This, in turn, reduces the neuronal excitability and nociceptive impulses of pain.[17] They are used successfully for the relief of acute pain.[18,19]

Perhaps combining an opioid (codeine) and a nonopioid analgesic (acetaminophen) can induce analgesia through both peripheral and central mechanisms[17] at a higher level than that possible by using either drug alone. This could help reduce the risk and intensity of bleaching-induced TS. Indeed, some studies in the dental field that used this combination for treatment of irreversible pulpitis have shown good results in terms of pain prevention.[20,21]

Therefore, this parallel, triple-masked, randomized clinical trial aimed to evaluate the effect of an acetaminophen/codeine analgesic, administered perioperatively for 48 hours, on the risk and intensity of bleaching-induced TS. The color change was also evaluated as a secondary outcome. The null hypothesis of this study was that combined acetaminophen/codeine administered perioperatively for 48 hours does not reduce the risk and intensity of bleaching-induced TS.

METHODS AND MATERIALS

Ethics Approval

This clinical investigation was approved (protocol number 890.207) by the scientific review committee and by the committee for the protection of human subjects of the State University of Ponta Grossa (Ponta Grossa, Brazil). This study was registered in the Brazilian clinical trials registry under the identification number RBR-4jsgbc (http://www.ensaiosclinicos.gov.br). This clinical report follows the protocol established by the Consolidated Standards of Reporting Trials statement.[22]

Trial Design, Settings, and Locations of Data Collection

This was a randomized, parallel, placebo-controlled, triple-mask clinical trial in which the patient, operator, and evaluator were masked to the group assignment. A third researcher, not involved in the evaluation process, was responsible for the randomization process and delivery and guidance on the administration of the drugs. This study was performed from July to December 2015 in Ponta Grossa and Cascavel, Brazil. All bleaching procedures were carried out within the Clinics of the Dental School of the State University of Ponta Grossa, Ponta Grossa, and at Paranaense University, Cascavel, Brazil. Both centers are located in the state of Paraná, Brazil.

Recruitment

Two weeks before the bleaching procedures, all the volunteers, who were patients in search of treatment in the clinics of the respective dental schools, signed an informed consent form and received a dental prophylaxis with pumice and water in a rubber cup.

Eligibility Criteria

Patients included in this clinical trial were at least 18 years old and had good general and oral health and did not report any type of TS. The participants were required to have six caries-free maxillary anterior teeth without restorations and no periodontal disease and must have reviewed and signed the informed consent form. The central incisors were shade A2 or darker as judged by comparison with a value-oriented shade guide (Vita Classical, Vita Zahnfabrik, Bad Säckingen, Germany).

Two calibrated investigators in each dental school independently performed the color evaluation with a value-oriented shade guide. The two examiners, masked to the allocation assignment, scheduled these patients for bleaching and evaluated their teeth against the shade guide at baseline and one month after the procedure. The two examiners were required to have an agreement of at least 85% (kappa statistic) before beginning the study evaluation. For this purpose, the evaluators independently registered the color of 10 patients before and after bleaching to record the interexaminer agreement by kappa statistics. After reaching this level of agreement, they were considered trained to start the color measurements in the present clinical trial.

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

Patients who reported some earlier or present stomach, heart, kidney, or liver problems and participants reporting continuous use of anti-inflammatory and/or analgesic drugs were excluded. Additionally, patients with diabetes, hypertension, or known allergies to acetaminophen/codeine and lactose were excluded from the study.

Sample Size Calculation

The primary outcome of this study was the absolute risk of TS. The absolute risk of TS was reported to be approximately 87%[14,23] for the bleaching product Whiteness HP Maxx (FGM Prod. Odont. Ltda, Joinville, Brazil). Thus, a minimum sample size of 92 patients was required to have an 80% chance of detecting, as significant at the two-sided 5% level, a decrease in the primary outcome measure from 86% in the control group to 61% in the experimental group (which represents a difference of 25% in the absolute risk of TS). Sample size was increased by approximately 15% to compensate for the eventual loss of patients.

Random Sequence Generation and Allocation Concealment

We used blocked randomization (block sizes of 2 and 4) with an equal allocation ratio. The randomization process was performed using software freely available on the Internet (http://www.sealedenvelope.com). Opaque and sealed envelopes containing the

identification of the groups were prepared by a third party not involved in the study intervention. This third researcher, not involved in the evaluation process, was responsible for the randomization process, delivery, and guidance on the administration of the drugs. A single random sequence was performed for both centers.

Study Intervention

Patients were divided into acetaminophen/codeine and placebo groups. All patients received the same bleaching treatment, which was performed by a single operator in each dental school. One hour before in-office bleaching, patients received either the acetaminophen (500 mg)/codeine (30 mg) (Tylex, 30 mg; Janssen-Cilag Farmacêutica Ltda, São José dos Campos, Brazil) or the placebo in identical capsules. The operator administered the first dose of drug one hour before the protocol, and extra doses were administered every six hours for 48 hours to keep a safe maximum daily dosage of 240 mg of codeine and 4000 mg of acetaminophen.[24]

The tablets of Tylex (combination of acetaminophen and codeine) were removed from their original packaging and inserted whole into empty capsules (identified by green and white color). We stored the capsules in individual vials containing eight capsules that would be required for each bleaching session. We prepared the placebo capsules in the same way described above. The capsules contained the same components of the Tylex 30-mg drug (starch, cellulose, sodium dioctyl sulfosuccinate/sodium benzoate, and magnesium stearate) except for the active ingredient.

One hour before starting the bleaching application, the masked researcher responsible for drug administration gave the first dose to the patient. Then they isolated the gingival tissue of the teeth to be bleached using a light-cured resin dam (Top Dam, FGM), and each tooth was light cured for 10 seconds (Radii-cal, SDI, Victoria, Australia). After placement of a lip retractor (Arcflex, FGM), the researcher used the 35% HP gel (Whiteness HP Maxx, FGM) in three 15-minute applications for both groups in accordance with the manufacturer's directions. The researcher refreshed the in-office bleaching agent every 15 minutes during the 45-minute application period. Two bleaching sessions were performed one week apart. All participants were instructed to brush their teeth regularly using fluoridated toothpaste.

TS Evaluation

TS was evaluated during bleaching up to one hour, from one hour up to 24 hours, and from 24 hours up to 48 hours postbleaching in both sessions. The patient was asked to indicate the numerical value of the degree of sensitivity for each one of the periods above using a five-point numeric rating scale (NRS) where 0 = none, 1 = mild, 2 = moderate, 3 = considerable, and 4 = severe.[4,6,13,14,23,25,26]

Additionally, the participants were instructed to record the pain intensity using the visual analog scale (VAS).[1,27,28] This scale is a 10-cm horizontal line with scores of 0 and 10 at their ends, where 0 = no sensitivity and 10 = severe sensitivity. The patient marked with a vertical line across the horizontal line of the scale the intensity of the TS. Then the distance in millimeters from the zero ends was measured with the aid of a millimeter ruler.

First, the data of TS from both bleaching sessions were merged. For this purpose, the worst score (NRS) and numerical value (VAS) obtained in both bleaching sessions in each assessment period was considered for statistical purposes and for the determination of the overall risk and intensity of TS.

If a patient scored zero (no sensitivity) in all time assessments from both bleaching sessions, this patient was considered to be insensitive to the bleaching protocol. In all other circumstances, the patients were considered to have sensitivity to the bleaching procedure. This dichotomization allowed us to calculate the absolute risk of TS, which represented the percentage of patients who reported TS at least once during treatment. We also calculated the overall TS intensity.

Color Evaluation

Color evaluation was recorded before and one month after dental bleaching. Color evaluation was never performed immediately after each bleaching session so that the effect of dehydration and demineralization on color measures could be prevented. The measurement area of interest for shade matching was the middle one-third of the facial surface of the anterior central incisor, according to the American Dental Association guidelines.[3,23,29] The color evaluation was performed with the value-oriented shade guide Vita Classical (Vita Zahnfabrik) and the Vita Bleachedguide 3D-MASTER (Vita Zahnfabrik). Additionally, an objective color evaluation was performed with the spectrophotometer Vita Easyshade (Vita Zahnfabrik).

For the subjective examination, with shade guide Vita Classical, the shade guide's 16 tabs were arranged from the highest (B1) to the lowest (C4) value. Although this scale is not linear in the truest sense, we treated the changes as representing a continuous and approximately linear ranking for the purpose of analysis, as already performed in several published studies.[1,4,14,25,26,28-30] The Vita Bleached-guide 3D-MASTER contains lighter shade tabs and is already organized from the highest (0M1) to the lowest (5M3) value.[5,30,31]

The two examiners, masked to the allocation assignment, scheduled these patients for bleaching and evaluated their teeth against the shade guide at the different time assessments. Color changes were calculated from the beginning of the active phase through to the individual recall times by calculating the change in the number of SGU (ΔSGU), which occurred toward the lighter end of the value-oriented list of shade tabs. In the event of disagreements between the examiners during shade evaluation, a consensus was reached.

For the objective examination, before the spectrophotometer measurement, an impression of the maxillary arch was taken with dense silicone paste (Zetaplus and Oranwash Kit, Zhermack, Italy). The impression was extended to the maxillary canine and served as a standard color measurement guide for the spectrophotometer. For each dental component to be evaluated, a window was created on the labial surface of the molded silicone guide using a metal device with a radius of 3 mm and well-formed borders.[25] The shade was determined using the parameters of the Easyshade device where it 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 is given by differences between the two colors (ΔE), calculated using the formula $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.[32]

Blinding

This was a triple-mask study in which the patient, operator, and evaluator were blinded to the group assignment.

Statistical Analysis

The analysis followed the intention-to-treat protocol and involved all participants who were randomly assigned.[22] In case of missing data, the last observation was carried forward. A preliminary statistical analysis was conducted to evaluate if the study center had any influence on the results. The absolute risk of pain of each group in both centers was compared by using the Fisher exact test. We also compared the TS intensity of both centers (within group) at each assessment time using the Mann-Whitney *U*-test. As we did not observe any statistical difference between study centers ($p>0.05$ for any comparison), the data from both centers (that come from the same population of the state of Paraná, Brazil) were merged and evaluated together.

The absolute risk of TS (including data from both centers) of both groups was compared using the Fisher exact test ($\alpha=0.05$). The relative risk as well as the confidence interval for the effect size were calculated.

Comparison of the TS intensity (NRS data) of the two groups (from the two centers) at the two different assessment points was performed using the Mann-Whitney *U*-test. Comparisons between times within each group were performed using the Friedman test. The comparison of the TS intensity obtained with the VAS was performed with two-way repeated measures analysis of variance and the Tukey test for pairwise comparison. The color changes between groups (ΔSGU and ΔE between baseline vs one month postbleaching) were compared using a Student *t*-test. In all statistical tests, the significance level was 5%. We performed all analyses by using the software SigmaPlot version 11.0 (Systat Software, San Jose, CA, USA).

RESULTS

Baseline Data and Characteristics of Included Participants

A total of 200 participants were examined in a dental chair to check whether they met the inclusion and exclusion criteria (Figure 1). In-office bleaching was performed in 105 patients out of these 200 examined participants. The baseline color (mean±standard deviation) of the participants was similar (placebo=6.2±2.0 and acetaminophen/codeine=6.0±2.0). Similarly, the mean age (years) of participants from both groups was equivalent (placebo=22.5±5.0 and acetaminophen/codeine: 22.3±6.3), ranging from 18 to 46 years old. Nineteen participants (37%) from the placebo group and nine participants (17%) from the acetaminophen/codeine group were male.

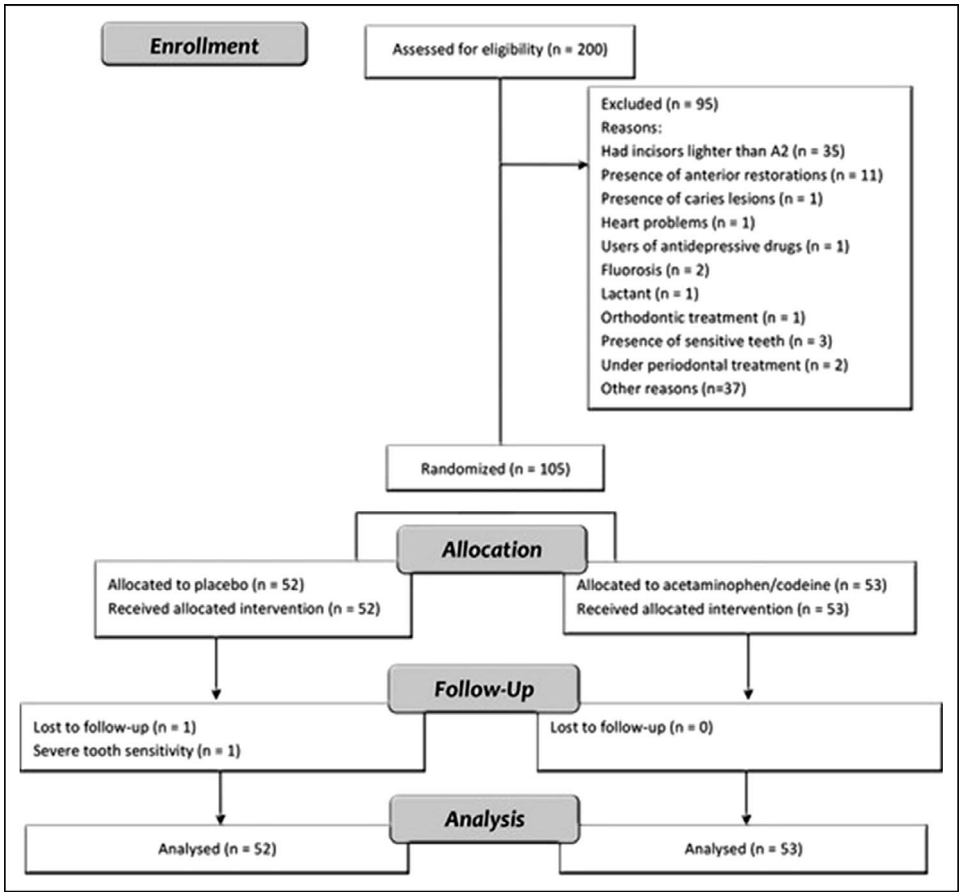

Figure 1. *Flow diagram of the clinical trial including detailed information on the excluded participants.*

Adherence to the Protocol and Dropouts

One patient from the placebo group discontinued intervention in this clinical investigation due to intense pain. This patient performed only the first bleaching session. All participants attended the recall visits one month postbleaching, except the participant from the placebo group who discontinued treatment. For this participant, the last observation was carried forward for statistical purposes to keep the intention-to-treat analysis.[22]

Two patients from the placebo group reported not taking one and six capsules after the first bleaching session: one forgot how to take it, and the other presented adverse effects (described later). Three patients from the acetaminophen/codeine group did not take three, five, and six capsules after the first bleaching session due to the development of adverse effects. Figure 1 depicts the participant flow diagram in the different phases of the study design.

TS

Three patients from the placebo group and one from the acetaminophen/codeine group took an analgesic (rescue medication) to alleviate the bleaching-induced TS (Tylenol, Janssen-Cilag Farmacêutica Ltda, São José dos Campos, Brazil). The data of these patients were included in the data analysis following the intention-to-treat protocol.

In regard to the absolute risk of TS, no significant difference was observed between groups as seen in Table 1 ($p=0.61$). The relative risk, along with the 95% confidence interval, is also evidence that the use of the acetaminophen/codeine drug had no effect on the reduction of TS. In regard to TS intensity (Tables 2 and 3), the groups did not differ statistically under the two pain scales used in this study ($p>0.05$). The very low mean difference between the two groups also shows that the observed difference is not clinically important (Table 3). But we could clearly observe that bleaching-induced TS was significantly more severe in the first 24 hours ($p<0.05$) and that it reduced considerably 48 hours after bleaching (Tables 2 and 3).

Color Evaluation

Significant whitening was observed in both study groups under the subjective and objective evaluation

Table 1: *Comparison of the Number of Patients Who Experienced Tooth Sensitivity (TS) During the Bleaching Regimen in Both Groups Along With Absolute Risk and Risk Ratio*[a]

Treatment	TS (Number of Participants)		Absolute Risk (95% Confidence Interval)	Risk Ratio (95% Confidence Interval)
	Yes	No		
Placebo	52	1	96.2 (87.2 to 98.9)	1.0 (0.92 to 1.07)
Acetaminophen/codeine	50	2	96.2 (87.0 to 98.4)	

[a] *Fisher's exact test (p = 0.61).*

methods ($p<0.05$). At the end of the bleaching protocol, a whitening of approximately 4 to 6 SGU was detected for both groups, and the ΔE varied by approximately 4.0 units (Table 4). The statistical analysis of the subjective (Vita Classical and Vita Bleachedguide) and the objective evaluation with the spectrophotometer matched the hypothesis of equality between the groups after bleaching ($p>0.05$). The very low mean difference between color change for each color measurement instrument highlights the lack of clinical importance of the observed difference (Table 4).

Adverse Effects

Five patients from the acetaminophen/codeine group and one patient from the placebo group presented nausea, vomiting, and malaise after the first bleaching session. For this reason, medication was discontinued, but the patient finished the bleaching protocol. In one patient from the placebo group and two patients from the acetaminophen/codeine group, symptoms disappeared with the discontinuation of the drugs. However, another patient from the acetaminophen/codeine group had to have the support of a physician who prescribed him an antiemetic drug.

DISCUSSION

According to the Vita Classical shade guide, the degree of whitening observed in this study, for both groups, was approximately 5 SGU. Although comparison with other studies is quite difficult due to different bleaching products and protocols,[33] studies that performed two in-office bleaching sessions with 35% HP yielded similar results.[3,23,34,35]

The Vita Classical shade guide is the most frequently used subjective tool for evaluation of color changes in bleaching studies,[5,33,36] but it presents a nonlinear arrangement of colors, as it was not designed for whitening evaluation. For this reason, we also employed the Bleachedguide VITA 3D-MASTER shade guide and an objective tool, the Easyshade spectrophotometer.

The Bleachedguide VITA 3D-MASTER shade guide was developed for evaluation of color changes in bleaching studies; it presents more uniform color distribution compared to Vita Classical,[37] and it is already organized from low to high value. It has the disadvantage of being rarely employed,[5,31,38-42] preventing authors from comparing these results with the literature. In the light of that, the authors of this study discourage the sole use of this tool.

Regardless of the instrument used for color measurement, all were convergent in the findings that groups were statistically similar, meaning that the perioperative use of acetaminophen/codeine did not jeopardize the whitening efficacy of the bleaching procedures. Intraoral use of different drugs has been used to prevent bleaching-induced TS, but based on the present study and on the findings from earlier studies, we reached the overall conclusion that the use of intraoral drugs did not affect whitening efficacy.[5,13-15]

Table 2: *Comparison of Tooth Sensitivity Intensity Experienced by Patients From the Treatment Groups at Different Assessment Points Using the Numeric Rating Scale (NRS)*[a]

Time Assessments	NRS[b]		Number of Scores 0/1/2/3/4	
	Placebo	Acetaminophen/Codeine	Placebo	Acetaminophen/Codeine
Up to 1 h	2 (1/3) Aa	2 (1/3) Xa	1/12/18/18/5	5/13/17/12/5
Up to 24 h	1 (0/2) Bb	1 (0/2) Yb	14/18/11/10/0	20/10/11/8/3
Up to 48 h	0 (0/0) Cc	0 (0/0) Zc	45/6/2/0/0	43/8/1/0/0

[a] *At each period, differences are represented by different lowercase letters (Mann-Whitney U-test for the NRS scale). For each treatment, different uppercase letters represent statistically significant means (Friedman test for the NRS scale).*
[b] *Medians (interquartile range).*

Table 3: *Comparison of Tooth Sensitivity Intensity Experienced by Patients From the Treatment Groups at Different Assessment Points Using the Visual Analog Scale (VAS)*[a]

Time Assessments	VAS[b]		Mean Difference (95% Confidence Interval)
	Placebo	Acetaminophen/Codeine	
Up to 1 h	3.8 ± 2.8 Aa	3.6 ± 2.7 Xa	0.2 (−0.9 to 1.3)
Up to 24 h	2.3 ± 2.4 Bb	2.6 ± 2.9 Yb	−0.3 (−1.3 to 0.8)
Up to 48 h	0.2 ± 0.7 Cc	0.3 ± 0.8 Zc	−0.1 (−0.4 to 0.2)

[a] *At each period, differences are represented by different lowercase letters (two-way analysis of variance for the VAS scale). For each treatment, different uppercase letters represent statistically significant means (repeated measures one-way analysis of variance for the VAS scale).*
[b] *Means and standard deviations.*

It is believed that TS that originates from bleaching is an inflammatory response of the dental pulp after the application of HP[12,43] with the liberation of bradykinin[44] and substance P, which are involved in the process of inflammation and pulp pain.[45,46] The release of substance P sends the nociceptive pain signals that indicate to the central nervous system a local proinflammatory action.[47]

The majority of the common opioids are agonists and work by stimulating the different opioid receptors (MOP, KOP, DOP, and so on), all of which are G-protein–coupled receptors. Within the central nervous system, activation of MOP receptors in the midbrain is thought to play the most important role on the opioid-induced analgesia.[48] Thus, theoretically, the net effect is the reduction of neuronal excitability, resulting in decreased neurotransmission of nociceptive impulses.[49] This could have been reinforced by the peripheral action of the acetaminophen, which acts by inhibiting cyclooxygenase release.[47]

We administered the medicines one hour before starting the bleaching procedure, as the drug takes around 30 to 60 minutes after ingestion to reach pharmacological plasmatic levels.[24] Extra doses of the medicine were administered every six hours up to 48 hours. Although the combination of codeine and acetaminophen (Tylex, 30 mg) is used in cases of severe pain, this combination reduced neither the risk nor the intensity of TS experienced by these patients. A high risk of TS was observed for both groups, corroborating the findings of previous studies.[5,7,13,14,28] Approximately 96% of participants in both groups reported TS at some stage of bleaching.

The ineffectiveness of this drug combination suggests that there are other mechanisms involved in the development of bleaching-induced TS. Previous studies that tested drugs with other mechanisms of action, such as nonsteroidal anti-inflammatory drugs,[13,15] selective anti-inflammatory medicine,[14] and even corticosteroids,[5] have also reached the same conclusion that they are not capable of preventing bleaching-induced TS. As wisely described by de Paula,[6] several factors may modulate the amount of drug that reaches the plasma and extracellular fluid and in the pulp chamber when administered per the oral route, such as the presence of the immune system, lymphatic drainage, urinary excretion, and morphological characteristics of the dentin substrate.[6]

Another factor that may explain the lack of effectiveness of this medicine combination is that codeine, which is the prototype of the weak opioid analgesics, has a weak affinity to MOP opioid receptors. Additionally, and contrary to other opioids, codeine needs to first be metabolized to morphine for production of its analgesic effect.[48] Between 5% and 10% of the population is estimated to lack the ability to perform this conversion, limiting the pain relief produced by such a drug.[48]

Table 4: *Means ± Standard Deviations of Differences Between Colors (ΔE) and Change in the Number of Shade Guide Units (ΔSGU) Obtained With the Vita Classical and Vita Bleached Guides Between Baseline and One Month Postbleaching*

Color Instruments	Groups		Mean Difference (95% Confidence Interval)	*p*-Value[a]
	Placebo	Acetaminophen/Codeine		
ΔSGU Vita Classical	5.0 ± 1.8	4.6 ± 2.0	0.4 (−0.3 to 1.1)	0.29
ΔSGU Vita Bleached	6.0 ± 3.1	5.7 ± 3.3	0.3 (−0.9 to 1.5)	0.70
ΔE	4.1 ± 1.7	4.1 ± 1.6	0.0 (−0.6 to 0.6)	0.94

[a] *Unpaired* t*-test.*

So far, minimization of the bleaching-induced TS was successfully observed only when desensitizing agents were applied topically.[7,23,28] This may suggest that perhaps these medicines are not reaching the plasmatic tissue of the pulp at the time that bleaching is performed. More basic studies should be performed to elucidate the mechanism of pain generated during bleaching and yield more appropriate approaches for the management of such an undesirable side effect.

Finally, the limitations of this study should be reported. Although our sample size was larger than most studies that evaluate anti-inflammatory drugs for the reduction of TS, the sample size was calculated only for the detection of high effect sizes for the risk of TS. Therefore, we cannot rule out the fact that smaller effect sizes might exist. The use of the same experimental design to conduct studies with larger sample sizes should be encouraged to rule out this hypothesis.

CONCLUSION

The use of an acetaminophen/codeine combination (Tylex, 30 mg) is not recommended because it does not decrease the risk and intensity of bleaching-induced TS and also causes a high number of adverse effects.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the State University of Ponta Grossa. The approval code for this study is 890.207.

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.

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Laboratory Research

Longitudinal Evaluation of Radiopacity of Resin Composites: Influence of Photoactivation and Accelerated Photoaging

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Clinical Relevance

Radiopacity has been considered an unchangeable property with great significance for assessing the quality of a restoration by radiographic images. Any changes in this property over time, under the influence of photoactivation and photoaging, might affect radiographic diagnosis.

SUMMARY

This study aimed to assess longitudinally the radiopacity of resin composites under the influence of photoactivation and photoaging processes. Ten specimens (1 mm thick and 4 mm in diameter) of three different microhybrid resin composites, Filtek Z250 XT (R1), TPH 3 Spectrum (R2), and Opallis (R3), were prepared for this study. For longitudinal assessment of radiopacity, radiographic images were obtained five times. The first time (T1), the specimens were not photoactivated; the second time (T2), the specimens were photoactivated; and the next three times, photoaging was carried out, with images obtained at 24 hours (T3), 48 hours (T4), and 72 hours (T5) after this process. The photoaging was conducted using LED light (700 lumens, 9 W, 6400 k) under controlled environmental conditions at 37°C (±1°C) and 65% (±5%) relative humidity. The digital system DIGORA Optime was used. The digital images were measured using the histogram function, and then the pixel intensity values were converted into mmAl (the standard unit of radiopacity) using a linear regression function, with minimal adjustment of $R^2 \geq 0.9$. Data in mmAl were statistically analyzed using an analysis of variance (α=0.05). R2 resin composite showed higher values of radiopacity, R1 resin composite showed intermediate values, and R3 resin composite showed lower values. Only at T1 did

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Table 1: *List of Materials Tested in This Study*

Material	Abbreviation	Manufacturer	Shade
Filtek Z250 XT	R1	3M ESPE, St Paul, MN, USA	A3
TPH 3 Spectrum	R2	Dentsply International Inc, Petropolis, RJ, Brazil	A3
Opallis	R3	FGM Produtos Odontológicos, Joinville, SC, Brazil	A3

the higher radiopacity of R2 composite differ significantly from other groups ($p = 0.0000$). After application of treatments (photoactivation and photoaging), all radiopacity values were similar (p-values to T2=0.0507, T3=0.0536, T4=0.0502, T5=0.0501) due to consecutive increase of radiopacity of R1 and R3 composites from T2. Photoactivation and photoaging processes influenced the radiopacity, but changes occurring in the degree of radiopacity were dependent on the composition and chemical characteristics of each composite used.

INTRODUCTION

Conventional two-dimensional radiographic images have been routinely used as the method of choice for oral diagnoses in general dentistry. However, despite the current revolution in radiology that has been driven by the advent of digital systems, some limitations remain unchanged because radiographic diagnosis, a "visual specialty," continues to depend on the observer's ability to interpret radiographic images.[1]

Radiographic images are maps of x-ray attenuation coefficients, which largely depend on the physical and chemical properties of the radiographed objects[2] in addition to their three-dimensional characteristics. Radiographic diagnosis is very challenging because there is a wide range of attenuation coefficients that cause dissimilar contrasts in images formed by different complex objects from facial areas, including restorative materials, lesions, and material prosthetics. Any change in x-ray attenuation coefficients can cause a variation in the resulting image and, consequently, could affect the observer's performance in a specific diagnosis. In the study of Cruz and others,[3] it was observed that radiopacity from materials with attenuation was statistically similar, ranging from dentin to enamel, which can promote a subjective influence on the diagnosis of secondary caries–like lesions, with the highest radiopacity, closer to the enamel, causing a negative influence.

There are numerous commercially available resin composites with different compositions that generate dissimilar radiopacities.[3-5] According to draft technical regulations,[6] the radiopacity of a composite material needs to be equal to or greater than that produced by a reference of aluminum (Al) of the same thickness (mmAl is the standard unit of radiopacity) and cannot vary 0.5 mmAl below any value. The Al used to produce the image reference shall have a minimum technical purity of 98% (less than 0.1% copper and less than 1% iron). Nevertheless, in spite of these draft technical regulations, the degree of radiopacity of resin composites clinically identified as the best for radiographic diagnosis has not yet been established.

Thus, considering that the radiographic diagnosis can be affected by changes in radiopacity, the aim of this study was to assess longitudinally the radiopacity of resin composites under the influence of photoactivation and photoaging processes. The radiopacity was initially considered as an unchangeable property; thus, the null hypothesis was that neither photoactivation nor photoaging would likely influence radiopacity in any way.

METHODS AND MATERIALS

The materials evaluated in this study (listed in Table 1) consisted of three different dental microhybrid resin composites, as follows: Filtek Z250 XT (R1; 3M ESPE, St Paul, MN, USA), TPH 3 Spectrum (R2; Dentsply International Inc, Petropolis, RJ, Brazil), and Opallis (R3; FGM Produtos Odontológicos, Joinville, SC, Brazil). The information about the components is listed in Table 2.

For preparation of 10 specimens of each material, resin composites were inserted into 1-mm-thick stainless steel ring molds with an internal 4-mm-diameter hole. In order to achieve uniformly smooth surfaces, the molds were placed between two glass slides covered with Mylar strips and then submitted to 1 kg/cm^2 pressure for 1 minute to remove the excess material. The target was to obtain the first digital radiographs of these resin composites without the action of polymerization process for the longitudinal assessment. Therefore, at the first time (T1), the resin composites were not photoactivated as described in Table 3. At the next step, the second

Table 2: *Composition/Information on Ingredients of the Studied Materials Cited in Material Safety Data Sheet Available*

Material's Abbreviation	Ingredient	CAS No.	Percentage[a]
R1	Silane-treated ceramic	444758-98-9	65–90
	Bis-GMA	1565-94-2	1–10
	Silane-treated silica	248596-91-0	1–10
	Bis-EMA	41637-38-1	1–10
	UDMA	72869-86-4	1–10
R2	Fiberglass wool	65997-17-3	<50
	Lead bisilicate	65997-18-4	<30
	TEGDMA	109-16-0	<10
	Bis-EMA	24448-20-2	<10
	Urethane modified bis-GMA dimethacrylate	—	<10
	Siloxanes and silicones, di-Me, reaction products with silica	67762-90-7	<3
	Silane, dichlorodimethyl-, reaction products with silica	68611-44-9	<3
	titanium dioxide	13463-67-7	<1
	Colorants	—	—
	Inorganic iron oxides	—	—
R3	Silane-treated ceramic	444758-98-9	65 – 75
	Bis-GMA	1565-94-2	6 – 8
	Bis-EMA	24448-20-2	5 – 10
	Silane-treated silica	248596-91-0	5 – 10
	Bis(2-methacryloxyethyl)-N,N′-1,9-nonylene biscarbamate	41137-60-4	5 – 10
	TEGDMA	109-16-0	<5
	Ethyl 4-dimethylaminobenzoate	10287-53-3	<1
	DL-camphorquinone	465-29-2	<1

Abbreviations: Bis-EMA, bisphenol-A ethoxylate dimethacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; di-ME, Dimethylsilicone; R1, Filtek Z250 XT; R2, TPH 3 Spectrum; R3, Opallis; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *Specific chemical identity and/or exact percentage (concentration) of all chemical components of the resin composites are not available due to trade secrets.*

time of treatment (T2), the photoactivation process occurred. Thus, these resin composites were photoactivated for 20 seconds, according to the manufacturer's instructions, using an Elipar S10 (1200 mW/cm^2) LED curing light unit (3M ESPE). The light intensity of the LED curing device was measured after every five uses by radiometer (SDI LED Radiometer/ SDI Victoria, Australia) at a distance of 0 mm, checking for any variation. Then, the Mylar strips were removed. Immediately after photoactivation, the second digital radiographs of the same resin composites were obtained. In the next three steps, involving the photoaging process and occurring over a period of 72 hours, the digital radiographs were obtained at 24 hours (T3), 48 hours (T4), and 72 hours (T5) after the photoaging process of the same resin composites. The photoaging was conducted using an LED light (Superled Ouro 100, Ourolux, Brazil) at luminous efficacy of 700 lumens, color temperature of 6400 k, power of 9 W, under controlled environmental conditions at 37°C (±1°C) and 65% (±5%) relative humidity.

The radiographic images (Figure 1) were obtained using a digital system using a receptor of the photostimulable phosphor plate system (DIGORA Optime; Soredex, Milwaukee, WI, USA) in a Helio-

Table 3: *Treatments Applied for Each Specimen*

Sequence of Treatment	Abbreviation	Photoactivation	Photoaging	Radiography
First time	T1	No	No	Yes
Second time	T2	Yes	No	Yes
Third time	T3	Yes	Yes for 24 h	Yes
Fourth time	T4	Yes	Yes for 48 h	Yes
Fifth time	T5	Yes	Yes for 72 h	Yes

Figure 1. *Illustration of radiographic technique. (A): Front view of set of objects, cylindrical locator device of the x-ray tube and acrylic plate, (B): a top view of set of aluminum (Al) wedge and stainless steel ring molds with resin specimens, (C): the Al wedge, (D): a card bite cover of the photostimulable phosphor plate, with (E): active side and (F): opposite side.*

dent 60B x-ray machine (Siemens, Erlangen, Germany) that operated at 60 kVp, 10 mA, 40-cm focus–receptor distance, and 0.12-second exposure time. A 1.2-cm-thick acrylic plate was placed between the objects and the cylindrical locator device of the x-ray tube to replicate the soft tissue.

For calculating the radiopacity of the resin specimens, in terms of their Al-equivalent thicknesses (mmAl), one Al wedge (99.8% purity) with 12 steps, each 1-mm thick, was used as the internal standard of radiopacity. Thus, all radiographic images were obtained from the set of a specimen of each material and the Al step wedge (Figure 2). Each set was radiographed three times.

The digital radiographs were measured using the histogram function of the ImageJ 1.43u software (Wayne Rasband, National Institutes of Health,

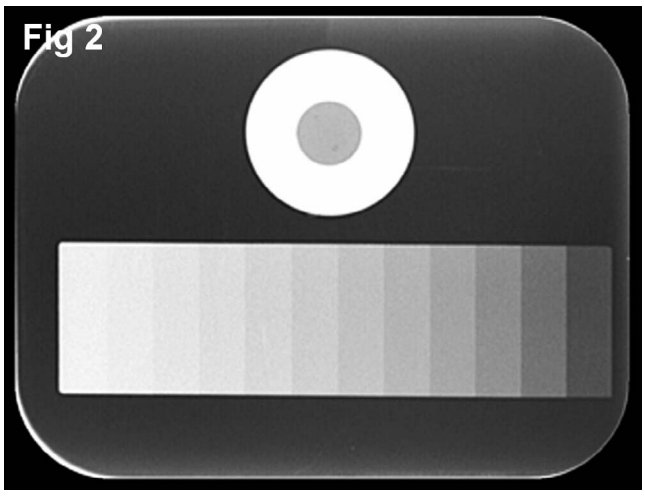

Figure 2. *Sample of image with set of specimen and aluminum step wedge used in this study.*

Table 4: *The Mean and Standard Deviation (SD) Values in Aluminum- (Al-) Equivalent Thickness (mmAl) of Each Material Tested in Accordance With Treatment Applied*[a]

Materials	Treatment				
	T1	T2	T3	T4	T5
R1	1.974 (0.548) B	2.574 (0.481) AB	3.032 (0.398) A	3.246 (0.685) A	3.441 (0.341) A
R2	3.731 (0.455) A	3.252 (0.588) A	3.322 (0.480) A	3.556 (0.602) A	3.531 (0.405) A
R3	1.486 (0.150) B	2.445 (0.390) AB	2.468 (0.481) AB	2.530 (0.402) AB	2.626 (0.365) A

Abbreviations: R1, Filtek Z250 XT; R2, TPH 3 Spectrum; R3, Opallis; T, time.
[a] *Statistically significant difference ($p < 0.05$, by the analysis of variance [ANOVA]–Tukey test) within a column between different materials. Dissimilar letters in one row designate radiopacities that differed ($p < 0.05$, by the ANOVA–Tukey test) between different treatments.*

Bethesda, MD, USA). A trained evaluator collected three areas of the same size in regions of interest (ROIs) on the center of each resin specimen and on each step of the wedge. For an accurate delimitation of each ROI, the image was enlarged. All data relating to pixel intensity values were tabulated and then converted into mmAl using a linear regression function. The minimal adjustment was found to be $R^2 \geq 0.9$.

Data in mmAl were tabulated and then statistically analyzed using an analysis of variance (ANOVA). For statistical analysis, there was no dissociation of polymerization and photoaging factors once the methodology was longitudinal for each specimen. All statistical analyses were conducted with a significance level setting of 5% (α=0.05).

RESULTS

Table 4 shows the values of radiopacity from different resin composites in terms of their Al-equivalent thicknesses. TPH 3 Spectrum (R2) resin composite showed higher values of radiopacity, Filtek Z250 XT (R1) resin composite showed intermediate values, and Opallis (R3) resin composite showed lower values. However, despite the differences in radiopacity values, only at the first time (T1) did the higher values of radiopacity of the TPH 3 Spectrum resin composite differ statistically significantly (p=0.0000) from others. After application of the treatments (from T2 on), all radiopacity values were brought closer (p-values to T2=0.0507, T3=0.0536, T4=0.0502, T5=0.0501) because resin composites Filtek Z250 XT (R1) and Opallis (R3) had a variation in radiopacity by a consecutive increase from the first time (T1). In Filtek Z250 XT (R1), this increase in radiopacity was statistically significant (p=0.0318) compared with that at the third time (T3). In Opallis (R3), the radiopacity was statistically significant (p=0.0447) at the fifth time (T5). Conversely, in TPH 3 Spectrum (R2), the radiopacity was more stable after the treatments, without significant differences (p>0.05).

DISCUSSION

In this short-term laboratory study, the radiopacities from different microhybrid resin composites were longitudinally evaluated under the action of time, from the first uncured stage to further late stages under polymerization and with induced photoaging. This methodology for evaluation of radiopacity differed from other previous studies commonly found in the literature[3,5,7-10] because radiopacity has always been evaluated in a static mode, transversely. In the current study, conversely, radiopacity was assessed more dynamically under the action of treatments over time. Moreover, against expectations and partially rejecting the null hypothesis, this property was changeable with the type of composite used and underwent changes according to the properties of each material. Thus, under the action of polymerization and photoaging, this property may be subject to changes that typically vary according to the chemical characteristics of each material.

Radiopacifying agents, which differ in their constituents, combinations, concentrations, and particle sizes, possess distinct x-ray attenuation coefficients that cause variation in radiopacity.[3-5,8,9,11-13] An ideal radiopacifier should be inert to content and be nonhazardous, possessing an adequate attenuation coefficient to enable its visualization on radiographic images without compromising other material properties such as wear resistance, degree of conversion, and polymerization shrinkage.[11,14-16] However, due to trade secrets, the specific chemical identity and/or exact percentage (concentration) of each of these chemical components of commercially available resin composites is not always accessible.

Fundamentally, all dental resin composites contain an organic matrix and inorganic fillers as ingredients. All physical properties of the resin composite are critically influenced by both the chemical structure of the monomers used in the matrix phase and the properties of their fillers. The final radiopacity of the composite is therefore largely

derived as a result of the sum of radiopacities from these different ingredients,[14] with the inorganic fillers causing a higher attenuation of x-rays. Amirouche and others[14] described all the theoretical background information about attenuation coefficients of x-rays by the dental materials according to the atomic number of their radiopacifying agent and their spatial distribution, where a high atomic number results in radiopaque images and their high concentration per area results in more radiopacity in the image. Valente and others[11] reported that the size of the inorganic fillers could not change the radiopacity when comparing dental resin composites of similar elemental composition of the filler systems but with micron- and submicron-sized monomodal glass filler particles. However, these authors emphasized that in spite of a similar elemental composition, there was a higher concentration of filler in the submicron composite. It is probable that this increase in concentration of the submicron particles promoted a compensation in attenuation, which was consequently able to promote similar radiopacity to that caused by the higher particles. In the current study, all materials evaluated were commercially available microhybrid resin composites, which are still very popular and widely used in several countries. They had particles relatively similar in size, with average values that vary from 0.1 to 0.6 μm, but had dissimilarities in elemental compositions. There are no studies in the available literature using such an approach comparing microhybrid, nanohybrid, and nanofilled resin composites, preventing any such supposition in this regard. Thus, for a better understanding on this subject, further study, using not only microhybrid, which was a limitation of the present study, but also nanohybrid or nanofilled composites, should be performed.

In the current study, the material with higher radiopacity was TPH 3 Spectrum (R2), probably due to titanium dioxide and inorganic iron oxides, which have higher atomic numbers. Silica and ceramic, the primary radiopacifying agents of resin composites Filtek Z250 XT (R1) and Opallis (R3), have the lowest atomic numbers, causing the lowest x-ray attenuation. However, in the current study, statistically significant differences in radiopacity were observed only at the first time (T1), when the composites were yet uncured. After polymerization, composites Filtek Z250 XT (R1) and Opallis (R3) did not differ significantly from TPH 3 Spectrum (R2). Thus, after polymerization, all radiopacities were statistically similar. The same situation was seen in previous studies[3,8,10,12,17] in which, despite clinical radiopacity having had variation and causing changes in the diagnostic accuracy,[3] no statistically significant difference was observed among them.

The increase in radiopacity observed in composites Filtek Z250 XT (R1) and Opallis (R3) after polymerization could be justified by polymerization shrinkage that causes closer spatial approximation between particles of radiopacifiers. The polymerization process can promote an overall reduction in the distance between the molecules of matrix because monomers react to form a covalent bond resulting in volumetric shrinkage in the final polymer network.[15] Evidence has been reported in the literature[11,15,18,19] that the volumetric shrinkage of composites is proportional to its degree of conversion. The ideal for mechanical properties would be a composite having a minimal polymerization shrinkage with an optimal degree of conversion. Unfortunately, however, the degree of conversion depends on several complex interconnected factors, ranging from chemical characteristics of the resin composites used to methodological and environmental interferences. The inorganic compounds have important roles in determining the physical and mechanical properties of resin composites.

In relation to the behavior of attenuation of radiation by resin composites over time, under the influence of induced photoaging, two distinct patterns observed in the current study can be described. One pattern was shown by composites Filtek Z250 XT (R1) and Opallis (R3), whose behavior was the increase in radiopacity over time, from the first stage after polymerization. This behavior of increasing radiopacity over time could be initially justified by the polymerization shrinkage described before that causes a closer approximation between particles, promoting the highest attenuation of radiation. In the study by Lau and others[16] it was observed that there is a continuing shrinkage even after the end of the 40-second photoactivation, with the shrinkage strains measured at 10 minutes being significantly greater than those measured at 40 seconds. However, in addition to initial polymerization shrinkage, a more important cause of dimensional change could be a bias of methodology, which was caused during the photoaging process because the specimens were maintained under a condition of relative humidity and were exposed to light. In this case, the resin composites Filtek Z250 XT (R1) and Opallis (R3), both with bisphenol-A glycidyl methacrylate, a hydrophilic compound, might have had a higher dimensional change by the absence of immersion in

water added at dehydration caused by the environment with 65% (±5%) relative humidity and by the light exposure, which may interfere in the hygroscopic expansion causing an over-shrinkage.[20,21] The other pattern was shown by composite TPH 3 Spectrum (R2), in which the radiopacity remained constant over time after applying the treatments. This behavior, on the contrary, could be justified by the highest proportion of inorganic fillers in the matrix, which might cause a higher tolerance to water sorption[20] and, consequently, smaller dimensional changes due to bias of methodology, being therefore more stable to treatments.

Furthermore, it must be emphasized that the results of laboratory evaluations are not directly applicable to the oral environment; there are several huge challenges in which dental restorative materials must withstand the widely varying conditions, including temperature fluctuations, continuous exposure to moisture, mechanical stresses, and a more extensive aging protocol. Thus, further studies comparing longitudinally the radiopacity of different materials under the influence of hydrothermal cycling and controlled clinical trials are necessary to substantiate the validity of the present results. However, an implicated direct outcome of the current study is related to the time for evaluation of radiopacity in future studies. Thus, time is a variable that should be strictly controlled between preparation of specimens and analysis of radiopacity in all groups.

CONCLUSION

The null hypothesis of the present study was partially rejected for the reason that photoactivation and photoaging processes influenced the radiopacity, but changes occurring in the degree of radiopacity were dependent on the composition and chemical characteristics of each composite used. Thus, an increase of resin composite radiopacity over time may occur, causing interference in radiographic diagnosis using digital radiographic images.

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.

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Effect of Chlorhexidine Treatment Prior to Fiber Post Cementation on Long-Term Resin Cement Bond Strength

M Durski • M Metz • G Crim • S Hass • R Mazur • S Vieira

Clinical Relevance

The addition of chlorhexidine application prior to adhesive cementation appears to be effective in enhancing the long-term push-out strength with glass fiber posts in all root thirds *in vitro*.

SUMMARY

The purpose of this study was to evaluate the push-out bond strength of two different adhesive cements (total etch and self-adhesive) for glass fiber post (GFP) cementation in simulated, long-term service (thermocycling) when the root canal is treated with chlorhexidine before cementation. One hundred twenty premolar specimens with a single root canal were selected, endodontically treated, and shaped for GFP cementation (n=120). The specimens were randomly placed into one of 12 groups (10 specimens each) according to cement (T = total-etch RelyX ARC or S = self-adhesive RelyX Unicem), treatment with chlorhexidine (N or Y: without or with), and number of thermal cycles (00, 20, or 40: 0, or 20,000 or 40,000 cycles): 1. TN00, 2. TN20, 3. TN40, 4. TY00, 5. TY20, 6. TY40, 7. SN00, 8. SN20, 9. SN40, 10. SY00, 11. SY20, 12. SY40. The root of each specimen was cut perpendicular to the vertical axis, yielding six 1.0 mm-thick sections. A push-out bond strength test was performed followed by statistical analysis using a factorial analysis of variance. Pairwise comparisons of significant factor interactions were adjusted using the Tukey test. Significant differences of push-out bond strengths were found in the four main effects (resin cement [$p<0.0001$], treatment with chlorhexidine [$p<0.0001$], number of cycles [$p<0.0001$], and root third [$p<0.0001$]) and all interactions ($p<0.05$ for

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all). Both resin cements produced higher bond strength in the cervical third followed by the middle third, and lower values were detected in the apical third. Additionally, the results suggest that the use of an additional disinfection treatment with chlorhexidine before the cement application produced the highest push-out bond strength regardless of root third. Further, the thermocycling simulation decreased the bond strength for both resin cements long-term when the chlorhexidine was not applied before cementation. However, when the root canal was treated with chlorhexidine and the fiber post was cemented with self-adhesive cement, the bond strength increased after 0, 20,000 and 40,000 cycles.

INTRODUCTION

The cement preferred for glass fiber post (GFP) cementation is dual-cured resin cement. In addition to having the advantages of light-cure activation, dual-cured cements also contain chemical initiators for deep areas where light access is difficult to achieve.[1,2] However, there have been some problems (incomplete post seating, debonding,[3,4] and defects such as pores, voids, and cracks[5,6]) reported regarding the complicated technique. To decrease the number of steps and simplify the cementation process, self-adhesive dual-cured cement was developed. This cement has one step (no need for conditioning or bonding system) and has demonstrated similar[2,7] or better[8] bonding performance compared with other resin-based cements.

Current literature has suggested that the decrease in adhesive bond strength might be caused by the degradation of the exposed collagen fibrils due to incomplete resin-infiltrated acid-etched dentin.[9] This degradation is induced by an endogenous proteolytic mechanism involving the activity of matrix metalloproteinases (MMPs).[10] Nevertheless, 2% chlorhexidine has been shown to inhibit intrinsic factors such as MMPs.[11,12] Elimination of MMPs can stabilize the composite-dentin hybridization.[13] However, there is no consensus if chlorhexidine treatment before cementation improves the bond strength or for how long. Thermocycling simulates the aging process, and it has been suggested that 10,000 cycles is comparable to 1 year of temperature changes in the mouth.[14] The literature indicates that a wide range of cycles (4000-40,000) has been used.[15] A decrease,[16] increase,[17] or no change[18] in long-term bond strength has been reported but not with the chlorhexidine interaction.

The aim of this study was to evaluate the push-out bond strength of two different adhesive cements (total etch and self-adhesive) for GFP cementation in long-term (after thermocycling simulation of 2 and 4 years = 20,000 and 40,000 cycles) when the root canal is treated with or without chlorhexidine prior to cementation.

The null hypotheses tested were: (1) there will be no statistically significant difference in GFP push-out strength between the total-etch and the self-adhesive resin cements, (2) there will be no statistically significant difference between the different resin cement GFP push-out strengths among different root thirds, (3) there will be no statistically significant difference between root canals treated without or with chlorhexidine, (4) there will be no difference in GFP push-out bond strength in long-term simulation comparing 0, 20,000 and 40,000 cycles, and (5) there will be no statistically significant difference in GFP push-out strength after the interaction of all variables.

METHODS AND MATERIALS

One hundred twenty human premolars (n=120) meeting the inclusion criteria with a ≥18-mm single root canal were selected for the study. Exclusion criteria were the presence of resorption, caries, root fractures, or previous endodontic treatment. The teeth were numbered, radiographed, and stored in 0.1% thymol.

The root canal systems were accessed through the occlusal surface under copious amounts of water and located using a K#15 file (Senseus K-Flexofile, Dentsply, Maillefer, Switzerland). Working length of the root system was determined to be 1 mm from the root apex and verified with an apex locator. The root canals were shaped by rotary instruments (ProTaper system, Dentsply) and sequenced in order (SX, S1, S2, F1, F2), applying the crown-down technique. Irrigation was performed with 1 mL of 6% sodium hypochlorite (NaOCl) solution (Vista Dental, Racine, WI, USA) using a syringe and a 27-gauge needle throughout progression of file sizes. Final irrigation was done with 17% ethylene diamine tetra acetic acid (EDTA) (Vista Dental) for 1 minute followed by 6% NaOCl solution for 1 minute. The root canals were obturated using a warm vertical condensation technique performed with gutta percha cones (Protaper F2, Dentsply DeTrey Gmbh, Konstanz, Germany) and root canal sealer (AH-Plus, Dentsply DeTrey).

A careful demineralization and tubule opening of the root dentin, with ultrasonic instrumentation in

Table 1: *Groups, Composition, Root Canal Treatment and Techniques of the Cement Application for All Materials Used in the Present Study*

Adhesive Cement	Cement Composition	Treatment with 2% Chlorhexidine	Procedures	Cement Application	Thermocycling
RelyX ARC (T) 3M ESPE, St Paul, MN, USA	HEMA, bisGMA, dimethacrylate resins, methacrylatemodified polycarboxylic acid copolymer, photoinitiator/water, ethanol	Yes group No group	Etching with 37% phosphoric acid for 15 s. Rinsing with 10 mL distilled water. Removing water excess with paper point #80 Application of multisteps adhesive system (Single Bond—3M)	Application of the adhesive cement into the root canal with a cylindrical tip microbrush. Application of the adhesive cement on the post. Insertion of post into the root canal. Light cure for 60 s.	0 cycles 20,000 cycles 40,000 cycles
RelyX Unicem **(S)** 3M ESPE, St. Paul, USA	Methacrylated phosphoric esters, dimethacrylates, acetate, initiators, stabilizers, glass fillers, silica, calcium hydroxide	Yes group No group	Rinsing with 10 mL distilled water. Removing water excess with paper point #80. Capsule activation.	Application of the adhesive cement into the root canal with an elongated tip. Insertion of post into the root canal. Light cure for 60 s.	0 cycles 20,000 cycles 40,000 cycles

association with EDTA, was performed. Post spaces were prepared to depths of 10 mm, leaving an apical seal of 5 mm of gutta-percha in the canal space. A series of sequential reamers provided for the GFP No. 2 (Rely-X Fiber Post, 3M ESPE, St Paul, MN, USA) were used for this process. The specimens were randomly placed into one of 12 groups (10 specimens per group) according to cement (T or S: total etch RelyX ARC or self- adhesive RelyX Unicem, respectively), treated with chlorhexidine (N or Y: without or with), and number of thermal cycles (00, 20 or 40: 0, 20,000, or 40,000 cycles) as follows: 1.TN00, 2.TN20, 3.TN40, 4.TY00, 5.TY20, 6.TY40, 7.SN00, 8.SN20, 9.SN40, 10.SY00, 11.SY20, 12.SY40.

All GFP surfaces were cleaned with 70% isopropyl alcohol (Cumberland Swan, Smyrna, TN, USA) and air-dried. Half the groups were not treated with chlorhexidine before cementation. The root canals of the other groups were treated with a 2% chlorhexidine solution (VEDCO, St Joseph, MO, USA), which was used to irrigate the post space using a syringe and a 27-gauge needle, and left for 60 seconds. After the waiting time, the root canal was irrigated with water and slightly dried before cementing. All manufacturer recommendations were followed for each material according to Table 1. After cementation, all teeth were light-cured for 60 seconds (Optilux 500, Kerr, Orange, CA, USA) from the occlusal direction. Thermocycling was used to simulate the aging process. The groups subjected to the 2- and 4-year aging simulation were held in 5°C cold water and then in 55°C hot water, 30 seconds in each bath, for 20,000 and 40,000 cycles.

The roots of each specimen were immediately sectioned perpendicular to the vertical axis by means of a low-speed diamond saw (Isomet, Bueher, Lake Buff, IL, USA) under copious amounts of water. Six 1.0-mm slices for each root were obtained, which were then separated into three subgroups according to specimen area (two cervical, two middle, and two apical; Figure 1).

The push-out test for each specimen section was performed with a universal testing machine (No. 8841, Instron, Canton, MA, USA) at a crosshead speed of 0.5 mm/min. The push-out test load was applied in an apical-to-cervical direction until the post became dislodged from the specimen. The push-out strength values were measured at failure and recorded in MPa ± standard deviations. A mean average for each section tested was recorded for each of the subgroups.

Statistical Analysis

The push-out strengths were measured at failure and recorded in MPa ± standard deviation. Inferential statistical analysis of the data was evaluated using factorial analysis of variance (ANOVA) and the Tukey test (α=0.05) to compare resin cement, treatment with chlorhexidine, number of cycles, and root third (SPSS version 20.0, SPSS, Chicago, IL, USA).

RESULTS

When comparing different resin cements (total-etch and self-adhesive), we found a significant difference ($p<0.05$), independent of treatment with chlorhexi-

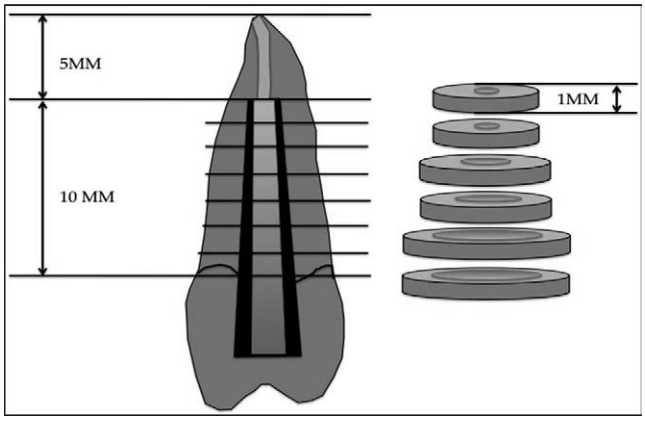

Figure 1. *Sectioning of the cemented glass fiber posts into six slices (two cervical, two middle, and two apical).*

dine and thermocycling. The self-adhesive cement had higher bond strengths than did the total-etch resin cement in all thirds (Figure 2).

When we evaluated root thirds (cervical, middle, and apical), a significant difference was determined ($p<0.05$), independent of cement, treatment with chlorhexidine, or thermocycling. The cervical third displayed the highest push-out strength, followed by the middle third, and the apical third showed the lowest push-out strength.

When comparing the chlorhexidine treatment (without and with), we found a significant difference ($p<0.05$), independent of cement, thirds, or thermocycling. The push-out strength was generally higher for both the total-etch and self-adhesive resin cement when the chlorhexidine was applied in the root canal before cementation.

In evaluating thermocycling, we found a statistically significant difference ($p>0.05$) for the self-adhesive resin cement. For this cement, thermocycling increased bond strength after 20,000 and 40,000 cycles. On the other hand, no significant statistical difference was found for the total-etch resin cement after thermocycling ($p>0.05$).

The factorial ANOVA comparing multiple independent and dependent variables showed statistically significant differences among all groups independent of cement type, root third, treatment with chlorhexidine, or thermocycling ($p<0.05$). Descriptive statistics are given in Table 2 with statistical differences noted by different uppercase letters in columns and different lowercase letters in rows.

DISCUSSION

The teeth were stored in 0.1% thymol, which has no effect on either the organic or inorganic content of dentin,[19] during the study for sterilization/disinfection purposes, and to prevent dehydration.

This research included a meticulous analysis of many factors that can improve the long-term bond strength of GFP cementation and increase the retention of the future restoration[20] of a tooth with considerable loss of coronal structure.[21]

Prefabricated GFPs were used in this study because of their lower modulus of elasticity and inherent flexibility, which help distribute stress

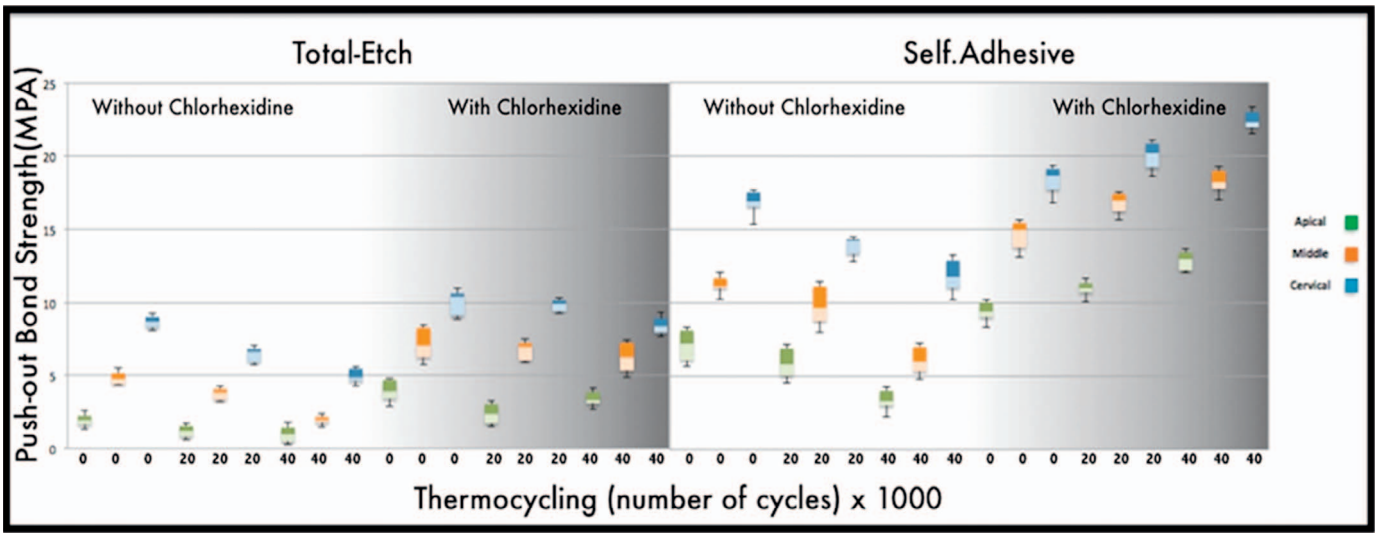

Figure 2. *Push-out bond strength means (MPa) with standard error bars of the total-etch and self-adhesive resin cements, chlorhexidine treatment, and thermocycling stratified by root thirds.*

Table 2: *Descriptive Statistics (Mean Group Push-Out Strength [MPa] ± Standard Deviation [MPa]) With Statistical Differences Noted by Different Uppercase Letters in Columns and Different Lowercase Letters in Rows*[a]

	Root Third		
	Apical Third	**Middle Third**	**Cervical Third**
Total-etch			
0 Cycles (n = 20)			
Without chlorhexidine	2.14 ± 1.09 Kc	5.55 ± 1.27 Lb	8.71 ± 0.91 La
With chlorhexidine	3.95 ± 1.82 Kc	7.09 ± 2.06 Kb	10.44 ± 1.89 Ka
20,000 cycles (n = 20)			
Without chlorhexidine	1.22 ± 0.88 Jc	3.87 ± 0.79 Jb	6.64 ± 1.15 Ja
With chlorhexidine	3.10 ± 1.23 Ic	6.99 ± 1.29 Ib	9.93 ± 0.95 Ia
40,000 cycles (n = 20)			
Without chlorhexidine	1.01 ± 0.96 Hb	2.00 ± 0.92 Hb	4.39 ± 1.12 Ha
With chlorhexidine	3.33 ± 1.15 Gc	6.29 ± 2.02 Gb	8.38 ± 1.11 Ga
Self-adhesive			
0 Cycles (n = 20)			
Without chlorhexidine	7.23 ± 1.60 Fc	11.14 ± 0.99 Fb	16.91 ± 1.72 Fa
With chlorhexidine	9.42 ± 1.21 Ec	14.97 ± 1.94 Eb	18.68 ± 2.01 Ea
20,000 cycles (n = 20)			
Without chlorhexidine	5.81 ± 1.33 Dc	8.67 ± 0.76 Db	14.23 ± 1.48 Da
With chlorhexidine	11.00 ± 1.00 Cc	16.98 ± 1.43 Cb	20.20 ± 1.66 Ca
40,000 cycles (n = 20)			
Without Chlorhexidine	3.31 ± 1.19 Bc	6.00 ± 1.26 Bb	11.76 ± 1.67 Ba
With Chlorhexidine	12.99 ± 0.92 Ac	18.22 ± 1.28 Ab	22.31 ± 0.86 Aa

[a] *Groups connected by different uppercase letters in columns and different lowercase letters in lines represent statistical differences* ($p < 0.05$).

more uniformly to the entire root surface.[22] A double-tapered GFP system was the shape selected. Schmage and others reported that precise fitting of the tapered GFP into the endodontic canal avoids the need of a wider preparation, which preserves dental hard structure in the apical third where the anatomical root form narrows the most.[23]

A careful removal of gutta-percha and tubule opening of the root dentin with ultrasonic instrumentation in association with EDTA was performed.[24] Aggressive removal of the gutta-percha during the post-space preparation often leads to an oversized post-space and can be a cause of adhesive or cohesive failure.[23] Final irrigation was performed with a 2% chlorhexidine solution, which has demonstrated antibacterial properties in the current literature, restricting microbial ingress into dentinal tubules.[25] Chlorhexidine has been shown to have the property of inhibiting matrix metalloproteinase (MMP), preserving composite-dentin hybridization over time.[26] Chlorhexidine in this study, was used in a manner similar to other antimicrobial agents, which are rinsed after use to avoid possible interference with the resin's ability to micromechanically bond to dentin. A prior study tested the chlorhexidine application and did not find any statistical difference between investigators that rinsed and those who did not rinse the solution.[27] GFPs were disinfected with alcohol before cementation to reduce the likelihood of surface damage to the glass fibers, which would have affected the integrity of the post.[26]

Considering the structural variability of the dentinal substrate inside the root canal, the push-out strength test allows for a more accurate analysis of the overall bonding mechanism and the ability to better simulate a clinical scenario.[26] Bond strength longevity can be measured after a thermocycling simulation. Thermocycling simulates the aging process, and it has been suggested that 10,000 cycles is comparable to 1 year clinically.[14] This process imitates the thermal changes that occur inside the mouth by drinking, eating, and breathing. The international standard for testing bond strength long-term stipulates an aging procedure in which test specimens are held in 5°C cold water and then in 55°C hot water, 30 seconds in each bath, for a large number of cycles. Investigators have used between 4000 and 40,000 cycles.[15] In our study, 20,000 and 40,000 cycles were used to simulate 2 and 4 years, respectively.

Considering the push-out strength of the two different resin cements, the first null hypothesis was rejected. The results showed that the total-etch adhesive cement (T) had lower push-out strength values to dentin compared with the self-adhesive cement (S), similar to the findings of previous studies.[28-31] However, other studies have reported higher bond strengths for the total-etch adhesive cement compared with the self-adhesive cement.[32-34] Conflicting results between the various luting cements might be explained by variability in research methodology.[7] Additionally, the complexity of clinical technique using multiple steps may cause inherent problems including the incorporation of air trapped in the cement layer. The self-adhesive cements are only mildly acidic, resulting in limited demineralization and hybridization of the root dentin.[35] However, even with limited hybridization,[36] the push-out strengths in this study for self-etching cement are statistically higher than those of the total-etch adhesive cements. These results may be explained by the fact that the chemical interactions between the adhesive cement and hydroxyapatite are more important for root dentin bonding than is the ability to hybridize dentin.[28] Published in the current literature, this interaction is based on calcium ion chelation by acidic groups from the self-adhesive cements, producing a chemical interaction with the dentin hydroxyapatite.[37] Despite the fact that the hybrid layer makes an important contribution to micromechanical bonding, the chemical interaction and the simplicity of application may contribute to the success of self-adhesive cements.

Considering the push-out strength between the different root thirds, the second null hypothesis for this study was rejected. The total-etch and the self-adhesive resin cement showed higher cervical third values compared with the middle third, while the apical third presented significantly lower push-out strength. The results agree with those reported in the current literature.[31,38] However, some investigators found equal bond strength values for the total-etch and self-adhesive cements in different root thirds.[39,40] Additionally, some researchers disagree, reporting higher push-out strengths in the apical third for the self-adhesive cements.[39,41] The lower push-out bond strength in the apical third, compared with the middle and cervical thirds, can be explained. Some explanations are: (1) difficulty accessing the narrow and deep areas, (2) incomplete removal of the smear layer before cementation, and (3) poor cement penetration into the root canal dentin.[7] Factors that should also be considered are the difficulty of phosphoric acid demineralization in deep areas and maintenance of ideal moisture before cementing.[37] In addition, those regions farthest from the curing light access likely affect the degree of conversion of the resin cement. Dual polymerization has better conversion values when light activation is used during polymerization.[7,42] Goracci and Ferrari used a tapered, translucent GFP to improve light penetration in the apical third of the adhesive cement.[43]

Considering the push-out strength between a root canal treated without or with chlorhexidine, we rejected the third null hypothesis. The results obtained in this study, for both cements, indicate that the immediate push-out strength is lower for the groups treated without chlorhexidine compared with those treated with chlorhexidine. For some authors, in general, the chlorhexidine method either did not demonstrate a significant difference among groups[44] or it negatively affected the bond strength of the adhesive systems to dentin.[45] These findings are supported by the fact that the antibacterial effects of the chlorhexidine solution alone does not seriously affect the bond strength of resin cements. The higher push-out strengths obtained in this study for both the total-etch and self-adhesive, when the root canal was treated with chlorhexidine prior the cementation, may be explained by the fact that other irrigating solutions were used in combination with the chlorhexidine. Sodium hypochlorite (NaOCl), the first solution used, can dissolve organic tissue for its antimicrobial properties. However, because NaOCl influences only the organic components of the smear layer, a demineralizing agent such as EDTA is indicated to supplement the NaOCl action. The 2% chlorhexidine, which has been recommended as an irrigant in endodontic treatment, is also indicated as an additional step because of its antibacterial properties.[11] When a NaOCl + EDTA combination supplemented with chlorhexidine is employed, a higher bond strength between the root canal dentin and the resin cement can be achieved.[46] Another possible explanation is that the adsorption of chlorhexidine by dentin favors the resin's infiltrating into the dentinal tubules, which might explain the high bond strengths obtained in this study. Certain properties of chlorhexidine (a strong positive ionic charge, ready binding to the phosphate groups, a strong affinity to tooth surfaces that are increased by acid etching, and increases in the surface-free energy of enamel and perhaps that of dentin) could lead to the assumption that its application after dentin acid etching will increase the dentin-wetting ability of primers, thus improving adhesion.[27]

In considering the push-out, long-term bond strength of 0, 20,000 and 40,000 cycles, we rejected the fourth null hypothesis. The literature reports a decrease in bond strength after thermocycling simulation.[15] A possible explanation for the lower bond strength of the resin cement after thermocycling is the hydrolysis effect at the cement interface. One reason might be the lack of treating the root canal with 2% chlorhexidine, whose action, when applied before bonding, inhibits the MMPs,[12] preserving the composite-dentin hybridization long-term.[13] This action can be verified by the presence of "water trees," which is the water path inside the hybrid layer. Studies have shown that the hybrid layer is conserved for up to 12 months[16] but is decreased after this period of time. The lower long-term bond strength values for the total-etch cements might be explained by the complexity of the clinical technique, which has multiple steps. This complexity may cause inherent problems, including incorporation of air in the cement layer and gaps,[8] creating access to the water trees. On the other hand, the increase in bond strength of the self-adhesive found in this study might be explained by the calcium ion chelation by acidic groups, which produces a very close chemical interaction with the dentin hydroxyapatite. As a result, a homogenous interface is produced between the GPF and the dentin in the root canal, blocking the water trees' access.

A limitation in this study was using root thirds as testing specimens. Although we determined the weakest link of the individual root thirds, a collective bond strength was not determined. Due to the taper of the canal and post, one would have to push from the apex toward the crown. A pilot study is currently under way for this type of analysis. In this study, unfortunately, simply adding the three mean root thirds together to obtain a full root push-out would produce erroneous data.

CONCLUSIONS

Within the limitations of this *in vitro* study, the results suggest that the self-adhesive cement has higher push-out strengths in all thirds compared with total-etch cement, and when treated with chlorhexidine before cementation, these numbers are greater, immediately and in long term.

Regulatory Statement

This study was conducted in accordance with all the provisions of the human subjects oversight committee guidelines and policies of the University of Louisville. The approval code for this study was IRB Exempt 14.1063.

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 presented in this article.

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Fatigue Failure Load of Restored Premolars: Effect of Etching the Intaglio Surface of Ceramic Inlays With Hydrofluoric Acid at Different Concentrations

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Clinical Relevance

Etching with hydrofluoric acid at different concentrations (1%, 5%, 10%) does not affect the fatigue failure load of premolars restored with feldspar inlays (milled by a computer-aided design/computer-aided manufacturing system). Thus, those acids might be used for ceramic etching.

SUMMARY

The aim of this study was to evaluate the effect of etching, with different hydrofluoric acid concentrations at the intaglio surface of feldspathic ceramic inlays, on the fatigue failure load of restored premolars. A total of 60 upper premolars were embedded in plastic cylinders with acrylic resin (up to 3 mm below the cement-enamel junction) and prepared using a device specially designed for that purpose. Teeth were randomly assigned to three groups (n=20): HF1, HF5, and HF10 (etching with hydrofluoric acid for 60 seconds at concentrations of 1%, 5%, and 10%, respectively). Preparations were scanned and restorations were milled by a computer-aided design/computer-aided manufacturing system. The inner surfaces of the inlays were etched and received an application of a silane coupling agent; the dentin and enamel were treated appropriately for the luting system (RelyX ARC, 3M-ESPE).

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The restorations were cemented and the fatigue failure load (in N) was determined using the staircase method (10 Hz; 10^5 cycles in each step). The initial load (585.5 N) was applied on the slopes of the cusps (labial and palatal/lingual, simultaneously) through a cylinder attached to the test machine (Instron Electro-Puls E3000). The tested samples were analyzed under a stereomicroscope for failure analysis. Fatigue data were analyzed by one-way analysis of variance. There was no statistical difference among the fatigue failure loads (in N): HF1 (448.5±79.1), HF5 (360.7±55.4), and HF10 (409.5±121.1). Regarding the fracture mode, there was a predominance of interfacial fracture (50%), followed by cusp fracture (34.6%). It may be concluded that the etching with hydrofluoric acid at the tested concentrations (1%, 5%, and 10%) does not influence the fatigue failure load of feldspathic ceramic inlays cemented on premolars.

INTRODUCTION

Esthetic and minimally invasive restorations are made possible mainly through adhesive dentistry and ceramic advancement. Despite the fact that inlays may be considered a conservative restorative alternative when compared with traditional crowns,[1] their preparation still leads to enamel and dentin loss. This may decrease strength, specifically of premolars, because it increases cusp deflection under occlusal load.[2] Costa and others[3] showed that the cavity size is a significant factor, because it influences the stress distribution and fracture strength of premolars (ie, higher cavity size leads to a higher stress concentration, triggering fracture under smaller loads).

Another factor that may influence the strength of teeth restored with inlays is the type of restoration. According to Lee and others,[4] indirect restorations decrease the cusp deflection due to the absence of polymerization shrinkage of the restorative material in the oral environment. On the other hand, shrinkage in direct restorations results in a large degree of cusp deflection, creating microcracks in the tooth structure. Thus, feldspathic ceramic is widely used for indirect restorations such as inlays, onlays, veneers, and covering ceramics of fixed dental prostheses (FDPs). Furthermore, this ceramic presents increased wear strength, has high survival rates, and withstand high values of compression.[5,6]

The clinical success of ceramic inlay restorations is based on promoting a durable adhesion among resin cement, ceramic, and tooth structure,[7,8] especially because the divergent preparation for such restorations generates a very low friction effect between the preparation wall and the inner surfaces of the restoration. From the adhesion-effect standpoint, the higher the bond strength between the tooth and the restoration, the lower the cusp deflection.[3]

Moreover, feldspathic ceramics are classified as acid sensitive due to the presence of silica in their composition.[9-12] Therefore, the recommended surface treatment is the conditioning of the intaglio surface of the ceramic with hydrofluoric acid, which promotes topographical changes and allows mechanical interlocking of the ceramic with resin cement.[13]

The changes in the ceramic surface topography, however, depend not only on the ceramic composition, but also on the acid concentration and etching time.[14] Venturini and others[15] reported that 3%, 5%, and 10% hydrofluoric acid promoted a higher and a more stable bond strength of resin cement to feldspathic ceramic after long-term aging, in comparison with 1% hydrofluoric acid. These results show that resin adhesion to this ceramic material seems to be dependent on microtopographical changes. Higher acid concentrations produce more intense surface changes, leading to greater mechanical interlocking.[16,17] However, when the flexural strength of feldspathic ceramic was tested with the same hydrofluoric acid concentrations, Venturini and others[18] found a weakening effect, regardless of the concentration used, when compared with nonetched ceramic. Consequently, there is a modification of the "defects" at the ceramic surface and subsurface, which could affect the mechanical behavior of the feldspathic restorations when exposed to cyclic intermittent loading.

When evaluating the effect of hydrofluoric acid at different concentrations on mechanical fatigue behavior of feldspathic ceramic crowns, Venturini and others[19] depicted that 5% hydrofluoric acid had a negative effect on the fatigue failure load of the crowns, whereas 1% and 10% hydrofluoric acids did not change the fatigue resistance. All of these findings highlight that the threshold breakdown during clinical function of ceramic restorations might be caused by micromorphological alterations of the inner surface of the restorations.

Besides, the quality of the adhesive interface may have an impact on bond strength values, leading to a higher or lower cusp deflection.[3] Whereas hydrofluoric acid etching of the feldspathic ceramic

Table 1: *Experimental Design*

Group	Ceramic surface treatment
HF1	Etching with 1% hydrofluoric acid[a]
HF5	Etching with 5% hydrofluoric acid[a]
HF10	Etching with 10% hydrofluoric acid[b]

[a] *Experimentally formulated by FGM.*
[b] *Condac Porcelana, FGM, Santa Catarina, Brazil.*

promoted appropriate bond strength values in previous studies,[17,20] Addison and others[21] showed that this process may weaken the ceramic surface depending on the acid concentration. Thus, it is important to find the hydrofluoric acid concentration that simultaneously enhances adhesion to this ceramic and does not weaken the ceramic material. Moreover, the aforementioned weakening effect associated with the cyclic loading of chewing may lead to fatigue failure of the restoration that occurs when the final loading cycle exceeds the mechanical capacity of the material or restored tooth.[22]

Therefore, with regard to a test setup that simulates the real restorative scenario with ceramic inlays under cyclic mechanical loading, there is no study indicating the optimal concentration of hydrofluoric acid to promote stable adhesion without weakening the restoration. Costa and others[3] reported that the etching procedure has a significant impact on the adhesive interface and that it may influence the fatigue failure load of teeth restored with ceramic inlays.

Thus, the objective of this study was to evaluate the effect of etching the intaglio surface of inlay restorations with hydrofluoric acid at different concentrations on the fatigue failure load of premolars restored with feldspathic ceramic inlays milled by a computer-aided design/computer-aided manufacturing (CAD/CAM) system, as well as to evaluate the mode of failure of the restored premolars. The null hypothesis tested was that the hydrofluoric acid at different concentrations would not influence the fatigue failure load of teeth restored with ceramic inlays.

METHODS AND MATERIALS

Experimental Design

A total of 60 extracted upper human premolars were selected following the inclusion criteria of absence of visible cracks or caries under visual examination. The specimens were randomly assigned to three groups (n=20; Table 1). After tooth randomization (http://www.randomizer.org), buccolingual and mesiodistal measurements for each tooth were performed with a digital caliper (Starrett 727, Starrett, Itu, Brazil), aiming to verify the homogeneity of teeth size in each group through the Levene test, which showed that the randomization had worked properly.

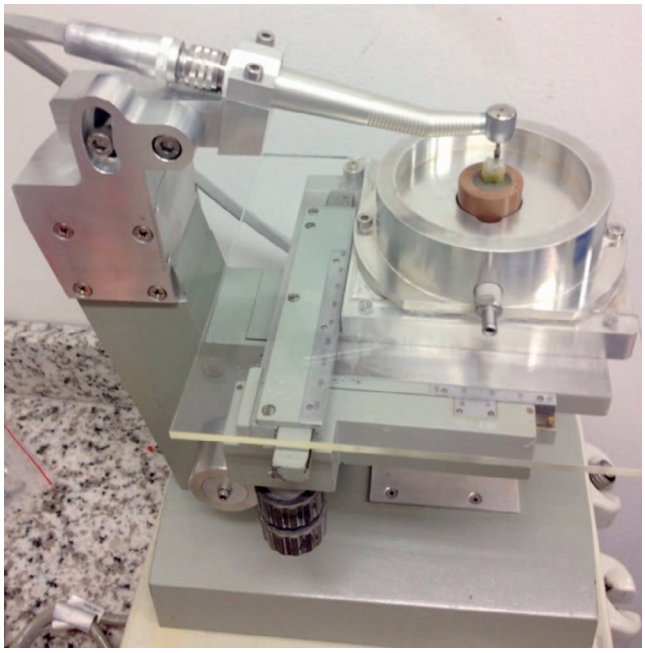

Figure 1. *Adapted device to perform standardized preparations.*

Afterward, teeth were embedded in plastic cylinders (h=14 mm, Ø=25 mm) containing chemically cured acrylic resin (Dencrilay, Dencril, Caieiras, Brazil) up to 3 mm below the cement-enamel junction, with the occlusal surface parallel to the horizontal plane.

Tooth Preparation

Standardized cavity preparations (inlay type) were performed on all teeth, using a conical-trunk diamond bur with rounded angles (KG Sorensen 3131, Barueri, Brazil). Burs were mounted on a high-speed handpiece fixed to a modified optical microscope (Figure 1).

At first, a mesio-occlusal-distal cavity was prepared to a depth of 2 mm under cool water. Then, the proximal boxes with a 2 mm depth were executed, taking into consideration the already prepared pulpal wall. Preparations had the following final dimensions: occlusal box depth = 2 mm; proximal box depth = 4 mm; occlusal isthmus determined by the bur diameter; and rounded internal line angles. Each diamond bur was used to prepare five teeth. Following, all preparations were finished with diamond burs with the same shape as the first one

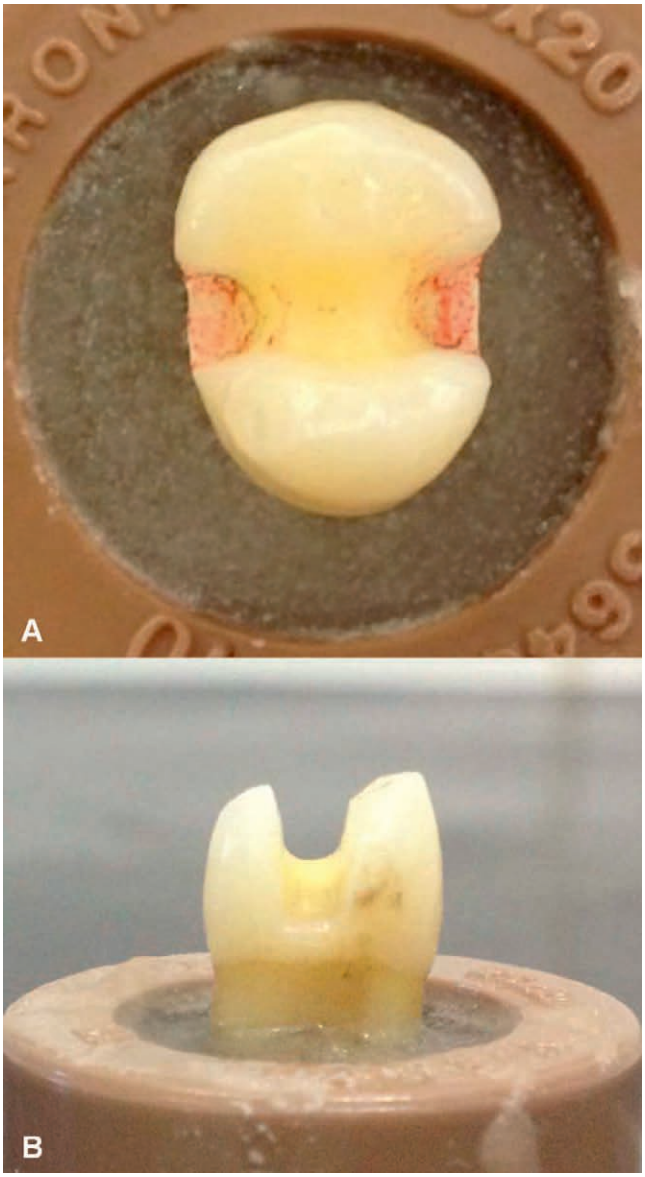

Figure 2. *Prepared teeth for inlay restoration: A) Occlusal view; B) Mesial view.*

but with lower grit size (extrafine diamond bur, KG Sorensen 3131FF) (Figure 2).

Production/Milling of Inlays

Cavities were impressed using polyvinyl siloxane by a one-step impression technique (Elite HD, Batch No. 122842, Zhermack, Badia Polesine, Italy). Impressions were poured using type IV die stone (Durone IV, Dentsply, Petropolis, Brazil). Then, master dies were sprayed with a scanning powder (Optispray CEREC, Sirona, Benshein, Germany) and optically captured by scanning (inEos Blue, Sirona). The generated image was digitally worked in specific software, which formed a three-dimensional virtual model. The cement space in the software of the CAD/CAM system was preestablished and standardized at 90 μm. After inlay design, restorations were milled in the CEREC inLab milling machine (Cerec MC XL, Sirona) from feldspathic ceramic blocks (Vita Mark II for Cerec/inLab, 2M2C/I12, and 2M3C/I12 Vita Zahnfabrik, Bad Säckingen, Germany).

Ceramic Surface Treatment and Cementation

The intaglio surfaces of the inlays were etched by hydrofluoric acid at different concentrations: 1%, 5%, and 10% (FGM, Joinville, Brazil). The etching protocol was the same for all groups: etching time of 60 seconds, rinsing with air-water spray for 30 seconds, and air-drying for 30 seconds. Then, a silane coupling agent (ESPE-Sil, 3M ESPE, Seefeld, Germany) was applied; the surface was kept untouched for 5 minutes (to allow ethanol evaporation, as recommended by the manufacturer). Tooth surfaces were conditioned with 37% phosphoric acid (Atacktec CAITHEC, São José dos Pinhais, Brazil) for 20 seconds, followed by washing and drying. The adhesive system (Single Bond, 3M ESPE) was applied on the surfaces for 20 seconds, lightly air-dried, and then light-cured (Radii Cal, SDI, Bayswater, Australia) for 20 seconds. The resin cement (RelyX ARC, 3M ESPE) was mixed as recommended and applied to the intaglio surface of the ceramic inlay. The restoration was then seated on the preparation, and a load of 750 g was applied over the occlusal inlay surface for 1 minute. The excess resin cement was removed, and photo-curing (Radiical, SDI) was performed for 20 seconds on each surface. After cementation, the specimens were stored in distilled water at 37°C for at least seven days before conducting the fatigue tests (staircase method).

Fatigue Failure Load (via Staircase approach)

First, a monotonic compression test was performed with two teeth in a universal testing machine (DL-2000, EMIC, São José dos Pinhais, Brazil) with the same piston that was later used for the fatigue test; a mean load-to-failure of 975 N was obtained. Then, the fatigue test was conducted under water, at room temperature, according to the staircase (up-and-down) method,[23] in an electrodynamic testing machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, USA).

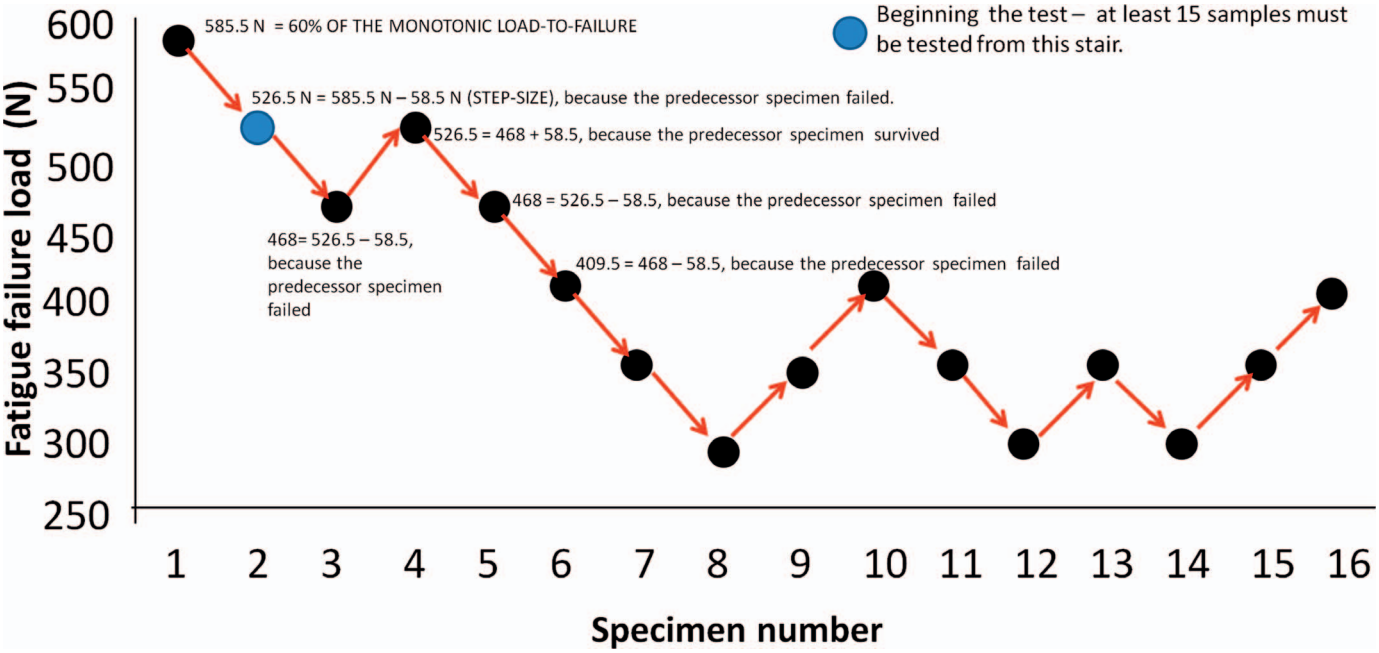

Figure 3. *Illustrative diagram of the staircase test.*

The initial load of the fatigue tests was defined as 60% of the mean monotonic load-to-failure (585.5 N).[23] According to Collins,[23] the initial load should be defined in a range where the fatigue failure load is expected to be. In this sense, previous studies usually assume values ranging from 50%-70% of the mean monotonic load-to-failure. In addition, Collins[23] defined that the staircase test would start only after the first inversion of the stair and that a minimum of 15 samples should be tested after this inversion. So, the first specimen was tested, and depending on the observation of survival or fracture, one increment (step size preestablished at 10% of the initial load; ie, 58.5 N) was increased or decreased, respectively, for the next specimen tested. The test progressed in this manner, with each succeeding specimen being tested at a load level corresponding to one increment above or below its predecessor, depending on whether it survived or fractured, until at least 15 samples were tested after the first inversion of the stair (Figure 3).

For both tests (monotonic and fatigue) the samples were placed on a metal platform at an angle of 90°, in which a cylinder piston (Ø=8 mm) applied a load only on the cusp slope, without contact with the restoration. An occlusal marker (21-μm thick carbon paper film; Accu-S017, Parkell, Farmingdale, New York, USA) was used to ensure that the piston did not touch the inlays.[1,24-26] The fatigue failure load was determined after 10^5 cycles at a frequency of 10 Hz.

Failure Analysis

After visual examination, fractures were classified on a stereomicroscope (Discovery V20, Carl Zeiss, Gottingen, Germany) according to the following criteria: cusp failure, small fractures and/or cracks in the tooth structure; restoration failure, fractures and/or cracks most evident in the restoration, and interfacial failure, fracture and/or crack at the interface with propagation through the restoration.

Topographical Analysis Under Scanning Electron Microscopy

Eight additional milled restorations (n=2) were manufactured and conditioned with 1%, 5%, or 10% hydrofluoric acid, following the aforementioned etching procedures; in addition, two machined restorations remained as milled (control to topographical analysis). Then, a micromorphological analysis under scanning electron microscopy (FE-SEM, Inspect F50, FEI, Hillsboro, OR, USA) was executed in two samples of each group. For that, the specimens were subjected to sputter-coating with gold-palladium alloy, and images were obtained with 500×, 1000×, and 2000× magnification.

Table 2: *Fatigue Failure Load (Lf) and Standard Deviation (SD) for Different Groups*

Group	Fatigue failure load, N (±SD)
HF1	448.5 (±79.1)
HF5	360.75 (±55.4)
HF10	409.5 (±121.2)

Abbreviation: HF = hydrofluoric acid.

Data Analysis

The mean fatigue failure load (Lf) and standard deviation (SD) were determined using equations 1 and 2, respectively, according to Collins[23]:

$$\mathrm{Lf} = \mathrm{Lf}_{X0} + d\left(\frac{\sum in_i}{\sum n_i} \pm 0.5\right) \quad (1)$$

$$SD = 1.62d\left(\frac{\sum n_i \sum i^2 n_i - \left(\sum in_i\right)^2}{\left(\sum n_i\right)^2} + 0.029\right), \quad (2)$$

where Lf_{X0} is the lower load considered in the analysis and d is the fixed increment (step size). In order to determine the fatigue failure load, analyses were based on data of less frequent events (survival or failure). In equation 1, the negative sign was used if the less frequent event was failure, and the positive sign was used when survival was the less frequent event. The lowest load level considered was designated $i = 0$, the next was $i = 1$, and so forth, and n_i was the number of failures or survivals at a given load level.

In addition, a one-way analysis of variance (ANOVA) test ($p<0.05$) was used to analyze the fatigue failure load data.

RESULTS

One-way ANOVA revealed no significant differences among the tested groups ($p=0.14$) (Table 2). Survival was the less frequent event for the HF5 and HF10 groups, whereas failure was the less frequent event for the HF1 group (Figure 4). Most failures had their origin at the interface between tooth and restoration (Figure 5; Table 3). Only one irreparable fracture was observed, 3 mm below the cement-enamel junction in one specimen of the HF10 group.

Topographical analysis under FE-SEM showed noticeably different surface patterns according to the different acid concentrations, especially in comparison to the untreated ceramic surface (Figure 6). However, all surfaces presented a waved pattern that can be seen in the micrographs of 500× magnification (Figure 6A,D,G). This pattern may have been caused by the machining performed by the CAD/CAM system.

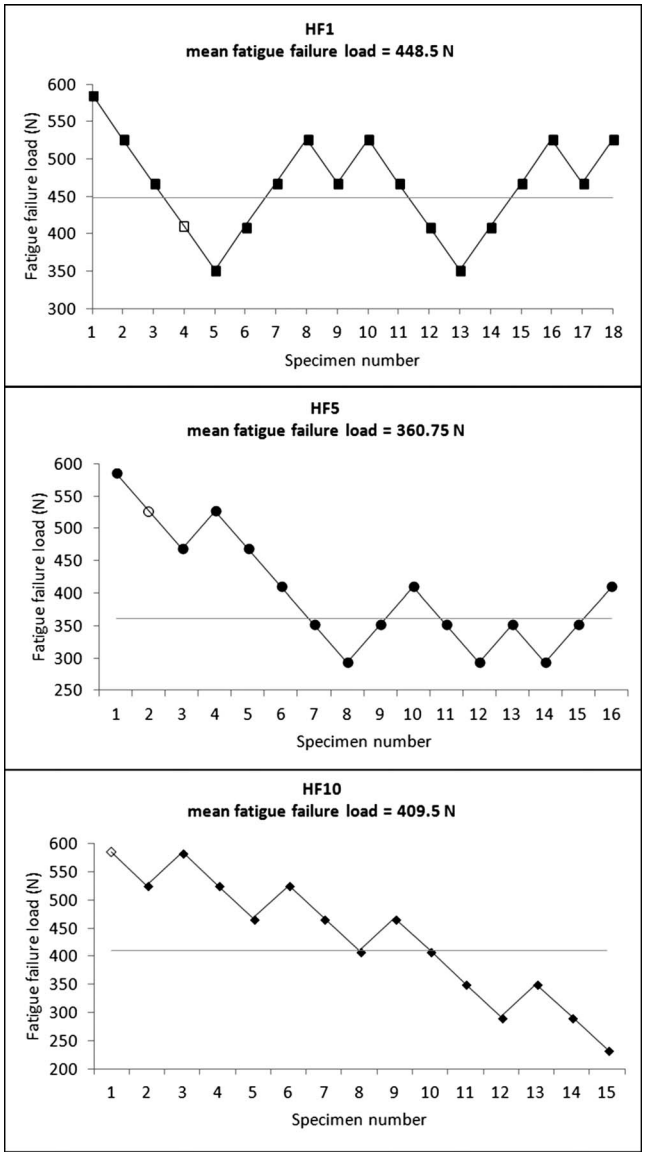

Figure 4. *Fatigue failure load events for each experimental group. The empty scorers refers to the beginning of the test.*

DISCUSSION

Our findings support that the fatigue failure load of premolars restored with feldspathic ceramic inlays was not influenced by etching with hydrofluoric acid at different concentrations (1%, 5%, and 10%), and, therefore, the null hypothesis was accepted.

Plausible explanations for those findings are: 1) the use of a silane coupling agent and 2) the variability in roughness after machining of the glass

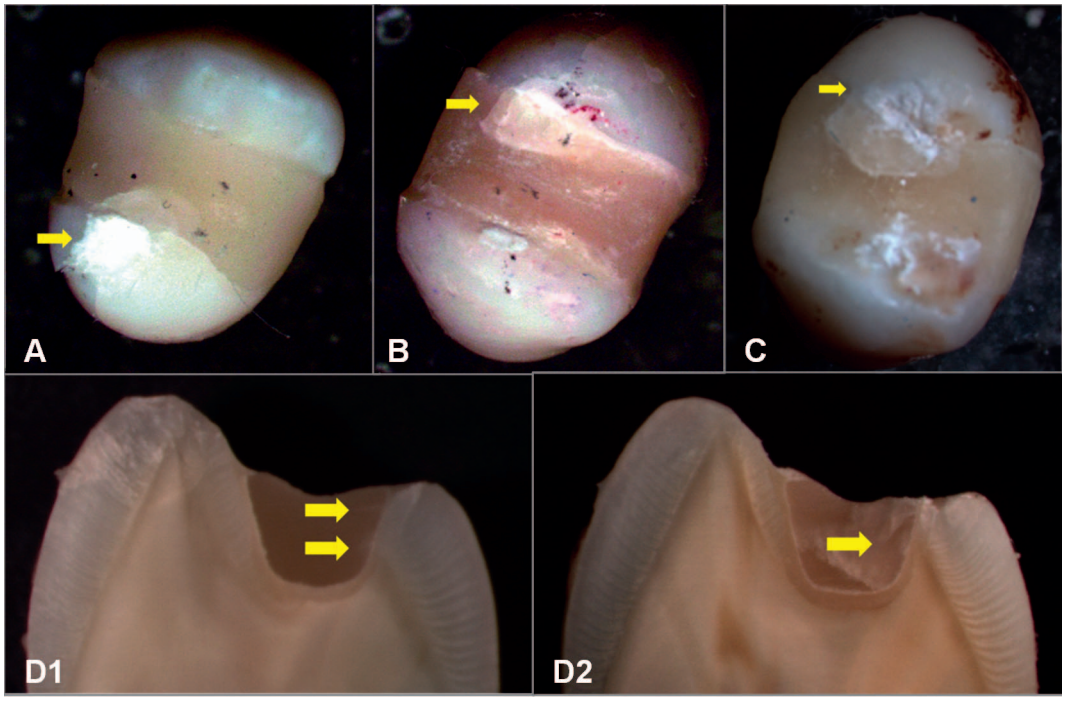

Figure 5. *Representative images of the failure modes obtained with stereomicroscopy. A- Cusp failure: small cracks and/or fractures in the tooth structure; B- Restoration failure: fractures and/or cracks in the restoration; C- Interface failure: cracks and fractures with origin at the interface. D- Cross-section of a restored tooth with interface failure; the indicators show that the origin of the failure seems to start at the interface tooth/restoration.*

ceramic, which may have enhanced mechanical interlocking; consequently, the bond strength via chemical and mechanical bonding between the resin cement and the ceramic material could be improved.[9,10,14] According to Fraga and others,[27] a variability in roughness might be expected after machining, in response to the wear of the grinding tool. In this way, they stated that the differences in roughness generated (ie, the restoration machining order) affected the mechanical properties of leucite glass-ceramics. Moreover, it is known that the use of a resin cement may increase the fracture resistance by means of filling the defects of ceramic restorations, especially the ones introduced by etching.[28,29,30] Thus, the association of those three factors could justify the similar fatigue failure load among the tested groups, given that topographical pattern alterations alone, noticed from the use of different acid concentrations, were not enough to affect the fatigue performance.

Table 3: *Failure Mode Distributions for Each Tested Group*

Group	Failure mode			
	Cusp	Rest	Interf	Total
HF1	3 (50%)	1 (16.66%)	2 (33.33%)	6 (100%)
HF5	1 (11.11%)	1 (11.11%)	7 (77.77%)	9 (100%)
HF10	5 (45.45%)	2 (18.18%)	4 (36.36%)	11 (100%)
Total	9 (34.61%)	4 (15.38%)	13 (50%)	26 (100%)

Abbreviations: Cusp = small cracks and/or fractures in tooth structure; HF = hydrofluoric acid; Interf: cracks and fracture with the origin at the interface; Rest = fractures and/or cracks in restoration.

Besides, cusp deflection is also influenced by an adequate bonding, and the interaction of those factors are also related to the final resistance to fracture.[3] The type of cement used and the quality of the bonding interface (tooth-restoration) may affect the displacement values: the higher the adhesive strength, the lower the cusp deflection.[3] In terms of adhesion, hydrofluoric acid etching is indispensable to promote effective bond strength between resin cements and silica-based ceramic surfaces.[31] Basically, the hydrofluoric acid changes the ceramic surface, increasing roughness and creating a topography for micromechanical interlocking, and the silane coupling agent interacts with the oxides, enabling chemical adhesion when associated with the use of a resin cement.[32-34]

On the other hand, some studies have suggested that hydrofluoric acid at different concentrations and etching times may weaken the ceramic material because they may introduce defects with different sizes and shapes that may not be completely filled by the cement layer.[14,35,36] Those studies[14,35,36] state

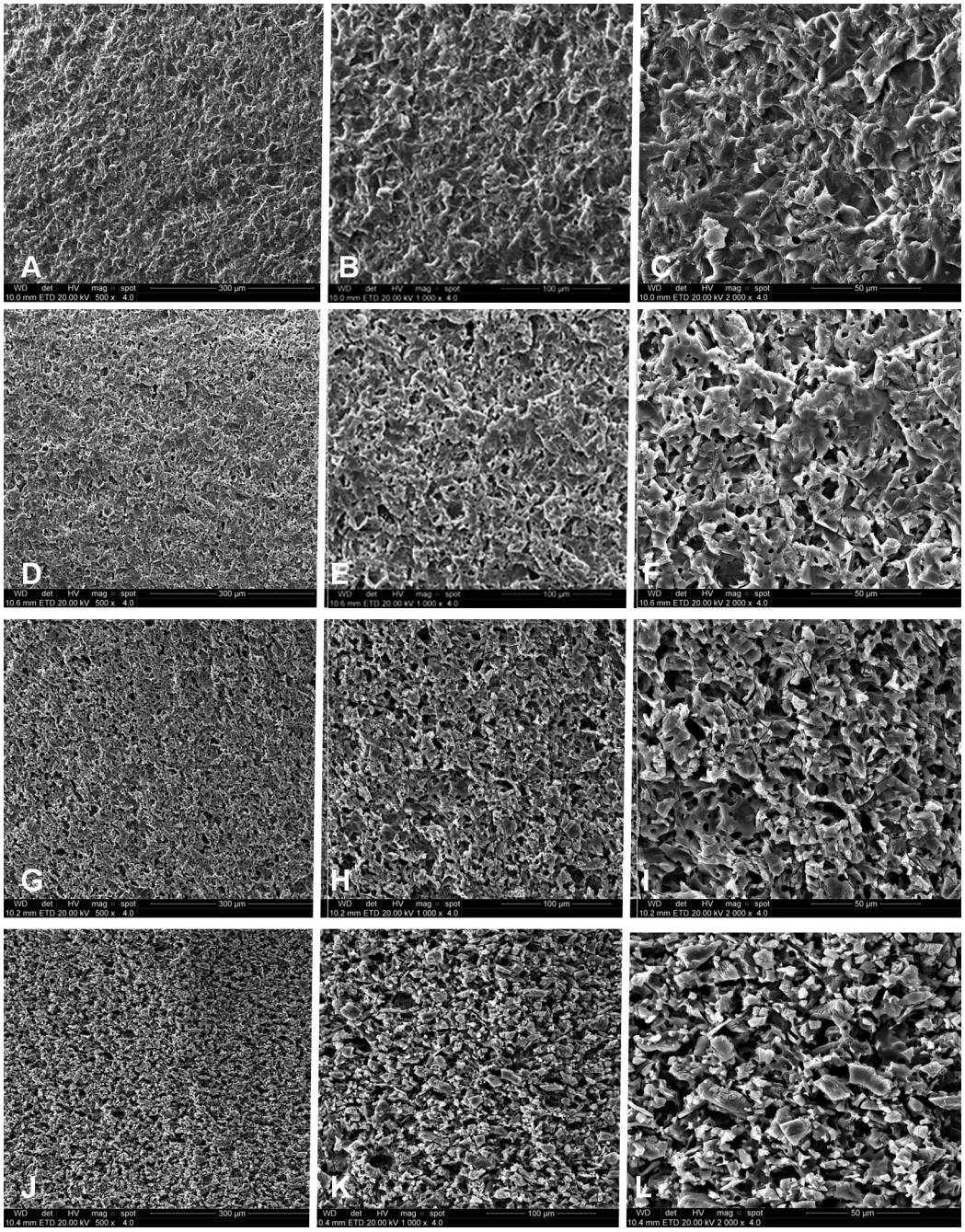

Figure 6. *Representative micrographs of ceramic surface after different acid etching treatments compared with untreated surface. From left to right: 500x, 1000x, 2000x magnifications. (A-C) untreated surface; (D-F) treated with 1% hydrofluoric acid; (G-I) treated with 5% hydrofluoric acid; (J-L) treated with 10% hydrofluoric acid. The surface patterns of the etched surfaces were noticeably different. Higher hydrofluoric acid concentrations promoted deeper and more evident craters and pits, while slight topographic changes were created for the HF1 group (D-F images).*

that when stress is applied on the ceramic material, it will concentrate around those defects, leading to premature failure under smaller loads. However, Venturini and others,[18] who tested the effect of the same acid concentrations used in this study, found no statistical difference for flexural strength among different hydrofluoric acid concentrations, and only a reduction in strength was observed when compared with the untreated ceramic condition. They suggested that the pores promoted after hydrofluoric acid etching could act as a source of crack initiation.

Regarding the failure analysis, we noticed a predominance of interface failure (50%), followed by cuspal failure (34.6%). It should be highlighted

that in the HF5 group, more than 50% of the failures were at the tooth/inlay interface, which presented failures where the crack propagated along the restoration interface (77.7%) (Table 3). These results might corroborate the aforementioned concept where etching (in this scenario with 5% hydrofluoric acid) created superficial defects that the resin cement could not fill properly due to its viscosity, creating areas of discontinuity at the interface. These areas might, consequently, act as regions of tensile stress concentration when submitted to mechanical stimuli, and thereby be responsible for the crack initiation and propagation that leads to failure.[19]

As for the incidence of cuspal failures (major failure type for HF1 and HF10 groups; 50% and 45.45%, respectively), they may be assigned to the device setup used for the fatigue testing, which led to stress concentration on the cusp surface[37] through compression load application, given that the piston was near to cusp tips.[1] This is consistent with a previous report[3] which indicated, using finite element analyses, that fractures started on the occlusal surface (at the load point) and propagated in a cervical direction.

SEM images clearly revealed the progressive effect of different hydrofluoric acid concentrations on the ceramic microstructure compared with untreated ceramic surface (Figure 6A-C), showing greater dissolution of the glassy matrix and the presence of pores in the surface after etching. These pores could act as sources of crack initiation. However, although SEM micrographs demonstrated more intense alterations in the ceramic surface topography as a result of higher hydrofluoric acid concentrations, no significant difference in the fatigue failure load of inlays was noted for the tested hydrofluoric acid concentrations.

One of the most-used approaches for inducing fatigue of dental ceramics in an accelerated and precise manner consists of the staircase method proposed by Collins.[23] Therefore, that method was chosen due to its viability and low variability.[29] Collins[23] states that to guarantee a precise estimation, 15 to 30 specimens are required. It has to be emphasized that the test only starts after the beginning of the up-and-down pattern (ie, point where the first reversal occurs) (Figure 3).[23]

The staircase method is a very useful approach for determining the mean and variance of fatigue strength over any specified lifetime; in this sense, the term *fatigue failure load* identifies the maximum load that the material may support with an increased predictability of survival (low risk of failure) at the specified lifetime. The term *fatigue limit* represents the stress below which the material supports an infinite number of cycles without failure.[23] So, our data support that none of the hydrofluoric acid concentrations (1%, 5%, and 10%) influenced the fatigue failure loads from the statistical viewpoint, which means that the surface of the feldspathic ceramic may be treated with any of these acid concentrations because they do not seem to weaken the inlay restoration. However, we must highlight that those findings should be taken with caution, given that Venturini and others[15] found a decrease in bond strength values when feldspathic ceramic was etched with 1% hydrofluoric acid, after aging. Also, we did not subject the samples to long-term aging under water, which could lead to degradation of the interface, affecting the bond durability, which could affect the fatigue behavior of restored premolars.

Following the aforementioned concepts, the current research applied an *in vitro* design, approaching parameters to approximate a clinical scenario (load application and testing setup bringing about fatigue), inducing failures that are typically observed clinically.[37] Cautions were taken for the load to be applied to the cusp slopes only (not on the interface), because our aim was also to verify whether the different etching scenarios and the quality of bonding between tooth and ceramic inlay would influence the cusp deflection.

A monotonic test with small-diameter ball indenters creates a stress state that results primarily in surface damage, which is not seen as part of a typical clinical failure.[37] Our study used restored premolars subjected to cyclic loading resembling the clinical situation. This type of load occurs in the mouth and may be simulated in laboratories with controlled parameters such as load, total number of cycles, and frequency.[1,24-26,38] Fatigue load may be considered one of the main reasons for failure of restorations clinically, and its concept is defined as the fracture of a material due to progressive brittle cracking under repeated cyclic stresses of intensity below the material's normal strength.[22,23,37]

Besides, *in vitro* studies have inherent limitations and may not fully simulate some clinical conditions that might damage the restoring assembly, such as bacteria accumulation and its toxins, temperature changes, humid environment, and sliding contact during chewing and clenching. In this sense, in our testing scenario we applied axial loading only; thus, it did not simulate sliding conditions where forces with various incidence angles take place. Thus, the

cyclic fatigue method should be viewed as an efficient screening tool for evaluating dental materials rather than as a simulation of actual dental function.[39]

Another important limitation is the difficulty for standardization of the dental substrate regarding functional age of teeth, morphologic variations of the pulp, and abnormalities in dentin composition before extraction.[40,41] Again, we must emphasize that the results of this study require careful interpretation. Further studies could investigate other testing conditions, such as different cavity size, groups of teeth, type of restorations (onlays), ceramic materials, and long-term aging under water, as well as using other methodologies for lifetime prediction.

CONCLUSION

The hydrofluoric acids at the tested concentrations (1%, 5%, and 10%) do not affect the fatigue failure load of the premolars restored with feldspathic ceramic inlays.

Acknowledgement

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Santa Maria. The approval code for this study is 1.178.683.

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.

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Degradation of Multimode Adhesive System Bond Strength to Artificial Caries-Affected Dentin Due to Water Storage

AC Follak • LL Miotti • TL Lenzi • RO Rocha • FZM Soares

Clinical Relevance

When using multimode adhesives on sound dentin, clinicians can choose to use either an etch-and-rinse or a self-etch strategy in terms of bond strength; however, on caries-affected dentin, bonding degradation will be greater irrespective of etching mode.

SUMMARY

The purpose of this study was to evaluate the influence of water storage on bond strength of multimode adhesive systems to artificially induced caries-affected dentin. One hundred twelve sound bovine incisors were randomly assigned to 16 groups (n=7) according to the dentin condition (sound; SND, artificially induced caries-affected dentin; CAD, cariogenic challenge by pH cycling for 14 days); the adhesive system (SU, Scotchbond Universal Adhesive; AB, All-Bond Universal; PB, Prime & Bond Elect; SB, Adper Single Bond 2; and CS, Clearfil SE Bond), and the etching strategy (etch-and-rinse and self-etch). All adhesive systems were applied under manufacturer's instructions to flat dentin surfaces, and a composite block was built up on each dentin surface. After 24 hours of water storage, the specimens were sectioned into stick-shaped specimens (0.8 mm^2) and submitted to a microtensile test immediately (24 hours) or after six months of water storage. Bond strength data (MPa) were analyzed using three-way repeated-measures analysis of variance and *post hoc* Tukey test (α=5%), considering each substrate separately (SND and CAD). The etching strategy did not influence the bond strength of multimode adhesives, irrespective of the den-

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tin condition. Water storage only reduced significantly the bond strength to CAD. The degradation of bond strength due to water storage was more pronounced in CAD, regardless of the etching strategy.

INTRODUCTION

To simplify the adhesive technique and improve its clinical versatility, materials referred to as universal or multimode adhesives have been developed. They generally consist of one-bottle adhesives that can be used both in etch-and-rinse and self-etch strategies. Moreover, manufacturers and previous studies recommend an alternative protocol: the selective enamel etching technique, which combines the advantages of acid etching on enamel with the simplified self-etching technique in dentin.[1,2]

Moreover, several multimode adhesives present phosphate monomers (such as 10-MDP)[3] in the composition in an attempt to improve bond strength longevity by chemical adhesion to hydroxyapatite. As the hybrid layer is prone to both collagen and resin matrix hydrolytic degradation,[4-6] it is extremely important to evaluate the bond stability achieved with multimode adhesive systems on different substrates.

Recent studies have evaluated different multimode adhesive systems, comparing the performance of etch-and-rinse and self-etch strategies.[7-16] A systematic review with meta-analysis demonstrated that the etch-and-rinse strategy produces higher values for enamel; however, on dentin, for the majority of the multimode adhesive systems included, both strategies produced similar values.[17]

High immediate bond strength values have been found for multimode adhesives[7-9,11,12,14,15]; however, the question about the long-term bonding effectiveness to dentin remains controversial.[7,10,11,13-16] Moreover, the influence of a substrate condition such as caries-affected dentin on the bond strength of multimode adhesives is unclear. In caries-affected dentin of primary molars, the effect of etching strategy was substrate dependent,[18,19] whereas in permanent dentin, the effect of approach strategy was not significant.[20]

Caries-affected dentin left after selective removal of carious tissue is a clinically relevant substrate in everyday dental practice. The selective removal of carious tissue can significantly reduce the risk of pulp exposure and postoperative pulp symptoms, which is advantageous for deep caries treatment.[21] However, caries-affected dentin differs in composition and microstructure compared with sound dentin,[22] and lower bond strengths have been reported when bonding to this substrate, regardless of the adhesive system tested.[22-28] It is also known that hybrid layer degradation is more pronounced on caries-affected dentin due to substrate-intrinsic characteristics.[27] Based on this, laboratory assessments of bonding to caries-affected dentin is relevant,[22] including the performance of universal adhesive systems on this kind of substrate.

Therefore, the aim of this *in vitro* study was to evaluate the influence of water storage on bond strength of multimode adhesive systems to artificially induced caries-affected dentin. The null hypotheses tested were as follows: 1) water aging has no effect on bond strength of multimode adhesives systems to sound and artificially induced caries-affected dentin, irrespective of the etching strategies, and 2) the multimode adhesives present similar bonding, independent of the etching strategies on either sound or artificially induced caries-affected dentin.

METHODS AND MATERIALS

Tooth Selection and Preparation

One hundred twelve freshly extracted bovine incisors were stored in 0.5% aqueous chloramine T at 4°C for a maximum of 30 days and used in this study. The teeth were divided into dentin substrates (sound and artificially induced caries affected), and for each substrate, teeth were allocated into eight groups (n=7). The root portion was removed using a diamond disc in a low-speed hand piece. The buccal surfaces were ground under water cooling using a 100-grit SiC paper in a polishing machine (EcoMet 250, Buehler, Lake Bluff, IL, USA) to expose and obtain flat dentin surfaces. Further, for both substrates (sound dentin and artificially induced caries-affected dentin), buccal surfaces were ground manually using 600-grit SiC paper for 60 seconds to create a standardized smear layer.

Artificial Caries Induction

The teeth were randomly assigned in two groups according to the substrate condition: sound (SND) and artificially induced caries-affected dentin (CAD). After procedures to obtain a standardized smear layer, teeth allocated to the sound dentin group were kept in distilled water and those to the artificially induced caries-affected dentin group were submitted to artificial caries induction by a pH-cycling model.[29,30] Teeth were individually submitted to immer-

sion for eight hours in 10 mL of demineralizing solution (2.2 mM $CaCl_2$, 2.2 mM NaH_2PO_4, and 50 mM acetic acid, adjusted pH of 4.8 with 1 M KOH) and for 16 hours in the same volume of remineralizing solution (1.5 mM $CaCl_2$, 0.9 mM NaH_2PO_4, and 0.15 mM KCl with adjusted pH of 7.0). Solutions were changed at every cycle for 14 days, and the solution pH was confirmed on each cycle using a digital pH meter (Digimed, DM22, ServMed Analítica, Guarulhos, SP, Brazil).

Bonding and Restorative Procedures

Teeth from each dentin substrate (sound dentin and artificially induced caries-affected dentin) were randomly reallocated into eight groups according to the adhesive system and etching strategy (n=7). The three multimode adhesives systems evaluated were as follows: Scotchbond Universal (SU; 3M ESPE, St Paul, MN, USA), All-Bond Universal (AB; Bisco, Shaumburg, IL, USA), and Prime & Bond Elect (PB; Dentsply Caulk, Milford, DE, USA). All materials were applied on dentin surfaces in either a self-etch (SE) or etch-and-rinse (ER) protocol. As control groups for each strategy, a two-step etch-and-rinse Adper Single Bond 2 (SB; 3M ESPE) and a two-step self-etch Clearfil SE Bond (CS; Kuraray Noritake Dental, Tokyo, Japan) were used. A single trained operator applied the adhesive systems on dentin surfaces strictly under the manufacturers' instructions (Table 1).

After hybridization, a block (~10 × 7 × 5 mm) of resin composite (Filtek Z250, shade A2, 3M ESPE) was incrementally built up on dentin surfaces, and each increment was light cured for 20 seconds using a light-emitting diode curing unit (Emitter B, Schuster, Santa Maria, RS, Brazil) delivering 800 mW/cm^2 and checked with a radiometer (Demetron Research Corp, Danbury, CT, USA) every three blocks. All specimens were stored in distilled water at 37°C for 24 hours.

Microtensile Bond Strength (μTBS)

Specimens were sectioned in two perpendicular axes with an underwater cooled diamond saw in a cutting machine (Labcut 1010, Extec Co, Enfield, CT, USA) by a single blinded operator, obtaining stick-shaped specimens with a cross-sectional area of approximately 0.8 mm^2 measured individually with a digital caliper (Carbografite, Petrópolis, RJ, Brazil). Then, one half of the obtained specimens from each tooth were randomly assigned to be tested immediately (24 hours) and the other half after six months (6Mos) of water storage.

For microtensile testing, specimens were fixed to metallic devices (Odeme Medical and Dental, Joaçaba, SC, Brazil) with cyanoacrylate glue (Three Bond Super Gel, ThreeBond, Diadema, SP, Brazil) and submitted to the microtensile test in a universal testing machine (EMIC DL in 1000, Equipment and Systems Ltda, São José dos Pinhais, PR, Brazil) at a crosshead speed of 1 mm/min until fracture. Specimens that failed prior to the test or during cutting or fixing procedures were recorded as pretesting failures (PTFs) and included in the bond strength means. A single blinded operator performed the microtensile test. All fractured specimens were observed under 40× magnifying stereoscope (Discovery.v20, Zeiss, Oberkochen, Germany) to identify and classify the type of failure as adhesive/mixed (failure at the resin–dentin interface or mixed with cohesive failure of the neighboring substrate) or cohesive (dentin or resin).

Scanning Electron Microscopy

Representative specimens from each dentin substrate (sound and artificially induced caries-affected dentin) were gold sputtered and analyzed with scanning electron microscopy (SEM; Jeol-JSM-T330A, JEOL, Tokyo, Japan) operated in the secondary electron mode with 5 kV.

Statistical Analysis

The experimental unit in the study was the tooth. Thus, the means of μTBS (MPa) values of specimens tested at 24 hours or 6Mos were averaged for statistical purposes. The sample size had been determined that, considering a mean difference of 20% among groups and expecting a variation coefficient of 20%, a minimum of seven teeth per group was required to achieve a power of 0.8 and an α error probability of 5%.

Three factors were considered in statistical analysis: adhesive system (AB, PB, SU, SB, and CS), etching strategy (ER and SE), and evaluation time (24 hours and 6Mos). Analyses were performed for each substrate separately (SND and CAD).

A normal distribution of the data was confirmed by the Kolmogorov-Smirnov test. Data were analyzed by a three-way repeated-measures analysis of variance (ANOVA) and *post hoc* Tukey tests at a significance level of 0.05, using a statistical software package (Minitab, Minitab Inc, State College, PA, USA).

PTFs were included in the bond strength means with a value of 0.[31,32]

Table 1: *Adhesive systems (manufacturers and batch number), composition, and application mode*[a]

Adhesive System/Batch	Composition	Application Mode
All Bond Universal (Bisco Inc, Schaumburg, IL, USA) (1500000055)	Bis-GMA, 10-MDP, HEMA, ethanol, initiators, water	SE: 1. Dispense one to two drops into a clean well. 2. Apply two separate coats, scrubbing the preparation with a microbrush for 10-15 seconds per coat. Do not light cure between coats. 3. Evaporate excess solvent by thoroughly air-drying with an air syringe for at least 10 seconds, there should be no visible movement of the adhesive. The surface should have a uniform glossy appearance. 4. Light cure for 10 seconds. ER: 1. Etch enamel and dentin using an etchant for 15 seconds. Rinse thoroughly. Remove excess water by blotting the surface with an absorbent pellet or high volume evacuation for one to two seconds, leaving the preparation visibly moist. 2. Apply adhesive as self-etch technique.
Prime Bond Elect (Dentsply Caulk, Milford, DE, USA) (140304)	Mono-, di- and trimethacrylate resins, PENTA, diketone, organic phosphine oxide, stabilizers, cetylamine hydrofluoride, acetone, water	SE: 1. Place two to three drops into a clean well. Immediately apply generous amounts of adhesive to thoroughly wet all the tooth surfaces. Agitate the applied adhesive for 20 seconds. Re-wetting of the microbrush may be required in order to coat the preparation for the full 20 seconds. 2. Remove excess solvent by gently drying with clean, dry air from a dental syringe for at least five seconds. Surface should have a uniform glossy appearance. 3. Light cure for 10 seconds. ER: 1. Apply Caulk 34% tooth conditioner gel. Condition enamel for at least 15 seconds and dentin for 15 seconds or less. Remove gel with aspirator and/or vigorous water spray and rinse conditioned areas thoroughly for at least 15 seconds. Remove rinsing water completely by blowing gently with an air syringe or by blot drying with a cotton pellet. 2. Apply adhesive as self-etch strategy.
Scotchbond Universal (3M-ESPE, St. Paul, MN, USA) (509806)	MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond Copolymer, Filler, Ethanol, Water, Initiators, Silane	SE: 1. Apply the adhesive to the entire preparation with a microbrush and rub it in for 20 seconds. 2. Direct a gentle stream of air over the liquid for about five seconds until it no longer moves and the solvent is evaporated completely. 3. Light-cure for 10 seconds. ER: 1. Apply etchant for 15 seconds. Rinse thoroughly and air dry or cotton pellet. Do not overdry! 2. Apply adhesive as in the self-etch strategy.

Table 1: Continued.

Adhesive System/Batch	Composition	Application Mode
Adper Single Bond 2 (3M-ESPE, St. Paul, MN, USA) (N520165)	Dimethacrylate resins, HEMA, Vitrebond Copolymer, Filler, Ethanol, Water, Initiators	1. Apply etchant for 15 seconds. Rinse for 10 seconds. Blot excess water using a cotton pellet or mini-sponge. The surface should appear glistening without pooling of water. 2. Immediately after blotting, apply two to three consecutive coats of adhesive for 15 seconds with gentle agitation using a fully saturated applicator. Gently air thin for five seconds to evaporate solvents. 3. Light cure for 10 seconds.
Clearfil SE Bond (Kuraray Noritake Dental Inc., Tokyo, Japan) (Primer: 01233A Bond: 01865A)	PRIMER: 10-MDP, HEMA, Hydrophilic aliphatic dimethacrylate, dl-Camphorquinone, *N,N*-Diethanol-p-toluidine, Water BOND: 10-MDP, Bis-GMA, HEMA Hydrophobic aliphatic dimethacrylate, dl-Camphorquinone, *N,N*-Diethanol-p-toluidine, Colloidal silica	PRIMER: 1. Dispense the necessary amount of PRIMER into a well of the mixing dish immediately before application. 2. Apply PRIMER to the entire cavity wall with a sponge or a disposable brush tip. Leave it in place for 20 seconds. Use caution not to allow saliva or exudate to contact the treated surfaces for at least 20 seconds. 3. After conditioning the tooth surface for 20 seconds, evaporate the volatile ingredients with a mild oil-free air stream. BOND: 1. Dispense the necessary amount of BOND into a well of the mixing dish 2. Apply BOND to the entire surface of the cavity with a sponge or a disposable brush tip. 3. After application, make the bond film as uniform as possible using a gentle oil-free air stream. 4. Light-cure the BOND for 10 seconds with a dental curing light.

Abbreviations: bis-GMA, bisphenyl-glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl-dihydrogen-phosphate.
[a] *According to information provided by manufacturers.*

RESULTS

Statistical analyses were performed separately for each substrate (SND and CAD). Significant cross-interaction among the three factors (adhesive system × strategy × time) was found on both SDN ($p=0.005$) and CAD ($p=0.012$) analyses. Table 2 presents the μTBS values (means and standard deviation) and contrasts found in the interactions separately for each substrate.

On SND, the etching strategy did not influence the bond strength for any tested universal adhesive, regardless the water storage. After 6Mos of water storage, all materials showed a trend of reducing numerical values, but with no significant differences, except the AB with ER strategy, which showed a significant degradation of bond strength values.

Etching strategies also produced similar values when compared among each other on CAD. However, considering degradation over time on CAD, different behavior was observed because a significant reduction in bond strength values was found, except for AB on ER strategy and PB on SE strategy.

Bond strength values of multimode adhesive systems in the ER strategy were similar to SB, which was used as the control etch-and-rinse system. Only PB–ER after 6Mos of water storage presented lower values than the control. The same was found for multimode systems used in the self-etching strategy on sound dentin; no significant differences were found compared with CS, the self-etch system that was used as a control. On CAD, significant differences were only found for bond strength values of PB and SU using the SE strategy, with values that

Table 2: *Bond strength mean values in MPa (standard deviation) for experimental groups[a] [tested sps/pretest failures]*

Material	Strategy	Sound Dentin		Caries-Affected Dentin	
		24 Hours	Six Months	24 Hours	Six Months
AB	ER	50.3 (12.4) A [52/0]	28.6 (11.3) B,C,D [57/0]	13.1 (2.7) b,c,d [46/14]	2.5 (0.3) d [49/2]
	SE	34.7 (7.5) A,B,C,D [53/2]	34.1 (10.8) A,B,C,D [57/0]	22.5 (4.4) a,b [55/5]	3.7 (2.4) d [58/0]
PB	ER	42.3 (8.3) A,B,C [62/2]	24.7 (8.1) C,D [71/0]	19.5 (6.1) a,b,c [49/10]	3.9 (1.6) d [58/0]
	SE	26.2 (17.1) C,D [48/14]	20.0 (5.7) D [53/0]	16.3 (10.9) b,c [41/22]	9.4 (7.9) c,d [55/0]
SU	ER	48.0 (14.2) A,B [53/4]	33.6 (15.4) A,B,C,D [58/0]	21.3 (6.1) a,b [56/5]	2.4 (0.2) d [63/0]
	SE	40.1 (8.9) A,B,C,D [55/2]	34.7 (8.8) A,B,C,D [59/0]	17.7 (4.4) b,c [55/5]	4.7 (3.2) d [58/0]
SB		52.6 (11.8) A [51/1]	47.3 (10.4) A,B [61/0]	22.4 (6.4) a,b [60/6]	3.4 (1.8) d [64/0]
CS		26.5 (9.6) C,D [52/18]	23.2 (7.7) C,D [54/0]	29.2 (7.3) a [58/4]	12.5 (11.3) b,c,d [60/0]

[a] *Different letters indicate statistically significant differences ($p<0.05$). Uppercase letters for sound dentin and lowercase for artificially induced caries-affected dentin.*

were lower than the control at immediate evaluation.

Significant differences were found between control adhesives at immediate and 6Mos of water storage because the etch-and-rinse system (SB) presented higher values than the self-etch adhesive (CS).

Adhesive/mixed failure patterns were predominant for all experimental groups, except for SB ER on SND after 6Mos of water storage. Cohesive failures (resin or dentin) seemed to be more frequent in sound dentin and increased in the 6Mos groups (Figure 1). PTFs were numerically more evident in CAD compared with SND (Table 2).

DISCUSSION

Caries-affected dentin has been a common substrate through direct restoration procedures in a minimally invasive adhesive dentistry approach. Several studies tested different adhesive systems, with different strategies in an attempt to achieve better performance on this kind of substrate.[18,22-26,28,33-35] Intrinsic characteristics of caries-affected dentin usually lead to lower bond strength compared with sound dentin.[22,26,27,33] Thus, in our study, artificially induced caries-affected dentin was considered separately in the statistical analyses, enabling us to evaluate three universal adhesive systems while discriminating both etching strategy and storage time for each dentin condition. This is one of the first studies that assessed the long-term bond strength of different multimode adhesives to caries-affected dentin.

The bond strength values to CAD were clearly lower compared with SND. Carious dentin is more porous than sound dentin due to the demineralization process.[22] Lower mineral content in CAD can induce a decrease in bond strength.[27] A previous study reported that such a decrease could be the consequence of the collapse of collagen fibrils that prevents proper penetration of adhesive resin.[36] This behavior on substrates such as caries-affected dentin is well stated in the literature for self-etch and etch-and-rinse systems.[22-26,28]

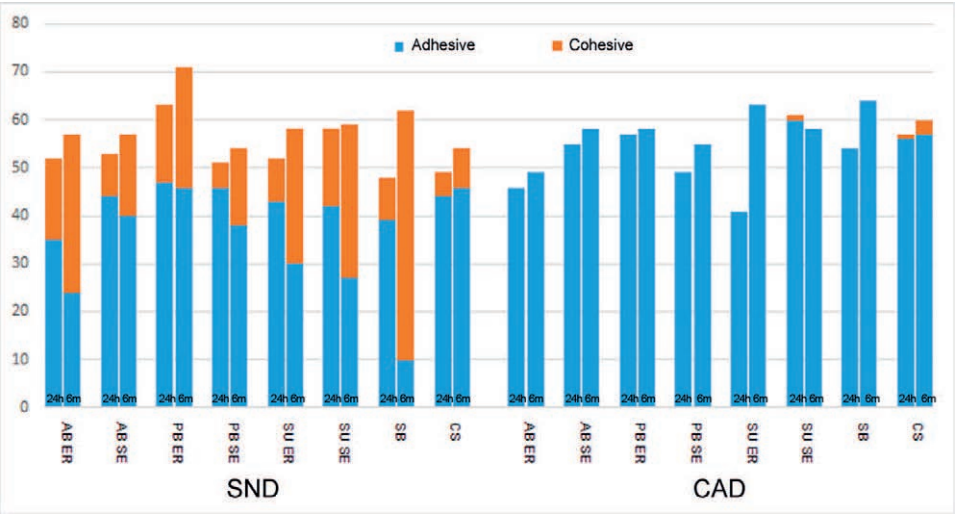


Figure 1. *Fracture type distribution per experimental group.*

The bond strength values remained stable in sound dentin after 6Mos of water storage, irrespective of the etching strategy, except for All Bond Universal using the etch-and-rinse strategy, which presented significant bond strength reduction after storage.

On the other hand, bonding to CAD presented a significant reduction for most experimental groups. There was an evident numerical drop in bond strength values for all the adhesive systems tested on this substrate. Only All Bond Universal using the ER strategy and Prime & Bond Elect using the SE strategy presented similar values after water aging on CAD. It is worth mentioning that these two adhesives, using their respective strategies, already produced the lowest immediate bond strength to CAD. Less mineral content, wettability, and other morphologic and chemical characteristics apparently had a strong influence on resin–CAD bond longevity due to higher permeability and a poor-quality hybrid layer.[22,26,37] In addition, the action of matrix metalloproteinase (MMPs) is more intense in caries-affected dentin.[27] The susceptibility to degradation of the interfaces created on CAD was reported in other studies evaluating self-etch and etch-and-rinse systems[33,34] and confirmed in our study, including the universal adhesive systems. Despite using a relatively short-term water storage, our study was able to demonstrate a more pronounced decrease in bond strength in caries-affected dentin, probably because of the substrate characteristics.

The etching strategy did not influence the immediate bond strength values for the multimode adhesives, regardless of the dentin condition and storage time, which is in accordance with previous studies that also evaluated both sound[8,11,12,15] and caries-affected dentin.[18]

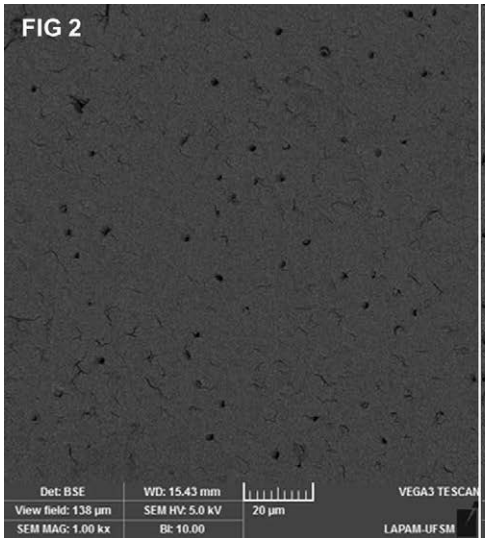
The three multimode adhesives evaluated in this study present different compositions. Two of them (All Bond Universal and Scotchbond Universal) contain a monomer able to chemically bond to the dentin substrate (10-MDP). In addition, Scotchbond Universal incorporates a polyalkenoic acid copolymer (PAC) that also chemically interacts with the calcium of hydroxyapatite. Several studies showed that MDP-mediated chemical bonds maintained stable adhesion and prevented bond degradation when using the two-step self-etch adhesive Clearfil SE Bond, which was used as control in our study for SE strategy.[38-40]

Considering the presence of MPD in the universal adhesives evaluated in this study, Scotchbond Universal (on both strategies) and All Bond Universal (on SE strategy) maintained bond strength after storage. Yoshida and others[3] showed that MDP-containing adhesives were able to form a nano-layer at the adhesive interface in different degrees, depending on the adhesive composition. They speculated that compositional differences and possibly different MDP concentrations could explain the distinct behavior of the MDP-containing adhesives. Moreover, they hypothesized that the presence of other components such as PAC or 2-hydroxyethyl methacrylate (HEMA) may compete with MDP on bonding sites to the calcium of hydroxyapatite.[3] These characteristics could explain the behavior of the MDP-containing adhesives tested in our study.

Similar performance was observed by Muñoz and others,[13] which supports the speculation that the presence of PAC is more important for etch-and-rinse adhesives as only All Bond Universal on ER strategy showed bond strength reduction after 6Mos. They indicated that PAC improves stability on a moist substrate, an important factor for etch-and-rinse adhesives. We hypothesized that the decreased performance of All Bond Universal on ER after water storage could be due to the etching strategy. The removal of available calcium by acid etching might have prevented any potential chemical bonding mediated by MDP since All Bond Universal with the SE strategy maintained bond strength values after 6Mos.

The other universal adhesive evaluated (Prime & Bond Elect) is HEMA free and acetone solvated. Zhang and others[16] explained that the absence of degradation observed for this system might be related to the higher vapor pressure of acetone (compared with ethanol) that improves solvent evaporation and may lead to less retention of residual water within the adhesive. A recent study concluded that entrapment of residual water in resin–dentin bonds could compromise the performance of universal adhesives.[9] Furthermore, Prime & Bond Elect does not contain HEMA in composition, making it less hydrophilic. To avoid or reduce hybrid layer hydrolytic degradation, researchers have been developing less hydrophilic adhesives such as HEMA-free adhesives.[5,41] Therefore, we hypothesized that these characteristics may explain Prime & Bond Elect performance on dentin after 6Mos of aging.

The adhesive systems used as controls performed differently on SND. The etch-and-rinse system used as a control (Adper Single Bond 2) showed higher bond strength values than the self-etch system



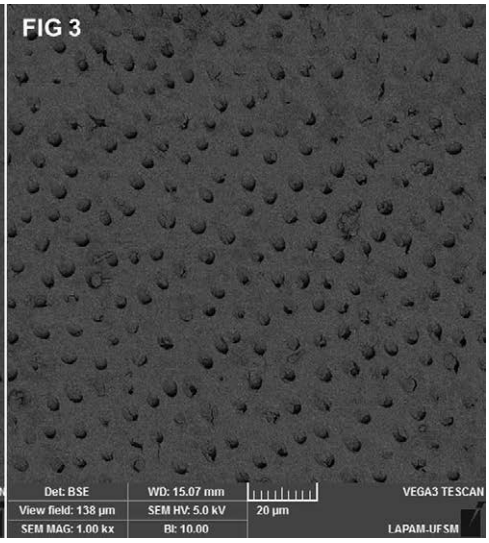


Figure 2. *SEM image of representative specimen from sound dentin.*

Figure 3. *SEM image of representative specimen from artificially induced caries-affected dentin.*

(Clearfil SE Bond); results have been previously reported.[42-44] We speculated that the results obtained with the self-etch system used as a control compared with the ER adhesive might be related to the fact that Clearfil SE Bond is the only tested adhesive that manufacturers do not recommend for active application. Several studies demonstrated that the active application enhanced immediate and long-term bond strength of self-etch adhesives.[45-48] The fact that Clearfil SE Bond has been available on the market for a long time could explain the manufacturer's instructions without using active application.[39]

This study used bovine dentin, which presents similarity compared with human dentin in several characteristics,[49,50] enabling its use for this purpose. A recent systematic review with meta-analysis supported the use of bovine tooth as a substitute to the human tooth in bond strength studies.[51] To confirm the differences for dentin substrates (SND and CAD), representative specimens for sound and artificially induced caries-affected dentin were analyzed by SEM (Figures 2 and 3).

Considering the obtained results, we failed to reject the first null hypothesis because the universal adhesives tested showed similar performance for both strategies on SND and CAD. Moreover, we partially rejected the second null hypothesis as water aging had no effect on SND for both strategies, and degradation over time was observed on CAD.

CONCLUSIONS

Premature bond strength degradation occurred only to artificially induced caries-affected dentin. Etching strategy did not influence the bond longevity of universal adhesives tested to artificial caries-affected dentin.

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Regulatory Statement

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

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.

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