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Clinical Technique/Case Report

A Clinical Presentation of the Direct Gold/Composite Sandwich Restoration

DB Henry

Clinical Relevance

Studies indicate the failure of posterior composite restorations, where bonding to dentin is required, continues to be an issue. The results are leading to the need to replace and/or repair existing restorations earlier than has been the experience using traditional restorative materials.

SUMMARY

The intent of this paper is to present a new idea for increasing the life expectancy of class II composite restorations where the proximal marginal seal is compromised by the necessity to rely on dentin bonding. As implied by the Clinical Relevance statement, studies show that bonding to dentin in areas with high levels of bacterial action, combined with sustained high plaque formation, tends to be the "Achilles heel" with regard to sustained long-term restorations. Therefore, this paper will present a thought experiment, combined with clinical evidence, for combining gold foil with composite in these areas for the class two composite restoration. The results, if proven viable, will be to develop a procedure utilizing the properties of gold foil that make it one of the longest-lasting restorative materials with the recent development of modern cosmetic materials for a truly long-lasting and healthy class II restoration.

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INTRODUCTION

The results of relying upon dentin bonding in proximal and subgingival areas have shown decreased longevity for class two composite restorations as compared to previous restorative materials used in the past 80 years.[1-6] The results suggest the need to replace and/or repair existing restorations earlier than has been the experience when using traditional restorative materials.[5,6] The central problems arise from leakage and subsequent breakdown of the bond at the gingival and subgingival dentinal margins for class 2 and class 5 procedures (personal correspondence between Dr Lloyd Baum and Dr Giancarlo Gallo).[3,4,7] Due primarily to bonding properties associated with the organic and water composition of dentin being at 50% as compared to 12% for enamel, dentin bonding characteristics are not ideal for long-term durability.[1-9]

The clinical use of gold foil to seal dentin margins in class 2 and class 5 gingival prep areas has proven to be one of the most predictable restorative techniques with a long history of success.[5,10-12]

The properties of gold foil indicate it is an ideal material to both create a seal and maintain that seal over time in the gingival and subgingival environments encountered in class 2 and class 5 restorations.[13-16] These properties include: (1) a marginal fit approaching 1 micron, (2) a coefficient of expansion close to tooth structure, (3) no corrosion, and (4) an oligodynamic effect. The first three are important; and when combined with the oligodynamic effect of interfering with cell membrane transport, one has a material with a disinfecting property when placed in areas where pathogenic organisms can lead to margin failure.[5,6,13,14,16-18]

This paper describes a clinical restorative technique to address the limitations of dentin bonding in the class 2 composite dental restoration. It is a procedure that combines the proven long-term application of gold foil with the cosmetic aspects of composite resin by removing the need to bond to dentin in the proximal box, where leakage and breakdown occur at an accelerated rate due to dentin bonding issues. In addition, this is a clinical presentation and will not engage in debate about the use of available direct gold options or the use of different composites/bonding techniques. It is a first look at the possibility for combining two proven materials and techniques utilizing the long-term sealing ability of gold foil and the long-term enamel bonding ability of composite for the benefit of patients.

A CLINICAL DECISION PROCESS

Approximately eight years ago, while completing class 2 foils in my private practice, there was an emergency patient who needed immediate care. The interruption necessitated the need to temporize the two class 2 foils that were approximately at mid-completion (Figure 1). The proximal boxes in both foils had been completed. Both proximal boxes were filled with E-Z Gold (Lloyd Baum Dental Center, Loma Linda, CA, USA) to the level of the occlusal floor in the preparation. E-Z Gold is the author's choice for bulk fill when doing a gold foil. Due to the ease of use and faster build up, E-Z Gold is the gold of choice within a busy private dental practice where gold foil is routinely placed. The E-Z Gold is veneered with #4 gold foil when the restoration is to be completed with direct gold.

In this case, to temporize the restorations, the foil was micro etched with 50-micron aluminum oxide then completed utilizing a total etch (Ultra-Etch, Ultradent, South Jordan, UT, USA) and resin bonding (3M Universal Scotchbond) material to the remaining tooth structure and the micro-etched foil. A posterior composite (3M Silux Plus) was used at that time to complete the restoration. (Figure 2) The patient was to

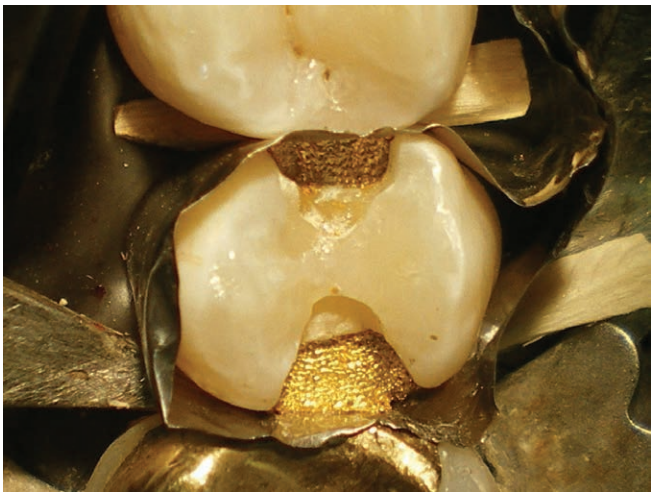

Figure 1. *Two class 2 foils in tooth 4 (2012).*

return for removal of the composite and completion of the foil later. However, the patient did not return for six months, at which time the restoration was evaluated and found to be doing well clinically. The decision was made not to replace the composite. The restoration is now six to eight years in function. No clinical photos exist.

Following the original restoration and upon discussions with Dr Clyde Roggenkamp of Loma Linda, CA, it was discovered that Dr Lloyd Baum had conceived of the idea for combining foil and composite and had discussed this with his friend Dr Giancarlo Gallo of Italy. Thanks to Dr Roggenkamp, the correspondence from 1992 to 1997 between Dr Lloyd Baum of Loma Linda University and Dr Giancarlo Gallo of Alba, Italy, was forwarded for review. Their discussion centered around the concept of utilizing a sandwich technique combining gold foil and composite

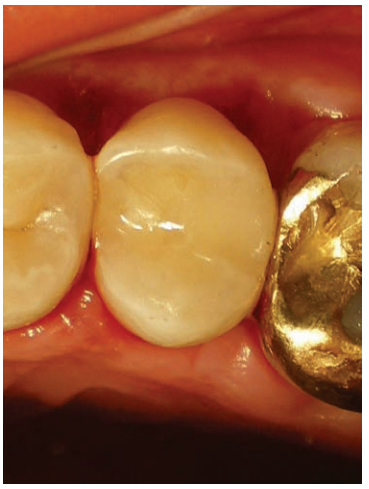

Figure 2. *Final restoration of foil and composite in tooth 4 (2012)*

resin for the class 5 restoration. Their designs can be seen in the hand drawings from their correspondence in Figure 3 (reprinted with permission from Dr Roggenkamp).

Combining what was learned from Dr Baum and Dr Gallo, a refinement of the technique was developed to be applied to a class 2 foil restoration. The refinements included micro etching the gold and remaining prep with 50-micron aluminum oxide, then applying Metaltite (Tokuyama Dental America, Inc, Encinitas, CA, USA) to the gold via manufacturer's directions. Metaltite MTU-6, a thiouracil monomer, which, according to the manufacturer, "enhances a tenacious chemical bond between resins and precious metals" (Figure 4). In addition, it was decided to place E-Z Gold into the proximal box of the class 2 prep to the level of the occlusal floor of the prep to create the contact in gold for the final restoration (Figure 1).

The author's reasoning for the procedure was to enhance the longevity of posterior composite class 2 restorations by utilizing the properties of E-Z Gold for stabilization of the proximal area and the adjacent contact over time. The result is a restoration that combines the use of E-Z Gold in the proximal box to facilitate longevity with the aesthetic appeal of

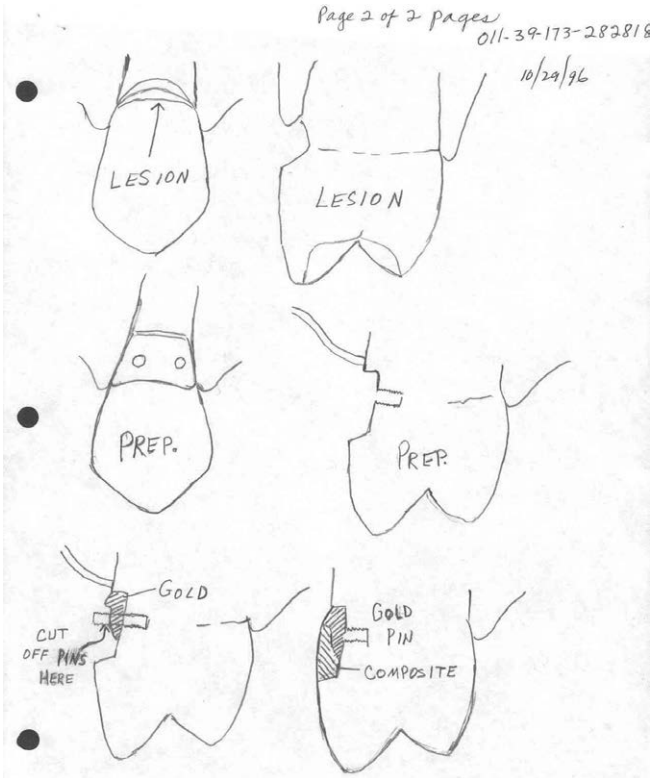

Figure 3. *Original drawings from correspondence between Dr Lloyd Baum of Loma Linda University and Dr Giancarlo Gallo of Alba, Italy.*

Figure 4. *Metaltite by Tokuyama Dental America Inc.*

composite where effective bonding to enamel has been proven. This restorative combination takes into consideration concerns that the patient, the operator, or both might have with the cosmetic appearance of gold in areas where it can be seen during normal function. Also, it is hoped that this paper could begin to re-assert the value of utilizing direct gold to enhance the so-called "bondodontic" explosion within dentistry and prioritize restorative outcomes and longevity in selecting restorative options.

CLINICAL CASES

Three cases are presented; first a class 2 DO/MO in a maxillary second bicuspid (Figures 1-2). The second case is a DO class 2 on tooth 13 (Figures 5-13). The third case is a DO class 2 on tooth 4 (Figures 14-23).

CLINICAL PROCEDURES

Clinical Case 1: Class 2 in Tooth 4, DO and MO Class 2 Foil-Composite Sandwich

As stated previously, the class 2 foil was terminated at the point of completion of the gold placement in the proximal boxes of both class 2 restorations in tooth 4 (Figure 1). The photo shows the completion of E-Z Gold placement in both proximal boxes and the placement of a GI liner proximal to the gold.

Following this, the gold was micro etched with 50-micron aluminum oxide, washed and dried, then a total etch with 35% phosphoric acid (Ultra-Etch, Ultradent) then bonded with 3M Universal Scotchbond (3M Oral Care, St Paul, MN, USA). Finally, 3M Silux Plus (repeat manufacturer's name here) composite was

used to complete the restoration. This procedure was originally intended to be a temporary fix (Figure 2).

Following reevaluation of the restoration, it was decided to continue using the procedure with the modifications of adding Metaltite (Tokuyama Dental America, Inc) to increase bond efficiency between gold and composite resin plus filling the proximal box with E-Z Gold to ensure no dentin bonding and to make the contact in gold. All personal observations are from the author's private practice.

Clinical Case 2: Class 2 DO #13 Foil-Composite Sandwich

The procedure consists of removing all caries, then filling voids created with GI (3M ESPE Fil Quick Aplicap (Need manufacturer's name and location unless same manufacturer has already been noted). This is followed by preparing a classic class 2 preparation for placing gold foil or amalgam under a rubber dam (Figures 5-13).

The initial penetration into tooth 13 was with a 169 bur (Brasseler) to determine the extent of the carious lesion (Figure 5) This was followed by removal of all caries. Healthy tooth structure is not removed; therefore, Black's rules are not observed at this point. Following the insertion of the GI the preparation is completed to Black's specifications.

Figure 6 shows the placement of the remaining glass ionomer for the deep caries destruction and the final prep with E-Z Gold placed in the proximal box. The glass ionomer proximal to the foil acts as a liner and thermal insulator in cases where deep caries penetration into the dentin occurs (Figure 6).

A dead-soft Tofflemire matrix band is placed (HO Band), wedges inserted, and the band is burnished prior to placement of E-Z Gold (Lloyd Baum Dental Center) into the proximal box. In placing class 2 foils, the author normally uses no band (Figure 16, Case 3). However, a brass T band can be used as well, depending on the situation. Following the placement of E-Z Gold to the level of the occlusal floor of the prep (Figures 6-10), the foil and remaining tooth structure were micro-etched (Figure 7), utilizing a chairside micro etcher and 50-micron aluminum oxide.

The preparation and gold were then washed and dried to clear the aluminum oxide prior to etching. In addition, the prep was treated with 2% chlorhexidine (Consepsis, Ultradent) for 60 seconds prior to total etch. A total etch with 35% phosphoric acid for 30 seconds was completed. In this case, Ultra-Etch by Ultradent was used.

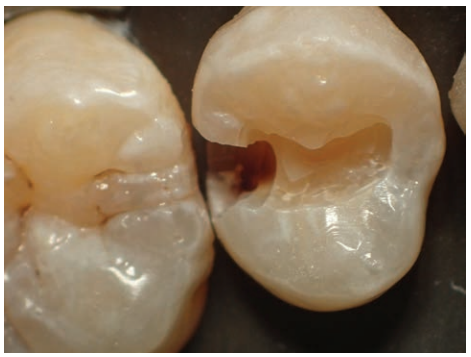

Figure 5. *Initial bur penetration into carious lesion distal-proximal on tooth 13.*

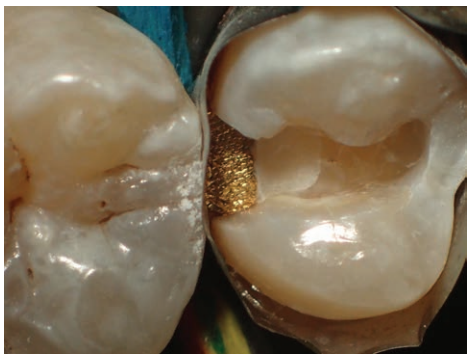

Figure 6. *Distal box filled with EZ Gold, also showing the glass ionomer liner located proximally.*

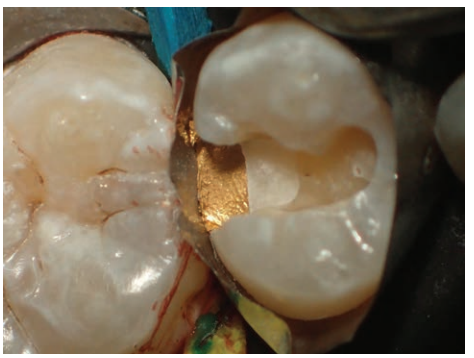

Figure 7. *Foil and prep after micro etching with 50-micron aluminum oxide.*

Figure 8. *Foil treated with two layers of metaltite metal bonding resin.*

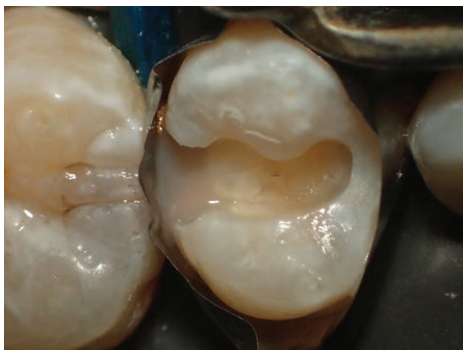

Figure 9. *Initial layer covering gold with shade A2.5 3M Filtek flowable composite.*

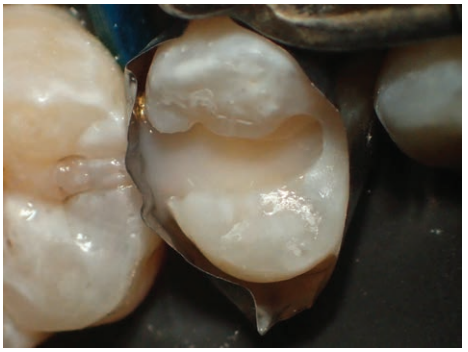

Figure 10. *Middle layer of shade A-2 3M Filtek flowable composite.*

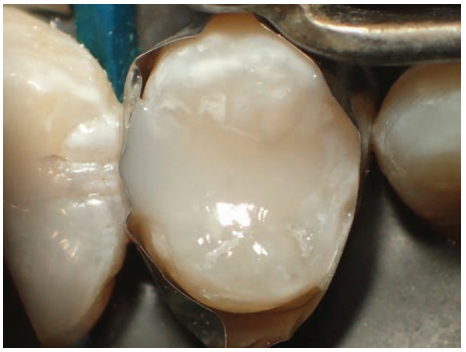

Figure 11. *Final layer of shade A-1 3M Filtek flowable composite.*

These steps were followed by applying Metaltite (Tokuyama) via a micro brush to the gold only (Figure 8). Manufacturer's directions were followed with air drying of one-two layers of Metaltite. Again, the procedure was completed under rubber dam.

Following the treatment of the E-Z Gold with Metaltite, 3M universal bonding was applied to the complete prep and cured. Next, 3M Filtek flowable composite was layered into the final preparation and cured in multiple increments until the restoration was completed (Figures 9-12). A darker or more opaque shade of composite was used in the first layer to mask the color of the gold, in this case, Shade A-2.5 (Figure 9) . Finishing of the composite was completed using increasingly lighter shades layered and cured (Figures 9-11). Polishing points

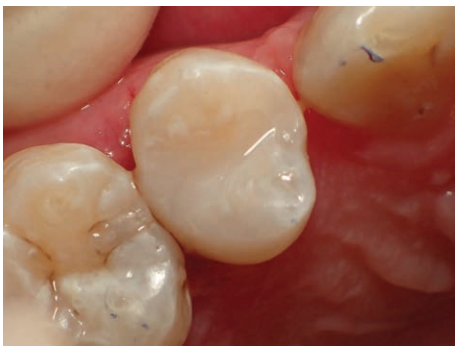

Figure 12. *Completed DO class 2 foil/composite sandwich on tooth 13.*

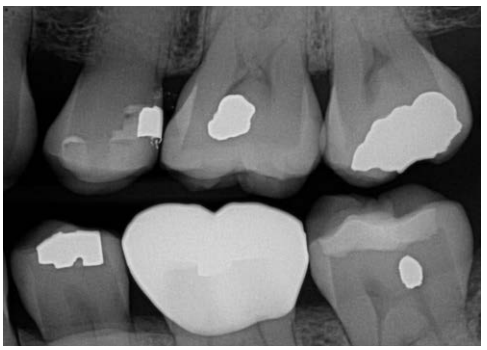

Figure 13. *Radiograph showing the placement of the foil in the proximal box distal on tooth 13.*

and 3M disc and finishing diamonds were used to shape and finish the final restoration (Figure 12). Figure 13 is a radiograph showing the class 2 foil placement in the gingival 1/2 for the completed foil/composite sandwich restoration in tooth 13. The presence of a GI liner can be visually differentiated beneath the composite as well. There appears to be a small radiolucency within the GI. It is not known if this is an artifact or a small void. Because damage to the foil would likely occur with removal and replacement of the GI, it was decided to watch over time.

Case 3: DO Virgin Caries Tooth 4

A penetration cut with a 169 bur (Brasseler) showing proximal caries at DO 4 is shown in Figure 14. This is followed by Figure 15, showing the placement of GI following complete caries removal. Figure 16 shows the final DO prep ready with E-Z Gold in place. In this case E-Z Gold was placed without a matrix band. This is the usual procedure when class 2 foils are completed by the author. This ensures maximal contact and gives better access to the proximal gingival floor for gold placement. After finishing of the gold contact utilizing VisionFlex Diamond Strips (Brasseler) a dead soft matrix band was placed prior to micro etching (Figure 17). This was to prevent etching the adjacent tooth or restoration.

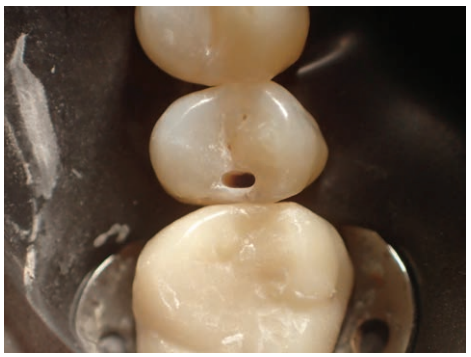

Figure 14. *DO 4 169 bur penetration to see caries.*

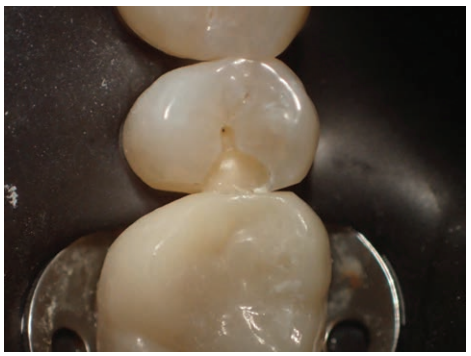

Figure 15. *DO 4 caries removed and GI placed prior to final prep.*

Figure 16. DO 4 With EZ Gold Placed to the Level of the Occlusal Floor of Prep Creating the Contact in Gold. Gold Placed Without Matrix Band to Ensure Easier Access to Gingival Margin and a Tight Contact.

Figures 18 and 19 show the micro etching of the gold and prep followed by treating the gold with Metaltite (Tokuyama). Figures 19 through 22 show the placement of the composite bonding and layers of 3M Filtek flowable composite. A final veneer of compactable composite can be used in heavy occlusion cases.

Figure 23 shows the final radiograph of the DO restoration with the placement of foil, GI, and composite.

CONCLUSIONS

This presentation is intended to be a thought exercise to demonstrate one possible solution to improving the success for class 2 posterior composite resin procedures.

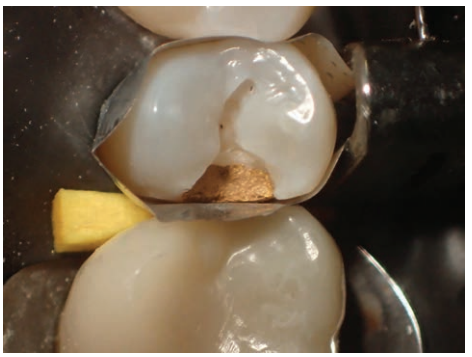

Figure 17. *DO #4 micro etched with 50-micron al oxide and dead soft matrix band used to prevent micro etch 3 and control composite placement. contact is already established in gold.*

Figure 18. *DO 4 treated with two layers of Metaltite (Tokuyama).*

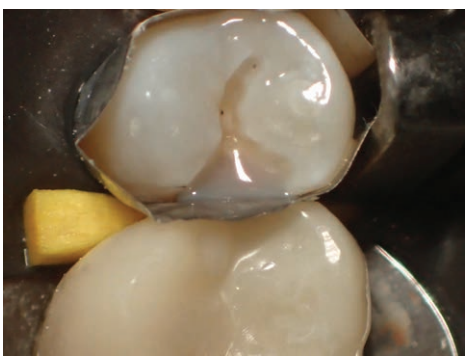

Figure 19. *First layer shade 3.5 to cover the Gold 3M Filtek.*

Figure 20. *Second layer flowable shade 2.0 applied and cured (3M Filtek).*

Figure 21. *Final layer a-1 laid in and cured (3M Filtek).*

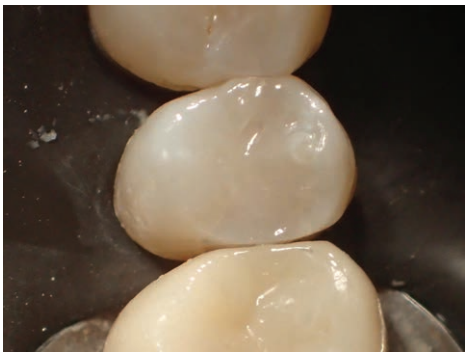

Figure 22. *Completed restoration DO 4 foil-composite sandwich.*

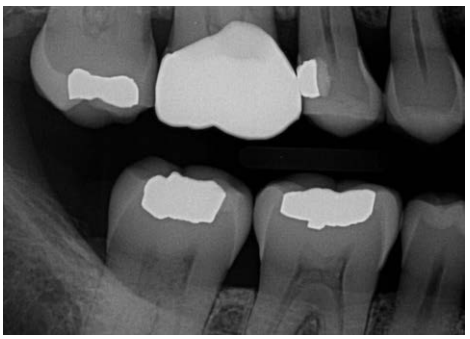

Figure 23. *Radiograph showing completed gold GI composite DO 4.*

More research is needed, especially to observe what is happening at the bond interface between the composite resin and gold. A comprehensive evaluation of the overall success of adding foil to this procedure would also be beneficial. This is a project that should be completed in an academic setting. Obviously, anyone attempting this procedure is expected to be proficient in the placement of gold foil in a clinical setting. A start would be to contact the American Academy of Gold Foil Operators. The author is also personally available for comment.

Acknowledgements

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Conflict of Interest

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

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Three-year Follow-up of Conservative Direct Composite Veneers on Eroded Teeth

RQ Ramos • NF Coelho • GC Lopes

Clinical Relevance

The direct resin composite veneer is a conservative procedure to restore eroded teeth that results in satisfactory outcomes. A polishing protocol appears to be important for both esthetic and periodontal reasons.

SUMMARY

This clinical case describes an esthetic rehabilitation of a young patient presenting with erosive tooth wear. The etiological factors for the erosion in this clinical case was excessive carbonated beverages and lemon water intake. The patient's main complaint was the yellowish aspect of her smile. The treatment procedure selected was direct resin composite veneers in the six maxillary anterior teeth. A three-year follow-up of the case is presented. The three-year follow-up showed a successful clinical performance of the treatment procedure after a finishing/polishing protocol.

INTRODUCTION

Dental erosion is a multifactorial condition defined as loss of dental hard tissue due to exogenous or endogenous acids without bacterial involvement.[1-6] Based on the etiology, erosion can be classified as intrinsic or extrinsic.[6-9] Intrinsic dental erosion results from regurgitation of stomach contents due to gastroesophageal reflux disease or eating disorders, like anorexia nervosa, bulimia nervosa, and rumination.[2,4,8-10] Extrinsic dental erosion is caused by the regular consumption of carbonated beverages, natural citrus fruits, low pH foods and candies, intake of some medications and dietary supplements or occupational factors, such as professional wine tasting, regular swimming in pools with low pH water or workers who are exposed to acidic liquids or vapors.[2,4-7,9]

The initial aspect of dental erosion is softening of the enamel surface.[2,7,10,11] The softened enamel structure is vulnerable to mechanical abrasive forces, such as tooth brushing, the movement of the tongue, and bruxism.[2-4,7,12] This combination of factors leads to tooth wear with dental erosion as the primary etiology,

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which is defined as erosive tooth wear (ETW).[1,3,4,7] Patients are often late in perceiving that they suffer from ETW.[11] The clinical appearance is the most important feature for dental professionals to diagnose ETW, even though the early clinical signs of ETW may be difficult to diagnose.[3,10] The typical signs of ETW that may be evident at an early stage include a silky-glazed or a smooth dull enamel surface, yellowing of the teeth (due to enamel loss), increased incisal translucency, and cupping and grooving on the occlusal surfaces.[2-4,8,10,13] In the more advanced stages, the convex areas on smooth surfaces flatten, concavities may become present in intact enamel along the gingival margin, restorations may stand above the level of the adjacent tooth surface, and a rounding of the cusps or even hollowed out surfaces can develop on the occlusal surface of the posterior teeth.[2,9,3,5,13]

It is important to detect ETW as early as possible to prevent further progression.[5] After identifying all the possible etiological factors, a preventive program and a treatment plan based on dental tissue wear severity should be suggested to the patient.[2,9,11,14] If a restorative treatment is necessary it should be as minimally invasive as possible, ie, additive adhesively bonded resin composite restorations.[2,8,11] This case report demonstrates a conservative approach for restoring esthetics and function with direct resin composite veneers in the six maxillary anterior teeth in a young patient with initial ETW with three-years of follow-up. Also, a finishing/polishing step-by-step protocol is presented to establish high-gloss resin composite surfaces at baseline and also at follow-up appointments.

CASE REPORT

A 32-year-old woman presented at a clinical appointment complaining about dentin hypersensitivity and the yellowish aspect of her smile. An extensive patient history revealed that the patient consumed excessive acidic beverages, including an isotonic sport drink (Gatorade) and energy drink (Red Bull). Gatorade has a pH value of 2.7,[15] and Red Bull has pH value of 3.08.[16] Also, the patient routinely drank lemon water early in the morning followed by tooth brushing. The initial clinical examination revealed that the patient presented ETW involving mainly the cervical third (on the facial surface) and incisal third (on the palatal surface) of the six maxillary anterior teeth due to extrinsic acid intake and bruxism. A more severe erosive wear was observed on both maxillary central incisors. Enamel loss, probably due to resin remnant removal after orthodontic bracket debonding and pre-existing resin composite restorations, were also seen on the facial surfaces of the six maxillary anterior teeth (Figure 1). All anterior teeth responded positively to a pulp sensibility test (cold test).

The patient received professional education regarding the importance of her habits in relation to her tooth wear condition. Also, the patient was educated about the etiological factors to treat and control the sequelae of ETW. The proposed treatment plan to the patient was direct resin composite veneers in the six maxillary anterior teeth to protect enamel and dentin from further ETW, to prevent dentin hypersensitivity, and to restore dental esthetics. After dental prophylaxis using a rubber cup and a prophylaxis paste (Odahcam, Dentsply Sirona, York, PA, USA), a mock-up procedure (Figure 2) was performed using a micro-hybrid resin composite (Essentia, GC Corp, Tokyo, Japan). The similar resin composite flexural strength and elastic moduli after 24 hours in distilled water or 30 days in a soft drink (Coke, Coca-Cola Company, Atlanta, Georgia, USA),[17] and the high polishing surface with low surface porosities analyzed under scanning electron microscope helped in the restorative material selection.[18] The mock-up restorative procedure allowed the correct resin composite shade selection. Two composite shade were

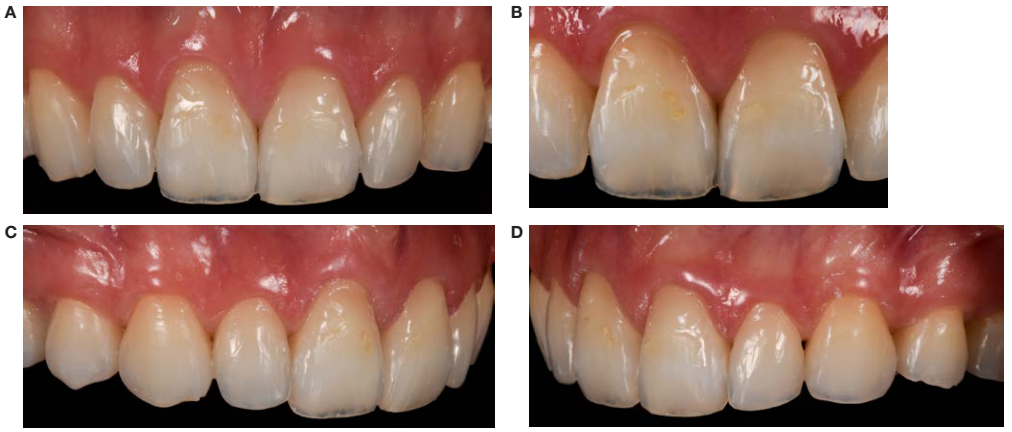

Figure 1. *Initial intraoral aspect of the maxillary anterior teeth: (A) Tooth erosion and loss of dental structure due to resin clean up after orthodontic bracket debonding; (B) Pre-existing resin composite restorations and tooth erosion exposing cervical dentin; (C) Right side view; (D) Left side view.*

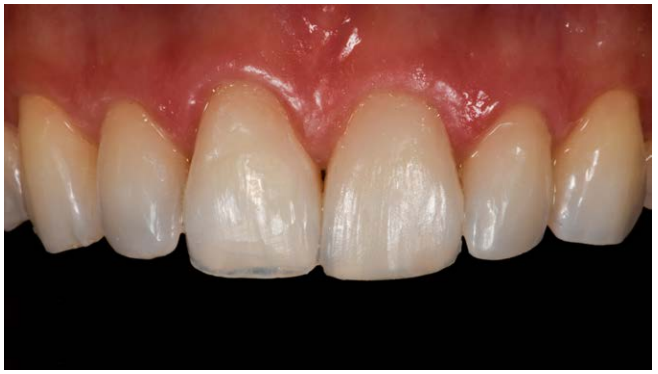

Figure 2. *Resin composite mock-up to choose resin composite shades.*

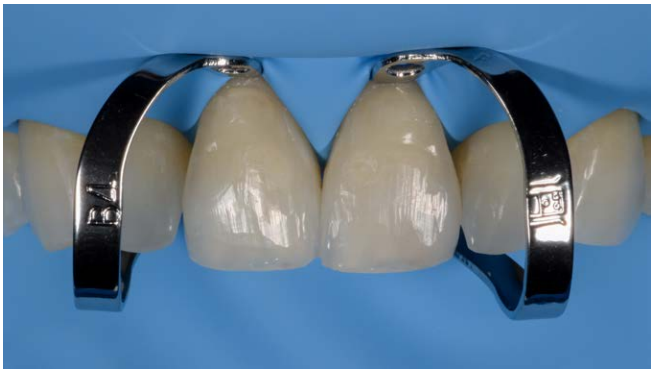

Figure 3. *Rubber dam isolation using dental retainers (Hygenic Brinker B4, Coltène/Whaledent Inc). Note that the existing resin composite restorations were carefully removed. No tooth structure was removed to perform the restorative treatment.*

selected: medium dentin (MD), as dentin substitute; and light enamel (LE), as enamel substitute.

All restorative procedures were done under rubber dam isolation (Figure 3). Dental retainers (Hygenic Brinker B4, Coltène/Whaledent Inc, Cuyahoga Falls, OH, USA) were used to reveal the cavities cervical margins. The restorative procedures were performed two teeth at a time: first the two maxillary central incisors, followed by the two maxillary lateral incisors, then the two maxillary canines. Prior to the phosphoric acid etching step, a 7.0-mm metal matrix band was positioned in the proximal surfaces to avoid acid-etching the surfaces of other teeth. Then, 35% phosphoric acid (Ultra-etch, Ultradent, South Jordan, UT, USA) was applied for 15 seconds on dentin and 30 seconds on enamel and rinsed off using copious amounts of water from dental syringe. Tooth moisture contamination was controlled using an endodontic aspirator. A two-step etch-and-rinse adhesive system (Single Bond Plus, 3M Oral Care, St Paul, MN, USA) was applied on both enamel and visibly moist dentin using a disposable brush, air-dried and light-activated for 10 seconds using a LED light-curing unit (VALO Cordless, Ultradent) with output of approximately 1,000 mW/cm^2. On the two maxillary central incisors and on the two maxillary canines, a first increment of resin composite (shade MD, Essentia, GC Corp) was applied on the cervical third and light-activated for 20 seconds. Afterwards, a final increment of resin composite (shade LE, Essentia, GC Corp) was applied over the entire buccal surface of the teeth and light-activated for 20 seconds. On the two maxillary lateral incisors, a single increment of resin composite (shade LE, Essentia, GC Corp) was applied on the entire facial surface of the teeth and light-activated for 20 seconds. A dental composite brush (#3, Cosmedent Inc, Chicago, IL, USA) was used in the last increment of all restored teeth in order to adapt, shape, and get a smooth surface of the resin composite. The use of a dental composite brush from the cervical third towards the incisal third of the teeth helped to sculpt the composite similar to natural tooth volume, removing the excess of resin composite. This is an important step, to obviate necessity of using rotatory finishing instruments at the restorative procedure appointment. Delaying finishing/polishing with rotary instruments for 24 hours improves the marginal seal with less microleakage compared to immediate finishing.[19] A final light-activation was performed for 60 seconds on the facial surface of each restored teeth. Finally, occlusal adjustment was performed, verifying proper contacts in protrusive and lateral excursive movements of the mandible.

Finishing and polishing of the composite restorations were performed under constant water cooling after 24 hours of the restorative procedure as follows: 1) a fine LTA Lamineer tip (Dentatus, Spånga, Sweden) mounted on a Profin contra-angle (W&H, Bürmoos, Austria) was used to remove resin composite overhangs close to the gingival margins (Figure 4A); 2) 3/8-inch medium abrasive disks were used to reduce surface roughness and to shape marginal recontours (2381M Sof-lex, 3M Oral Care) (Figure 4B); 3) finishing strips (Epitex, GC Corp) were used for interproximal finishing in decreasing abrasive grade (from coarse to extra fine) (Figure 4C); 4) a finishing silicone rubber point (Astropol F, Ivoclar Vivadent, Schaan, Liechtenstein) was used to refine surface contours and to remove marginal composite excess (Figure 4D); 5) a polishing silicone rubber point (Astropol P, Ivoclar Vivadent) (Figure 4E) followed by a rubber finishing cup (Blue FlexiCups, Cosmedent) were used to smooth the resin composite surfaces (Figure 4F); 6) a super fine rubber polishing cup (Pink FlexiCups, Cosmedent) (Figure 4G) followed by a spiral shaped diamond polisher (DT-DCP14f, Diacomp Plus Twist, EVE, Keltern, Germany) were used to establish a high gloss composite surface

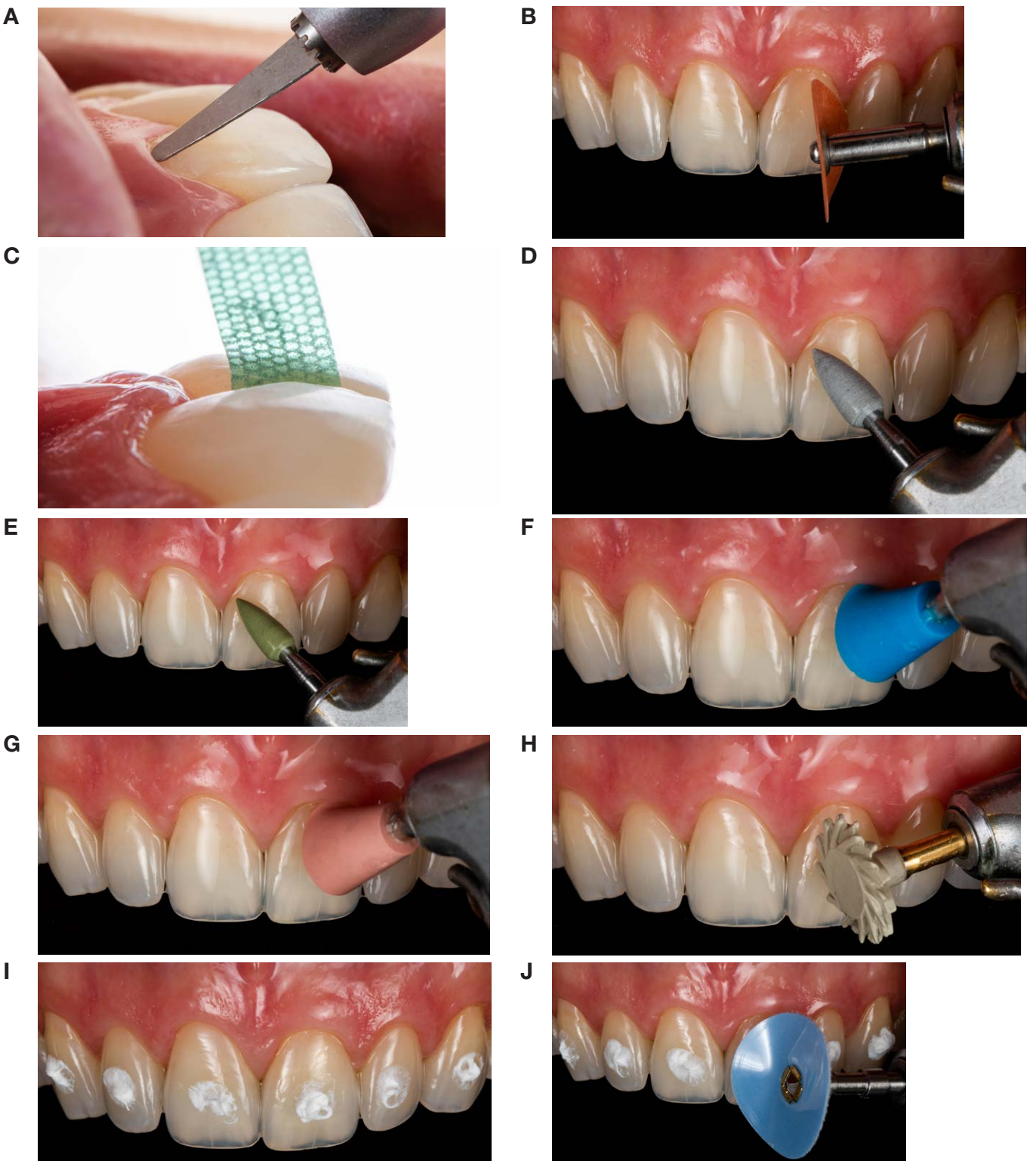

Figure 4. *Finishing and polishing procedures: (A) Overhangs removal at gingival margin; (B) Shaping and contouring the restorations; (C) Finishing strip for interproximal area; (D) Contouring and refining the margins of the restorations; (E) Smoothing the surfaces of the restorations, step 1; (F) Smoothing the surfaces of the restorations, step 2; Note that this rubber finishing cup (Blue FlexiCups) can easily polish gingival margins due to its flexibility; (G) Polishing the margins of the restorations; (H) Polishing the facial surface of the restorations; (I) Polishing paste on facial surfaces of the restorations; (J) Final polishing step of the restorations with polishing paste and felt disk.*

(Figure 4H); 7) an aluminum oxide polishing paste (Enamelize, Cosmedent) was applied (Figure 4I) before using a felt disk (FlexiBuff, Cosmedent) (Figure 4J) for final polishing purposes to increase the gloss, luster and surface smoothness of the composite restorations (Figure 4I). The final aspect of the resin composite veneers shows a high-gloss surface (Figure 5).

When the patient was recalled each year, polishing was performed as mentioned in steps 6 and 7 to provide maintenance of the composite restorations (Figure 6 A-C). At the three-year appointment, a satisfactory appearance of the patient's smile was still seen, showing the stable performance of the composite restorations (esthetic success) and absence of additional ETW (biological success) (Figure 7A). However, some dental plaque and chipping at gingival margins, mild inflammation of the gums, and a dull surface in all resin composite veneers could be seen (Figure 7A). Also, some wear at the lingual surface at the incisal third of the teeth 6, 8, 9, and 12 was observed. At this appointment, resin composite restorations were placed on the lingual surfaces at the incisal third of teeth 6, 8, 9, and 12 using the already mentioned etch-and-rinse adhesive system (Single Bond Plus, 3M Oral Care) and resin composite (Essentia, shade LE, GC Corp). Then, the finishing and polishing procedure of the resin composite veneers (all steps except step number 2) and the lingual composite restorations (all steps) were performed. In this way it was possible to refine the margins and to reestablish the polishing luster of the restorations. Figure 7B shows the final aspect one week after the polishing procedure from the three-year follow-up.

DISCUSSION

Ideally ETW etiological factors should be controlled prior to restorative procedures, since tissue loss stops progressing only when the cause is eliminated.[11,13] For this reason, from the moment that ETW has been detected, the patient should be informed about his or her condition, encouraged to follow a preventive program, and receive a treatment plan based on dental tissue wear severity.[2,9,11,14] The importance of the preventive program should be highlighted. The patient must be aware that ETW progression will continue to occur if the beginning of the preventive program is postponed.[5]

As with all dental operative procedures, longevity of a treatment involving teeth with ETW can only have a positive prognosis with accompanying preventive measures, so regular follow-ups are mandatory.[2,3,11] In the present case report, the annual follow-ups had two main objectives: monitoring the management of ETW and evaluating the resin composite veneers. The monitoring of the management of ETW at each follow-up was done by investigating the patient's dietary habits, history of dentin hypersensitivity, and examining unrestored tooth surfaces for absence of progressive or

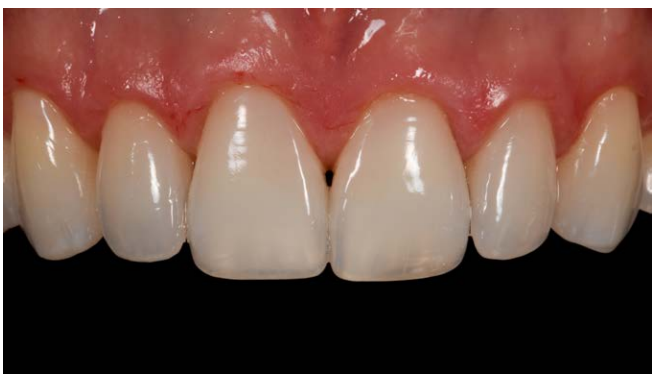

Figure 5. *Final aspect of the direct resin composite veneers immediately after finishing and polishing.*

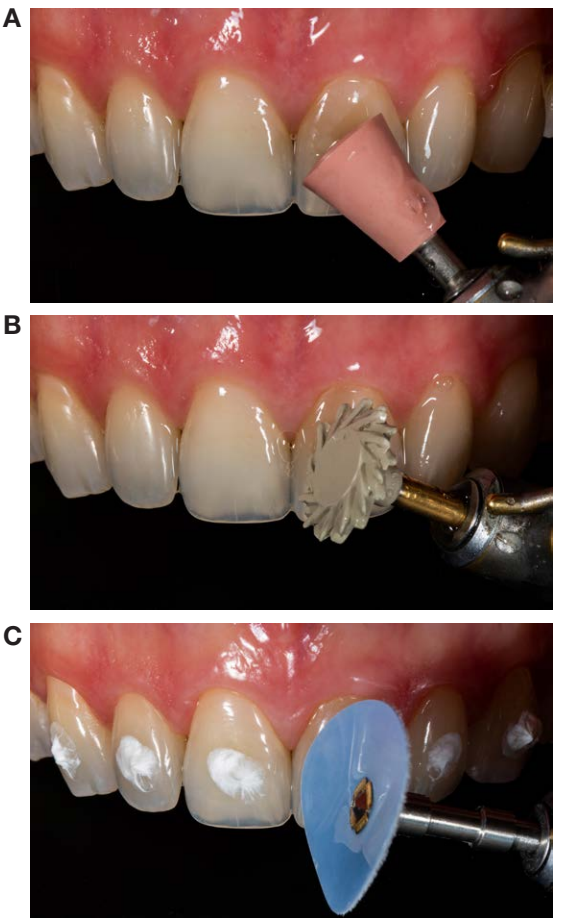

Figure 6. *Annual polishing maintenance sequence: (A) Polishing of margins and facial surfaces of the restorations (Pink FlexiCups); (B) Polishing of buccal surface of the restorations (DT-DCP14f, Diacomp Plus Twist); (C) Polishing paste and felt disk.*

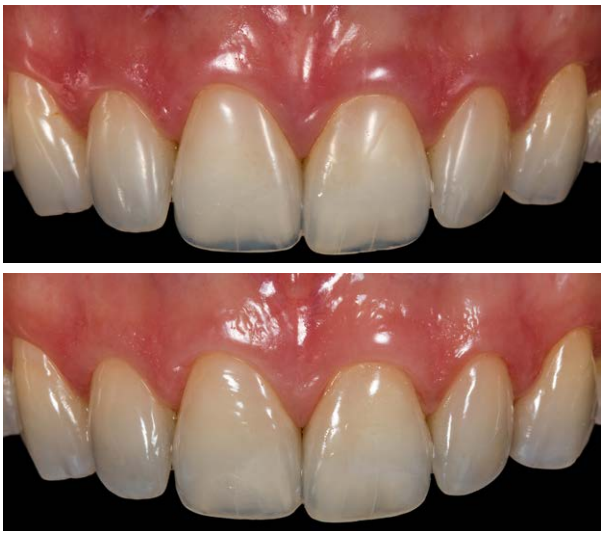

Figure 7. *Three-year follow-up: (A) Initial aspect. Presence of dental plaque, mild inflammation of the gums, and absence of surface luster of the restorations; (B) One week after resin composite repair at lingual surface at incisal third (teeth 6, 8, 9, and 12) and finishing and polishing sequence. Note the absence of dental plaque, the healthy periodontal aspect, and that the surface luster of the restorations was restored.*

early signs of ETW.[2,3,7,8,10] The resin composite veneers were examined following six main criteria: marginal integrity (marginal adaptation), marginal staining, surface staining, surface gloss/luster and roughness, incisal wear, and maintenance of periodontal health.[20]

A highly polished resin composite surface is important for both esthetic and periodontal reasons, since it helps to maintain the surface luster and color, enhancing the longevity of the resin composite restoration procedure, and reduces plaque accumulation, avoiding periodontal inflammation.[21-24] At the three-year follow-up clinical examination, the consequences of a rough resin composite surface could be seen as a visible biofilm and mild inflammation of the gums detected along the gingival margins (Figure 7A). One week after finishing and polishing steps, good periodontal health and surface luster of the composite restorations were seen (Figure 7B), showing the importance of maintenance polishing of resin composite restorations.

Selecting the least invasive restorative treatment plan should always be considered to manage ETW.[2,3,11] The objectives of the restorative treatment are: 1) to diminish or stop ETW progression, 2) to reduce or stop dentin hypersensitivity, 3) to restore esthetics, and 4) to restore dental function.[2,3,9] Direct resin composite restorations or sealing of eroded posterior teeth generally are indicated in cases of slight or moderate tooth wear.[9,11,13,25] In cases of extensive tooth wear, an indirect approach or a combination of indirect and direct restorative procedures may be necessary.[8,25] The possibility of using direct restorative materials should always be considered, since they allow a minimally invasive treatment that replaces only the lost dental tissues without the use of diamond burs for tooth preparation.

CONCLUSION

Direct resin composite restoration in anterior eroded teeth affords practical, feasible, and conservative dental treatment. Besides monitoring the ETW management, the key to success is the polishing aspects: select resin composite material with high polishing properties, perform finishing and polishing steps properly, and establish a strict polishing protocol over each follow-up session, if needed.

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

Longevity of Direct Resin Composite Restorations in Maxillary Anterior Crown Fractures: A 4-year Clinical Evaluation

B Korkut • M Özcan

Clinical Relevance

A monochromatic composite layering technique can meet the esthetic and functional expectations over 4-years, even when using microhybrid resins.

SUMMARY

Objectives: To investigate the longevity of direct composites for Class IV restorations and the possible reasons of failure.

Methods and Materials: The longevity of 168 Class IV restorations in 50 adult patients was evaluated, in terms of modified United States Public Health Service criteria, for 4 years. Restorations were performed using a monochromatic layered microhybrid, resin-based composite (RBC) (Essentia, Universal Shade, GC Corporation, Japan; n=76) and polychromatic layered micro/nanohybrid (MD and LE shades, Essentia, GC Corporation, Japan; n=92) RBCs, by a single operator.

Results: The majority of the teeth (n=156) remained acceptable at the end of 4 years, and the overall survival (OS) rate was considered as 92.86%. Survival rates for the monochromatic layering technique (MLT) and polychromatic layering technique (PLT) were 90.8% and 94.6%, respectively. Mean survival was 46 months for MLT and 47 months for PLT, indicating no significant difference (p=0.343). Fracture of the restoration was the most common reason for failure (4.2% out of 7.1% of general failures) for both the layering techniques.

Conclusions: Under the conditions of this mid-term clinical study, MLT and PLT as well as microhybrid and nanohybrid resin composite materials, showed similar clinical durability. In terms of simplicity, monochromatic layering can be preferred for Class IV restorations, when the right indication criteria are met.

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INTRODUCTION

Clinical conditions such as caries lesions, discoloration, diastemas, crown fractures, and misaligned teeth may cause an undesirable esthetic appearance and smile.[1] Two main esthetic treatment options are available to solve these problems: indirect ceramic and direct resin-based composite (RBC) restorations.[2] With recent developments in adhesive dentistry materials and techniques, direct RBC restoration is now considered a good minimally, or even noninvasive, option.[3,4] Compared to indirect ceramic restorations, direct RBCs have the advantages of single-visit treatment, less preparation time, durability, and repairability.[4] However, periodic checks are mandatory to ensure durability of resin composites, and more of these checks are needed as compared to ceramics. In cases of fracture or chipping of the composite, a simple repair protocol is used to extend the life of the original restoration.[5] Direct restorations reportedly have successful short-term clinical results.[6] Appropriate indications, effective isolation, good optical and mechanical properties of the resin composite, operator experience and skill, accurate shade selection, successful finishing and polishing of the direct restorations, and frequent checkups are needed for long-lasting functional and esthetic outcomes.[4,7] In the literature, there is a lack of long-term evidence of the clinical efficacy of direct RBC restorations placed in the anterior teeth. The most common reason for failure of direct composite build-ups is fracture of the RBC.[6,8] According to previous studies, the 3- to 5-year anterior restoration survival rate varies between 79% and 89%.[5,6,9] Limited longevity has been reported for composite laminate veneers due to their susceptibility to staining, wear, and fracture.[10] However, the potential influence of chemical and physical properties of the resin composite, the size of restoration, and patient- and dentist factors still remain to be determined, especially in long-term clinical trials. A need for information regarding the potential factors influencing long-term failure clearly remains. Kubo and others[11] investigated the main factors associated with the longevity of Class III-V composite restorations, including cavity type, gender, age, dentist factors, and the requirement for retreatment. Dentist factors, cavity type, and retreatment significantly influenced the survival rate.

Two types of veneers comprised of different microhybrid resin composite materials were compared by Gresnigt and others[12], and no significant difference in 3- or 5-year survival was found. A meta-analysis of prospective studies on anterior composite restorations reported median survival rates of 95% and 90% for Class II and IV restorations, respectively, after 10 years.[13] Since esthetics is one of the main concerns regarding anterior teeth, some researchers have recommended using resin composites with a smaller filler size (nanofilled composites) to produce a smoother surface.[14] However, a systematic review comparing nanofilled and submicron composites to microhybrid composites reported no improvement in surface smoothness with use of nanofilled composites.[15]

The aim of this clinical study was to determine the mid-term survival rate of Class IV composite restorations of maxillary anterior teeth and to investigate the possible reasons for failure.

METHODS AND MATERIALS

Study Design and Participants

Patients who had received Class IV restoration(s) of maxillary anterior teeth were selected for this 4-year clinical follow-up study. Fifty patients (22 males and 28 females; total of 168 Class IV restorations) aged between 18 and 56 years (mean age, 31.1 years) were included. A flow diagram of the restorations is shown in Figure 1. All patients provided written informed consent before the restorative procedures. Class IV restorations of maxillary teeth, which were conducted at least 4 years ago, were included. Baseline (1 week) and 1-, 2-, 3-, and 4-year follow-up data were evaluated by two experienced restorative dentistry specialists. The distribution of the restorations according to the layering technique, composite filler type, and tooth number is provided in Table 1.

Inclusion and Exclusion Criteria

The medical and clinical history of the patients was taken, and they attended 1-week and 1-, 2-, 3-, and 4-year follow-up appointments. The 168 Class IV restorations of maxillary anterior teeth were done between June 2014 and July 2015. Restorations that were extracted, replaced, repaired, or repolished during this period were not excluded from the study, but were considered failures. Teeth that underwent root canal treatment (RCT) at baseline were excluded from the study; however, endodontically treated teeth were included. The necessity for RCT after treatment was determined based on the assessment results. All patients had full anterior dentition and normal occlusion without generalized periodontal disease, as verified by clinical and radiographic records. The reasons for the Class IV restorations were all uncomplicated crown fractures. Before the restorations, minimally invasive removal of any residues of former restorations and related secondary caries was conducted. Presence of bruxism was also diagnosed based on medical and clinical

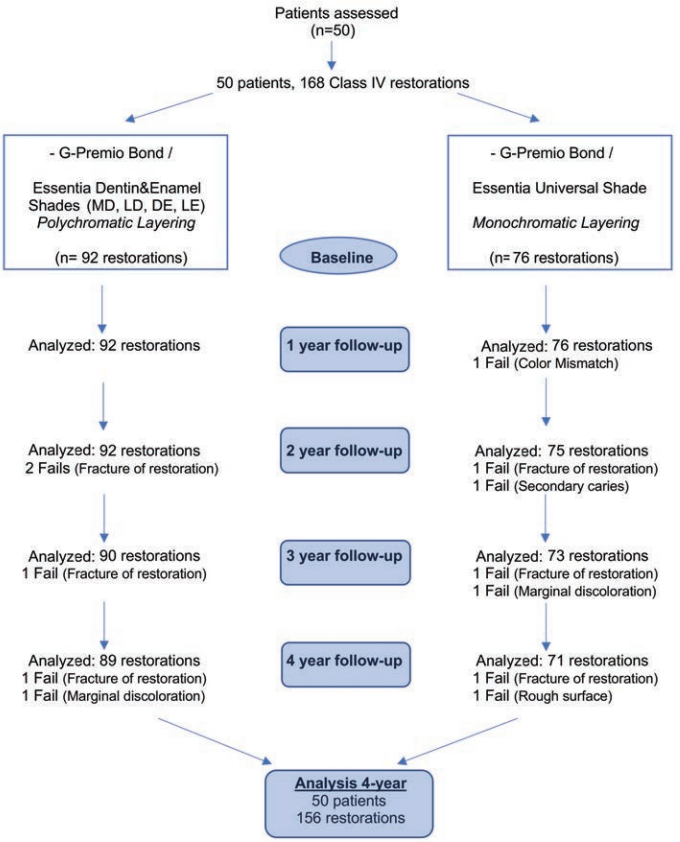

Figure 1. *Flow diagram of history of restorations.*

history. Thirty-seven restorations of eleven patients were not included for the analysis, due to the presence of bruxism.

Restorative Procedures

All restorations were performed using the same procedures by a restorative dentistry specialist in an academic university clinic. A silicone key constructed via either diagnostic wax-up or direct mock-up was used for all restorations. The principle of minimally invasive dentistry guided the preparations. Before tooth preparation, shade matching was performed with the button technique using a digital camera (D750 ; Nikon, Tokyo, Japan) with a 105-mm macro lens (Nikon), the R1C1 Wireless Close-up Speedlight System (Nikon), a flash mounting bracket (Owlbrckt C, Torun & Torun, Ankara, Turkey), and the polar_eyes cross-polarization filter (PhotoMed International, Van Nuys, CA, USA). The most appropriate enamel and dentin shades of the selected composite resin were placed on the labial surfaces of the adjacent teeth and polymerized,

Table 1: *Distribution of Class IV Restorations According to Layering Type, Composite Filler Type, and Tooth Number*

Layering Technique	Composite Filler Type	n	Tooth Number					
			6	7	8	9	10	11
Monochromatic Essentia (Universal shade)	Microhybrid	76	18	9	12	10	11	16
Polychromatic Essentia (MD and LE shades)	Micro-/ nanohybrid	92	5	17	21	24	19	6
Total		168	23	26	33	34	30	22

following which photographs were taken and evaluated by the operator. The selected shades and initial dental photographs were recorded for each patient. Following shade selection, teeth #5-12 were isolated with a rubber dam (Nic Tone, Bucharest, Romania). The teeth to be restored were retracted with either dental floss or rubber dam clamps (Hygenic Brinker Clamps; Coltene, Altstatten, Switzerland). Minimally invasive removal of the former restorations was performed in all cases. Then, 45° beveling of the buccal surface of the fracture lines (including all enamel and up to half of the exposed dentin) was performed using a red diamond needle-shaped bur (Dentsply Maillefer, Ballaigues, Switzerland) under constant water cooling. Following this, 35% phosphoric acid gel (Ultra-Etch, Ultradent Products, Inc, South Jordan, UT, USA) was applied to all of the prepared enamel surfaces for 30 seconds, water-rinsed for 10 seconds, and gently spray-dried. Then a one-step adhesive system (G-Premio Bond, GC Corporation, Tokyo, Japan) was applied to all of the etched enamel and nonetched dentin surfaces, as per the manufacturer's instructions. The adhesive was left undisturbed for 10 seconds after rubbing and air-dried under maximum air pressure for 5 seconds. Polymerization was performed using a light-curing unit (wave length: 430-480 nm; Elipar DeepCure-S LED, 3M Oral Care, Maplewood, MN, USA), applied for 20 seconds at a light intensity of 1370 mW/cm^2 with an irradiated diameter of 10 mm.

Regarding the restorative materials and technique, a microhybrid RBC (Universal Shade Essentia, GC Corporation) with a chameleon effect was used for monochromatic Class IV restorations (n=76). A combination of microhybrid and nanohybrid RBCs (Medium Dentin [MD] and Light Enamel [LE] Essentia shades, GC Corporation) were used for polychromatic Class IV restorations (n=92). The brand, type, manufacturer, and chemical compositions of the materials are listed in Table 2. Mono- or polychromatic layering was performed according to the necessity for incisal translucency of the incisal edge. If the adjacent or symmetrical tooth had these features, the polychromatic layering technique (PLT) was considered. All restorations were gradually built-up under silicone index guidance. The incremental layering technique (≤2-mm thickness) was used for monochromatic layering of the microhybrid resin and for polychromatic layering of the micro/nanohybrid resin. A nanohybrid translucent shade (LE) was used to mimic the natural enamel tissue, whereas microhybrid, opaque, and chromatic shades (MD) were used to mimic the natural dentin tissue. Marginal walls of the restorations were completed using self-contoured, kidney-shaped posterior metal matrix bands (No. 1298, Tor VM, Moscow, Russia). All shades were polymerized for 20 seconds at an irradiation of 1370 mW/cm^2. The light intensity of the curing unit was evaluated before each restoration using a radiometer (Hilux Curing Light Meter, Benlioglu Dental, Ankara, Turkey). The final labial surface layers of the restorations were polymerized under a glycerin gel cover (Air Barrier, GC Corporation) to eliminate the oxygen inhibition layer.

The final occlusion was adjusted by protrusive and lateral movements of the mandible. Interproximal surfaces were polished with interdental polishing strips (Epitex strips; GC Corporation) with three different grits (medium #500, fine #800, and extrafine #1200). Labial and incisal embrasures were adjusted using aluminum oxide-embedded abrasive polishing discs (Sof-Lex, 3M Oral Care) with three different grains (medium [40 μm], fine [24 μm], and superfine [8 μm]) under dry conditions at 15,000 rpm, as per the manufacturer's instructions. Finishing of the restorations was performed using a 12-blade bur (Diatech, Dental AC, Heerbrugg, Switzerland) at 30,000 rpm under water cooling. Diamond particle-embedded medium- and fine-grit rubber wheels (Twist Dia; Kuraray Noritake Dental, Tokyo, Japan) were operated at 10,000 rpm without water cooling to polish the labial surfaces. Additional polishing was performed using a medium-grit diamond bur (Diatech), operated horizontally at 5000 rpm. All patients were scheduled for repolishing 24 hours later. Only the high-shine (fine-grit) polisher was used for final surface polishing. Patient's medical/dental histories, as well as dental photographs, and radiography records, if necessary, were collected at the 1-week and 1-, 2-, 3-, and 4-year follow-up appointments. No repair or repolishing procedure was performed at any of follow-up visits.

Evaluations and Statistical Analyses

Medical history, radiographic and clinical data, were collected for each patient by the operator. Variables such as age and gender were recorded. Patients were also questioned regarding postoperative sensitivity. Radiographs were only taken when indicated by clinical examination, and when it was a necessity to complete the examination, to minimize radiation exposure. The necessity was judged by the operator during the annual follow-up visits. Intraoral frontal bite, frontal view with contrast enhancement (Owlcntrst, Torun & Torun), frontal close-up view, and occlusal photographs were taken using the equipment described in Section 2.3. The arms of the mounting bracket were set at a 45° angle for all photographs. All photographs were taken under the same conditions (1/250 shutter speed, f:28

Table 2: *Brands, Types, Manufacturers, and Chemical Compositions of the Materials*

Brand	Type	Manufacturer	Chemical Composition
Essentia Universal Shade	Microhybrid composite	GC Corp, Japan	Matrix: UDMA, Bis-MEPP, Bis-EMA, Bis-GMA, TEGDMA Filler: prepolymerised fillers (17 μm): strontium glass (400nm), lanthanide fluoride (100nm), fumed silica (16 nm) FAISi glass (850 nm) [81 wt%]
Essentia Medium Dentin (MD) Shade	Microhybrid composite	GC Corp, Japan	Matrix: UDMA, Bis-MEPP, Bis-EMA, Bis-GMA, TEGDMA Filler: prepolymerised fillers (10 μm):barium glass (300nm), fumed silica (16 nm), silica glass (850 nm) [76 wt%]
Essentia Light Enamel (LE) Shade	Nanohybrid composite	GC Corp, Japan	Matrix: UDMA, Bis-MEPP, Bis-EMA, Bis-GMA, TEGDMA Filler: prepolymerised fillers (10 nm): barium glass (300 nm), fumed silica (16 nm) [81 wt%]
G-Premio Bond	Self-etch / Universal adhesive agent	GC Corp, Japan	4-MET, MDP, MDTP, dimethacrylate monomers, water, acetone, silicone dioxide, photoinitiators
Ultra-Etch	Etching gel	Ultradent Products, US	35% phosphoric acid
Twist Dia Prepolisher	Polishing material / rubber spirals	Kuraray Noritake, Japan	Diamond grains embedded synthetic rubber spirals. Medium grit (325-400 mesh)
Twist Dia High-shine Polisher	Polishing material / rubber spirals	Kuraray Noritake, Japan	Diamond grains embeded synthetic rubber spirals. Fine grit (4-8 μm)
Sof-Lex Discs	Four step polishing discs	3M Oral Care, Japan	Aluminium oxide paticles embedded round polishing discs in different girts. (Coarse: 55 μm; Medium: 40 μm; Fine: 24 μm; Ultrafine: 8 μm)
Epitex Polishing Strips	Four step interdental polishing strips	GC Corp, Japan	Diamond particles embeded interdental polishing strips in 4 different grains. (Coarse #300; Medium #500; Fine #800; Extra fine #1200)
Air Barrier	Oxygen inhibition layer inhibitator	GC Corp, Japan	Glycerine gel in high viscosity

Abbreviations: MDP, methacryloyloxydecyl dihydrogen phosphate; 4-MET, methacryloyloxyethyl trimellitic acid; MDTP, thiophosphate ester monomer; Bis-GMA, bisphenol A diglycidl ether dimethacrylate; UDMA, diurethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

diaphragm, ISO 200); the distance to the patient was also kept constant. The white balance was adjusted for each patient using gray paper. The surfaces of the teeth and restorations were spray-dried before the photographs were taken. The photographs were taken as quickly as possible to avoid de-hydration for precise shade matching. Frontal photographs were also taken to aid in shade matching and identification of any discoloration. The photographs were saved as JPEG and RAW files.

The 168 Class IV restorations were evaluated between August and November 2018 by two experienced and fully blinded examiners using a dental mirror and explorer. Before evaluating the data, the examiners were provided with a set of reference photographs illustrating the scoring criteria. Cohen kappa coefficient (κ) was calculated as a measure of observer agreement. The intraobserver (κ=0.74 and 0.77) and interobserver (κ=0.67) agreements were both substantial. The restorations were examined and scored individually in accordance with modified United States Public Health Service (USPHS) criteria at the 1 week and 1-, 2-, 3-, and 4-year follow-ups.[16] The patient and restoration histories were obtained from the dental records. Failed

restorations were excluded from the analysis, and reasons for failure were recorded. Caries in nonfilled tooth surfaces with acceptable composite resin restoration were not considered as reasons for failure.

Data collection and statistical analysis were performed using software SPSS Statistics for Windows (Version 23.0; IBM Corporation, Armonk, NY, USA). Descriptive statistics for the evaluation criteria and failure rates were generated. Qualitative analysis based on the modified USPHS criteria was performed separately for each of the nine clinical characteristics evaluated. Kaplan-Meier survival analysis was performed to obtain survival curves for the two layering techniques. An additional survival analysis of the restorations was performed using Cox regression analysis. The associations of survival with factors including tooth number, patient age and gender, and layering technique (independent variables) were evaluated. The layering techniques were compared in terms of the proportion of acceptable USPHS scores by year using the chi-squared test and Cochran Q test.

RESULTS

In total, the outcomes of 156 teeth were acceptable after 4 years, and the overall survival (OS) rate was 92.86%. The failure rates for the monochromatic layering technique (MLT) and PLT were 9.2% and 5.4%, respectively. The survival rate for the first year was 99% for MLT and 100% for PLT, and 99% overall; the respective rates for the second year were 96%, 98%, and 97%, while those for the third year were 93%, 97%, and 95%, and those for the fourth year were 91%, 95%, and 93%. Restorations requiring any repair or replacement were considered as failures. Repolishing was not performed during the follow-up period. Of the 168 restorations, 12 (7.14%) were failures. No restoration had more than one clinically unacceptable score, and no patients were lost to follow-up, so the number of unacceptable scores was equal to the number of failed restorations. The reasons for failure included fractured restoration (n=7), marginal discoloration (n=2), color mismatch (n=1), surface roughness (n=1), and caries (n=1). Fracture occurred in seven restorations (4.2% of the 7.1% of restorations that failed) and was the most common reason for failure in both the MLT (3.9%) and PLT (4.3%) groups. Only 2 teeth (2.6%) in the MLT group and 21 (22.3%) in the PLT group showed no detectable changes (score of 0). In 145 (86.3%) restorations, at least one change was detected (score of 1-4). Postoperative sensitivity (USPHS score of 1) was noted in 20 restorations (11.9%) in only eight patients, all at baseline (1 week after the restoration); all of these were considered recovered at the first-year follow-up.

Figure 2 shows the Kaplan-Meier survival curves for the restorations performed with the two layering techniques. The MLT (microhybrid RBC) and PLT (micro/nanohybrid RBC) groups showed no significant difference in mean survival time (p=0.343). The mean survival time was 46.026 and 46.957 months for the MLT and PLT groups, respectively. According to the chi-squared (χ^2) test, the proportion of acceptable USPHS scores did not differ between the two layering techniques in any year ($p \geq 0.05$); this was also the case in the Cochran Q test analysis ($p \geq 0.05$). There was no significant difference in failure rate among years in the MLT group ($p \geq 0.05$), whereas, in the PLT group there was a significant difference, attributable to the rate in the second year (p=0.042). For all restorations, second-year scores (p=0.018) differed significantly from the first-, third-, and fourth-year scores (p=0.433, p=0.151, p=0.302, and $p<0.05$, respectively).

Cox regression analysis of the restorations was also performed to evaluate the effect of four independent variables (tooth number, patient age and gender, and layering technique). None of the variables were associated with survival ($p \geq 0.05$) (Table 3).

DISCUSSION

In this clinical follow-up study, the long-term performance of maxillary Class IV composite restorations was investigated. The restoration outcomes using two layering techniques (MLT and PLT) were

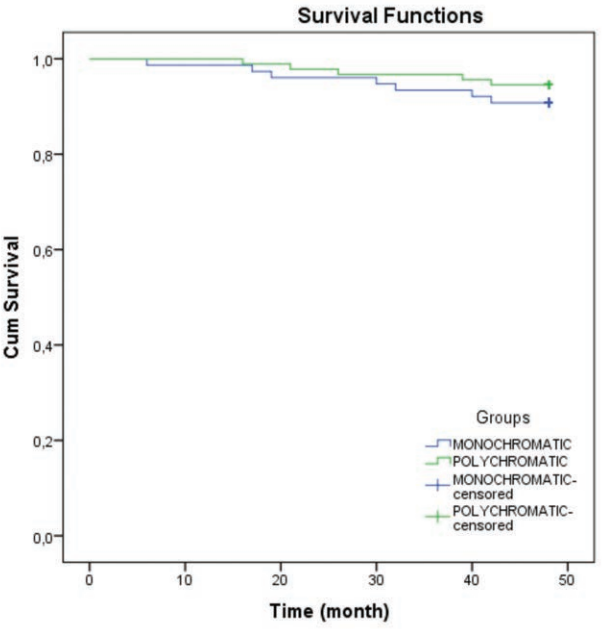

Figure 2. *Kaplan-Meier survival curve for survival of restorations with monochrome and polychrome layering techniques during the mean observation period.*

Table 3: *Cox Regression Analysis of the Restorations Regarding the Independent Variables*

	OR (95% CI)	*p*
Tooth number	0.985 (0.881-1.101)	0.788
Age	1.047 (0.992-1.106)	0.096
Layering technique	0.807 (0.237-2.752)	0.732
Gender	1.147 (0.363-3.62)	0.815

compared over a 4-year period. The methodology employed has been used in many other clinical studies.[7,11,17,18] Also, the clinical evaluations in our study were performed by two independent observers, similar to previous studies.[1,19,20] Challenges to clinical studies include standardization of indications and treatment protocols, achieving operator agreement, and dealing with missing data.[16] As the cases in this study were to be used in laboratory demonstrations for undergraduate and postgraduate students, a high-quality operator was required; the operator was a university instructor with 15 years of clinical experience specializing in restorative dentistry. The modified USPHS criteria were used for evaluating the selected restorations in this study, allowing for a standardized and detailed longitudinal assessment of the restorations. Although there is a need for a more definitive method,[21] this evaluation method has nevertheless been used in many clinical studies, to which our results could thus be compared.[11,12,16,22,23]

Substantial intraobserver (κ=0.74 and κ=0.77) and interobserver (κ=0.67) agreements were obtained for the fully blinded observers. The observers agreed with all the unacceptable scores, and the disagreements were not related to the acceptable/unacceptable decisions. Disagreements occurred regarding the acceptable scores of 0 and 1 for marginal discoloration, color match, and surface roughness criteria. It might be difficult to make a decision between these scores using only dental photographs, particularly for segmental deterioations from ideal in some of these criteria. Previously, Peumans and others[9] reported that the photographic evaluation may mask imperfections on the restoration surface and thereby lead to misjudgements, especially for the assessment of color match. Therefore, the obtained minor disagreements were considered to not effect the targeted outcomes of the present study. The evaluations of the first observer were taken into consideration for the statistical analyses.

A high OS rate of 92.84% after 4 years was obtained for the Class IV restorations in this study. For survival analysis, the Kaplan-Meier method is the gold standard and was therefore used in this study instead of the log-rank test, which has limited utility for analyzing multivariate datasets. Nevertheless, an additional survival analysis (Cox regression) of the restorations was performed. Four independent variables (tooth number, patient age and gender, and layering technique) were not associated with survival ($p \geq 0.05$) (Table 3). The OS rate observed in our study was higher than that in the studies of Coelho-de-Souza and others[1] (3.5-year survival rate of 80.1%), Frese and others[3] (5-year survival rate of 84.6%), Lempel and others[16] (7-year survival rate of 88.3%), Khayatt and others[24] (7-year survival rate of 85%), and Gresnigt and others[12] (3.5-year survival rate of 87.5%). This may be because all of the restorations in our study were done by a single restorative dentistry specialist under the same clinical conditions, and not by undergraduate students or inexperienced operators (which can affect restoration longevity).[20,21] Although all of the patients in this study were dental school applicants with low socioeconomic status, this was not associated with negative outcomes in our study, unlike some other clinical studies.[1,21]

In this study, a microhybrid RBC (81 wt%) was used in conjunction with the MLT, and a combination of microhybrid and nanohybrid RBC (76 wt%) in conjunction with the PLT. Coelho-de-Souza and others[1] reported that microfilled composite veneers had better surface gloss, color matching, anatomical, marginal adaptation, and surface staining properties compared to universal composites. However, nanofilled resin was not evaluated. Accordingly, gloss retention and polishability were previously reported to be better for resins including nanofiller (0.005-0.01 μm) compared to those including microfiller (0.01-0.1 μm).[14,25] More incisal chipping and a 3.7-fold higher risk of failure were reported by Lempel and others[16] for build-up restorations of anterior teeth when using microhybrid resin compared to nanofilled resin. Massano and others[23] reported good clinical performance of Class III and Class IV restorations using nanofilled resin over a 2.7-year period (failure rate of 2.4%). However, microfilled resins were also reported to have the advantages of high surface hardness and high resistance to wear, fracture, and shrinkage.[19,26,27] As well as the size, both the shape and amounts of particles were reported to affect the performance of resin composites.[7]

In the present study, the respective failure rates when using the MLT and PLT were 9.2% and 5.4%. The Kaplan-Meier survival analysis revealed that the mean survival durations of the restorations were not different at 46.026 and 46.957 months, respectively, (p=0.343) nor were the survival rates for each year or the OS rate (based on the proportion of acceptable USPHS scores; $p \geq 0.05$). Both RBCs (microhybrid and nanohybrid) applied to the top surface layer of the

restorations exhibited satisfactory performance. While nanofilled composites only use nanosized particles, nanohybrids combine nano- and microsized particles, similar to microhybrid composites.[14] Thus, becoming microhybrid or nanohybrid is directly related to the distribution of the nano- and microparticles.[26] Moraes and others[14] reported that microhybrid and nanofilled composites with similar matrix components yielded similar polymer network structures and thus similar hardness despite noticeable differences in filler size. According to their results, the behavior of nanohybrid composites was more similar to that of microhybrid, rather than nanofilled composites. Also, in previous studies, nano- and microhybrid resins were reported to have similar physical characteristics, depending on the filler content.[14,27] The RBCs used in this study were micro- and nanohybrids of the same brand, with similar contents including almost the same filler type and the same amount of filler particles (81 wt% for both) (Table 2). Therefore, this similarity might be the reason for no significant difference between the restorative materials, for marginal discoloration, color matching, surface roughness, and restoration wear in our study. It may also explain the lack of difference in mean survival duration at any time point or in OS between the two resin composites used. In addition, performing the restorations by a single specialist, under the same restoration protocols and clinical conditions, might be related to the similar clinical performances of the restorations with different RBC materials.[20]

The restoration failure rates of the two layering techniques used in this study did not differ by time point ($p \geq 0.05$), except for the rate at the 2-year follow-up in the PLT group ($p=0.042$). Considering both groups together, the failure rate at the 2-year follow-up was different to those of the other time points ($p=0.018$). Two unacceptable "fractured restoration" USPHS scores during the second year may explain this result. Fracture occurred in seven restorations and was the most common reason for failure when using either the MLT (3.9%) or PLT (4.3%). These results were similar to those of previous clinical trials.[5,21]

Fracture and chipping were the most frequent reasons for failure in microhybrid anterior RBC restorations in the studies of Frese and others,[3] van Dijken and others,[8] Coelho-de-Souza and others,[1] Gresnigt and others,[12] and Milosevic and Burnside.[28] In their systematic review Heintze and others[13] reported that Class IV restorations, including of the incisal edge, had a higher risk of failure compared to Class III restorations. All of the fractured restorations had a USPHS score of 2 ("partial fracture in restoration>1/4"). The fracture rate was not statistically different between the two layering techniques ($p \geq 0.05$). No fracture was observed using either technique during the first 2 years of follow-up.

Marginal discoloration, the second most common reason for failure, was observed in two cases in each layering technique group, all of which had a USPHS score of 2 ("obvious staining could not be polished away"). The rate of marginal discoloration was not significantly different between the layering techniques ($p \geq 0.05$). Heintze and others[13] and Lempel and others[16] reported that adhesion to enamel and 37% phosphoric acid etching were important for good sealing and prevention of discoloration. Selective enamel etching was performed for all restorations in our study. Additionally, 45° beveling of the labial surface of the teeth prior to conditioning the enamel was performed, to ensure that the transition between the restoration and enamel was not visible. Beveling prevents marginal staining[16,23,29] and improves fracture resistance at the tooth-restoration interface.[16,29]

Color mismatch was the least common reason for failure in this study and the rate thereof did not differ between the two layering techniques ($p \geq 0.05$). Only one restoration in the MLT group had an unacceptable ("slight mismatch in color or shade") USPHS color mismatch score; 51 restorations in the MLT group and 32 in the PLT group had a score of 1 ("good color match") during the 4-year follow-up period. Nasim and others[26] reported that the rate of discoloration was the highest for nanofilled RBCs among the microhybrid and microfilled RBCs tested. Tekçe and others[30] reported similar findings *in vitro*. Superficial degradation of restorative materials and absorption of staining agents are responsible for discoloration.[16] Vichi and others[25] reported that low triethylene glycol dimethacrylate (TEGDMA) content in the resin matrix may limit water uptake and, by extension, the color variation induced by absorption of the staining solution. In this study, both RBCs contained TEGDMA, which may explain the staining results. Additionally, filler particle type, size, and distribution are important physical properties of composite fillers[27] and may affect color stability. A previous study reported that smaller filler particle size led to low visual opacity,[27] while, in another study, it decreased staining and enhanced esthetics.[25] Lempel and others[16] reported no long-term positive effects of nanoparticles on color stability or surface gloss *in vivo*. This was supported by a recent systematic review, which concluded that nanofilled and submicron RBCs did not yield superior color stability or gloss retention outcomes compared to microhybrids.[15] In addition to material factors, patient factors (such as diet) and operator factors (operating environment, isolation,

adhesion, finishing and polishing protocols, and recall frequency) may also influence RBC staining outcomes. In our study, the experienced operator, standardized restorative technique, and high patient motivation may have been responsible for the very low rate of color mismatch failures.

The surface roughness USPHS score was unacceptable only in one case, at the 4-year follow-up in the MLT group. In total, 36 restorations performed using the MLT, and 32 using the PLT, had a score of 1 ("slightly rough or pitted") on the surface roughness USPHS criterion during the 4-year follow-up period. Repolishing was not performed for any restoration. There was no statistically significant difference in surface roughness between the two layering techniques ($p \geq 0.05$). Caries related to the restoration was considered unacceptable in only one case and no significant difference in caries was found between the two layering techniques ($p \geq 0.05$).

While clinical examinations were performed only during the fourth year of follow-up, postoperative sensitivity data were obtained from the medical histories of the patients at baseline; 20 restorations (11.9%) in only eight patients had a score of 1 for this USPHS criterion, all of which had recovered at the first-year follow-up. As the etching of dentin with phosphoric acid is considered a risk factor for postoperative sensitivity,[31] the use of the selective etch technique in this study may explain the low postoperative sensitivity scores, which also showed no difference between the layering techniques ($p \geq 0.05$). In accordance with the results of Gresnigt and others[12] and Lempel and others[16] regarding restoration wear, no wear was detected in our cases.

Some researchers have suggested that the failure criteria should be revised, where some repaired restorations remain functional and therefore should not be considered as complete failures.[16,32,33] Those studies concluded that if repaired restorations are not classified as failures, annual failure rates would drop, such that repairability could be considered as a predictor of better survival of RBC restorations.[16,34] Frese and others[3] classified repaired cases as restoration survival rather than failure. Reparability of the RBC materials was considered the most important factor in extending the life of their restorations, which had a functional survival rate of 100%. Van de Sande and others[34] reported 69% survival and 2.4% annual failure rates for Class III and IV restorations, respectively, when repair was not considered as failure, compared to 64% and 2.9%, respectively, when it was considered as failure. Composite repair is a suitable alternative to Class III-IV and veneer restorations, since it may increase the survival rate of anterior restorations.[16,34] However, in our study, restorations needing repair, retreatment, or even repolishing were considered as failures. Considering this, the 92.84% OS rate can be considered very high.

There were some limitations to our survival analysis, including the relatively low number of cases, mid-term follow-up period, and lack of generalizability, as only one operator was involved. The results of survival analyses for different dental materials should be interpreted with care, as the numbers of cases (including failures) and follow-up periods tend to be limited.[1] Demarco and others[5] noted a lack of long-term clinical results regarding the performance of anterior RBC restorations in a systematic review. The reasons for this include poor patient compliance and follow-up visit attendance.[22] Regarding our results, in case of a long-term evaluation period, perceptible major differences might have occurred in nano- and microhybrid restorations, therefore similar longevity outcome might have also changed. Recently, Dietschi and others[20] identified several factors influencing outcomes in a systematic review including; patient hygiene, caries risk, age, socioeconomic status, operator characteristics, treatment environment, observation period, and evaluation method. Use of composite filler materials and the type of curing light had little to no impact on clinical success at any time point, whereas treatment environment and number of operators affected the restoration failure rate. According to their results, a single operator yielded the optimum results.[20] In the present study, all restorations were performed by a single operator at the same clinic under consistent conditions and using the same materials. However, the effect of operator's skill, experience, and the operation environment still remained unclear. The outcomes of this study represent patients without bruxism. Therefore, the high success rate might also be associated with patients with low-risk factors. In spite of that, some patients might have developed slight or severe bruxism during its course, and this was not assessed. Differences among patients in oral parafunctions, malocclusion, dietary habits, and oral hygiene might have also affected the outcome. Because bruxism is a self-reported behavior that is difficult for patients to identify, diagnoses based on patient histories can be inaccurate. There is evidence that bruxism is a major risk factor for fracture.[8] However, in many other clinical trials, bruxism was not associated with survival. Coehlo-de-Souza and others[1] reported no correlation between tooth fracture and the longevity of build-up restorations. In the study of Milosevic and Burnside,[28] bruxism was not associated with tooth fracture or restoration failure. Further clinical long-term studies

are needed to assess the effect of bruxism on survival rate. Also, studies including more than one operator, larger sample size, and a variety of RBC materials are necessary to verify the findings of this study.

CONCLUSIONS

From this study, the following conclusions were drawn:

1. Class IV direct composite resin restorations showed good clinical outcomes, with a survival rate of 92.84% after 4 years.
2. Use of both the MLT and PLT for Class IV anterior restorations provided acceptable durability, with mean survival periods of 46 and 47 months, respectively.
3. Fracture was the most common reason for restoration failure in both the MLT (3.9%) and PLT (4.3%) groups.
4. Micro-/nanohybrid composite restorations showed a slightly higher survival rate (94.6%) than the microhybrid composite restorations (90.8% survival), but the difference was not statistically significant.
5. Monochromatic layered microhybrid and polychromatic layered micro-/nanohybrid Class IV restorations showed no significant difference in optical properties over the 4-year study period.

Regulatory Statement

This retrospective study was approved by Ethics Committee of Marmara University Faculty of Dentistry (Approval no: 2018-198 and approval date: 24.05.2018).The approval code issued for this study is 2018-198.

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 manuscript.

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Literature Review

Color Change of Resin-based Composites After *In Vitro* Bleaching Protocols: A Systematic Review and Meta-analysis

ML Vidal • OE Pecho • K Collares • SBN Brandeburski • A Della Bona

Clinical Relevance

Dental bleaching alters the color of resin-based composites, but the color change is clinically acceptable. Different bleaching protocols produce similar color changes on resin-based composites.

SUMMARY

Objectives: To systematically review the literature on color stability of resin-based composites (RBC) after *in vitro* bleaching protocols and to assess the influence of bleaching protocols by meta-regression analysis on RBC color stability, and the association with clinical and experimental characteristics.

Methods: The electronic search was conducted in MEDLINE/PubMed, Scopus, and Web of Science databases and included English language studies that evaluated and reported color differences (CIELAB values) of RBC after *in vitro* bleaching procedures using hydrogen peroxide and/or carbamide peroxide.

Results: Database search for color change of RBC after bleaching retrieved 1335 eligible papers after removing duplicates. After initial screening, 66 articles were assessed for full-text reading with final inclusion of 23 published papers. A meta-regression analysis showed that storage time ($p \leq 0.01$), color measuring device ($p \leq 0.01$), and background color ($p \leq 0.01$) had influenced on color changes of RBC. The bleaching protocol (bleaching agent and time of application) did not influence on color changes of RBC ($p > 0.01$).

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Conclusions: There is evidence that RBC change color after bleaching, but the change is not clinically significant.

INTRODUCTION

Resin-based composites (RBC) were introduced in the 1960s. Since then, RBC experienced several improvements of the organic and inorganic phases, such as the progressive reduction of filler particle size,[1] the development and inclusion of new monomers,[2] and monomer combinations.[3] As a result, RBC have shown good mechanical properties[1] and long-term clinical performance.[4] Furthermore, the increasing dissemination of minimally invasive dentistry,[5] concerns about amalgam toxicity,[6] and esthetic demands[7] promoted RBC to the most used dental material for direct restorations. Yet, the replacement of anterior RBC restorations because of esthetic failures, including lack of color stability, have been reported.[8,9]

Considering esthetic materials in dentistry, color stability is the material's ability to maintain its color after aging (in service), staining, or bleaching.[10] The CIELAB (ΔE^*_{ab}) metric is recommended to calculate the color stability.[11,12] As color is a psychophysical property, the observers' interpretation should be considered when evaluating color stability.[10] In addition, the color difference (ΔE^*_{ab}) alone has little to no clinical meaning, and, therefore, the color stability should be evaluated through the perceptibility (PT) and acceptability (AT) thresholds for CIELAB color differences (ΔE^*_{ab}).[11,13] Further, the CIELAB color space is a tridimensional space that considers changes in the color coordinates (L*, a*, and b*), which makes it impossible to determine how much "whiter" an object has become.[14] For this reason, some studies proposed whiteness indexes such as WI,[15] WIC,[16] or WI_D.[17]

Dental bleaching is one of the most popular esthetic procedures in dentistry,[18,19] with three possible protocols: in-office (performed by a professional); at-home (prescribed by a professional but performed by the patient at home); and over the counter (no professional prescription or follow-up).[20] As a result, different application times and concentrations of carbamide peroxide and hydrogen peroxide agents can be used for in-office and at-home bleaching protocols.[10,21,22]

Patients submitted to dental bleaching often have restored teeth, and the effect of bleaching agents on RBC is not completely understood. Studies reported on oxidation of amine compounds and pigments[23] and chemical bond degradation[24] by bleaching agents that may alter the color perception of RBC[21] and leading to esthetic complaints from patients. Nevertheless, the color change of RBC after bleaching is a controversial subject,[10,25,26] and the influence of the RBC type and the bleaching protocol are inconclusive.[8,10,27] Therefore, the aim of this study was to systematically review the literature on color stability of RBC after *in vitro* bleaching protocols and to assess by meta-regression analysis the influence of bleaching protocols on RBC color stability and the association with different covariates (RBC type, storage time, background color, and color measuring device). The hypothesis tested was that bleaching results in color alteration on RBC below the AT.

METHODS

This review was performed according to the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) statement.[28] The research question was: Is color change of RBC after bleaching clinically significant?

Search Strategy

Electronic searches were conducted in three different databases (MEDLINE/PubMed, Scopus, and Web of Science), with no restriction for publication date with the last search performed on March 15th, 2020. The search strategy was designed to find articles written in English that evaluated *in vitro* color changes in RBC after bleaching, as described in Table 1.

Study Selection

Search results were duplicated using Mendeley software. Two trained reviewers (SBNB and MLV, both PhD students) independently selected the studies by title and abstract, according to the eligibility criteria. Records meeting the criteria or classified as unclear were retrieved for full-text analysis, which was performed independently by the same reviewers. Whenever necessary, screening discrepancies were resolved with the assistance of a third senior reviewer (ÁDB). During full-text reading, exclusion reasons were recorded.

Eligibility Criteria

The present study included published *in vitro* studies written in the English language that evaluated the color change of RBC after different bleaching procedures. In contrast, the exclusion criteria were as follows:

- Type of study: case report, technical report, literature review, questionnaire-based studies, animal studies, and *in vivo* studies.
- Materials: studies that only evaluated nonmethacrylate-based composite resins (silorane and ormocer), studies that did not use hydrogen peroxide and/or carbamide peroxide bleaching agents.

Table 1: *Structured Search Strategy Carried Out in MEDLINE/PubMed Database*[a]

Search	Topic and Terms
#4	Search #1 AND #2 AND #3 AND
#3	Color change: "color" [Mesh] OR "colour" OR "color stability" OR "colour stability" OR "color difference" OR "color differences" OR "colour difference" OR "colour differences" OR "color-difference" OR "color-differences" OR "colour-difference" OR "colour-differences" OR "color change" OR "color changes" OR "colour change" OR "colour changes"
#2	Bleaching procedures: "tooth bleaching" [Mesh] OR "tooth bleaching agents" [Mesh] OR "tooth-bleaching" OR "teeth bleaching" OR "tooth whitening" OR "teeth whitening" OR "whitening" OR "dental bleaching" OR "home bleaching" OR "at-home bleaching" OR "office bleaching" OR "in-office bleaching" OR "in-office dental bleaching" OR "in-office tooth bleaching" OR "tooth-whitening" OR "hydrogen peroxide" OR "carbamide peroxide"
#1	Composite resin: "composite resins" [Mesh] OR "composite resin" OR "resins" OR "composite" OR "resin-based composite" OR "resin-based composites" OR "resin based composite" OR "resin based composites" OR "resin composite" OR "resin-composite" OR "resin-based restoration" OR "resin-based restorations" OR "dental composite"

[a] *Searches in Scopus and Web of Science were adapted according to each database.*

- Methodology: studies that used colored solutions; studies that stored specimens for more than 16 days to perform the final color measurement or used artificial accelerated aging; studies that did not polish the specimens; studies that did not calculate color changes or did not use the CIELAB color difference equation.
- Outcome: studies that did not report color difference and standard deviation values.

Data Collection

Articles meeting the inclusion criteria were subjected to critical appraisal, which was carried out by two reviewers (SBNB and MLV) independently. A standardized data extraction form was created using Excel software (Microsoft Corporation, Redmond, WA, EUA), collecting the following data:

- Study characteristics: Author, publication year, objectives.
- Materials characteristics: Resin composite type, bleaching agent type, and concentration.
- Methods characteristics: Storage time and medium, bleaching agent protocol, color measuring device, background, color difference formula, color difference (ΔE) values, standard deviation, and sample size (n).

These data were collected and categorized for meta-regression analysis. The primary outcome was the CIELAB color difference (ΔE^*_{ab}) after application of bleaching agents (hydrogen peroxide and/or carbamide peroxide) onto the RBC, stored in a colorless substance for up to 16 days. The secondary outcomes included the influence of covariates such as the RBC filler portion, storage time, bleaching protocol, background color, and color measuring instrument.

The following experimental groups from selected studies were excluded from the present review: control group using no bleaching protocols, dry storage, storage longer than 16 days, storage in coloring substances, and color changes performed using CIEDE2000 color difference formula. Missing data from published papers were requested up to three times to the corresponding author. If data was not informed, the article was excluded from analysis.

Data Synthesis

A descriptive analysis of the findings summarized the data. Mean and standard deviation values, and sample size (n) were used to obtain the 95% confidence intervals (95% CI) for each group. Mean color difference (ΔE^*_{ab}) values and 95% CI from all groups of the included studies were used in a linear meta-analysis of random effects.[29] A reference category for each variable was arbitrarily selected and used for comparison against other categories. The presence of heterogeneity was tested using I2 statistic and Chi-square tests, with Chi-square p-value < 0.05 or I2 $>$ 50%, indicating high heterogeneity.[30] As heterogeneity was high throughout all analyses, random effects

models were used. Outliers and normality of residuals were checked by diagnostic procedures.

Meta-regression was performed to verify the impact of collected characteristics on color change. Bleaching procedure and further covariates (RBC, storage, color measuring device, background color, and bleaching protocol) were entered into the model, and a backward stepwise approach was applied for variables selection, keeping variables with a p-value ≤ 0.2. Analyses were performed using Stata 14 (StataCorp LP, College Station, TX, USA).

RESULTS

The search resulted in 1335 studies after removing duplicates. After the evaluation of titles and abstracts, 66 full-text studies were assessed for eligibility, and 24 papers were selected for the meta-analysis. Additional information on reasons for exclusion is shown in Figure 1.

To improve the accuracy of the model, the residuals were analyzed, and one study with outliers was excluded.[31] Thus, 23 papers and 126 experimental groups were eligible, and data was collected. Experimental groups that did not match the inclusion criteria were excluded for the following reasons: nonbleached groups,[22,26,32-40] dry-stored samples,[41] nonmethacrylate resins (silorane or ormocer),[33,34,39,42] nonhydrogen peroxide, or noncarbamide peroxide bleaching agents,[35] color changes calculated with CIEDE2000 metric,[12] and samples submitted to accelerated artificial aging.[43]

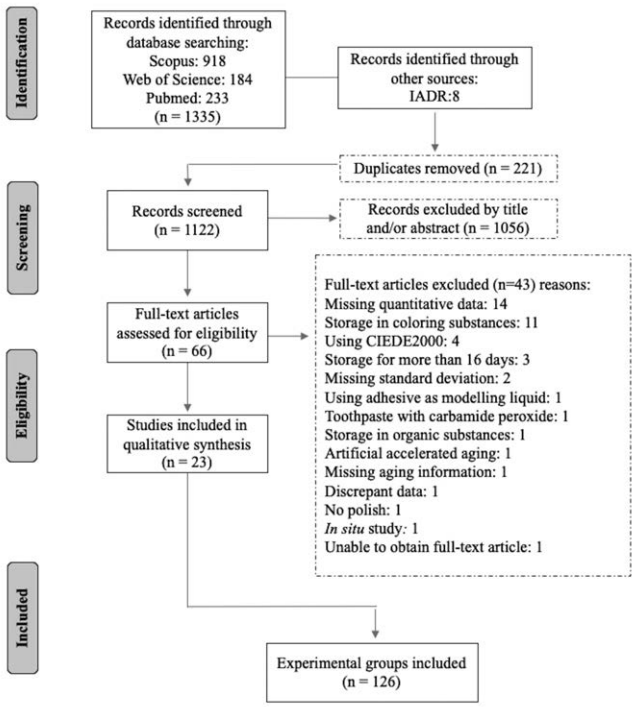

Figure 1. *Flow diagram of the systematic review.*

Table 2 shows the included studies after systematic search and the collected variables of interest. They were organized by alphabetical order and type of background. Most articles evaluated the following type of RBC: nanofilled (32%), microhybrid (27%), and nanohybrid (15%). The majority of the studies used a spectrophotometer (87%) as the color measuring instrument, and the samples were placed on a white background (48%). Carbamide peroxide (57%) and hydrogen peroxide (43%) were the bleaching products reported in the studies. Studies on in-office bleaching reported 20-60 minutes (52%) and 90-180 minutes (48%) of bleaching agent application time, while studies on at-home bleaching reported 7-21 hours (43%) and 28-147 hours (57%) of bleaching agent application time. Most studies reported a storage time of 1-2 days (75%). These data are summarized in Figure 2.

CIELAB metric, and its ΔE^*_{ab} equation, has been the most prevalent approach to calculate color difference values in dentistry. Thus, all studies included in the present review used CIELAB metric.[21,22,25,26,32-50] Figure 3 shows the mean values of color differences and 95% confidence intervals (95% CI) for all experimental groups included in the present review. The overall color difference value was 2.02 ΔE^*_{ab} units that is between PT (ΔE^*_{ab}=1.22) and AT (ΔE^*_{ab}=2.66) values, which means an acceptable color match.

Results from the meta-regression analysis are presented in Table 3. Nanohybrid composites showed greater color changes after bleaching procedures when compared to nanofilled composites (p=0.004). Storage time (p£0.01), color measuring device (p≤0.01), and background (p≤0.01) also influenced color changes. Only the bleaching protocol (bleaching agent and time of application) did not influence color changes (p>0.01).

DISCUSSION

The present systematic review and meta-analysis was designed to examine the controversial issue of RBC color changes after bleaching and its clinical significance. The results confirmed the study hypothesis that RBC color changes resulting from bleaching are below the AT.

As the human eye does not perceive small color differences,[14] a single analysis of such differences may not be clinically significant.[13] Therefore, the PT and AT were introduced to bring clinical relevance to visual color assessments.[51] Thus, if the color difference is at or below PT, it represents an excellent match between color before and after a procedure, such as bleaching. If the color difference is between PT and AT values, as

Table 2: *Descriptive Characteristics of Studies Included in the Meta-regression Analysis*

Study	Objective	n	Study Groups[a]	Resin-based Composite	Bleaching Agent (Total Application Time)	CMD	Background Color
Dziedzic & others[44]	To investigate the effect of in-office bleaching material on color changes of two tooth-colored restorative materials	12	2	Nanohybrid (1)	35% HP (90 and 45 min)	SP	White
Farinon & others[41]	To verify whether tooth whitening alters the color of a universal nanocomposite	5	3	Nanofilled (1)	10% CP (28, 56, and 112 h)	SP	White
Hubbezoglu & others[42]	To compare color changes of a microfill-, a microhybrid-, and an ormocer-based resin composite exposed to bleaching agents	5	6	Microfilled (1) Microhybrid (1)	16% CP (42 h) 37% CP (1 h) 35% HP (30 min)	C	White
Kamangar & others[33]	To compare the effect of two bleaching agents on three dental composites with different resin composition, volume, and type of filler particles	6	4	Microhybrid (1) Nanofilled (1)	15% CP (56 h) 40% HP (20 min)	SP	White
Kamangar & others[35]	To assess the effects of two bleaching agents on methacrylate-based composites with different filler sizes compared to a silorane-based composite	6	4	Microhybrid (1) Nanofilled (1)	16% CP (56 h) 35% CP (40 min)	SP	White
Kurtulmus-Yilmaz & others[26]	To evaluate the color change of five different resin composites after two bleaching gels application	10	8	Nanohybrid (3) Microhybrid (1)	10% CP (112 h) 10% HP (14 h)	SP	White
Mohammadi & others[45]	To evaluate the effect of a carbamide peroxide bleaching gel on color stability of a giomer and a microflled composite resin	20	1	Microfilled (1)	15% CP (112 h)	SP	White
Poggio & others [36]	To evaluate the effects on surface discoloration of eight composite resins, after staining and bleaching procedures	10	8	Microhybrid (3) Nanohybrid (5)	17% CP (28 h)	SP	White
Reinhardt & others[37]	To confirm and measure staining of a composite resin and to determine the degree of lightening by using self-applied at-home whitening products	5	2	Microhybrid (1)	15% CP (70 h) 6% HP (21 h)	SP	White
Rodrigues & others[38]	To evaluate color stability of two composite resins exposed to bleaching procedures and aged in staining beverage	7	4	Microhybrid (1) Nanofilled (1)	35% HP (2 h) 16% CP (56 h)	SP	White

Table 2: *Descriptive Characteristics of Studies Included in the Meta-regression Analysis (cont.)*

Xing & others[46]	To evaluate the effect of two in-office bleaching agents on the color changes of two ceromers and one direct composite resin after staining	4	2	Microhybrid (1)	35% HP (30 min) 38% HP (30 min)	SP	White
Farah & Elwi[47]	To evaluate color stability of two bleach-shade resin composites after the exposure to 3 storage solutions and the effect of 3 bleaching agents on the color stability and stain removal	3	6	Nanofilled (1) Nanohybrid (1)	10% CP (20 h) 16% CP (20 h) 25% HP (1 h)	SP	Grey
Kim & others[35]	To evaluate the effects of two types of home bleaching systems on changes on color and surface roughness of two resin composites	5	18	Nanofilled (2) Microhybrid (2) Hybrid (2)	18% CP (112 h) 6.5% HP (14 h) 3% HP (14 h)	SP	Dentin shade
Yu & others[40]	To investigated the effects of a bleaching gel on susceptibility of tooth-colored restorative materials to different staining solutions	6	2	Nanofilled (1) Packable (1)	15% CP (112 h)	SP	Dentin shade
Pecho & others[25]	To evaluate the influence of 35% hydrogen peroxide bleaching gel on color and whiteness of three resin-based composites	10	6	Nanohybrid (1) Microhybrid (1) Microfiller (1)	35% HP (45 and 90 min)	SP	Black
Anagnostou & others[21]	To evaluate the color changes of two resin composites after two bleaching products application	8	12	Hybrid (1) Nanohybrid (1)	14% HP (7 and 14 h) 6.5% HP (7 and 14 h) 10% CP (21 and 42 h)	C	INF
Çelik & others[32]	To evaluate the staining susceptibility and color stability of bleached restorative materials and subsequently immersed in different staining solutions	7	3	Nanofilled (2) Microhybrid (1)	20% CP (48 h)	SP	INF
Gouveia & others[43]	To evaluate the influence of at-home bleaching containing two different thickeners on the physical properties of a nanocomposite resin submitted or not to accelerated artificial aging	10	2	Nanofilled (1)	16% CP (56 h)	SP	INF
Gouveia & others[48]	To assess the effect of accelerated artificial aging, bleaching treatment, and staining agents on color of a nanocomposite resin	10	2	Nanofilled (1)	10% CP (84 h) 35% HP (45 min)	SP	INF

Table 2: *Descriptive Characteristics of Studies Included in the Meta-regression Analysis (cont.)*							
Halacoglu & others[49]	To evaluate the effect of different staining solutions and bleaching procedure on color stability of a resin composite with or without polishing	12	1	Nanofilled (1)	35% HP (24 min)	C	INF
Kwon & others[22]	To examine the effect of hydrogen peroxide on color change of three resin composites containing nanofillers from three different shades	5	18	Nanofilled (9)	15% CP (147 h) 35% HP (3 h)	SP	INF
Rao & others[50]	To evaluate the effect of three home bleaching agents on color stability of two resin composites and a glass ionomer cement	10	6	Microfilled (1) Nanofilled (1)	6% CP (560 min) 16% CP (14 h) 20% CP (14 h)	SP	INF
Yalcin & Gurgan[39]	To compare color changes of five different tooth-colored restoratives after two different bleaching regimens	5	6	Flowable (1) Packable (1) Hybrid (1)	10% CP (28 h) 6.5% HP (14 h)	SP	INF

Abbreviations: C, colorimeter; CMD, color measuring device; CP, carbamide peroxide; HP, hydrogen peroxide; INF, information not found; n, sample size; SP, spectrophotometer.

[a]*Number of experimental groups included in the present study.*

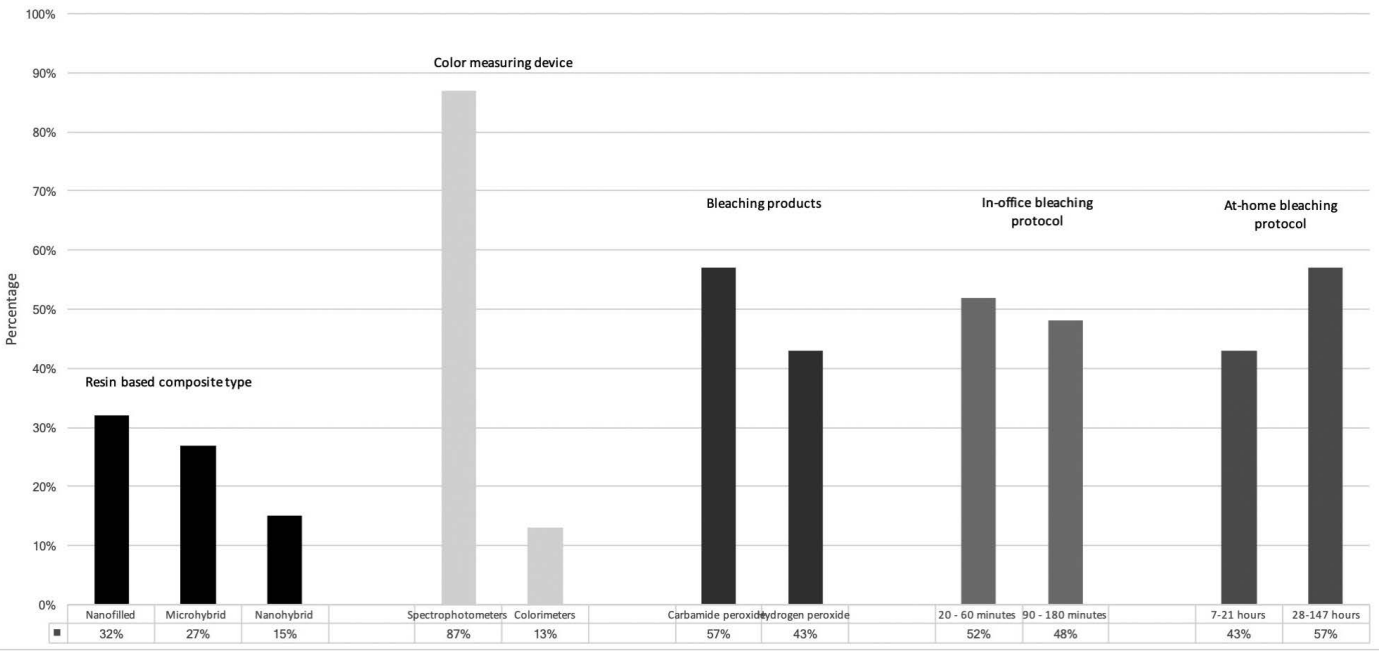

Figure 2. *Some of the covariates investigated and the data collected.*

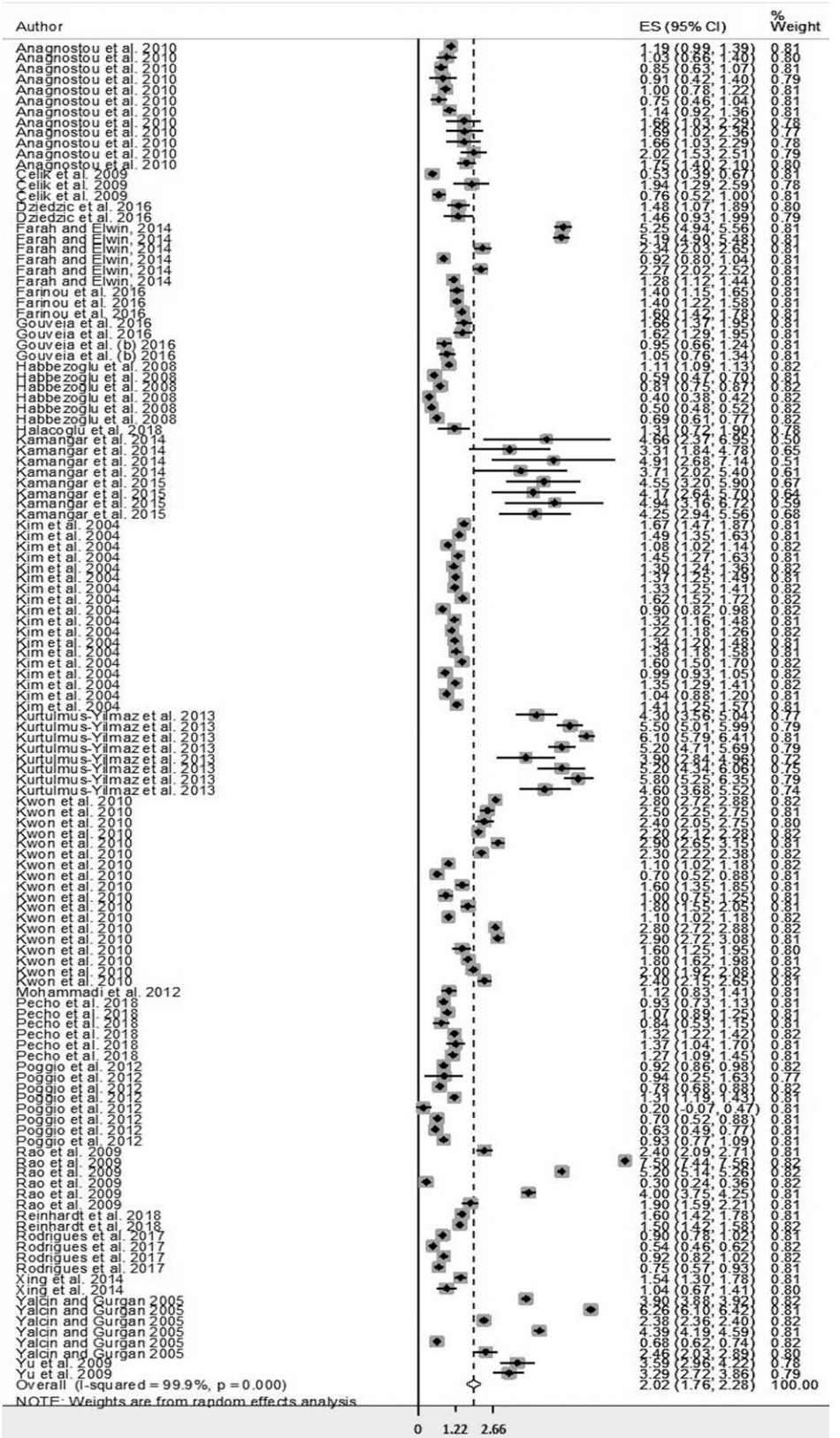

Figure 3. *Forest plot showing the mean color changes (ΔE^*_{ab}) reported in the experimental groups included in the present review. Dotted line represents the overall color difference value (2.02 ΔE^*_{ab} units). Values for the perceptibility (PT) (1.22) and acceptability (AT) (2.66) thresholds are indicated.*[13]

Table 3: *Meta-regression Analysis of Color Difference (ΔE) Values in the Final Multiple Variable Model*

Variables	**Success**	
	Regression Coefficient (95% CI)	***p*-value**
Composite (ref = nanofilled)		
Nanohybrid	1.12 (0.36 to 1.87)	0.004
Microfilled	0.49 (–0.61 to 1.59)	0.378
Microhybrid	–0.09 (–0.81 to 0.62)	0.799
Others	0.57 (–0.18 to 1.33)	0.132
Storage (ref = 1-2 days)		
7 days	0.32 (–0.89 to 1.53)	0.600
14-16 days	–2.25 (–3.09 to –1.41)	<0.001
Unknown	0.70 (–0.65 to –2.06)	0.305
Color measurement device (ref = colorimeter)		
Spectrophotometer	2.20 (1.42 to 2.98)	<0.001
Background (ref = white)		
Black	–2.12 (–3.32 to –0.93)	0.001
Dentin	–1.82 (–2.70 to –0.94)	<0.001
Grey	–1.27 (–2.93 to 0.40)	0.136
Unknown	–0.71 (–1.49 to 0.06)	0.072
Bleaching protocol (ref = office 20-60 minutes)		
In-office 90-180 minutes	–0.75 (–1.76 to 0.25)	0.139
At-home 7-21 hours	0.19 (–0.75 to 1.13)	0.690
At-home 28-147 hours	–0.25 (–1.11 to 0.60)	0.555

Abbreviations: CI, confidence interval; ref, reference.

found in this meta-analysis (ΔE^*_{ab}=2.02), it represents an acceptable color match. A color difference above AT represents an unacceptable color match.[11,12] As some studies,[13,51,52] using different methodologies, proposed different ΔE values for PT and AT, the ISO[11] published, in 2016, the threshold values (PT=1.22 ΔE^*_{ab} units, and AT=2.66 ΔE^*_{ab} units) for tooth-colored dental materials based on a multicenter study with different groups of observers.[13] The use of color thresholds is widespread and well accepted in dentistry. From the 23 articles included in the meta-analysis, three of them did not use any visual thresholds to evaluate the results.[33,34,40] Most papers published up to 2016 used AT = 3.3 ΔE^*_{ab} units.[a] The present systematic review analyzed all included data based on the visual thresholds published in the ISO/TR 28642:2016.[11]

Although ΔE^*_{ab} is commonly used to evaluate color after dental bleaching, there are more specific and useful parameters to evaluate "whiteness," such as the whiteness index (WI),[15] the CIE whiteness index (WIC),[16] and the whiteness index for dentistry (WI_D)[17] that is based on the CIELAB color space, and it was especially developed for dentistry. Only one study included in this review used a whiteness index.[25] In addition, a recent study[53] reported visual thresholds for WI_D [0.61 units for whiteness perceptibility threshold (WPT) and 2.90 units for whiteness acceptability threshold (WAT)]. Future studies on color changes after dental bleaching should consider complementing their data analysis for "whiteness" changes of teeth or dental materials using WI_D, WPT, and WAT.[14]

Previous studies explored the reasons for RBC changing color after dental bleaching. Some studies reported that free radicals available from high-concentration hydrogen peroxide can diffuse into the polymer,[39,42,54] others related color changes with longer bleaching exposure time, allowing for free radical

[a] *References 21,26,32,35,36,39,42,45,47,50.*

infiltration into the polymer.[8,47] Nevertheless, the present review found an overall color difference value of 2.02 ΔE^*_{ab} units after bleaching of RBC. Previous clinical studies on tooth bleaching efficacy reported values between 4.3-4.6[55] and 7.1-10.6 ΔE^*_{ab} units,[56] with no significant color changes after 1 year.[57] Some studies suggested that RBC do not get whiter after bleaching,[10,25] as teeth do.[58] At the outset, one may sense that RBC color stability after bleaching is a desirable characteristic. However, as RBC and teeth show different gradients of color changes after bleaching, the color difference between RBC restorations and teeth may become clinically unacceptable, and patients may demand new restorations after dental bleaching. Such clinical dilemmas should be further investigated to support evidence-based information to clinicians and patients.

Due to the methodological variability among the selected studies, a high heterogeneity (99.9%) was observed (Figure 3). Nevertheless, in addition to the primary outcome, the present study was able to evaluate the following variables: bleaching protocol, composite type, storage time, color measuring device, and background color. Thus, two bleaching protocols were considered: in-office bleaching and at-home bleaching. For the purposes of the present study, over-the-counter products were considered as at-home bleaching. It was not possible to determine any standardization of bleaching agent application protocol. There was a great variation on the total application time of the bleaching agent among the selected studies: in-office bleaching protocols were applied from 20 minutes to 180 minutes,[22,25,33,34,42,44,46-49] and at-home bleaching protocols were applied from 7 hours to 147 hours.[b] Yet, some studies used different concentrations of the bleaching agent for the same application time.[35,46,47,50] This meta-analysis shows insufficient evidence to infer that the type of bleaching protocol influences on RBC color change, which is probably associated to the great variability of dental bleaching protocols.

The association between filler content and color changes of RBC after dental bleaching is controversial. While some studies reported that the filler portion does not influence RBC color changes,[25,26,33,34,36,38] other studies showed the opposite.[21,32,42] In the nanohybrid composites the filler ratio is variable,[59] combining nanometric and larger particle size fillers.[1] Their morphology and size are product dependent,[59] and the nanoparticle concept is different from the nanofilled RBC.[60] Such heterogeneity in the filler portion can produce variable properties, especially related to the solvent stability.[59] Despite a similar chemical basis, variations in monomers[61] and photoinitiator systems[62] can change the color stability of RBC. The meta-regression showed that the filler portion influenced the outcome with a different behavior for nanohybrid RBC compared to other types of RBC. However, this color change is not clinically significant when considered by the color difference thresholds.

Studies have reported on several methods to store and age RBC samples, including dry[22] and relative humidity environments,[63] immersion in a dye solution[64] and colorless substances,[25,26] and artificial accelerated aging.[10,43,65] To standardize storage conditions, this review only included studies that used water as a storage medium and a maximum of 16 days for the storage time. In the present study, time of storage influenced the outcome, that is, storing for 14-16 days was significantly different from shorter storage times, which is not surprising since literature shows that RBC color changes are time related, even in colorless media.[66] This information is clinically relevant, although such covariates (storage time and media) should be carefully evaluated in *in vitro* studies, because none of the storage and aging methods can mimic the complex dynamics that occur in the oral environment.

Several factors can influence color measurements,[14] such as illuminant,[67] color measuring device,[68] color difference metric,[25,69,70] and color background.[71] The variables "color background" and "color measuring device" significantly influenced the outcome of this study. As RBC can exhibit different levels of translucency,[72] the background color can affect the scattering, absorption, and reflectance of the material.[14] On a white background the light can be reflected, and on a dark background the reflection is reduced.[73] Most studies included in this review used white background,[c] followed by the studies that did not report the background.[21,22,32,39,43,48-50] Studies that used white background showed a different behavior from those using a dentin shade[35,40] and black[25] backgrounds. Due to the variety of color backgrounds found in this systematic review along with the information that 87% of the studies reported using AT and PT, which were obtained from different studies over a single background, it is an urgent need for further studies on the influence of background color on visual thresholds.

Despite numerous types of instruments to evaluate and measure color in dentistry, the included studies used either colorimeters or spectrophotometers. Colorimeters measure tristimulus values of the light reflection after the light source passes through filters but do not measure the spectral reflectance of an object[74] as spectrophotometers do.[75,76] The real color of

[b] *References 21,22,26,32-43,45,47,48,50*

[c] *References 26,33,34,36-38,41,42,45,46*

an object cannot be determined, since there is no gold standard for a correct evaluation, due to the nature of color.[77] Thus, a way to measure the instrument trustworthiness is focused on the repeatability (when the same method, operator, and/or instrument is used) and reproducibility (different method, operator, and/or instrument).[68,74] Studies have shown that when compared to colorimeters, spectrophotometers have better repeatability and reproducibility, providing better results.[68,78,79] Regarding the amount of measurements and measuring moment, the present review showed that, in addition to before and after color measurements, some studies also measured the color difference during the bleaching protocol.[21,25,41,44] Considering the abovementioned information on the time dependency (aging) of RBC color changes, additional measurements would be relevant in studies using medium- to long-term aging.

This meta-analysis was designed to investigate the color change of RBC after bleaching and the clinical significance of it. Reports using different bleaching protocols, experimental methods, and study objectives were included, making for very heterogeneous data. Therefore, associated factors such as longer storage/aging periods, material degree of conversion, and surface polish aspects could not be considered. Lastly, as the collected data is from *in vitro* studies, the influence of clinical factors such as tooth brushing, staining food, and masticatory dynamics were not taken into account. Therefore, the above-mentioned factors are limitations of the present study.

CONCLUSIONS

Within the limitations of the present review and meta-analysis, it is concluded that RBC experience color change after bleaching, but it is clinically acceptable when considering the dental color thresholds. The type of bleaching protocol did not influence the color change of resin-based composite. Nanohybrid RBC showed a different color stability behavior. As methodological variables (background color, color measuring device, and storage time) influenced on color changes of RBC, it is an urgent need for standardization of experimental variables in laboratory studies. As several articles were excluded because of insufficient reported data, authors should be more careful to provide enough information in future publications so that clinical decisions could be based on scientific evidence.

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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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Laboratory Research

Effect of the Sample Preparation and Light-curing Unit on the Microhardness and Degree of Conversion of Bulk-fill Resin-based Composite Restorations

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Clinical Relevance

Resin composite properties are dependent on how the sample is prepared prior to testing. Clinicians should pay attention to the proximal boxes of bulk-fill resin composite restorations, as these areas may be inadequately polymerized.

SUMMARY

Objective: To evaluate the effect of the sample preparation and light-curing units (LCUs) on the Knoop hardness (KH, N/mm2) and degree of conversion (DC, %) of bulk-fill resin-based composite restorations.

Methods: Two molds were made using human molar teeth embedded in acrylic resin. One was a conventional tooth mold where the molar received a mesio-occluso-distal (MOD) preparation. In the other, the tooth was sectioned in three slices (buccal, middle, and lingual). The center slice received

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a MOD preparation similar to the conventional mold. Both tooth molds were placed in the second mandibular molar position in a Dentoform with a 44-mm interincisal opening. Restorations were made using Opus Bulk Fill (FGM) high viscosity bulk-fill resin-based composite (RBC) and light cured using two different lights: VALO Cordless (Ultradent) and Bluephase G2 (Ivoclar Vivadent). The RBC was placed in one increment that was light-cured for a total of 80 seconds (40 seconds at the occluso-mesial and occluso-distal locations). The RBC specimens were then prepared as follows: EmbPol – tooth mold specimen was embedded in polystyrene resin and polished before testing; Pol – tooth mold specimen was not embedded, but was polished before testing; NotPol – sectioned tooth mold, specimen not embedded nor polished before testing. The KH was measured in different depths and regions of the specimens, and the DC was measured using Raman spectroscopy.

Results: The results were analyzed using a 2-way analysis of variance (ANOVA) or repeated measures followed by the Tukey post-hoc test (α=0.05). The preparation method (p<0.001), depth of restoration (p<0.001), and the interaction between method and depth (p=0.003) all influenced the KH values. Preparation method (p<0.001), tooth region (p<0.001), and the interaction between method and tooth region (p=0.002) all influenced DC values. The KH values were reduced significantly from the top to the bottom of the restorations and also at the proximal box when compared with the occlusal region. This outcome was most significant in the proximal boxes. The NotPol method was the most effective method to detect the effect of differences in KH or DC within the restoration. A lower DC and KH were found at the gingival regions of the proximal boxes of the restorations. When the KH and DC values were compared, there were no significant differences between the LCUs (KH p=0.4 and DC p=0.317).

Conclusion: Preparation methods that embedded the samples in polystyrene resin and polished the specimens reduced the differences between the KH and DC values obtained by different preparation techniques. The NotPol method was better able to detect differences produced by light activation in deeper areas.

INTRODUCTION

Ideally, a resin-based composite (RBC) should require a relatively short exposure time, exhibit low shrinkage stress, and have a uniform conversion of monomers in all parts of the restoration.[1,2] The use of incrementally filled resin-based composites (RBC) can produce restorations that are harder, have a higher degree of conversion and lower shrinkage stress.[1] However, the use of the incremental layering and photo-curing technique is both time-consuming and more likely to incorporate voids or contamination between each increment of RBC.[2] The drive for faster strategies to restore deep cavities has led to the development of new materials that have an increased depth of cure and that can be light-cured in increments that are 4- to 5-mm thick.[3-5]

RBC restorations require appropriate polymerization of the material,[4] and the light-curing step is a critical procedure that is often overlooked when providing these RBC restorations.[7-10] When the light-curing process is incorrectly performed, this may lead to debonding, post-operative pain, discoloration, or premature failure of the RBC restoration.[11] Instead, the dentist may look towards using new bulk-fill RBCs that claim the RBC can be placed and adequately light-cured in 4- to 5-mm thick increments, and yet still achieve mechanical properties comparable to restorations made using the incremental filling and incremental light-curing technique.[6] However, the RBC, the light-curing unit (LCU), the restorative protocol, the size and location of the restoration, the emission spectrum from the LCU, and the radiant exposure received by the RBC will all affect the final polymerization of the RBC.[13-15]

Restorations in the posterior regions of the mouth are challenging for clinicians to light cure. Dental structures often get in the way, it is difficult to position the LCU tip perpendicular over the restorations in the mouth, and the type and opacity of restorative material can affect the ability to photo-cure the RBC.[9] For example, the greater the interincisal opening, the easier it is to position the tip of the LCU over the posterior teeth.[11,16] Children,[17] and patients with temporomandibular joint issues often have a limited mouth opening,[18] that will prevent adequate access of the LCU to the teeth. The design of the LCUs, its shape, and the angulation of the light tip, can also affect the ideal positioning of the LCU tip perpendicular to the surface of the restoration.[19] Limited mouth opening, the presence of matrix bands, or a poor design of the LCU may also lead to an increased distance between the restoration and LCU tips. This may introduce regions of the cavity that are in shadow and where less light is delivered. This will negatively influence the mechanical properties,

color stability, solubility, dimensional stability, and biocompatibility of the RBC.[11,20]

Microhardness tests (Knoop or Vickers) and Raman spectroscopy are often used to measure directly or indirectly the polymerization of RBCs.[21-23] However, the surface must be flat for hardness or degree of conversion (DC) measurements. Therefore, the restorations are frequently embedded in resin and then cut or polished using copious liquid coolant.[21,22] Unconverted monomers on the surface of the RBCs can be washed away and lost during the cutting, finishing, and polishing processes.[24,25] Also, the exothermic heat produced during polymerization of the embedding material and any heat produced during polishing may increase the polymerization of the RBC.

Few studies have analyzed the effect of the preparation method on the microhardness and DC analyses, or the effect of the LCU design when used in a posterior RBC restoration in a clinical simulation. Therefore, this study aimed to evaluate the effect of the sample preparation of bulk-fill posterior RBC restorations made in a dentoform that had a clinically relevant interincisal mouth opening.[17,26] The null hypotheses were: 1) The method of sample preparation would not affect the KH or the DC of the bulk-fill restorations, and 2) The choice of LC (pen-style vs. angled light guide) will have no influence on the KH and DC of two bulk-fill RBCs.

METHODS AND MATERIALS

Cavity Preparation

This study was approved by the local ethics committee (protocol number 2.985.056). Two extracted intact caries-free human mandibular molar teeth with an average dimension of 10-mm from mesial to distal and a 4.7-mm occluso-pulpal distance were used to make two different molds.[27,28] To prevent extraneous light exposure, for the conventional sample preparation method (Figure 1A), the molar tooth was embedded in red acrylic resin (Dencrilay, Dencril, Pirassununga, SP, Brazil) to a depth of 2.0 mm below the cementoenamel junction.[29] This allowed some light-curing below the cementoenamel junction (Figure 1B). Using a cavity preparation machine,[30] a standard class II mesial-occlusal-distal (MOD) cavity was prepared using a cylindrical round diamond bur #3146 (KG Sorensen, Barueri, SP, Brazil) in a high-speed handpiece (Kavo do Brasil, Joinville, SC, Brazil) with copious air and water irrigation. The preparations had a 6-degree divergence, approximately 4/5 of the intercuspal width, 4.0-mm deep in the occlusal-pulpal dimension, a proximal box that was 2.0-mm wide, and a further 1.0-mm deep, making the proximal boxes 5-mm deep (Figure 1B-D).

For the new sample preparation method, a three-part matrix was developed.[28] The molar tooth was fully embedded, leaving only the occlusal region exposed (Figure 1E) in chemically activated red acrylic resin (Dencrilay). The mold was then sectioned into three parts (buccal, middle, and lingual) (Figure 1F). An impression of the middle part was taken with addition vinyl polysiloxane (Scan Putty Regular, Yller, Pelotas, RS, Brazil) to record its mesial and distal contours (Figure 1G). The MOD cavity preparation was then prepared using the same parameters described for the conventional method (Figure 1F). The transverse surfaces of the buccal and lingual parts of the matrix were polished using silicon carbide abrasive paper of decreasing grit size (#1200, #1500, #2000 and #2500, Norton, Campinas, SP, Brazil) followed by polishing with diamond pastes (6 μm; 3 μm; 1 μm; 0.25 μm; Arotec, São Paulo, SP, Brazil) on felt discs. All three

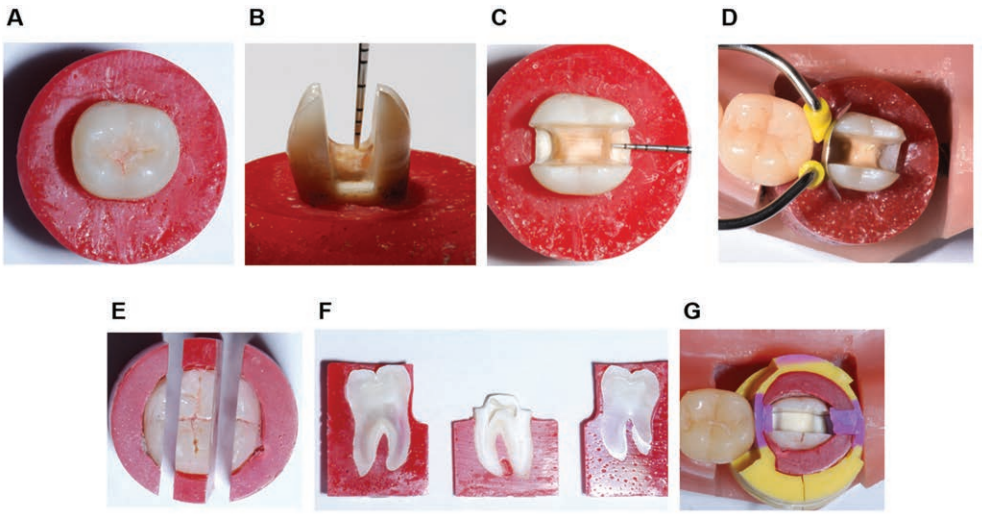

Figure 1. *Conventional matrix: (A) Molar tooth embedded in chemically activated acrylic resin in red colour. (B) Cavity preparation with 5 mm deep proximal boxes. (C) Occlusal view of the sample. (D) Sample showing the sectional matrix on one side. Three-part matrix: (E) Molar tooth totally embedded in chemically activated red acrylic resin, and sectioned in 3 parts (buccal, middle, and lingual). (F) View of the buccal, middle, and lingual parts. Middle part with no resin in the coronal third. (G) Sample showing the assembled mold.*

parts were then clamped together to make a MOD cavity (Figure 1F). This mold enabled the restoration to be easily removed by separating the three parts of the matrix after light-curing (Figure 1).

Development of a Buccal Opening Simulation Device

To better simulate the clinical environment, all restorations were light-cured with the tooth positioned in the second mandibular molar region. A dentoform (MOM, Marília, São Paulo, Brazil) was modified so that a cylinder of red acrylic resin (Dencrilay) containing the human molar tooth could be inserted. The prepared teeth were positioned to allow the proximal contact with its adjacent tooth (Figure 2A). Screws were used to fix and stabilize the resin cylinders in the correct position (Figure 2A). The dentoform was positioned in a dental patient simulator (MOM), and the interincisal mouth opening was fixed at 44 mm (Figure 2B).[17,26] This better simulated clinical reality compared to previous studies that used a tooth mold, but the tooth was not placed in a dentoform.[27, 28]

Restorative Procedure

A high viscosity bulk-fill RBC (Opus Bulk fill APS, FGM, Joinvile, SC, Brazil) and two multiple peak LCUs were used: VALO Cordless used on Standard power (Ultradent, South Jordan, UT, USA) and Bluephase G2 used on High power (Ivoclar Vivadent, Schaan, Liechtenstein). The irradiance (mW/cm^2), emission spectrum ($mW/cm^2/nm$), and radiant exposure (irradiance x time = energy/area = J/cm^2) emitted from the LCUs was measured five times using the MARC Resin Calibrator (BlueLight Analytics, Halifax, NS, Canada). The LCU's internal tip diameter was measured using a digital caliper (Mitutoyo, Mississauga, ON, Canada). A sectional matrix band (Unimatrix, TDV Dental Ltda, Pomerode, Santa Catarina, Brazil) was positioned and stabilized using an interdental wooden wedge at the mesial contact point in the groups that used the conventional tooth mold. The bulk-fill RBC was placed up to 5-mm thick, and the LCU was positioned 1 mm above the occlusal surface. The RBC was light-cured by hand for 40 seconds over the occluso-mesial and 40 seconds over the occluso-distal regions by a well-trained operator, following the manufacturer's recommendation to cover the whole of the restoration. The light-curing process was performed in a dark room with yellow light to avoid any possible light interfering with the RBC polymerization process. No adhesive system was applied so that the RBC sample could be removed from the mold. After light curing, the restorations were removed and stored in the dark and a controlled humidity at 37°C for 24h.

Preparation of the Samples for Microhardness Test (n=5)

The restorations were prepared following the three groups:

1. EmbPol – after removing from the mold, the restorations were embedded in polystyrene resin (Cristal, Piracicaba, SP, Brazil). Before testing, the RBC surfaces were finished with silicon-carbide paper (#1200, 1500, 2000, and 2500 grit sizes; Norton) followed by cleaning in an ultrasonic bath in distilled water for 5 minutes, and polished with metallographic diamond pastes (6-, 3-, 1-, and 1/4-μm sizes; Arotec) suspended in isopropyl alcohol.
2. Pol – the RBC restorations were not embedded in polystyrene resin, but were polished as described for the CEmPol Group and fixed on a glass coverslip using cyanoacrylate (Super Bonder Loctite, São Paulo, SP, Brazil).
3. NotPol – restorations made using the three-part matrix[28] were not polished nor embedded in polystyrene resin. Instead, the RBCs were removed from the tooth mold and stabilized with cyanoacrylate on a glass slide. The smooth RBC surface required for testing was obtained by the opposing polished surface of the tooth mold.

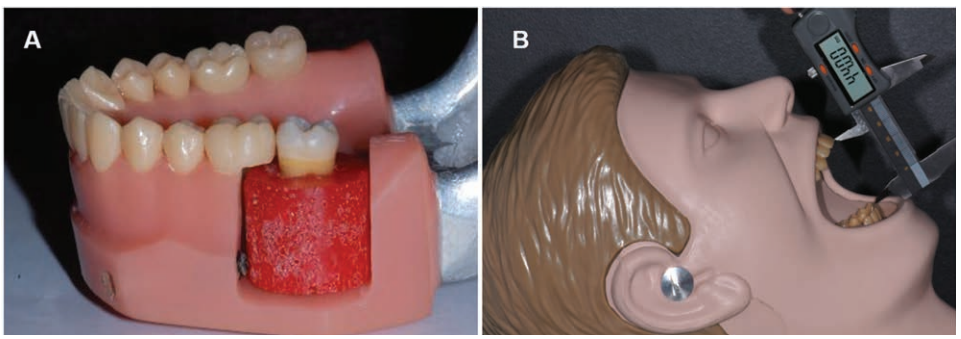

Figure 2. *Dentoform adapted to hold a cylindrical resin block in the second mandibular molar position (A) and sample showing its contact point. (B) The interincisal distance is set at 44 mm.*

Knoop Microhardness Test (KH)

Knoop microhardness indentations (Shimadzu HMV 2000; Shimadzu Corporation; Kyoto, Japan) were made in the transverse surface of the restorations using 50 g for 10 s at every 1.0 mm from the gingival and pulpal walls of the restorations. Ten indentations were made at the proximal box, and eight indentations were made at the occlusal box (A). The recorded KH data were plotted using Origin Pro 2020 (OriginLab, Northampton, MA, USA) software to produce hardness maps of the three groups.

Degree of Conversion (%)

The degree of conversion (DC) in the mesial and distal proximal boxes was evaluated at five locations: M1: occluso-mesial spot; M2: mesial proximal box spot; O: occlusal spot; D1: occluso-distal spot; D2: distal proximal box spot. The occlusal spot and a proximal box spot were at least 2-mm apart from each measurement point, Figure 3B). The DC was measured using a LabRam HR Evolution Raman spectrometer (Horiba LabRam, Villeneuve d'Ascq, France) and an excitation power of 17 mW. Using the radiation emitted by a He-Ne laser (633 nm), a Raman signal was acquired using a 600 line/mm grating centered between 1000 and 2000 cm^{-1} with a 200 μm confocal hole. These settings enabled spectra to be acquired with a resolution of 1.05 cm^{-1}/pixel. The spectra were then adjusted by polynomial function and by manual multiple point baseline correction. From the Raman vibrational modes, the areas of peaks: aliphatic (1638 cm^{-1}) and aromatic (1608 cm^{-1}) were calculated from polymerized (P) and unpolymerized (NP) bulk-fill RBC samples. The formula used to calculate the degree of conversion was: DC (%) = (1 - P / NP) x 100.

Statistical Analysis of Data

The KH and DC values were tested for normal distribution and equality of variances using Shapiro-Wilk and Levene tests. The data were then analyzed using two-way repeated-measures analysis of variance and Tukey post-hoc tests. The study factors were LCU type (2 levels), and sample preparation methods (3 levels), and the repetitions were considered the location of the restorations. All tests used a 0.05 level of statistical significance and were performed using Sigma Plot version 13.1 (Systat Software Inc, San Jose, CA, USA).

RESULTS

LCU Characterization

The mean and standard deviation of the tip irradiance values for the VALO Cordless was 1298 ± 3.3 mW/cm^2, and Bluephase G2 was 1394 ± 4.5 mW/cm^2. The emission spectrum for VALO Cordless ranged between 395-480 and Bluephase G2 between 385-515 nm. In 40 s, the occluso-mesial and at the occluso-distal regions of the restorations received a radiant exposure of 51.9 J/cm^2 from the VALO Cordless, and 55.8 J/cm^2 from the Bluephase G2 at each light-curing location: Thus, both lights delivered similar irradiances and radiant exposures.

Knoop Microhardness – KH (N/mm^2)

Means for KH values obtained in the specimens made using the two LCUs for each sample preparation method at various restoration depths are reported in Figure 4. ANOVA results demonstrated that the sample preparation method had a significant effect (p<0.001), the effects of the restoration depth were significant (p<0.001), and there was an interaction between sample preparation method and restoration depth (p=0.003).

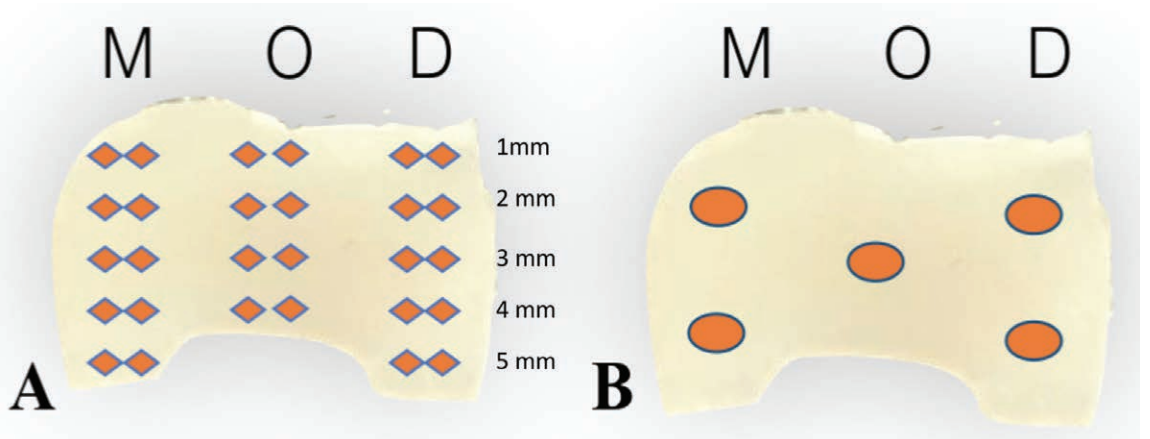

Figure 3. *Locations where the tests were performed: (A) Knoop microhardness indentations. (B) Degree of conversion measurement locations.*

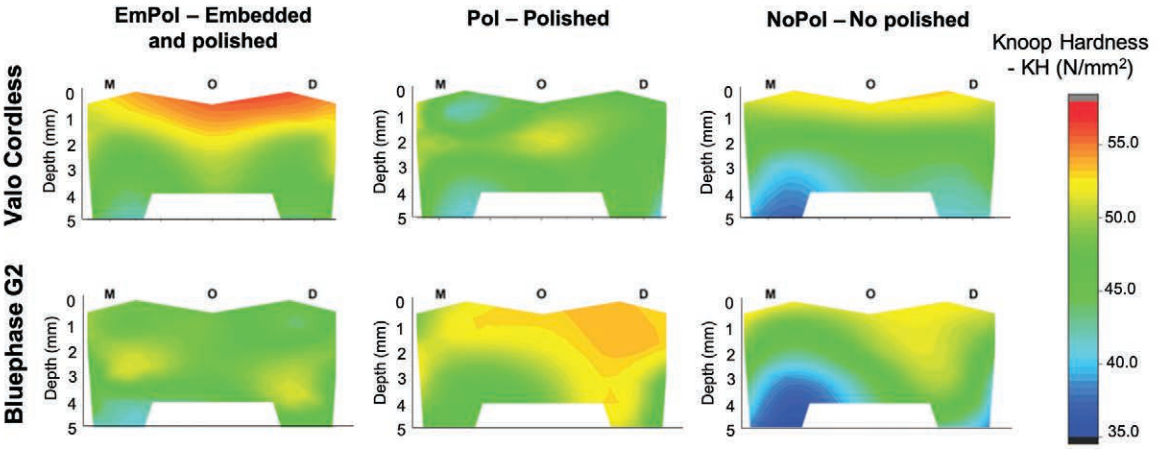

Figure 4. *Knoop hardness maps from the means obtained (n=5) comparing the three different methods used to prepare the samples and the two LCUs used.*

However, the choice of LCU had no significant effect (p=0.475), neither for interaction between sample preparation method and LCU (p=0.734), LCU and depth of restoration (p=0.700); and there was no interaction among sample preparation method, LCU and depth of restoration (p=0.766). The KH values were significantly reduced from the top to the bottom of the restoration. The location of the restoration influenced the KH values only in deeper regions. At 5 mm, the KH values were significantly lower in proximal boxes than at the occlusal region. This occurred mainly at the proximal boxes (p<0.05). The NotPol method was the most sensitive method at detecting the effects of restoration depth and tooth region.

Degree of Conversion - DC (%)

The means and standard deviations for the DC values obtained from the specimens made using the two LCUs for each sample preparation method at various restoration depths are reported in Figure 5. The ANOVA results showed that the sample preparation method (p<0.001), tooth region (p<0.001), and the interaction between sample preparation method and tooth region (p=0.002) were all significant. However, the LCU (p=0.127), sample preparation method and LCU (p=0.104), LCU and tooth region (p=0.114), sample preparation method, LCU and tooth region (p=0.154) all had no effect. The DC values were significantly lower at the gingival region of the proximal boxes compared to the measurements made on the top of the restorations, irrespective of the local, occlusal or distal areas. The DC measured on the M2 spot (gingival region) of the restoration's mesial proximal box for the NotPol method was the lowest compared to all the other methods.

DISCUSSION

The sample preparation method had a significant influence on the KH and DC results. Therefore, the first null hypothesis was rejected. When the RBC was light-cured for 40 seconds over the occluso-mesial and 40 seconds over the occluso-distal regions, the choice of LCU had no significant influence on these parameters. Thus, the second hypothesis was accepted.

In vitro studies usually perform restorations under ideal conditions, without any limitation on mouth opening or any difficulty when positioning the LCU over the restorations. Many studies also polish the RBC before testing.[24,31] Since the bottom or the sides of the restoration in contact with the tooth surface are never polished, and access to the restoration is often challenging in the posterior teeth,[23] these studies do not simulate clinical conditions. For this reason, the experimental design used in the present study was developed to better simulate the clinical condition.

The microhardness test method that is often used to assess the polymerization of RBCs requires a flat smooth surface. For this reason, the samples are frequently embedded and polished before hardness testing.[24] The polystyrene resin, commonly used for the embedment of specimens, has an exothermic polymerization reaction. The concern is that this exothermic reaction may heat the sample and thus increase the polymerization of the RBC.[32,33] The Pol and NotPol specimens made in the present study were not embedded and therefore received no additional heating effect. Since the Pol samples were not embedded in resin, the most likely explanation for the elevated KN and DC results observed in the EmbPol samples was this increase in the temperature of the RBC caused by the exothermic reaction of the polystyrene resin. The

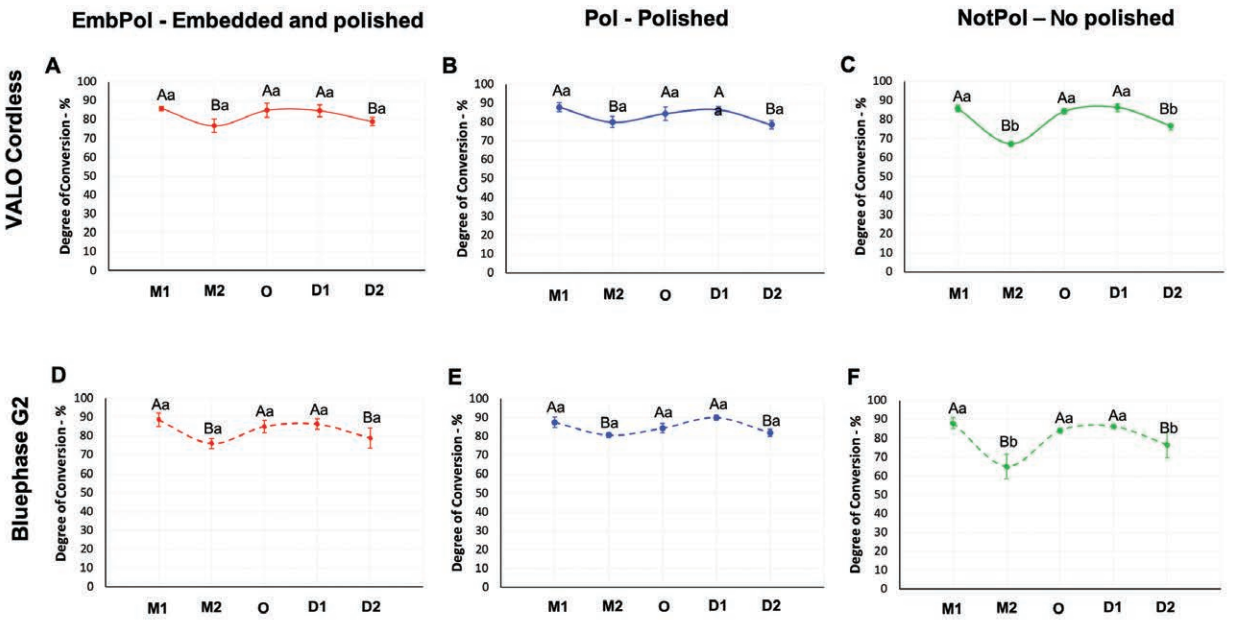

Figure 5. *Means and standard deviation of Degree of Conversion (%) comparing the three different sample preparation methods and the two LCUs (n=5). Different letters indicate a significant difference: uppercase letters are used for comparing the tooth region, and lowercase letters are used for comparing the sample preparation method (p<0.05). No significant difference was observed between the two LCUs. Abbreviations: M1, occluso-mesial spot; M2, mesial-proximal box spot; O, occlusal spot; D1, occluso-distal spot; D2, distal-proximal | box spot.*

DC in the gingival region of the proximal boxes of the restorations made using the NotPol method was lower than the DC measured in the restorations that had been polished. This probably occurred because the vinyl polyvinylsiloxane material present surrounding the proximal boxes would have reduced the amount of light received in these regions, but so would an opaque metal matrix. Thus, the three-part matrix method is recommended for future studies.[28]

Regarding the polishing process used before microhardness testing, the use of a copious amount of coolant to prevent local heat generation may also preferentially remove relatively hydrophilic, free monomers, such as residual triethylene glycol dimethacrylate (TEGDMA), or other low molecular weight monomers that have some degree of water solubility,[34-37] from surfaces of cut or polished specimens. In addition, some studies have used an alcohol-based polishing suspension.[38-40] In the present study, this suspension contained isopropyl alcohol. This treatment will likely remove more residual monomer and have an even greater effect on the RBC surface properties than polishing with an aqueous-based solution.[41-43]

The KH and DC tests should be performed at a standardized post-cured time because the restorations do not develop their final mechanical properties immediately after curing.[44,45] Therefore, in the present study, all the specimens were stored for 24 hours before testing. The KH and DC values were significantly reduced from the top to the bottom of the restoration, mainly at proximal boxes, which raises the concern of having a premature failure in those areas due to lack of adequate polymerization. Although the use of bulk-fill RBCs reduces the chair time, a lack of adequate polymerization along the bulk of the restoration may result in lower mechanical properties in some regions, compared to a highly filled nano-hybrid RBC restorations that were placed and light-cured using an incremental technique.[31]

In this study, even at the mesial proximal boxes of the NotPol group, the DC values were greater than 60%. The high DC could be attributed to the long exposure time of 80 seconds delivered to each RBC restoration, which resulted in the VALO Cordless delivering 51.9 J/cm^2, and the Bluephase G2 delivering 55.8 J/cm^2 at each light-curing location (occluso-mesial and occluso-distal regions). The DC values were higher at the surface locations of the RBC restorations (over 80%), which was different from the results of other studies that showed lower DC values.[3,5,6] This exposure time was chosen because the manufacturer (FGM) recommended 40 seconds of exposure time. However, to cover the whole tooth area is also recommended to light cure at more than one spot.[11]

Although it is recognized that specimens can be made in a metal, a Teflon mold,[30] or a silicone mold

between two polyester strips[4], these molds do not simulate the difficulties in the positioning of the LCUs over restorations that frequently occur in clinical conditions. In the present study, the specimens were made in a dentoform to simulate clinical conditions. The inter-incisal distance was set to 44 mm.[17,26] This mouth opening is close to the adult (18 to 70 years old) mouth opening that has been reported to range from 56.6 mm to 49.1 mm for men and from 49.8 mm to 44.4 mm for women.[46] Also, an Irish study reported a 43 mm of mouth opening for men and 41 mm for women,[47] which only slightly smaller than the mouth opening of Brazilian children.[17]

The mouth opening, the location of the cavity, and the operator experience all can affect the total energy delivered to RBCs.[16] The use of a patient simulator with an adjustable mouth opening enables *in vitro* studies to better replicate clinical reality. As shown by the results, the interincisal distance of 44 mm did not affect the results produced by either of the quality LCUs tested in this study. The Bluephase G2 has a curved tip, and the Valo Cordless has a straight design, but both showed similar KH and DC results. Therefore, for the mouth opening distance used in this study, both of these LCUs show similar results, which may not be valid for a patient who has a more limited mouth opening, or with LCUs that are not so well ergonomically designed.[11] The hardness maps (Figure 4) showed reduced polymerization at the bottom of the mesial proximal boxes than at the distal proximal boxes. This was photo-cured with the Bluephase G2. This may indicate the difficulty of the Bluephase G2 positioning due to the light tip angle even though the statistical analysis did not indicate any difference between the LCUs. Thus, clinicians should pay attention to the LCU shape and design because of the possibility of reducing the irradiance at the proximal boxes when LCUs with a higher light tip angle are used.[11] Delivering additional light exposure from the buccal and lingual regions of RBC restoration is recommended after removing the matrix band to compensate for the deficient polymerization in this situation. However, this should not be relied upon as the sole method of curing the RBC at the bottom of the proximal box because the significant amount of light attenuation through the tooth structure will reduce the impact of photo-curing through the tooth.[48]

Both LCUs are multi-peak broad-spectrum LED units, and both have been reported to deliver homogeneous beam profiles.[49,50] However, Valo Cordless was a pen style LCU and Bluephase G2 had a light guide. These two different designs were chosen to help elucidate if the tip shape and angulation factors should be considered when choosing the LCU, especially in areas where the position of the tip over the restoration could be affected. In a recent study comparing 22 contemporary light-curing units,[11] it was shown that the tip design can affect the ability to position the light tip at 90° to the posterior occlusal surface. However, further studies are encouraged to evaluate the effect of the mouth opening and different LCU designs on access to restorations in the mouth and irradiance on the beam profile from the LCUs. Further studies could use a mouth opening less than 44 mm, representing a child or a patient with limited mouth opening. Since the properties of the restorations such as DC and KH are dependent on the sample preparation method, the authors suggest that future *in vitro* studies should simulate restorations made under clinical conditions by using unpolished samples made in a dentoform and a three-part matrix.

CONCLUSION

Within the limitations of this *in vitro* study, it was concluded that sample preparation that embedded and polished specimens before testing reduced the differences between the KH and DC values of one bulk-fill RBC. The NotPol method was better able to detect differences produced by light curing in deeper areas of the restorations. When the RBC was light-cured for 40 seconds over the occluso-mesial and 40 seconds over the occluso-distal regions, no significant differences were found between the pen style Valo Cordless and Bluephase G2 that had a light guide when using a 44 mm interincisal mouth opening.

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Conflict of Interest

The authors have no financial interest in any of the companies or products mentioned in this article.

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Push-out Bond Strength of Two Fiber Posts in Composite Resin Using Different Types of Silanization

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Clinical Relevance

Silane surface treatment and type of glass fiber post influence bond strength of the post to a composite core.

SUMMARY

Objectives: The aim of this study was to evaluate the effect of different surface treatments and thermocycling (TC) on the push-out bond strength of two brands of glass fiber posts (GFPs) to composite resin. Methods: White Post DC (WP) (FGM Dental Group International, Joinville, Santa Catarina, BR) and Exacto (EC) (Angelus, Clinical Research Dental, Londrina, PR, Brazil). GFPs were cleaned with 70% alcohol and divided into five groups, according to the surface treatment (n=15): control (C), without treatment; prehydrolyzed silane (S-pre) (Prosil, FGM Dental Group International); 37% phosphoric acid + prehydrolyzed silane (AcS-pre); Scotchbond Universal Adhesive System (AdU), 3M Oral Care; two-bottle silane (S2B) (Dentsply Sirona Inc). The composite resin was inserted around the posts by using a split matrix. The samples were cut into 1-mm slices. Half of the samples were subjected to the push-out test immediately, and the other half underwent TC before the test. After failure analysis, the data were submitted to three-way analysis of variance (ANOVA) (α=0.05). Results: EC achieved higher bond strength than WP, regardless of TC (p<0.05). Regarding WP, surface treatments (p<0.001) and TC (p<0.001) influenced bonding strength. As for EC without TC, the highest bond strength (p<0.05)

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was found for C, then AcSpre, S-pre, AdU, and S2B. Application of TC resulted in a statistically higher bond strength values for the EC AcS-pre group ($p<0.05$), followed by S2B, S-pre, C, and AdU. The WP failures were predominantly cohesive, similar to the EC AdU and EC S2B groups. The other EC groups showed mostly mixed failures. Conclusions: Surface treatment and TC affected the bond strength to composite resin, depending on which post was used. It is important for dentists to understand the effects of different types of silanization on their chosen post.

INTRODUCTION

Glass fiber posts (GFPs) have been used as an alternative to metal posts when intracanal retention is needed, owing to their high esthetic potential, application technique, dentin-like elastic modulus, and cost.[1-3] The success of the final restoration of teeth treated with GFP depends on the remaining amount of dental structure, the condition of the supporting tissues, the esthetics of the restoration, and the chosen post.[1,4] Good post–composite resin interface quality is also needed for good bonding,[5] which allows post customization (by relining the posts) and the final restoration to be made suitably.[6,7]

However, it is common in clinical practice for failure to occur between the post and the composite resin. Therefore, it is fundamental to select an appropriate post and surface treatment to improve this bonding.[1,8,9]

The mechanical properties of GFP are affected by fiber arrangements inside the post.[4] A parallel fiber arrangement with the fiber orientation along the long axis of the tooth optimizes the flexural properties of the post.[10] The number and the thickness of these fibers also play a role in the strength and stiffness of the post. A higher fiber:matrix ratio leads to greater fracture resistance, whereas a higher number of fibers/mm^2 of post leads to a lower flexural modulus.[11] Exacto GFP (EC) (Angelus) and White Post DC (WP) (FGM Dental Group International) are two GFP options of double conicity—80% of their composition is parallel glass fibers, and 20% is epoxy resin. The two brands have a similar market value; however, they differ significantly in the number of fibers per post and fiber thickness. EC has a significantly greater number of fibers per post, compared to WP DC, but its fibers are thinner.[11] Furthermore, WP DC has "polymerization factors" that FGM Dental Group International failed to divulge when it was contacted; it merely informed the authors that these factors are a business secret. The EC post does not have these factors. Although these brands are macroscopically similar, it is unclear how their microscopic differences interfere in the final restoration.

Silane is commonly used in dentistry, since it is easy to use and to gain access to. It is formed by 3-trimethoxysilylpropylmethacrylate (MPS) diluted in a solution of water and ethanol, and has two functional groups of different polarities—one being alkoxy and the other, methacrylate.[12] The alkoxy group bonds chemically with the inorganic surface of the post, and the methacrylate group polymerizes with monomers of the composite resin. The reaction between the silane and the resinous monomers is induced by reactive free radicals that are created by the photoactivation of initiator components in the resin matrix.[13] It is also believed that silane can increase surface wettability, which helps form chemical bridges through covalent hydroxyl bonds with the substrates.[9,14,15] There are two conventional silane presentations: the prehydrolyzed (1-bottle) and the nonhydrolyzed (2-bottles) version; in the latter, the hydrolysis process occurs only when mixing the silane and an acid.[9,12] It is known that this last system has a longer life span and that atmospheric humidity acts against the prehydrolyzed form.[12] In addition, authors have found that this particular system can enhance the hydrolytic stability of the GFP–composite resin interface.[16] Notwithstanding this added feature, some studies have shown that both presentations act to increase the bond strength between post and resin,[12] whereas others deny this statement.[9,17] Some authors have proposed previous use of phosphoric acid to optimize its performance.[18] It is believed that this acid can increase the surface energy of the post by degreasing it, increase its wettability, and change its topography, resulting in greater contact surface between post and resin.[9] Other authors, however, did not find that bonding improved when the fiber post was treated with phosphoric acid prior to silanization.[19] The complete reaction mechanism of silane is an issue that is still not fully understood.[12]

There are also adhesives modified by silane. The Scotchbond Universal Adhesive System (3M ESPE, St Paul. MN, USA) is a self-etching adhesive system that contains silane in its composition.[20] According to its manufacturer, this adhesive can be applied using both the conventional and the self-etching techniques and is indicated for direct and indirect restorations on virtually any surface, whether enamel, dentin, zirconia, fiberglass, or ceramic. The silane component contained in this adhesive system may increase the adhesiveness of the post to composite resin.[17]

Thermocycling (TC) is normally used to simulate thermal changes that the oral cavity regularly undergoes in daily activities, like chewing food of varying temperatures, drinking fluids, and even breathing,

which can interfere with the adhesive interface of the restorations.[21] TC promotes the artificial aging of the sample, thereby allowing samples that have undergone this test to be compared with others that have not. This is an important resource, because it allows researchers to assess how the given surface treatment of a post can behave inside the oral cavity in the long term.[22,23]

The purpose of this study was to evaluate the bond strength of two commercial brands of GFP, submitted to different surface treatments as well as to analysis with and without TC. The tested null hypothesis was that the bond strength between the two commercial brands of GFP to composite resin would be the same, regardless of the surface treatment and application of TC.

METHODS AND MATERIALS

Ethical Aspects

This study was exempted from submission following Research Ethics Committee assessment (protocol number 2017/0859), since it does not involve human beings.

Experimental Design

- Type of study: *In vitro* study, with a completely random factorial structure (2×5×2).
- Experimental units: GFP (N=150, n=15).
- Factors under study:
  - Commercial brand of GFP on two levels: White Post (WP) DC3 GFP (FGM, Joinville, SC, Brazil).
  - Post surface treatment, on five levels: Prosil Silane, FGM Dental Group International (S-pre); phosphoric acid (37%)+Prosil Silane, FGM Dental Group International (AcS-pre); Universal Adhesive System containing silane in its composition, Scotchbond Universal, 3M ESPE (AdU); Silano two-bottle silane (primer and activator), Dentsply Sirona Inc (S2B); control (C), without surface treatment.
  - TC, on two levels: Without (control) and with (5000 thermal cycles).
- Response variable: Push-out bond strength (MPa)—quantitative and qualitative (percentage failure mode)
- Sample size calculation: The means and standard deviations were used to calculate the effect size (f=0.219), based on a pilot study carried out with three specimens. The G*Power 3.1.9.4 software program (Heinrich-Heine Universität, Dusseldorf, Germany) retrieved 13 specimens per group to detect the difference among the groups, at a 0.05 alpha level and 80% power. The final sample size per group was established at 15 specimens to account for potential losses during the study.

Preparation of the Specimens

All the posts were immersed in 70% alcohol for 1 minute. Seventy-five GFP specimens of each commercial brand were randomly divided into five groups, according to the surface treatment (n=15). The application of each treatment is detailed in Table 1.

The posts were fixed in a vertical position using a bisectioned metallic matrix (Figure 1), containing a main central cylindrical hole 10 mm high and 5 mm in diameter, and a secondary central also cylindrical hole 2 mm high and 2 mm in diameter. Opallis DA3 nanohybrid resin (FGM, Joinville, SC, Brazil) was used to fill the matrix; and an Ultraled (Dabi Atlante, Ribeirão Preto, SP, Brazil) light curing unit was applied with a minimal irradiance of 500 mW/cm^2. Then, 2 mm layers of the resin were inserted into the posts laterally, followed by light curing for 40 seconds each, until the entire length of the matrix (10 millimeters) was filled with composite resin.

After the matrix was completely filled, the specimens were removed (Figure 2), stored in 100% humidity, put aside for 1 week, and then sectioned transversely by using a cutting machine with a diamond cutting disc for 1/2" shafts, diameter 4"×0.012" thick in six 1 mm high samples.

Thermocycling

The samples were aged using the following process: 1 week after the samples were cut, three random sections were subjected to TC in an Elquip machine (model MSCT-3e, Salvador, Bahia, Brazil) from the Oral Biochemistry Laboratory of the Institute of Health Sciences, Federal University of Bahia, Brazil. A total of 5000 cycles were performed, with baths at temperatures of 5°C and 55°C, and with a dwell time of 30 seconds in each bath.

Push-out Test

The samples that did not undergo artificial aging were submitted to the push-out test 1 week after sectioning, and those that did were submitted to the test after TC. Each sample was submitted to the micro-push-out test (Figure 3), by using a Universal Testing Machine (model EMIC-DL 2000; EMIC - Instron, Salvador, Bahia, Brazil), and extrusion of the posts was evaluated with the microshear bonding test, showing the results in Newtons (N). The bond strength was calculated in megapascals (MPa) by dividing the maximum force

Table 1: *Materials Used and Method of Application*

Material/ Manufacturer	Composition	Application Mode[a]
White Post DC (WP) GPF - number 3 (FGM, Joinville, SC, Brazil)	Glass fibers (80±5%), epoxy resin (20±5%), inorganic filler, and promoters of polymerization	—
Exacto 3 (EC) GFP - number 3 (Angelus, Londrina PR, Brazil)	Glass fibers (80%) and epoxy resin (20%)	—
Opallis composite resin - shade DA3 (FGM, Joinville, SC, Brazil)	Monomeric matrix: Bis-GMA, Bis-EMA, UDMA, and TEGDMA. Fillers: barium aluminum, silanized silicate, nanoparticles of silicone dioxide camphorquinone as a photoinitiator, accelerators, stabilizers, and pigments. Composite particles range from 40 nm to 3.0 microns (average particle size: 0.5 microns). Inorganic filler loading is about 78.5% to 79.8% by weight and 57% to 58% by volume. Opallis is a nanohybrid resin.	—
Condac 37 phosphoric acid (FGM, Joinville, SC, Brazil)	37% phosphoric acid, thickener, dye, and deionized water	Apply for 30 seconds, wash for 30 seconds, and dry by air jet for 60 seconds
Prosil Silane (FGM, Joinville, SC, Brazil)	MDP, ethanol, and water	Apply a thin layer, wait for 60 seconds and dry with a light air jet for 30 seconds
Silano coupling agent (Dentsply Sirona Inc., Pirassununga, SP, Brazil)	Silane primer: 95% ethyl alcohol and Silane A 174. Activating silane: 95% ethyl alcohol and glacial acetic acid	Apply a thin layer of a 1:1 mixture of each silane after a 5 minute wait, and then dry by light air jet for 30 seconds
Scotchbond Universal Adhesive System (3M ESPE, St Paul, MN, EUA)	MDP phosphate monomer, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, and silane	Apply a thin layer, wait for 60 seconds and dry by light air jet for 30 seconds

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

[a] *According to manufacturer's instructions*

values in Newtons (N) by the area of the bonding interface. The following formula was used:

$$\frac{F\,(\mathrm{N})}{2\pi rh}$$

where F is the force obtained in Newtons, "π" is the constant value of 3.14, "r" is the radius of the post, and "h" is the height of each sample, obtained with a digital caliper.

Fracture Mode Analysis

After undergoing the push-out test, the specimens from each group were assessed with an optical microscope at 30× magnification to establish the failure types. The failures were classified as: 1) adhesive failure between resin cement and fiber post, 2) composite resin cohesive failure, 3) post cohesive failure, and 4) mixed failure when a combination of two or more of the failure types were found in the same sample (Figure 4).

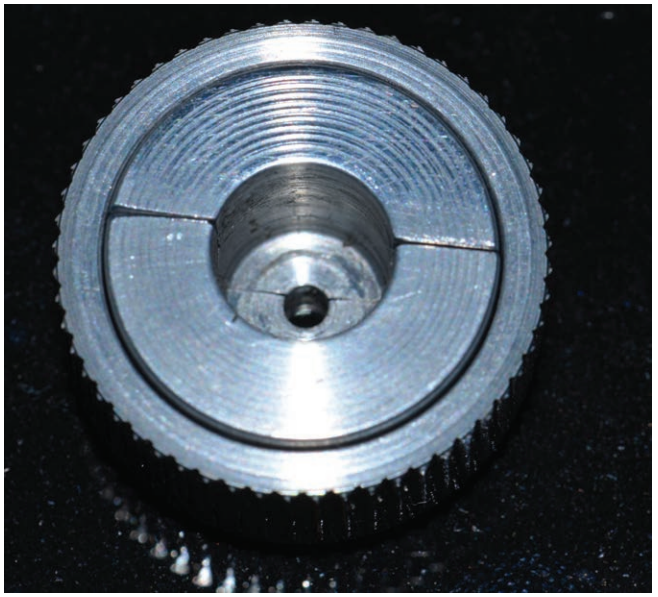

Figure 1. *Bisectioned metallic matrix.*

Statistical Analysis

The normality and homogeneity of variance were analyzed using the Shapiro–Wilk and the Levene tests, respectively.

Application of the three-way analysis of variance (ANOVA) test investigated the effects of the GFP brand, the surface treatment, and the TC, as well as the triple and double interactions among these three factors. Two-way ANOVA and Tukey tests were used to assess the separate parts of the interactions. The failure modes observed after the bond strength test were presented as relative frequency.

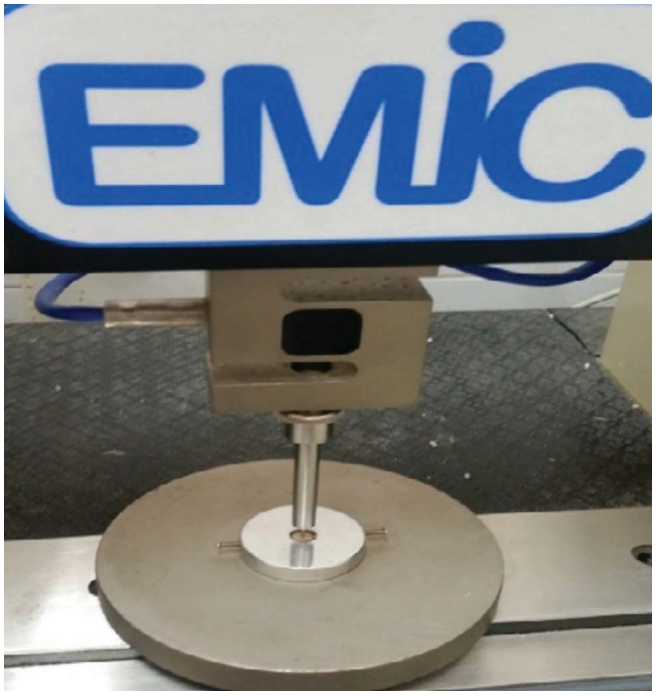

Figure 3. *Push-out test.*

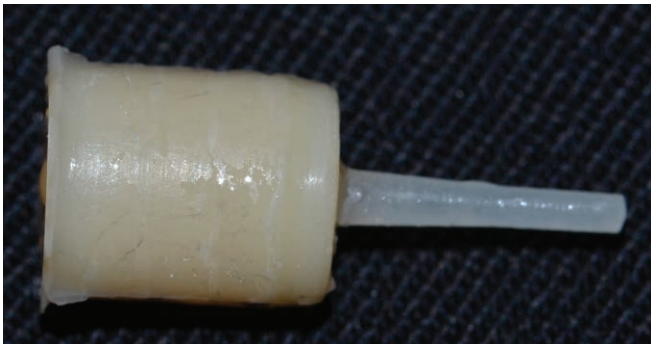

Figure 2. *Post bonded to composite resin before being sectioned transversally.*

The statistical calculations were performed using the IBM SPSS Statistics version 23 program (SPSS Inc., Chicago, IL, USA), adopting a significance level of 5%.

RESULTS

Three-way ANOVA revealed that the triple interaction between post versus surface treatment versus TC was significant (p=0.042). Two-way ANOVA and Tukey tests were used to evaluate the separate parts of the interaction; the findings are described below.

In regard to WP GFP, application of acid followed by silane and two-step silane were found to provide bond strength values significantly lower than those found for the control group, regardless of whether or not TC was performed (ANOVA: p=0.001). In regard to the universal adhesive system and the prehydrolyzed silane, intermediate values of bond strength were found and did not differ from those of any other treatment. In regard to TC, the bond strength values were significantly lower (ANOVA: p<0.001; Table 2), whether or not the

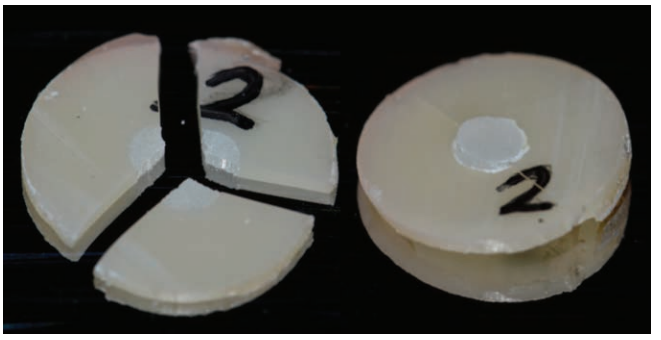

Figure 4. *Representation of the predominant failure types after push-out test. (A) Cohesive failure of post and composite resin; (B) mixed failure, adhesive+cohesive of composite resin.*

Table 2: *Average Values and Standard Deviations of White Post (WP) DC Bond Strength (MPa), According to Surface Treatment and Thermocycling (TC), and Triple Post versus Treatment versus TC Interaction*[a]

Main Factor	Comparison Between Groups				
Treatment	C 4.66 A (1.09)	S-pre 4.22 AB (0.73)	AcS-pre 4.19 B (0.73)	AdU 4.21 AB (0.48)	S2B 3.94 B (0.56)
Thermocycling (TC)	Without 4.62 a (0.79)	With 3.86 b (0.56)	—	—	—

Abbreviations: "—", no data; C, control; S-pre, Prosil silane; AcS-pre, phosphoric acid (37%)+Prosil silane; AdU, Scotchbond Universal; S2B, Silano two-bottle silane.

[a]*Different uppercase letters indicate statistical difference between treatments, regardless of TC. Different lowercase letters indicate statistical difference between the means of the specimens tested immediately and those submitted to TC, regardless of the surface treatment.*

different surface treatments were applied. The data for WP (Table 2) were grouped together (treatment regardless of TC and then TC regardless of treatment), because these factors did indeed produce an effect, even though there was no interaction between them. Comparatively, the data for EC (Table 3) indicates that there was an interaction between these factors.

Unlike WP GFP, EC GFP showed an interaction between the effects of surface treatment and TC (p=0.019). Analyzing these combined effects, primarily in the case without TC, the Tukey test revealed that the bond strength in the group whose post remained untreated was significantly greater than that achieved with the application of all other surface treatments, except for that in the group treated with phosphoric acid followed by silane. In contrast, this last group presented higher values only in comparison with the group that received the two-step silane, whose values did not differ significantly from those found for the EC GFP specimens treated with the universal adhesive system (Table 3).

Table 3: *Average Values and Standard Deviations of Exacto Post Bond Strength (MPa), According to Surface Treatment and Thermocycling (TC), Considering Triple Post versus Treatment versus Thermocycling (TC) Interaction Separately*[a]

Treatment	Thermocycling (TC)	
	Without	With
C	6.49 (0.96) Aa	5.84 (0.80) Cb
S-pre	6.15 (1.20) Ba	5.86 (1.02) Cb
AcS-pre	6.31 (0.83) ABa	6.54 (0.54) Aa
AdU	5.92 (0.81) BCa	5.02 (0.75) Db
S2B	5.82 (0.75) Cb	6.24 (0.94) Ba

Abbreviations: C, control; S-pre, Prosil silane; AcS-pre, phosphoric acid (37%)+Prosil silane; AdU, Scotchbond Universal; S2B, Silano two-bottle silane.

[a]*Different uppercase letters indicate statistical difference between treatments, considering testing with or without TC separately (comparisons in the same column). Different lowercase letters indicate statistical difference between the specimens tested immediately and those submitted to TC, considering each surface treatment separately (comparisons in the same row).*

In the case in which TC was performed, the bond strength value of the EC posts treated with phosphoric acid followed by silane exceeded the values found for all the other groups. The treatment that provided the second highest bond strength value for EC posts with TC was two-step silane. Significantly lower values were observed for the silanized (Prosil) and control groups, which did not differ from each other, but were higher than the values of the group that received the universal adhesive system for treatment of the EC post (Table 3).

TC caused a statistically significant reduction in the bond strength of the EC posts of the control group and those treated with silane (Prosil) or with the universal adhesive system. When the treatment consisted of phosphoric acid followed by silane, there was no statistically significant difference between the values obtained with or without TC. In the group that received two-step silane, the bond strength values were significantly higher in the case in which there was TC (Table 3).

Whether the samples were submitted to TC or not, the bond strength values achieved using EC GFP, overall, were significantly higher than those obtained with the WP GFP.

The normality of the data was based on the Shapiro–Wilk tests, which indicated a value of p>0.05 for the

variables of the EC post (p=0.788), AcS-pre-surface treatment (p=0.055), S-pre (p=0.096), and control (p=0.269), whereas p<0.05 was observed for the other variables and for the Levene test. The decision to use parametric statistical analysis allowed the authors to maintain the structure of the triple factorial design of the present study, and based it on a histogram-type graph and a Q-Q plot-type graph, which indicated normal data distribution.

As for the failure mode, as shown in Figure 5, WP had predominantly cohesive post and composite resin failures in all the groups, ranging from 80% to 96% of the failures, with the exception of the AdU and S2B groups with TC, in which case there were 100% cohesive failures. As for the EC post, the AdU and S2B groups followed the same pattern, and obtained predominantly cohesive failures (96% with TC and 98% without TC), but the other groups obtained mostly mixed failures, ranging from 51% to 67%.

DISCUSSION

The results of the present study showed a statistical difference among the groups, thus leading to rejection of the null hypothesis. Comparison of the two different posts tested shows that EC GFP resulted in significantly higher bond strength values than WP GFP. In addition, the posts behaved differently in regard to the surface treatments. Another difference and possible explanation for the results, other than the "polymerization factors" present in WP GFP and absent in EC, can be found in the number of fibers per post. EC has a significantly greater number of fibers (7951 fibers/post) compared with WP (2924 fibers/post).[11] The fibers of EC are thinner; therefore, the resin matrix space between them is smaller. Although the bond strength values were significantly higher for EC, it had more adhesive failures than WP, which had more cohesive failures. The resin matrix space in the GFP is where there is less flexural strength. The fact that this space is larger in WP may lead to cohesive failure of the post when subjected to the push-out test. Nonetheless, when comparing the different groups, this probably does not mean that the GFP is inferior. Because the GFP has thicker fibers, the flexural strength of this post, as a whole, is similar to that of the EC post when evaluated on its longitudinal axis, and its flexural modulus is significantly greater.[11] Both posts, however, have a parallel fiber arrangement. Wandscher and others[4] found that the parallel fiber arrangement in anterior teeth has less fracture resistance when supporting oblique occlusal loads. They also found that fracture occurred in cases associated with other factors, such as the absence of remaining coronal tooth structure and/or lack of occlusal stability, which reiterates the importance of these factors.

As for WP, it was observed in the research herein that the control group (only immersed in 70% alcohol) obtained the highest bond strength value, which was significantly higher than the groups treated with AcS-pre and S2B. No statistically significant difference was

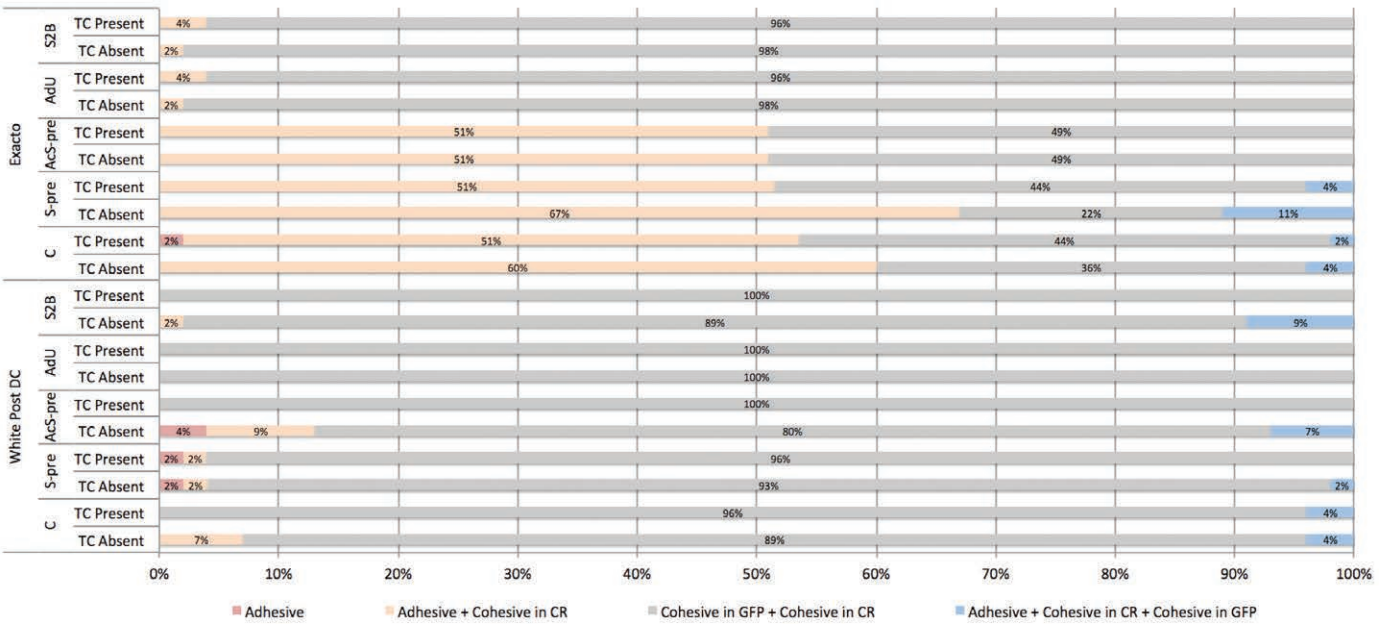

Figure 5. *Relative frequency (%) of failure modes after push-out bond strength testing of GFPs White Post and Exacto, subjected to different surface treatments, according to thermocycling.*

observed between these last two groups or between them and the other groups, with or without TC. When Belwalkar and others[14] tested different surface treatments to determine the bond strength of GFP, they observed that silanization alone did not increase the bond strength of prefabricated posts, compared with the nonsilanized group. They attributed this to the property of silane that increases the wettability of the surface of the post and consequently forms chemical bridges with the monomers of resin cements or composite resin. However, the authors added that the glass fibers in the post they used were protected superficially by epoxy resin (40% of the post), which interfered with this bridge formation. This also may have occurred with the posts in the present study. Furthermore, immersion of GFP in 70% alcohol for 1 minute performed herein was applied in all the groups, including the control group. The alcohol may have increased the surface energy of the GFP, thus increasing its wettability in relation to the composite resin. Since these posts are coated with a layer of epoxy resin, this increase may have been enough to enhance their adhesion. Faria and others[3] observed that using 70% alcohol prior to silane and adhesive promoted significantly higher bond strength results than using just silane and adhesive.

After TC, the bond strength values of the EC post control group decreased significantly, whereas those of the AcS-pre were maintained, and S2B increased significantly. In a systematic review and meta-analysis, Moraes and others[18] found that silanization improves the retention of the GFP only when the post surface is pretreated appropriately before application of silane. This explains why the AcS-pre group behaved the best. Corroborating these results, Li and others[15] found the best results after TC (5000 cycles) in the groups of posts treated with S2B and S-pre. The authors attributed this to the formation of covalent bonds (–Si–O–Si–) between post and resin. Similarly, Daneshkazemi and others[19] evaluated the effect of phosphoric acid, hydrogen peroxide, and silane on the adhesion of GFP to composite resin and found the best results for the group that was treated with phosphoric acid followed by silanization. AcS-pre was the best group for the EC post with or without TC (along with the control group without TC). The significant increase in bond strength values in the EC S2B group after TC may be attributed to induction of late polymerization of the composite resin. Ghavami-Lahiji and others[21] found that the degree of conversion of the tested composite resin after 4000 thermal cycling events increased significantly. The temperature increase provided by TC can promote a diffuse reaction where small molecules can enter the resinous polymer matrix and stimulate free radicals trapped during the resin vitrification phase, to induce late polymerization.[21] The reaction between silane and resinous monomers is induced by these reactive free radicals.[13] A study by Kim and others[16] found that two-step silanization produces a more stable hydrolytic bond between post and composite resin, compared with pre-hydrolyzed silane. This may explain why this study found a significant increase in the EC S2B group and not in the other groups. This more stable bond may have allowed new chemical bridges to be formed after stimulation of free radicals by TC. The same did not occur in the WP S2B group, possibly due to the "polymerization factors." These factors may have caused a higher conversion rate prior to TC, which led to decreased availability of free radicals. Acidic pH in universal adhesives induces a self-condensation reaction in the silanol groups over time, forming siloxane oligomers, which reduces the effectiveness of universal adhesives.[20] This is in line with the results for the EC post where the universal adhesive behaved worse regardless of TC.

The failure mode analysis of the present study showed almost all cohesive failures of the post and composite resin for all WP groups tested, as well as for the EC, AdU, and EC S2B groups. This corroborates the findings of França e Silva and others,[17] who found predominantly cohesive failures in almost all of their tested groups, including the group treated with the universal adhesive (Scotchbond Universal), which obtained 100% cohesive post and composite resin failures, similar to the equivalent group in the present study. However, the EC C, EC S-pre, and EC AcS-pre groups had more mixed failures (Adhesive+Cohesive of the composite resin). This is partially in line with the results of França e Silva and others,[17] who found mostly adhesive flaws in their control group, whereas the composite resin group remained intact. The present study used Opallis resin, whereas França e Silva and others[17] used Filtek Z250. The latter resin has significantly higher flexural strength values, compared with Opallis resin,[24] thus explaining the cohesive failure of the resin occurring herein and not therein. Since there were mostly cohesive failures of composite resin and post, it is understandable that the weak link of the samples was not the adhesive interface. Therefore, the values in MPa found by using the push-out test in this study were not representative of that area but must be viewed as the least of what can be expected.

The push-out test is appropriate to assess adhesive forces between GFPs and resinous materials, because it simulates clinical conditions better than the other tests.[25-28] However, the test has the disadvantage of

having nonuniform stress distribution.[28,29] This negative feature can be offset by cutting the slices in a thickness no greater than 1 mm,[28-30] as was done in the present study. This promotes less variability in the mechanical tests and a more homogeneous distribution of forces.[28, 29] This gives the test great clinical relevance,[28,29] which is why it was chosen as the method of evaluation in this study. Moreover, thicker slices may cause the bond strength to be overestimated.[28] However, push-out force is reported to decrease linearly with reduced thickness of the samples.[28] This may be because thin slices may cause a collapse in areas other than the adhesive interface, possibly explaining the high occurrence of cohesive failures seen in this and other like studies.

Normal occlusal forces during chewing range between 20 and 120 N.[31] Shear stress is a tangential force exerted on a contact surface. Considering that the whole post is at least 10× longer than the tested sections (at least 10-mm long), and that ideal conditions of fixation are in place, it can be concluded that under normal conditions shear stress would support at least 10 times the value of the pressure exerted for the rupture of a segment. Thus, analyzing the average values found in N for each of the tested groups (with and without TC), all tested adhesive systems are within the range of normal occlusal forces during chewing. It is worth mentioning that the density and stiffness of dentin is greater compared with composite resin evaluated in the present study, and so dentin is able to support and distribute greater occlusal forces than composites. Consequently, it is possible that under clinical conditions, the final treatment with a GFP, which has an intraradicular portion associated with the filling core, behaves better than that observed in the present study.

CONCLUSION

It can be concluded that immersion of the GFP in 70% alcohol before the insertion of the composite resin in the GFP increases its bond strength. Furthermore, the influence that the different types of surface treatments have on GFPs depends on the type of post used. For WP, no surface treatment other than the immersion in 70% alcohol had the highest bond strength results, while the group treated with 37% phosphoric acid prior to prehydrolyzed silane showed greater and more stable bond strength values for EC. TC influenced the results depending on the post and the surface treatment.

Regulatory Statement

Author represents that the study was performed in compliance with author's institution's appropriate policies. The approval code issued for this study is 2017/0859.

Conflict of Interest

The authors of the present study 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 the present article.

(Accepted 7 November 2020)

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Three-dimensional Quantification of Enamel Preservation in Tooth Preparation for Porcelain Laminate Veneers: A Fully Digital Workflow *In Vitro* Study

J Gao • L Jia • X Tan • H Yu

Clinical Relevance

We proposed a fully digital workflow to evaluate the preservation of enamel after tooth preparation at different depths, with the final objective of providing scientific guidelines for the digital analysis of the preparation depths for porcelain laminate veneers.

SUMMARY

Objective: This *in vitro* study aimed to evaluate the preservation of enamel after tooth preparation for porcelain laminate veneers (PLVs) at different preparation depths based on a fully digital workflow.

Methods and Materials: Sixty extracted human maxillary anterior teeth, including 20 maxillary central incisors (MCIs), 20 maxillary lateral incisors (MLIs), and 20 maxillary canines (MCs) underwent microcomputed tomography (CT) scanning, and were reconstructed as three-dimensional (3D) enamel and dentin models. Subsequently, the three-dimensional (3D) enamel models were imported into Materialise, where each enamel model underwent seven types of virtual preparation for PLVs at preparation depths

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at 0.1-mm increments from 0.1-0.3-0.5 mm (D1) to 0.7-0.9-1.1 mm (D7). The enamel surface was depicted by merging the virtual preparation and, respective, dentin models. The enamel area and prepared surface were measured to calculate the percentage of enamel (*R*%). The data were statistically analyzed using one-way analysis of variance (ANOVA) (α=0.05).

Results: The group-wise mean (standard deviation) *R* values for the MCIs were as follows: D1-D3: 100.00 (0) each, and D4-D7: 74.70 (2.45), 51.40 (5.12), 24.40 (3.06), and 0.00 (0), respectively. The group-wise mean *R* values for the MLIs were 100.00 (0), 73.70 (3.40), 53.50 (3.44), 25.20 (3.79), and 0.90 (0.99) for the D1-D5 groups, respectively; and 0.00 (0) each for the D6-D7 groups. The group-wise mean (standard deviations) *R* values for the MCs were as follows: D1-D3: 100.00 (0) each, and D4-D7: 99.00 (1.34), 77.10 (3.28), 74.20 (3.61), and 52.20 (4.09), respectively. The one-way ANOVA revealed significant differences between the seven groups in the MCIs, MLIs, and MCs (p<0.05).

Conclusions: Our results recommended preparation depths of up to 0.3-0.5-0.7 mm (MCIs), 0.1-0.3-0.5 mm (MLIs), and 0.4-0.6-0.8 mm (MCs) to facilitate complete intraenamel preparation. Moreover, 50% enamel was preserved at preparation depths of 0.5-0.7-0.9 mm (MCIs), 0.3-0.5-0.7 mm (MLIs), and 0.7-0.9-1.1 mm (MCs).

INTRODUCTION

The esthetic indications of porcelain laminate veneers (PLVs) have increased, because they provide clinicians with a more minimally invasive treatment method by allowing for greater preservation of tooth structure.[1] Since their retention relies solely on adhesion, a reliable bond strength between the veneer and tooth structures is critical for the clinical success of PLVs.[2] This bond strength is influenced by several factors, including the depths of tooth preparation and enamel preservation of the original tooth substrate.[3]

The preparation depth for PLVs is approximately 0.3-0.7 mm and varies from the incisal edge to the cervical margin.[4,5] Recently, minimally invasive preparations limited to within 0.3 mm or even nonreduction for ultrathin veneers has garnered considerable attention for the intraenamel preparation for PLVs.[6,7] Cherukara and others[8] found that tooth preparation at a depth of 0.5 mm was mainly intraenamel, except in the cervical region. Wang and others[9] established digital tooth models to indicate dentin exposure in standard tooth preparations for PLVs on maxillary central incisors (MCIs). LeSage[10] devised a classification to divide preparation depths, volume of remaining enamel, and percentage of dentin exposed. However, the preparation depths that facilitate complete intraenamel preparation for PLVs on maxillary anterior teeth have not been quantified.

Although intraenamel preparation is desired for PLVs, discolored or misaligned teeth may require a deeper reduction to improve the esthetic result, causing inevitable dentin exposure.[11] Enamel preservation is critical for the bond strength of PLVs. Öztürk and others[12] indicated that the bond strength of porcelain to dentin was 75% lower than that of porcelain and enamel. Gresnigt and others[13] have confirmed that 50% remaining enamel substrate demonstrated a significantly higher bond strength compared to a 25% residual enamel substrate, but there is a lack of quantitative analyses on the preparation depths that facilitate 50% enamel preparation for PLVs.

The purpose of this study was to quantitatively assess enamel preservation after tooth preparation at different preparation depths for PLVs for maxillary anterior teeth. The null hypothesis was that there would be no association between the preparation depths and enamel preservation in maxillary anterior teeth.

METHODS AND MATERIALS

Sample Collection

The protocol of this study was approved by the Ethics Committee of our institution (Approval Number: WCHSIRB-D-2019-122) (Figure 1). Sixty noncarious maxillary anterior teeth were extracted from patients (21-50 years old) within the last 6 months, including 20 MCIs, 20 maxillary lateral incisors (MLIs), and 20 maxillary canines (MCs). The inclusion criteria were as follows: normal crown shape, absence of dentin exposure or significant wear, and no history of root canal treatment or tooth fractures.

Digital Reconstruction of Teeth

All samples were thoroughly cleaned under the microscope, followed by scanning with microcomputed tomography (micro-CT) (scanning parameters: 80 Kv, 500 μA, 19.64 μm, and 800 ms), and the data were converted into the Digital Imaging and Communications in Medicine (DICOM) format. The DICOM files of the teeth were imported into a reverse engineering software (Mimics 17.0; Mimics), and the three-dimensional (3D) enamel and dentin models were reconstructed using the "adjust threshold," "region

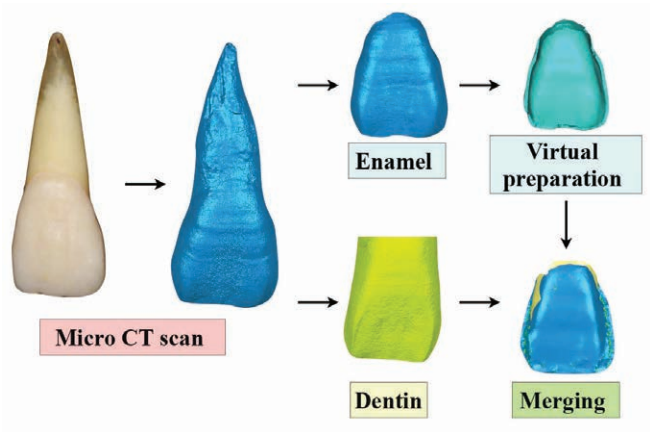

Figure 1. *Workflow diagram of this study.*

growth," and "calculate 3D" tools. These data were saved in the standard template library (STL) format.

Virtual Preparation

The enamel models of all 60 teeth were imported into the Materialise software (Magics 23; Materialise). The labial surface of each enamel model was selected and shifted inward by using the "Offset" tool to perform virtual preparation, as described by Gao and others.[14] The design of the virtual preparation was based on the standard clinical criteria of window preparation for PLVs, namely 0.3 mm, 0.5 mm, and 0.7 mm in cervical, middle, and incisal, respectively (0.3-0.5-0.7 mm); virtual preparations at seven different depths were performed on each enamel model, at 0.1-mm increments from 0.1-0.3-0.5 mm (D1) to 0.7-0.9-1.1 mm (D7) (Table 1). The virtually prepared surfaces, especially the transitional areas of different depths, were selected and smoothed using the "Smooth" tool.

Measurement of Enamel Substrate Area

All seven virtual preparation models and the respective dentin model of each tooth were imported into the Geomagic software (Studio12.0; Geomagic). The distributions of enamel and dentin substrates on the preparation surface were illustrated by merging the virtual preparation and dentin models. The enamel area was also measured (mm^2) using Geomagic. The surface was smoothed, the boundaries of the enamel surface and whole preparation surface were created using the "polygon" tool. The areas of the enamel surface (A_e) and whole preparation surface (A_w) were calculated with the "calculation" tool.

The percentage of enamel surface (R%) was calculated with the following equation: $R\% = A_e/A_w \times 100\%$.

The numerical (quantitative) data were presented as the mean and standard deviation. One-way analysis of variance (ANOVA) was used to statistically compare the percentage of enamel between multiple groups. The test standard was a two-tailed p-value of 0.05. The significance level was set at $\alpha = 0.05$. Statistical analyses were performed using the SPSS software (SPSS 25.0, SPSS).

Table 1: *Reduction Depths of Virtual Preparation in Seven Groups (mm)*

Groups	Cervical	Middle	Incisal
D1	0.1	0.3	0.5
D2	0.2	0.4	0.6
D3	0.3	0.5	0.7
D4	0.4	0.6	0.8
D5	0.5	0.7	0.9
D6	0.6	0.8	1.0
D7	0.7	0.9	1.1

RESULTS

The three-dimensional (3D) models of enamel and dentin for each sample tooth were reconstructed using micro-CT and the Mimics software (Figure 2).

The digital models for virtual preparation were created using the Materialise software. Seven types of virtual preparation were performed on the enamel model of each tooth. The distributions of the enamel and dentin surfaces were presented by superimposing the virtual preparation and dentin models (Figure 3). Figure 4 presents the distributions of the enamel surfaces with different preparation depths after tooth preparation for PLVs on the maxillary anterior teeth.

The percentages of the enamel substrate after virtual preparation of the maxillary anterior teeth are presented in Table 2. The preparation surface included only enamel in groups D1-3 in the MCIs. The percentage of the enamel surface was decreased significantly from group D4 to D7. In group D5, 50% enamel was preserved on the preparation surface. No enamel was preserved on the preparation surface in group D7. The entire preparation surface of the MLIs was composed of enamel substrate in group D1. Dentin exposure was 50% in group D3, and no enamel was preserved on the preparation surface in groups D5-D7. The preparation surface was composed entirely of enamel in groups D1-D4 in the MCs, while 50% of the surface enamel substrate was preserved in group D7. The one-way ANOVA revealed significant differences between the groups for each type of maxillary anterior tooth ($p<0.05$).

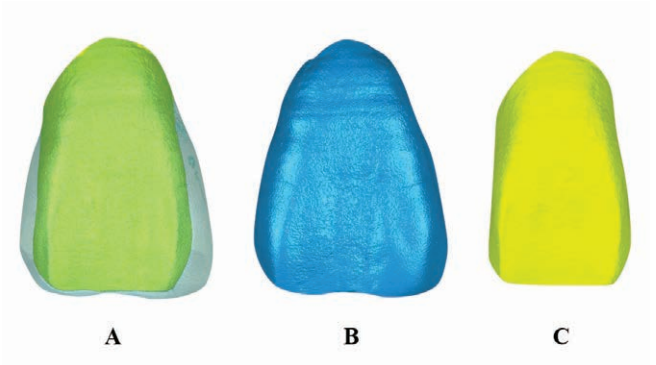

Figure 2. *Three-dimensional models of enamel and dentin: (A) enamel and dentin models, (B) enamel model, and (C) dentin model.*

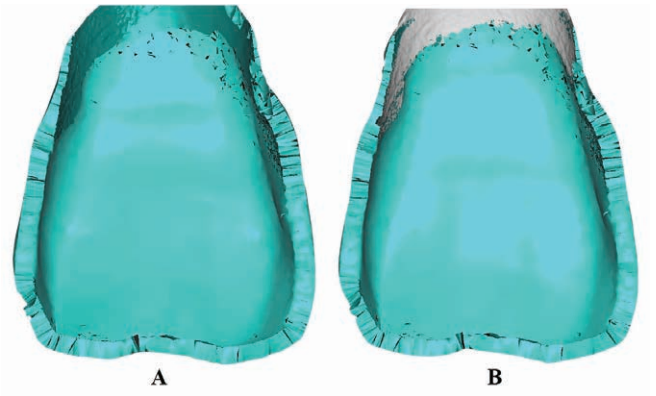

Figure 3. *Virtual preparation: (A) virtual preparation model and (B) superimposition of the virtual preparation and dentin models.*

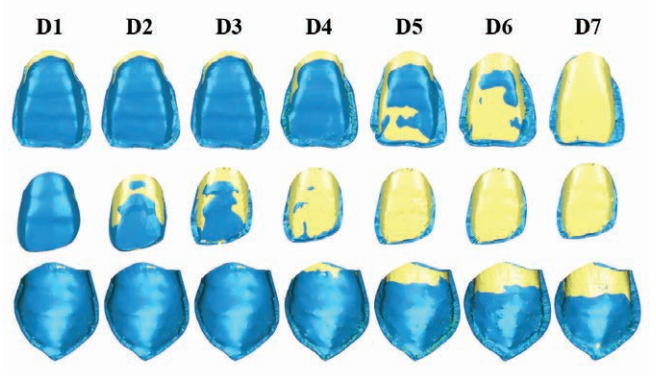

Figure 4. *Enamel distribution after preparation of the maxillary anterior teeth. Blue: Region of enamel, Yellow: Region of dentin.*

DISCUSSION

This study was the first to examine the preservation of enamel after tooth preparation for PLVs at different preparation depths using a fully digital workflow. The results of this study rejected the null hypothesis that there was no association between the preparation depths and enamel preservation in the maxillary anterior teeth.

In this study, 3D enamel and dentin models were reconstructed from the micro-CT scans of the teeth. Micro-CT has been proven to provide accurate 3D reconstructions of the scanned teeth.[9] Virtual preparations were performed on the 3D enamel models, which has been reported to control the preparation depths precisely.[15] The enamel and dentin surface were depicted by the superimposition of the virtual preparation and dentin models.[9] The fully digital workflow reduces the operative errors caused by manual preparation and limits the scanning error of the prepared tooth, and can thus be used to improve the accuracy of quantitative evaluation.[16,17]

We evaluated the preparation depths of the complete intraenamel preparation for PLVs. Complete intraenamel preparation for PLVs has garnered considerable attention owing to the concept of minimally invasive dentistry,[18] the analysis of targeted restorative space,[19] and the recommendations of tooth preparation guides.[20] Our results show that complete intraenamel preparation can be realized with preparation depths up to 0.3-0.5-0.7 mm in the MCIs, 0.1-0.3-0.5 mm in the MLIs, and 0.4-0.6-0.8 mm in the MCs. The enamel distribution of maxillary anterior teeth is uneven, with a mean thickness of 0.4 mm at the gingival-third, 0.9 mm at the middle- third, and 1.0 mm at the incisal-third[21]; thus, the preparation depths vary over the length of the tooth.

The preparation depths are also associated with the space required for the restoration, since its thickness should be sufficient to ensure mechanical durability. However, the preparation depths are critically limited by the thickness of the cervical enamel. Hence, special attention should be focused on the preparation depths in the cervical region, which should be within 0.3 mm for MCIs, 0.1 mm for MLIs, and 0.4 for MCs for the complete intraenamel preparation for PLVs. These findings are consistent with the results that maintaining cervical reduction within 0.3 mm provides complete intraenamel preparation for extrathin PLVs.[22,23] Considering all of these data and our results, it is reasonable to suggest that the preparation depths should be limited within 0.3-0.5-0.7 mm for MCIs, 0.1-0.3-0.5 mm for MLIs, and 0.4-0.6-0.8 mm for MCs, in order to facilitate complete intraenamel preparation.

This study also evaluated the preparation depths that facilitated the maintenance of 50% enamel substrate after tooth preparation for PLVs: 50% enamel reduction has been identified as the preparation criterion for PLVs,[21] as PLV debonding appears to occur if the remaining enamel substrate was less than 50%.[24-26] This study was the first to demonstrate that 50% of enamel

Table 2: *Percentages of Enamel Surfaces After Virtual Preparation on Maxillary Anterior Teeth: Mean, Standard Deviation (SD), and Respective Confidence Intervals (CI=95%)*[a]

Groups	Maxillary Central Incisors (MCIs)				Maxillary Lateral Incisors (MLIs)				Maxillary Canines (MCs)			
	N	Mean (SD)	95% CI for Mean		N	Mean (SD)	95% CI for Mean		N	Mean (SD)	95% CI for Mean	
			Lower	Upper			Lower	Upper			Lower	Upper
D1	20	100.00 (0) a	100.00	100.00	20	100.00 (0) A	100.00	100.00	20	100.00 (0)*	100.00	100.00
D2	20	100.00 (0) a	100.00	100.00	20	73.70 (3.40) B	72.11	75.29	20	100.00 (0)*	100.00	100.00
D3	20	100.00 (0) a	100.00	100.00	20	53.50 (3.44) C	51.89	55.11	20	100.00 (0)*	100.00	100.00
D4	20	74.70 (2.45) b	73.55	75.85	20	25.20 (3.79) D	23.43	26.97	20	99.00 (1.34)*	98.37	99.63
D5	20	51.40 (5.12) c	49.00	53.8	20	0.90 (0.97) E	0.447	1.353	20	77.10 (3.28)#	75.57	78.63
D6	20	24.40 (3.07) d	22.96	25.84	20	0.00 (0) E	0.00	0.00	20	74.20 (3.61)^	72.51	75.89
D7	20	0.00 (0) e	0.00	0.00	20	0.00 (0) E	0.00	0.00	20	52.20 (4.09)†	50.29	54.11

[a]*Different letters and symbols indicate statistically significant differences (p<0.05) among groups for each type of teeth.*

was preserved with preparation depths of 0.5-0.7-0.9 mm for MCIs, 0.3-0.5-0.7 mm for MLIs, and 0.7-0.9-1.1 mm for MCs. Previously, the degree of enamel preservation was evaluated visually after preparation with 34% phosphoric acid for 10 seconds; however, the preparation depths that allowed for 50% enamel preservation were unclear.[27] Recently, Farias-Neto and others[6] reported that tooth preparation at depths of 0.5-1.0 mm preserved approximately 50% to 80% of the enamel substrate. In this study, the distributions of the enamel surfaces indicated that dentin exposure occurs at the cervical area of the tooth, while the incisal preparation remains completely within the enamel at preparation depths of 0.5-1.0 mm. Thus, the preparation depths at the middle-third region are the most meaningful for 50% enamel reduction and should be maintained under 0.7 mm for MCIs, 0.5 mm for MLIs, and 0.9 mm for MCs.

These results provide new clinical methods for the analysis of PLVs using virtual preparation of the digital wax-up before the tooth preparation procedure.[14] The preparation depths can be measured by merging the virtual preparation and original tooth models. The preparation depths were further evaluated for the maintenance of 50% enamel surface or the preferred complete intraenamel preparation.

CONCLUSIONS

We arrived at the following conclusions within the limitations of this study.

1. Preparation depths can be measured by merging the virtual preparation and original tooth models to evaluate the maintenance of enamel surface after tooth preparation for PLVs.
2. Complete intraenamel preparation requires that the preparation depths should be limited within 0.3-0.5-0.7 mm for MCIs, 0.1-0.3-0.5 mm for MLIs, and 0.4-0.6-0.8 mm for MCs.
3. The maintenance of 50% enamel surface requires preparation depths of up to 0.5-0.7-0.9 mm for MCIs, 0.3-0.5-0.7 mm for MLIs, and 0.7-0.9-1.1 mm for MCs.

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Regulatory Statement

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

guidelines and policies of the Ethics Committee of Sichuan University (Approval Number: WCHSIRB-D-2019-122).

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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The Influence of Cement Removal Techniques on *In Situ* Bacterial Adhesion and Biodegradation at the Marginal Interface of Ceramic Laminates

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Clinical Relevance

The presence of excess cement at the marginal interface of ceramic materials may increase surface roughness and facilitate bacterial adhesion, leading to clinical failure.

SUMMARY

Objectives: This *in situ* study aimed to analyze the influence of different resin cement removal techniques on bacterial adhesion and biodegradation at the marginal interface of ceramic laminates.

Methods and Materials: Eighty feldspathic ceramic (F) blocks were prepared and cemented onto bovine enamel slabs (7×2.5×2 mm). Excess cement was removed using a microbrush (MBR), a scalpel blade (SCP), or a Teflon spatula (TSP). For the biodegradation analysis, 40 disc-shaped

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resin cement specimens were prepared (7×1.5 mm) using a Teflon mold. The specimens were randomly allocated into two groups: (1) No finishing procedure (only Mylar strip), and (2) with finishing and polishing procedures using the Jiffy system (Ultradent, South Jordan, UT, USA) (n=20). The *in situ* phase consisted of using an intraoral palatal device by 20 volunteers for 7 days. Each device contained five cylindrical wells (8×3 mm), where three dental blocks and two cement specimens were included in the wells. Surface roughness (R_a) was measured using a contact profilometer. A micromorphological analysis was performed under a stereomicroscope and a scanning electron microscope. Bacterial adhesion was quantitated based on the number of colony-forming units (CFU/mL) and their biofilm development potential.

Results: The cement removal techniques directly affected surface roughness at the marginal interface (p<0.001), and the SCP technique produced higher mean roughness, regardless of the surface area analyzed. Surface polishing protected cement specimens from further biodegradation (p=0.148). There were no differences in CFU counts between the groups after the *in situ* phase (p=0.96). All specimens showed CFU with a strong ability to develop a biofilm.

Conclusions: The techniques used for cement removal increased the surface roughness of ceramic laminates, particularly SCP, but they did not affect bacterial adhesion at the marginal interface. Surface polishing of the resin cement is recommended to mitigate biodegradation.

INTRODUCTION

Ceramic laminates have been successfully used as dental restorations, particularly when a minimally invasive esthetic procedure in anterior teeth is required.[1,2] Bond stability between the cement, ceramic material, and dental tissues is an important factor determining the clinical success of all-ceramic restorations.[3,4]

The longevity of indirect restorations can be compromised by a marginal misfit, the presence of surface irregularities, and the excess of luting cement, which may favor the accumulation of microorganisms at the marginal interface.[2] Thus, the increased surface roughness may result in more significant biofilm development, causing periodontal issues associated with esthetic impairment. Besides, it may also negatively affect the cement bond strength between the tooth and ceramic material.[2,5]

Early bacterial accumulation largely depends on the physical and chemical nature of the surface.[6,7] Overall, a mean surface roughness (R_a) of <0.2 μm is desirable for dental materials. A lower surface roughness seems to reduce biofilm accumulation significantly.[8] In contrast, rougher surfaces have niches that may protect the microorganisms from the mechanical forces of toothbrushing, muscle activity, and salivary flow.[9]

Clinically, the resin-based cement film in ceramic restorations is located in an area with a higher concentration of organic acids.[10] These acids are metabolized by cariogenic bacteria, which can degrade methacrylate-based polymers, thereby affecting surface hardness and increasing surface roughness. This process is known as biodegradation.[11]

Several techniques have been described considering the importance of avoiding excess cement material around the interfacial region of ceramic restorations.[2,12] Most *in vitro* studies evaluated the use of sharp scalpel blades (SCPs), microbrushes (MBR), or brushes, cotton balls, and plastic instruments. The use of MBR provided a homogeneous and regular interfacial area, while a Teflon spatula (TSP) showed surface irregularities with higher bacterial concentration compared to the MBR technique.[12] The partial photoactivation for 5 seconds before cement removal reduced the surface roughness, especially when using a blade or an explorer. From a topographical point of view, a smoother surface was observed. Regarding bacterial adhesion, the polishing technique reduced the colony-forming unit (CFU/mL) count, particularly when a MBR was used compared to the other removal devices.[2]

A previous study showed the influence of different dental materials' surface roughness on bacterial adhesion *in vitro*.[13] However, no *in vitro* tests are capable of reproducing the complexity of the biodegradation process.[11] *In situ* models are recognized as an experimental design to examine biofilms properly.[11,14-18]

Thus, this *in situ* study aimed to analyze the influence of different cement removal techniques on bacterial adhesion and biodegradation at the marginal interface of ceramic laminates. The null hypotheses tested were that (1) the cement removal technique does not affect bacterial adhesion, and that (2) surface polishing of the resin-based cement has no influence on material biodegradation within the oral milieu.

METHODS AND MATERIALS

Figure 1 shows a schematic illustration of the experimental design. All tested materials and their specifications are listed in Table 1.

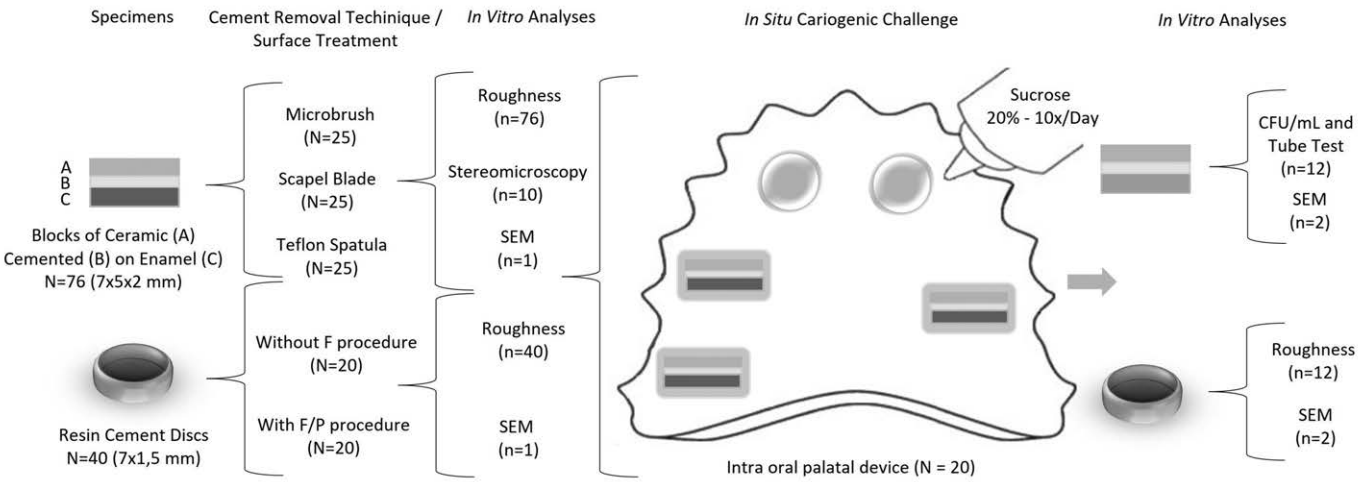

Figure 1. *Schematic illustration of the experimental design.*

For this *in situ* study, the sample size was calculated based on a previous study[15] in BioEstat 5.3 (Mamiraupa Sustainable Development Institute, Manaus, AM, Brazil), considering an α error of 0.05 and 0.8 statistical power. According to these parameters, a total of 17 volunteers were required to detect any significant differences. A final sample size of 20 volunteers was considered to compensate for possible outliers that could cause specimen loss.

Tooth Specimen Preparation

Eighty rectangular enamel slabs were obtained from extracted bovine incisors. The teeth were manually cleaned using periodontal curettes and a prophylaxis brush with pumice slurry and water. All cleaned teeth were stored in a 0.05% chloramine-T solution for disinfection.

The buccal surface of the tooth was ground with a silicon carbide paper (#600 and #1200) on a metallurgical polishing machine (METASERV 3000, Buehler, IL, USA) under constant water cooling. The tooth root was embedded into acrylic resin in a PVC mold (17×15 mm) to facilitate the handling. The tooth crown was longitudinally sectioned with a diamond saw (Isomet Diamond Wafering Blades - Buehler) in a low-speed precision cutting machine (Cutmaster Erios, São Paulo, SP, Brazil). The final dimensions of the enamel slab were obtained using diamond discs (7016, American Burs, Palhoças, SC, Brazil) mounted in a handpiece. The dentin was cut to obtain a block

Table 1: *Tested Materials, Composition, and Specifications*

Material (Color)	Composition	Manufacturer Batch Number
Duceram Kiss Bonding Porcelain –(A3)	Silicon Oxide, Aluminum Oxide, Potassium Peroxide, Sodium Oxide, Lithium Oxide, Barium Oxide, Boron Oxide, Calcium Peroxide, Titanium Oxide, Cerium Oxide, Tin Oxide, Phosphorus Oxide, Antimonious Oxide, Fluorine and Zirconium Oxide and pigments that are added in basic powders with variation between 1% and 10%	Dentsply Sirona Company (Hanau-Wolfgang, Germany) 118008
Tetric N Bond Universal	Methacrylates, ethanol, water, highly dispersed silicon dioxide, initiators, and stabilizers	Ivoclar Vivadent (Ontario, Canada) X25012
Variolink Esthetic LC (Light)	Monomers: BisGMA, UDMA, TEGDMA, HEMA, and GDMA (30 wt%) Inorganic Filler: ytterbium trifluoride and spheroid mixed oxide. Initiators, stabilizers, pigments and additional ingredients Filler loading (Wt%/Vol%)/size: (30%/38%)/0.04-0.2 µm	Ivoclar Vivadent (Ontario, Canada) Y05760

Abbreviations: Bis-GMA, bisphenol A glycidyl dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; and GDMA, glycidyl dimethacrylate.

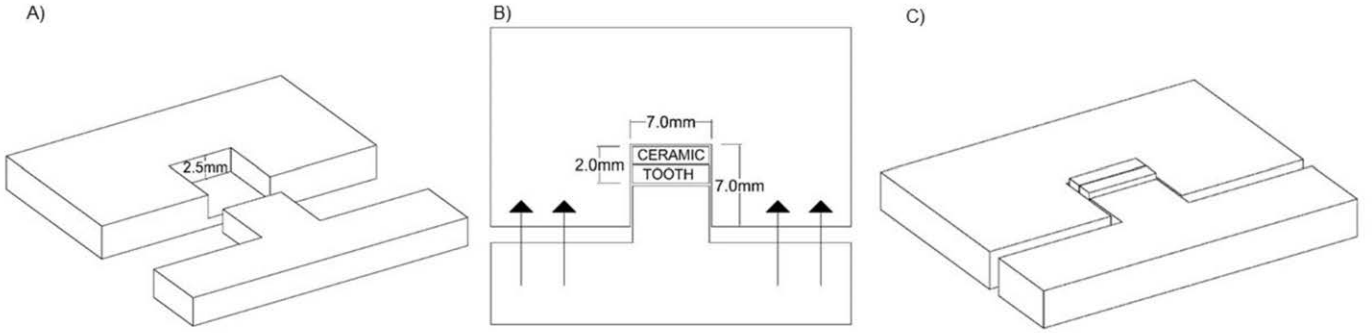

Figure 2. *Schematic illustration of the custom-made metal apparatus used for specimen fixture during cementation and cement removal technique. A) Side view of the metal apparatus; B) Front-view of the metal apparatus with the ceramic and teeth block. Black arrows indicate the direction of the parts; C) side view of the cemented block attached to the metal apparatus.*

with 7×2.5×2 mm using a digital caliper with 0.01-mm precision (Digimess, São Paulo, SP, Brazil). The slabs remained stored in distilled water at room temperature until the cementation procedure.

Ceramic Specimen Preparation

Eighty F blocks (Duceram Kiss Bonding Porcelain, Dentsply Sirona Company, Hanau-Wolfgang, Germany) were prepared according to the manufacturer's instructions. A rectangular stainless-steel split mold (25×2×2 mm) was filled in excess with the mixture, and the moisture was gently dried with absorbent paper. The ceramic blocks were submitted to a sintering cycle in an appropriate furnace (Multimat NTX Press, Dentsply). The blocks were sectioned using a handpiece with a diamond disc under constant water cooling to obtain the final dimension (7×2.5×2 mm) with the digital caliper. In addition to the cementation surface, a layer of glaze (InSync Glaze System, Chemichl AG Landstrasse, Vaduz, Liechtenstein) was applied onto each ceramic surface. The specimens were submitted to a second cycle in the furnace.

Ceramic Cementation and Cement Removal Techniques

Enamel surfaces were cleaned with pumice, and excess water was removed using an air-jet until dry. The cementation surface was etched with 5% hydrofluoric acid for 2 minutes (Condac Porcelana 5%, FGM Joinville, SC, Brazil), rinsed, and air-dried. The enamel surface was then actively etched with 37% phosphoric acid (Condac 37, FGM) for 30 seconds, rinsed, and air-dried. A layer of a silane coupling agent (Prosil, FGM) was applied onto the entire surface and left in contact for 2 minutes to promote water/alcohol evaporation.

A custom-made device was used to fix the specimens (Figure 2). Each enamel block was positioned and treated with 37% phosphoric acid for 30 seconds, rinsed for 15 seconds, and dried with air jets. An adhesive system (Tetric N Bond Universal, Ivoclar Vivadent, Ontario, Canada) was applied according to the manufacturer's instructions. A light-cured resin-based cement (Variolink LC Esthetic, Ivoclar Vivadent) was applied, followed by adapting the ceramic specimens.

A horizontal load was applied to fix the device, and maintain the correct position of the enamel and the ceramic blocks during excess cement removal (Table 2). Each specimen was cured at the marginal interface area using an LED light-curing unit (Radii-Cal, Dental Products, SDI, Baywater, Victoria, Australia) for 40 seconds with 1200 mW/cm² irradiance. After curing, the marginal interface of 10 specimens from each group was examined under a stereomicroscope (SteREO Discovery.V12, Carl Zeiss Microscopy GmbH, Jena, Germany) with 82× magnification.

Biodegradation of Resin Cement

The influence of polishing procedures on the biodegradation of resin cement materials was further examined. Briefly, 40 disc-shaped specimens (7×1.5 mm) were prepared. A Teflon mold was filled to excess with the resin cement, and a Mylar matrix strip under a microscope glass slab was placed on the top surface. Slight finger pressure was applied against the glass to minimize voids. Each cement specimen was cured at the central area, and the excess was removed using a sharp blade and silicon carbide papers (#600 and #1200).

The specimens were randomly allocated into two groups (n=20) according to the cement surface treatment: (1) no finishing procedure (light cured, Mylar strip) and (2) with finishing-polishing procedure by a single operator using Jiffy rubber points (Ultradent, South Jordan, UT, USA). Yellow and the white flame-shape points were used for 20 seconds each and then replaced after every five cycles.

Table 2: *Excess Cement Removal Technique*

Group	Removal Technique
Microbrush[A] (MBR)	A fine MBR was used in the cementation line in one direction before photoactivation (n=25)[D]
Scalpel blade[B] (SCP)	The excess of cement was displaced with a SCP after the first 5 seconds of curing and then continued the final photoactivation (n=25)
Teflon spatula[C] (TSP)	The excess of cement on the marginal interface was removed using a TSP before the photoactivation (n=25)

A: KG, Sorensen, Cotia, Brazil
B: Advantive (Sterilance, Sterilance Medical Inc. Suzhou, China)
C: Esthetic Plus, TDV, Pomerode, Santa Catarina, Brazil
D: Monowave LCU (Radii-Cal, SDI, Austrália)

Measurement of Surface Roughness

The R_a of the specimens was measured (μm) using a contact profilometer (SURFTEST SJ 310, Mitutoyo Corp, Kanagawa, Japan). For cemented blocks, the surface roughness was measured before the *in situ* phase. Ten successive in-line measurements were taken, with the needle in two different points of each predefined location: (1) ceramic surface; (2) ceramic surface, closer to the cement line; (3) cement line; (4) tooth, closer to the cement line, and (5) tooth surface (T). All measurements were performed in the specimen's long axis at a constant speed of 0.5 mm/s, with 0.7 load and 0.25 mm cut off.

For the biodegradation analysis, disc-shaped resin cement specimens were measured before and after the *in situ* phase. Three successive in-line traces were used to determine the mean surface roughness (R_a) from different angles. A trace length of 6.0 mm was used for both cemented blocks and cement disc specimens. A calibration step was performed periodically to monitor the device's performance.

Volunteer Selection

Twenty volunteers aged from 21 to 35 years, who were undergraduate and graduate dental students, participated in this study. The following inclusion criteria were considered: good systemic and oral health; no caries activity or any signs of gingivitis; and no use of antibiotics up to 2 months before the experimental phase or administration of any drugs that could affect salivary flow. Volunteers with poor oral hygiene, diagnosed with diabetes or chronic mouth breathing, with motor difficulties, palatal torus, denture use, or those wearing orthodontic appliances were not included in this study. A dentist carried out visual and oral examinations. All volunteers signed an informed consent form to authorize their participation. Before the experiment, the specimens were sterilized in a gamma radiation camera (25 kGy) for a period of 15 hours.

In Situ Experimental Phase

An acrylic custom-made palatal device was made for each volunteer. Each device contained five disc-shaped cavities (8×3 mm), to which three dental blocks and two cement specimens were fixed with wax (Figure 2). A plastic mesh was fixed over each cavity, maintaining a 1-mm space from the specimen surface to allow biofilm accumulation and to protect the specimens from mechanical disturbance.

During the 7-day experimental period, volunteers were instructed to brush their teeth with a regular fluoridated dentifrice three times per day (Colgate Maximum Cavity Protection—Palmolive Company, New York, NY, USA). There were no dietary restrictions during the experimental phase. The instructions were presented orally and written. Particular recommendations were given towards removing the device before eating or ingesting any food or beverages. In any case, the instruction was to keep the intraoral device constantly moistened in the plastic case provided by the authors.

The cariogenic challenge consisted of an extraoral application of one drop of a 20% sucrose solution onto each specimen 10 times per day at predetermined time intervals (8 am, 9:30 am, 11:00 am, 12:30 pm, 2:00 pm, 3:30 pm, 5:00 pm, 6:30 pm, 8:00 pm and 9:30 pm). The device was removed from the mouth, and excess saliva was cleaned with a gauze. Subsequently, a drop of sucrose was applied to the specimen. A 5 minute waiting period was established before the palatal device was repositioned in the mouth to enable sucrose diffusion into the biofilm.

After the experimental period, the devices were collected for further analysis. Cemented blocks were carefully removed from the devices and inserted into swab tubes (Absorve, Cral Artigos para Laboratório Ltda, San José, Cotia-SP, Brazil) containing 2 ml of Mueller Hinton broth. The disc-shaped specimens

were placed in tubes with sterile saline solution, washed in an ultrasonic bath for 30 minutes, and measured for their surface roughness.

Scanning Electron Microscopy (SEM)

One specimen from each group was selected for Scanning Electronic Microscopy (EVO LS 15, Carl Zeiss) analysis before and after the *in situ* phase. The specimens not submitted to *in situ* tests were dehydrated, dried (40°C/12 hours), and gold-sputtered (Q150T ES, Quorum Technologies Ltd, Laughton, UK) before SEM analysis. The specimens submitted to *in situ* tests were removed from the intraoral device and washed with 3 mL of sterile saline solution to remove nonadherent material from the surface.

Each specimen was placed in Eppendorf tubes containing a solution of glutaraldehyde (2.5%)/ paraformaldehyde (4%) in 0.1 M phosphate buffer (pH 7.2) for 2 hours at 4°C. The specimens were washed with the same solution and postfixed for 1 hour with osmium tetroxide in 0.1 M phosphate buffer (pH 7.2). Once again, they were washed and dehydrated with increasing concentrations of ethanol (30, 50, 70, 90, and 3× 100% for 30 minutes), dried using the critical point method, gold-sputtered, and observed under an SEM operated at 10kV with a working distance of 10 mm.

Colony-Forming Units Count (CFU/mL)

The cemented blocks from four volunteers were analyzed for CFU counting and biofilm formation. The specimens were removed from the swab, placed into tubes containing 2 mL of Mueller Hinton broth, and then sonicated for 30 seconds in a 50-60 W power ultrasonic homogenizer (Unique Ultrasonic Cleaner, USC-3300, São Paulo, SP, Brazil). A 1:1000 dilution was performed, and duplicate aliquots were seeded onto Muller Hinton agar. The plates were incubated at 37°C for 48 hours, and those containing 30-300 colonies were counted for CFU/mL.

Biofilm Formation

After CFU counting, bacterial colonies were also examined for their ability to develop a biofilm. Colonies were isolated from the specimens, and five colonies of each species were added to a Falcon tube containing 3 mL of saline solution. The tubes were vortexed, and the absorbance of the cell suspension was read at 600 nm (with a variation of 0.145-0.155). Then, 140 μL of Mueller Hinton culture medium, 20 μL of sterile distilled water, and 40 μL of the adjusted inoculum were added into a 96-well plate.

A standard bacterial colony was added as a biofilm starter (*Klebsiella pneumoniae*)—positive control. The absorbance was read at 600 nm at baseline (0 hour) and after 24 hours of incubation at 37°C. The supernatant was removed, and the plate was washed three times with sterile saline solution (0.85%) and then dried in an oven at 60°C for 60 minutes. Next, 200 μL of a violet crystal (0.4%) was added to the wells, and the plate was kept at room temperature for 15 minutes, followed by three washes under running water. Finally, 200 μL of ethanol (PA) was added to the wells, and the plate was kept for an additional 30 minutes at room temperature. The wells' absorbance was read at 570 nm, and the optical density was calculated and interpreted as follows: nonadherent, weakly adherent, moderately adherent, and strongly adherent, according to the methodology proposed by Stepanović and others (2000).[19]

Statistical Analysis

The data were analyzed descriptively and inferentially in SPSS version 21.0 (IBM Corporation). Shapiro–Wilk test was used to check for the normality of data distribution. Kruskal–Wallis test determined the difference between the groups, and the Mann–Whitney test was applied when significant differences were observed. In all tests, the significance level was set at $\alpha = 0.05$.

RESULTS

Analysis of Surface Roughness

Twenty volunteers were selected for this study, but only 18 completed the experimental phase. Two volunteers did not complete the established protocol and were excluded from the analysis. Surface roughness (R_a) measurements of the ceramic material after cementation are described in Table 3. Significant differences were observed between the techniques regarding the cement line ($p<0.001$), the area between the cement and the tooth surface ($p=0.002$), and the tooth surface ($p=0.003$). The mean roughness between the ceramic–cement area was nearly significant ($p=0.054$). The SCP removal technique produced the highest mean roughness, regardless of the surface area. Figure 3 shows the characteristics of the surfaces of different specimens, according to stereomicroscopy and SEM analysis.

Table 4 shows the contribution of surface finishing and polishing to the biodegradation of the resinous cement. The specimens without finishing procedure showed a significantly lower initial mean roughness (0.07 μm), which may be due to the Mylar strip's smoothness. However, after the *in situ* phase, this group showed a significant increase in surface roughness ($p<0.001$). For the specimens submitted to finishing and polishing procedures, no statistical difference was observed between evaluation periods ($p=0.148$).

Table 3: *Mean (SD) Roughness on the Surface and Interface in µm Following the Three Cement Removal Technique*[a]

Group	MBR	SCP	TSP	*p**
Ceramic surface	0.68 (0.38)	0.53 (0.25)	0.54 (0.32)	0.145
Ceramic/Cement	0.60 (0.33)	0.99 (0.61)	0.69 (0.37)	0.054
Cement line	0.86 (0.39) A	1.39 (0.42) B	0.97 (0.44) A	<0.001
Cement/Teeth	0.67 (0.29) A	1.30 (0.66) B	0.74 (0.54) A	0.002
Teeth surface	0.60 (0.28) A	0.49 (0.30) B	0.37 (0.18) C	0.003

Abbreviations: MBR, microbrushes; SCP, scalpel blades; TSP, Teflon spatula.

[a] *Different letters indicate statistical significance between groups through Mann–Whitney test. Uppercase letters indicate differences in each removal technique within the surface.*

**Kruskal–Wallis.*

Surface Micromorphology

Figure 3 shows images of the ceramic surface after block cementation for each cement removal technique. Excess cement can be seen at the cementation line in Figures 3A and 3B in specimens submitted to the MBR technique, with the presence of some irregularities and flaws (*red arrows*) in this area. The excess cement remaining after the use of SCP (3C and 3D) covered most of the feldspathic ceramic and tooth surface (*blue arrows*). The TSP removal technique (3E and 3F) seemed to have produced a smoother surface (*green arrow*), with fewer irregularities at the cementation line.

SEM images of the cement specimen submitted to *in situ* biodegradation are shown in Figure 4. The unpolished cement specimen (4A) showed rougher surface areas before the *in situ* phase and, therefore, exhibited a higher adhesion of bacterial colonies (4C). The polished specimen (4a) showed a smoother surface and promoted less bacterial adhesion after the *in situ* phase (4b and 4c).

CFU/mL Counting

The mean (±SD) CFU/mL (Log_{10}) is shown in Table 5. There was no statistical difference between the groups after the *in situ* phase (p=0.96).

Analysis of Biofilm Formation

The CFU counts of the specimens from four volunteers were determined, and volunteer number 2 showed the highest amount of isolated bacterial species (n=8). Table 6 shows the number of isolated bacterial species, biofilm formation analysis, and the Gram staining procedure for each strain.

When excess cement was removed using a MBR, two bacterial species were recovered from the specimens, as per the violet crystal technique. Both species were found to be Gram-negative and had a strong and moderate ability to form a biofilm.

When excess cement was removed using a TSP or a SCP, three bacterial species were recovered. Two species in the TSP group showed a strong potential

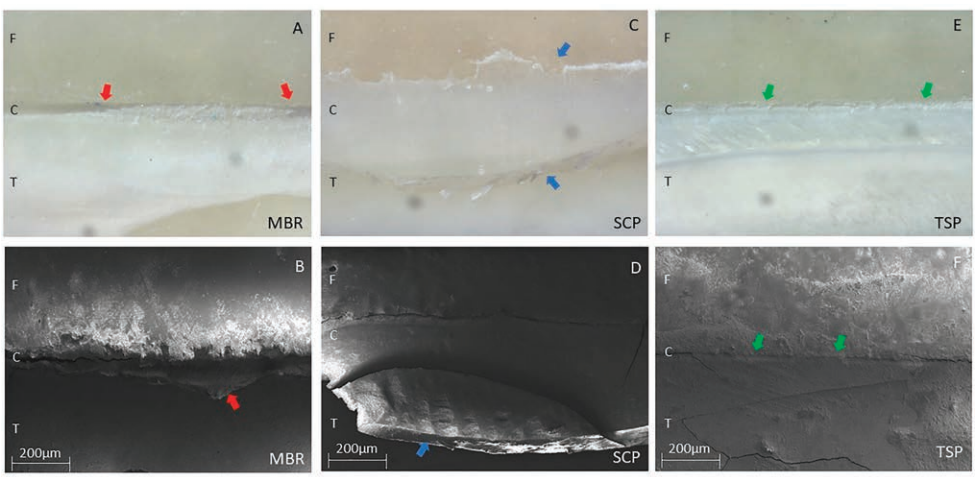

Figure 3. *Images A, C, E) in stereomicroscopy (82×); B, D, F) in SEM (60×) for the cement removal technique. (A/B) red arrow indicates the presence of irregularities and flaws at the cement line after cement removal with MBR; Blue arrow shows the excess of cement left after the use of SCP covering the feldspathic ceramic (F) and the tooth surface (T) (D/D). A more defined interface (green arrows) was observed after cement removal with TSP (Figures E and F).*

Table 4: *Mean (SD) Surface Roughness of Resin Cement Specimens in µm, Before and After Biodegradation*[a]

Group	Before	After	*p*[b]
Without finishing procedure	0.07 (0.02) Aa	0.36 (0.12) Ab	<0.001
With finishing/polishing procedure	0.19 (0.09) Ba	0.43 (0.22) Ba	0.148
p[c]	<0.001	<0.001	

[a] *Different letters indicate differences between groups.*
[b] *Wilcoxon signed-rank test was used to compare roughness in different moments of observation (lowercase letters in lines).*
[c] *According to the Mann–Whitney test (uppercase letters in columns).*

to form biofilm and were found to be Gram-negative, whereas one species showed a weak ability to form a biofilm. As for the SCP group, two species showed a moderate potential for biofilm formation, and another one showed a strong ability to do so. One of the species with moderate potential for biofilm formation was found to be gram-positive.

DISCUSSION

In our study, the cement removal technique did not significantly affect bacterial adhesion to the ceramic material, which confirms our first hypothesis. The results showed that bacterial adhesion was not associated with the excess cement removal technique. A previous study showed that surface roughness of up to 0.2 µm would accumulate less biofilm.[8] However, a recent systematic review[9] showed that a reduction in surface roughness (less than 0.2 µm) had no further impact on supra- or subgingival bacterial adhesion or biofilm composition compared to R_a above 0.2 µm, which is in agreement with others findings.[20-25]

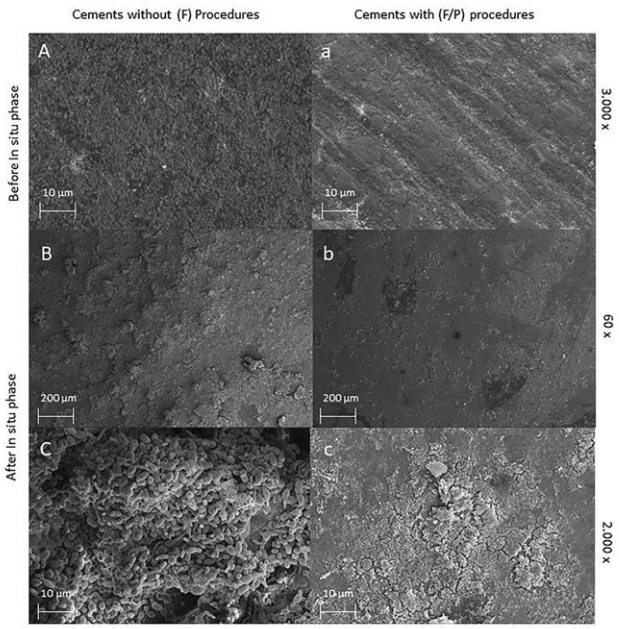

Figure 4. *SEM images of cement samples without finishing procedures (A,B,C) and cements sample with finishing-polishing procedures (a,b,c) in different magnifications.*

The bacterial adhesion was determined by analyzing the CFU/mL count. Several parameters may influence the bacterial adhesion, such as factors related to the microenvironment, surface characteristics, and the bacteria itself.[26] Among the factors related to the surface, surface roughness is one of them. However, in the present study, despite differences in surface roughness between groups, no differences were observed in the formation of CFU/mL. The surface roughness of each surface (tooth, resin cement, and ceramic) showed R_a means higher than 0.2 µm (Table 3), regardless of the removal device used.

In vitro studies previously demonstrated a significant association between the cement removal technique and bacterial adhesion onto the restorative material.[2,12] According to Anami and others,[12] the TSP technique showed the highest R_z value (arithmetic mean between the five highest peaks and five deepest valleys within a specific length), in addition to greater bacterial adhesion and biofilm volume. Pereira and others[2] found that the MBR removal technique was associated with lower CFU counts.

The clinical longevity of restorations is influenced by resin cement physical and mechanical properties and its ability to adhere to the dental structures. The outcomes of an *in vitro* study are generally more limited, because some factors are controlled, such as the type of bacterial inoculation, temperature, pH, and nutritional status.[9] Instead, *in situ* study designs are more versatile and can be used for various analytical purposes, such as assessing erosive or cariogenic potential.[27] Clinical and biological aspects such as temperature changes, salivary composition, and pH can contribute to the degradation phenomenon.[3] On the other hand, these factors may also be considered a limitation of *in situ* studies, as the oral milieu and the microbiome itself are specific to each volunteer.

For *in situ* studies, the cariogenic challenge's acceleration is commonly undertaken using 20% sucrose solutions 4×,[27] 8×,[28] or even 10× daily.[15] The time

Table 5: *Mean (SD) of Colony-forming Units (CFU/mL) log_{10} After* In Situ *Phase (n=4)*

Group	CFU/mL log_{10}	*p**
MBR	5.29 (0.19)	0.96
SCP	5.24 (0.18)	
TSP	5.26 (0.18)	

Abbreviations: MBR, microbrushes; SCP, scalpel blades; TSP, Teflon spatula.
**Kruskal–Wallis*

established for the duration of an *in situ* experiment is also highly variable. *In vitro* studies assessing bacterial adherence and colonization may have a duration of 24[12]-48 hours.[2] However, biofilm formation and maturation depends on the cohesion and coaggregation of different species and gene expression.[29] After 7 days, a climax community is established, having a dynamic balance with minor variations in species composition and proportion.[30] While extended *in situ* observation periods have been considered in the literature,[16] participant adherence to the protocol established in our study for more extensive periods may prove challenging to achieve and may be considered a limitation of our study.

The interaction of *Streptococcus mutans* with the surface of resinous materials promotes biodegradation. Organic acids produced by bacterial metabolism change the oral environment's pH (from 7.3 to 4.0), which may affect the surface of resinous materials.[31] An *in vitro* study[29] examined bacterial adhesion on the surface of resin composites using a 4 hour protocol. The authors found that early colonization of bacterial species is considered an essential factor for biofilm formation and maturation. Also, topographic characteristics and material composition affect only early bacterial adhesion but not biofilm maturation.[32-34]

The tube test is the most frequently used method to measure biofilm formation. Biofilm cultures may be formed on a culture tube and stained with a cationic dye or grown in a microtiter plate. The optical density of stained biofilm is assessed using a spectrophotometer.[19] The classification used herein to determine bacterial biofilm formation was based on a study of Christensen and others.[35] Here, all isolated species were adherent, and classified as moderate and strong biofilm-forming microorganisms, except for one species recovered from the TSP group, which showed a weak ability to form a biofilm.

The second tested hypothesis was that the cement polishing technique does not affect biodegradation in the oral environment. This hypothesis was rejected, as statistically significant differences were observed between baseline and final roughness measurements when no surface polishing was performed. Such a difference was not observed in the specimens submitted to finishing and polishing procedures. This phenomenon is frequently observed when metabolic acids from cariogenic bacteria cause surface damage, such as corrosion and increased roughness of restorative materials, but no *in vitro* test can reproduce the complex process of biodegradation.[36,37] Lactic acid is the most critical product metabolized by cariogenic bacteria, such as *S. mutans*, in the presence of sucrose.[38] However, the pH conditions in an *in vitro* environment may differ from those observed in oral conditions.

Table 6: *Analysis of Bacterial Biofilm Formation and Gram Test of the Isolated Bacteria in Each Sample Analyzed*

	Removal Technique	Biofilm Formation	Gram Staining
1	MBR	Strong (+++)	-
2		Moderate (++)	-
3	SCP	Moderate (++)	+
4		Moderate (++)	-
5		Strong (+++)	-
6	TSP	Strong (+++)	-
7		Weak (+)	-
8		Strong (+++)	-

Abbreviations: MBR, microbrushes; SCP, scalpel blades; TSP, Teflon spatula.

Although no differences in roughness measurements were observed before and after the polished specimens' cariogenic challenge, this does not imply that there was less bacterial adhesion. Other factors, such as the material's surface free energy, may also directly affect biofilm formation,[7,20] which could be confirmed in the micrographs shown in Figure 4. At the same magnification (2000×), more significant colonization of microbial species was observed than the specimens submitted to finishing and polishing procedures.

A positive correlation between increased surface roughness and bacterial adhesion was observed,[12,22,39,40] to the extent that it can even exceed other properties' influence, such as surface free energy.[32] Although the recommended (low) mean roughness measurement was obtained at baseline (<0.2 μm), polished cement specimens showed an increase in surface roughness over time due to the biodegradation of the polymeric matrix.[8]

The chemical composition of resinous materials is important for bacterial colonization. Monomer

polymerization is not fully complete, and approximately 5%-10% of unpolymerized content can be eluted. Some components present on the surface can favor or impair bacterial adhesion. The literature shows that the monomers ethylene glycol dimethacrylate (EGDMA) and triethylene glycol dimethacrylate (TEGDMA) are more easily released. These monomers can be used as carbon sources by anaerobic bacteria and are also known to increase cariogenic bacteria's viability.[30]

The Variolink resin cement contains bisphenol A glycidyl dimethacrylate (*Bis*-GMA), urethane dimethacrylate (UDMA), TEGDMA, 2-hydroxyethyl methacrylate (HEMA), and glycidyl dimethacrylate (GDMA) (30% wt) in its organic matrix composition. TEGDMA is a molecule that absorbs more water than *Bis*-GMA, leading to this material's higher solubility. In contrast, TEGDMA can modulate bacterial growth[41] and reduce surface degradation caused by acid exposure.[42]

The polishing procedure aims to improve the esthetic characteristics and durability of resinous materials by decreasing surface porosity and improving mechanical properties.[43] Furthermore, the organic matrix is removed, and exposure of inorganic particles avoids early degradation.[11]

Clinicians may choose to use more than one device for excess cement removal. However, the present study did not evaluate this synergistic effect. The combination of cement removal methods could lead to smoother surfaces, although time consuming. If the combination of methods is chosen, clinicians must be aware of maintaining the ceramic laminates in position, avoiding pressing and loosening the laminate to the prepared tooth, therefore, avoiding more outflow of the resin cement. Independent of solo or combined use, from our results, final polishing has shown a significant impact on the surface roughness of the resin cement. Further *in situ* studies are encouraged to determine the behavior of different resinous cements and preheated resin composites as luting agents for indirect restorations.

CONCLUSIONS

To conclude, our findings suggest that the three techniques used for cement removal increased the surface roughness of ceramic laminates, particularly with the scalpel blade (SCP). Still, they did not affect bacterial adhesion at the marginal interface. Finishing and polishing procedures at the cement interface should be periodically performed to minimize the biodegradation of the resinous interface.

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Regulatory Statement

This study was previously approved by the Institutional Research Ethics Committee, under protocol number 3.201.874. The approval code issued for this study is 08553219.3.0000.5207.

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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Surface Treatment and Cementation of Lithium Silicate Ceramics Containing ZrO_2

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CLINICAL RELEVANCE

Hydrofluoric acid followed by silanization or Monobond Etch & Prime is an efficient option for the cementation of lithium silicate and lithium disilicate glass ceramics.

SUMMARY

Objective: To evaluate the effect of different surface treatments on the shear bond strength (SBS) of lithium silicate (LS) and lithium disilicate (LD) ceramics, after thermocycling.

Methods and Materials: For SBS test, 72 ceramic blocks (18×14×2 mm) were made (24 blocks from each ceramic material): VITA Suprinity (LSS), Celtra Duo (LSC), and Lithium disilicate (LD). The blocks were polished with sandpaper of increasing grit (#280, #400, #800, and #1200) and embedded in chemically activated acrylic resin. Afterwards, they were randomly divided into 12 groups (6 blocks per group) according to: "Ceramic" (LD, LSC, and LSS) and "Surface treatment" (HFS: hydrofluoric acid + silane; MEP: Monobond Etch & Prime/Ivoclar). From each treated surface ceramic block, four dual-curing resin cement cylinders (RelyX U200, 3M Oral Care) were prepared using a Tygon tube (Ø=3 mm and h=2 mm) and light cured for 40 seconds (1000 mW/cm²) (N=288/n=24). All specimens were submitted

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to thermocycling (10,000 cycles, 5°C and 55°C, 30 seconds) and then to SBS test at a crosshead speed of 1 mm/min using a 50-kgf load cell. Forty-five additional blocks were made for roughness and SEM analysis. Failure mode was also performed. The data (MPa) were statistically analyzed by one-way analysis of variance (ANOVA), Tukey test (5%), and Weibull analysis. The R_a was analyzed by Kruskal–Wallis and Dunn Test (5%). The other variables were analyzed qualitatively.

Results: ANOVA revealed that "surface treatment" was significant for all ceramic materials ($p<0.05$). The LD-HFS (18.66±3.49), LSC-HFS (16.81±2.62), and LSS-HFS (16.33±3.08) groups had significantly higher SBS than the LD-MEP (7.00±4.2), LSC-MEP (14.12±3.51), and LSS-MEP (13.87±2.52) groups. Complete adhesive failures at the cement–dentin interface were more frequent. Weibull modulus was superior for the LD-HFS (6.22), LSC-HFS (8.8), and LSS-HFS (7.4) groups.

Conclusion: HF followed by silanization is the most suitable surface treatment for the cementation of LS and LD glass ceramics.

INTRODUCTION

Lithium silicate (LS) ceramics are presented, within the class of glass ceramics, for making indirect restorations, such as inlays,[1-3] onlays,[2,3] overlays, and crowns,[2-4] and according to manufacturers, provide a lower probability of fracture compared to conventional glass ceramics, and are also associated with high aesthetics.[3,5,6] Commercially, two types of LS ceramic materials are available, Vita Suprinity (Vita Zahnfabrik—Bad Sackingen, Germany) and Celtra DUO (Dentsply—Hanau Wolfgang, Germany), both of which are mainly made up of submicrometric lithium metasilicate (Li_2SO_3) crystals and orthophosphate nanometer lithium (Li_3PO_4) embedded in a glassy matrix with highly dispersed zirconium dioxide (ZrO_2) (±10 wt%).[2] The main difference between the two materials is the size of the Li_2SO_3 crystals (Li_2SO_3 phase), which appears to be larger in the Celtra Duo (up to 1 μm in length) than in the Suprinity (~0.5 μm).[2,7] Although it is reported that these ceramics are reinforced by zirconia,[5,6,8] it is not in its crystallized form, thus they are considered only glassy ceramics based on LS.[2]

Lithium disilicate (LD) ceramics, on the other hand, are composed in their crystallized phase by crystals of LD embedded in a glassy matrix and have a higher percentage of crystalline phase content (70%) compared to LS (40%-50%).[7] Despite the microstructural differences between these two types of ceramics, some studies report high clinical success rates, both for LS ceramics, reaching 98% for crowns or inlays after 3 years of follow-up,[9] and for LD ceramics that vary from 94.8% after 8 years[10] to 83.5% after 10 years.[11] However, failures in restorations such as fractures and debonding are still common regardless of the type of material, especially in clinical situations in which the substrate does not offer mechanical retention; also the cementation technique has a fundamental role in the clinical longevity of these indirect restorations.[9]

With regard to adhesion, *in vitro* studies have investigated different protocols for the surface treatment of these ceramics.[5,12] Among surface treatments, hydrofluoric acid etching (HF) followed by silanization has been proposed as the ideal treatment for all-glass ceramics.[13,14] However, some studies have reported that this protocol has some disadvantages, such as the high toxicity of HF,[5,15-17] different acid concentrations and variations in conditioning time between materials, besides difficulty in controlling the restoration exposure to acid, which can lead to overconditioning of the piece, most of the time decreasing the mechanical resistance of restorations[7,17-19] or impaired adhesion due to excessive glassy phase dissolution in some materials.[20,21] Thus, surface treatment alternatives to HF have also been investigated, such as airborne-particle abrasion with aluminum oxide (Al_2O_3),[5,22] silicatization associated with silanization,[5,23,24] or self-etching ceramic primer, such as Monobond Etch & Prime (MEP, Ivoclar Vivadent).[17,25]

A recent clinical option for conditioning and silanization in glass ceramic restorations is the self-etching ceramic primer (MEP). Because it contains in its single-bottle composition an aqueous acidic solution of ammonium polyfluoride and silane methacrylate, this primer allows the etching and silanization in a single step.[26] According to studies, this primer decreases the probability of excessive degradation of the silica glass matrix, and the toxic effect of HF, as well as presents a satisfactory clinical performance[17,27] and clinically stable adhesion.[28]

Despite several studies investigating glass ceramic surface treatments, there are still few studies that have used other surface treatments as an option to the use of HF, especially with MEP in LS ceramics. Associated with this, most studies, when investigating these protocols, did not use aging of the adhesive interface through thermocycling,[5,29] which has great clinical implications in long-term adhesion. Therefore, this study aimed to evaluate the effect of different types of surface treatments on the shear bond strength (SBS) of LS and LD ceramics to resin cement after

thermocycling. The hypotheses tested were: 1) the type of surface treatment does not influence the bond strength regardless of the type of ceramic; 2) the surface treatment influences the surface roughness, regardless of the type of ceramic.

METHODS AND MATERIALS

The materials used in this study, as well as their respective trademarks, manufacturers, and batches, are shown in Table 1. The flowchart of the design of this research is shown in Figure 1.

Production of Specimens

Computer-aided design—Computer-aided manufacturing (CAD–CAM) blocks of three ceramics (18×14×12 mm): Celtra Duo (LSC) (Dentsply—Hanau-Wolfgang, Hesse, Germany), VITA Suprinity (LSS) (Vita Zahnfabrik—Bad Sackingen, Baden-Württemberg, Germany), and IPS e.max CAD (LD) (Ivoclar Vivadent, Schaan, Liechtenstein) were sectioned on a precision saw (Isso Met 1000 Precision Saw, Buehler, Lake Buff - IL, USA) under constant irrigation, using Extec High Concentration Diamond Wafering Blades (Extec, Enfield, CT, USA), in smaller rectangular blocks (18×14×2 mm) for a total of 63 rectangular blocks (ISO/TS 11405). These blocks were polished with SiC abrasive papers (#280, #400, #800, and #1200, Norton Saint-Gobain, São Paulo, Brazil) and sintered according to the recommendation of each manufacturer in a specific oven. Thirty-six of these blocks were used for the SBS test and 27 blocks (9 blocks of each material) were used for Optical profilometry and Scanning Electronic Microscopy (SEM).

Preparation of Blocks

The blocks were embedded in chemically activated acrylic resin (Classic, São Paulo, Brazil), in a PVC tube (¾ inch). The exposed surface was covered with double-sided tape in order to avoid the covering of this surface by the resin.

After polymerization, all the blocks were ultrasonically cleaned with distilled water for 10 minutes (Vitasonic

Table 1: *Trademarks, Type of Material, Composition, Manufacturers, and Batch Numbers of Products Used in the Study*

Trademark	Type of Material	Composition	Manufacturer	Batch
Celtra DUO HT	Lithium silicate	Fully sintered lithium silicate/phosphate (LSP) glass-ceramic (SiO_2, P_2O_5, Al_2O_3, Li_2O, K_2O, ZrO_2, CeO_2, Na_2O, Tb_4O_7, V_2O_5, Pr_6O_{11}, Cr, Cu, Fe, Mg, Mn, Si, Zn, Ti, Zr, Al).	Dentsply, Hanau Wolfgang, Germany	18031266
Vita Suprinity HT	Lithium silicate	Lithium silicate glass-ceramic (SiO_2, Li_2O, K_2O, P_2O_5, Al_2O_3, ZrO_2, CeO_2)	Vita Zahnfabrik, Bad Säckingen, Germany	40020
IPS e.max CAD HT	Lithium disilicate	SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO and other oxides	Ivoclar Vivadent, Schaan, Liechtenstein	W04573
RelyX Ceramic Primer	Silane	Ethyl alcohol, water, methacryloxypropyltrimethoxysilane	3M, St Paul, MN, USA	N822741
Porcelain Etch 9%	9% Hydrofluoric acid	Hydrofluoric acid, water, thickener, surfactant, coloring	Ultradent, South Jordan, UT, USA	18005525512
Monobond Etch & Prime	Self-etching ceramic primer	Butanol, tetrabutylammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, trimethoxypropyl methacrylate monomer	Ivoclar Vivadent, AG, Schaan, Liechtenstein	V50443
Rely X U200	Self-adhesive resin cement	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers; Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers	3M, St. Paul, MN, USA	660958

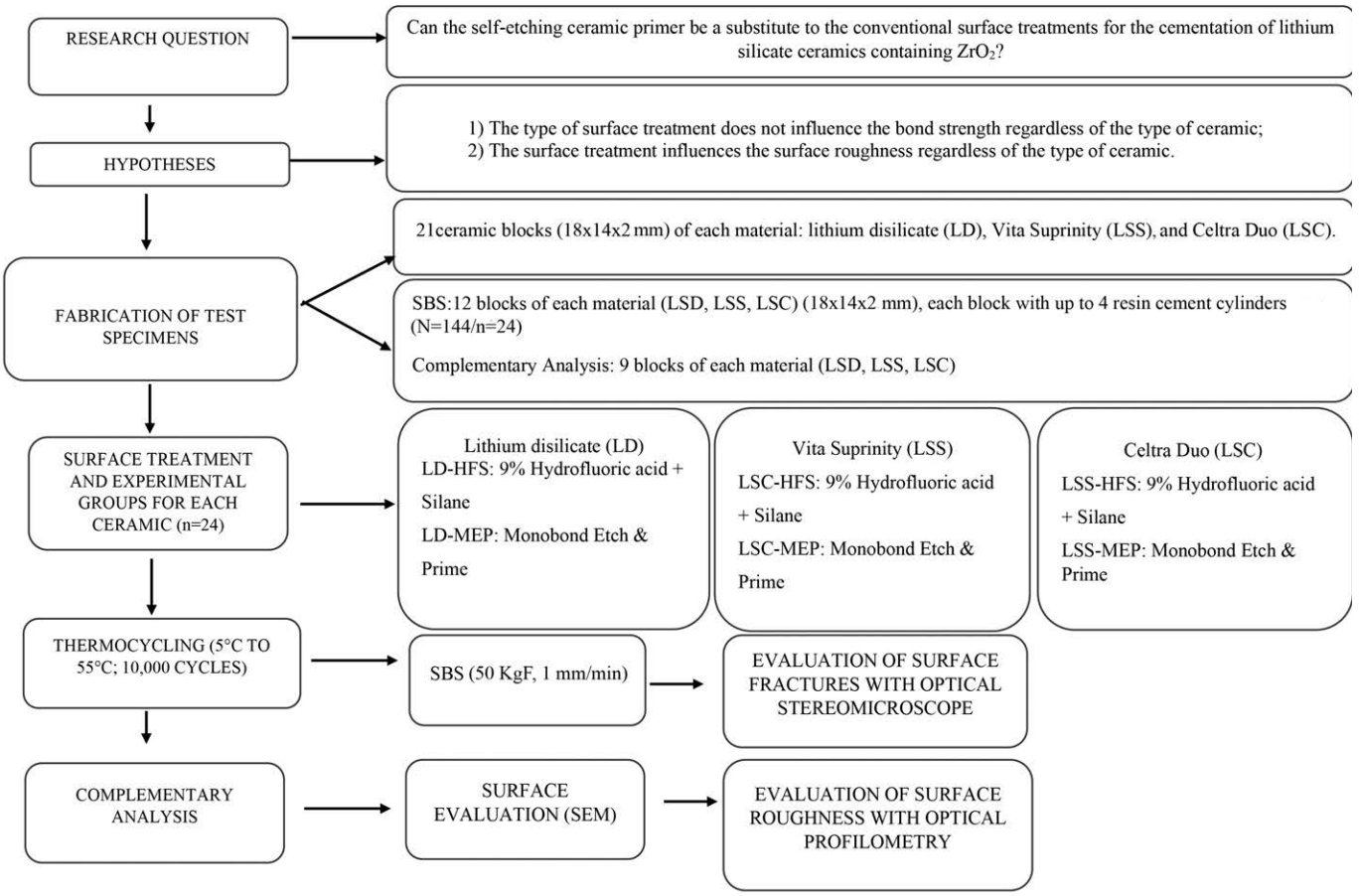

Figure 1. *Flowchart of study protocol. SBS: Shear Bond Strength. Abbreviations: TC, thermocycling; SEM, scanning electronic microscopy; LD, lithium disilicate; LSS, Vita Suprinity; LSC, Celtra Duo; SBS: Shear Bond Strength.*

II, Vita Zahnfabrik – Bad Sackingen, Baden-Württemberg, Germany) and divided into six groups (6 blocks per group). On each ceramic block, four resin cement cylinders were built-up to complete the 24 cylinders per group (N=144/n=24). The groups were divided according to: "glass ceramic" (LSS, LSC, LD) and "surface treatment" HFS: 9% Hydrofluoric acid (Ultradent—South Jordan, UT, USA) + RelyX Ceramic primer (3M Oral Care - St. Paul, MN, USA); MEP: Monobond Etch & Prime (Ivoclar Vivadent—Schaan, Liechtenstein).

Surface Treatments

Prior to surface treatments, all specimens were washed in an ultrasonic bath (Vitasonic II, Vita Zahnfabrik) in 99.8% isopropyl alcohol (Pharmacy of Homeopathy and Manipulation - Juiz de Fora - MG - Brazil) for 8 minutes. The surface treatments of the specimens were carried out by a single operator, on the entire surface of the block, and occurred according to the groups shown in Figure 1 as follows:

- 9% hydrofluoric acid (HFS): 9% Hydrofluoric acid was applied to the surface for 20 seconds for all ceramics, and then washed with air and water spray for 40 seconds, as instructed by the manufacturer. Then, a layer of RelyX Ceramic Primer was applied with the aid of a microbrush (Vigodent, Rio de Janeiro, RJ, Brazil), according to the manufacturer's recommendations.
- Monobond Etch & Prime (MEP): Monobond Etch & Prime was applied for 20 seconds by active friction with a microbrush, waiting 40 seconds for the material action, and then washed with air and water spray for 60 seconds. Finally, they were air dried until the moisture was eliminated. Due to the properties of this conditioning agent and the manufacturer's recommendations, no silane was applied afterwards.

Adhesive Cementation

After the surface treatment, up to four resin cement cylinders (n=24) (RelyX U200 (3M Oral Care) (Ø=3.0

mm, height: 2 mm) were made on the surfaces of the treated ceramics. For the standardization of the diameter of the adhesive area and the height of the cement increment, a matrix of flexible silicone tubing (Tygon tubing, Saint-Gobain Performance Plastic, Miami Lakes, FL)[30] was used. To fix the Tygon, a layer of wax n°7 (New wax, Technew, Rio de Janeiro, Rio de Janeiro, Brazil) was used with the aid of an electric dripper (Plaster, Caxias do Sul—RS, Brazil). Afterward, each resin cement cylinder was individually light cured for 40 sec (VALO/Ultradent) in the standard mode power energy (1000 mW/cm²). The power of the light–curing unit was measured by a single operator with a radiometer for LEDs (LED Radiometer–Kondortech, São Carlos, SP, Brazil). After photoactivation of each specimen, the Tygon was gently removed after 8-10 minutes, using a #15 scalpel blade, and then each specimen was light cured again, following the same previous protocol.

Thermocycling (TC) and Shear Bond Strength Test (SBS)

All specimens were subjected to thermal aging by means of a thermal cycler (521-D—Ethik Tecnology/Nova Ética—Vargem Grande Paulista—SP) with 10,000 cycles in distilled water at 55°C and 5°C for 30 seconds each, with an exchange interval of 5 seconds. Afterwards, the specimens were submitted to the mechanical shear test (SBS), in a universal testing machine (EMIC—Instron, São José dos Pinhais, PR, Brazil). The load was applied at the base of the cylinder on the adhesive interface using an orthodontic wire (0.4-mm diameter) at a speed of 1 mm/minute and load cell of 50 kgf until fracture of the specimen. The adhesive strength was calculated using the formula: $R = F/A$, where R = Adhesive strength (MPa); F = Force (N); A = Interfacial area (mm). The adhesive area of each block was defined by the area of a circle, calculated by the following formula: $A = \varpi r^2$, where $\varpi = 3.14$ and $r = 1.5$ mm.

Failure Analysis

After SBS testing, failure pattern analysis was performed on all specimens with a stereomicroscope (Carl Zeiss—Oberkochen, Germany) at 40× magnification, determining fractures classified as follows: A) Adhesive in ceramic–resin cement interface; C1) cohesive in ceramic; C2) cohesive in resin cement; mixed 1 (M1) adhesive in ceramic–resin cement interface + cohesive in resin cement; mixed 2 (M2): In ceramic–resin cement interface + cohesive in ceramic.

Optical Profilometry

Twenty-seven additional ceramic blocks, nine of each material (LSS, LSC, and LD), were prepared as previously described and subjected *to* the following surface treatments (n=3): HF, MEP, and control group (no treatment). Subsequently, the blocks were examined using a digital optical profilometer (Wyko, NT 1100, Veeco—Tucson, USA), connected to a computer with imaging software (Vision 32, Veeco—Tucson, USA) for obtaining 20× surface micrographs [qualitative analysis of three-dimensional (3D) geometry], and measurement of surface roughness (R_a). Five readings were performed on each specimen and an arithmetic mean (R_a) of the surface roughness was obtained using the proper system software.

Scanning Electron Microscopy (SEM)

The same surface specimens were examined at 2500× magnification in a TESCAN Scanning Electron Microscope (MEV-FEG, Model MIRA 3, Kohoutovice, Czech Republic) in high vacuum with the aid of a secondary electron Everhart–Thornley detector (ETD).

Statistical Analysis

The sample power calculation performed in this study used the mean and standard deviation of the groups and for this reason, it was performed after the SBS test. The following data were inserted for this calculation: confidence interval: 95%, the mean and standard deviation of the group that presented the higher mean, the mean and standard deviation of the group that presented the lower mean, and the number of tested specimens by group (Table 2). The data obtained from SBS were submitted to the statistical model of analysis of variance, after considering the distribution of residues (Levene test) using the Minitab software (Minitab, version 17, 2013). The residual values, resulting from the adjustment of the adopted model, were examined to assess the suitability of the model for valid statistical inferences, and it was determined that the original data provide an adequate adjustment when they adjusted to a normal probability distribution ($p>0.05$).

Analysis of variance (ANOVA—one way) and Tukey test (5%) were performed for the SBS test for each ceramic individually using the Statistix software (Analytical Software Inc., version 8.0, 2003). Shapiro–Wilk test was used to verify the normality of numerical roughness data, resulting in a nonparametric distribution ($p<0.05$). For these data, the Kruskal–Wallis test and Dunn multiple comparison test were performed using the GraphPad Prism software (GraphPad Software, San Diego, CA, USA). The probability value $p<0.05$ was considered as statistically significant. The failure analysis, SEM, EDS of the surface treatments were carried out through qualitative descriptive analyses.

Weibull analysis was performed to evaluate the

Table 2: *Number (N) and Percentage (%) of Pretest Failure (PTF) During Thermal Aging, Total Number of Specimens Submitted to the Shear Test and Failure Mode (%) of the Groups After SBS Test*

Ceramic	Surface Treatment	Groups	Number of Specimens	Number and Percentage of Spontaneous PTF During Aging	Number and Percentage of Tested Specimens	Percentage by Failure Mode					
						A	C1	C2	M1	M2	Total
Lithium Disilicate (e.max CAD)	HF+Silane	LD-HFS	24	0(0%)	24(100%)	100	—	—	—	—	100%
	MEP	LD-MEP	24	17(70.84%)	7(29.16%)	100	—	—	—	—	100%
Lithium Silicate (Suprinity)	HF+Silane	LSS-HFS	24	0(0%)	24(100%)	12.5	—	—	—	87.5	100%
	MEP	LSS-MEP	24	2 (8.33%)	22 (91.66%)	70.8	—	—	—	29.1	100%
LithiumSilicate (Celtra Duo)	HF+Silane	LSC-HFS	24	0(0%)	24 (100%)	12.5	—	—	—	87.5	100%
	MEP	LSC-MEP	24	5(20.83%)	19 (79.16%)	70.8	—	—	—	29.1	100%

Abbreviations: LD, Lithium disilicate; LSS, Vita Suprinity; LSC, Celtra Duo; HFS, 9% Hydrofluoric acid + Silane RelyX Ceramic primer; MEP, Monobond Etch & Prime; A, Adhesive in ceramic–resin cement interface; C1, Cohesive in ceramic; C2, Cohesive in resin cement; M1, Mixed 1, adhesive in ceramic–resin cement interface + cohesive in resin cement; M2, Mixed 2, adhesive cement–ceramic + cohesive ceramic.

reliability of the SBS, with the Weibull parameter (m) and the characteristic strength (σ_0), with a confidence interval of 95%, determined in a $\ln\sigma_c$–$\ln$ [$\ln 1/(1-F(\sigma_c)$] diagram (according to ENV 843-5):

$$\ln\ln\left(\frac{1}{1\text{-}F(\sigma_c)}\right)=m\ln\sigma_c\text{-}m\ln\sigma_o$$

The characteristic strength is the strength at a failure probability of approximately 63.3%, and the Weibull modulus m is used as a measure of the strength distribution, which expresses the structural homogeneity of the material. Statistical analysis was performed using Minitab software (version 17, 2013, Minitab, State College, PA). The level of significance was 5%.

RESULTS

Levene test was performed, and there was no significant difference amongst the standard deviations (p>0.05). These results report that the data follow a normal distribution. The sample power calculation was performed by comparing two averages, in which a sample power of 100% was obtained for all ceramics (LD, LSC, and LSS).

Shear Bond Strength Test (SBS)

Table 2 shows a higher number of pretest failures for ceramic: LD-MEP (70%). The means and standard deviations for SBS and Weibull modulus for each material and the comparison among experimental groups are shown in Table 3.

Lithium Disilicate (LD)—ANOVA revealed that the factor "surface treatment" (p<0.0001) significantly influenced SBS for LD. When comparing surface treatments, the HFS group (18.66±3.49 MPa) showed significantly greater bond strength.

The Weibull modulus (m) and characteristic strength (σ_0) of LD groups were not significant (p=0.05). The HFS showed statistical similarity from MEP. Weibull distributions are shown in Table 3 and Figure 2.

Vita Suprinity (LSS)—Regarding LLS, ANOVA revealed that the "surface treatment" (p=0.004) was statistically significant. When comparing the surface treatments, Tukey test (5%) revealed that the HFS group (16.33±3.08 MPa) also had a significantly higher mean than the MEP (13.87±2.52 MPa).

The Weibull modulus (m) of LSS groups was not significant (p=0.3). The characteristic strength (σ_0) of groups were significant (p=0.001). The HFS showed higher σ_0 and was statistically different from MEP. The HFS group also had a higher m but showed a statistical similarity from MEP. Weibull distributions are shown in Table 3 and Figure 3.

Celtra Duo (LSC)—For LSC, ANOVA revealed that the "surface treatment" (p<0.006) was also significant. In the comparison between groups, the HFS (16.81±2.62 MPa) had the highest mean of SBS and was statistically different in relation to the MEP groups (14.12±3.51 MPa) (Tukey test p<0.05).

The Weibull modulus (m) of LSC groups were significant (p=0.006). The HFS showed the highest (m) and was statistically different from MEP. The HFS group had the highest (σ_0); however, it did not show a statistical difference from MEP. Weibull distributions are shown in Table 3 and Figure 4.

Table 3: Tukey Test for the SBS (MPa) Means (Standard Deviations) and the Weibull Modulus (m), Characteristic Strength (σ_0), and, Respective CI (95%) for SBS of the Treatment Surfaces by Ceramic Material[a]

	Surface Treatment	Group Name	Shear Bond Strength (SBS) (MPa)	Weibull Modulus (*m*)	95% CI for *m*	Weibull Characteristic Strength (σ_o) (MPa)	95% CI for (σ_o)
Lithium Disilicate (e.max CAD)	HF+Silane	LD-HFS	18.67 ± 3.49 A	6.22 a	4.4-8.6	20.32 α	18.99-21.74
	MEP	LD-MEP	7.00±4.2 B	1.0 a	0.1-5.9	8.8 α	3.80-20.78
LithiumSilicate (Celtra Duo)	HF+Silane	LSC-HFS	16.81 ± 2.62 A	8.8 a	7.6-10.25	17.70 α	16.83-18.62
	MEP	LSC-MEP	14.12 ± 3.51 B	4.13 b	2.47-6.9	15.53 α	13.84-17.41
Lithium Silicate (Suprinity)	HF+Silane	LSS-HFS	16.33 ± 3.08 A	7.4 a	6.3-8.9	17.81 α	16.78-18.90
	MEP	LSS MEP	13.87 ± 2.52 B	6.6 a	5.3-8.1	15.30 β	14.32-16.35

Abbreviations: LSS, Vita Suprinity; LSC, Celtra duo; LD, Lithium disilicate.

[a] *Different uppercase letters indicate statistically significant difference for each material for the SBS. Different lowercase letters indicate statistically significant difference for each material for the Weibull modulus. Different Greek alphabet letters indicate statistically significant difference for each material for characteristic strength.*

Optical Profilometry

Kruskal–Wallis revealed that the "surface treatments" produced statistically different roughness according to each ceramic (p<0.0001). The HFS groups for all ceramics presented the highest mean roughness. Similarly, for all materials, MEP generated the lowest roughness values: LD-MEP (0.47±0.10 μm), LSS-MEP (0.16±0.01 μm), and LSC-MEP (0.39±0.08 μm). Means and standard deviation of roughness and the differences between the groups are expressed in Table 4.

The 3D surface topography images of the specimens subjected to all experimental surface treatment are illustrated in Figure 5.

Scanning Electronic Microscopy (SEM)

SEM-FEG images referring to LD, LSS, and LSC specimens without surface treatment (Figure 6) present a homogeneous surface with grooves due to the sanding process during preparation of blocks. For groups with HFS treatment (Figure 6), irregularities with numerous microporosities and grooves are seen as a result of the dissolution of the glassy phase, leading to an increase in surface roughness. For LSS and LSC, these features were more evident. On the other hand, MEP produced a smoother and more homogeneous surface without numerous microporosities, as observed by the HF groups (Figure 6).

Failure Analysis

The main failure type for the groups with the pretest failures during thermal aging was in the resin cement–ceramic interface (Score A). Complete adhesive failures at the cement–dentin interface (Score A) were more frequent for all groups of the LD ceramic (100%). For

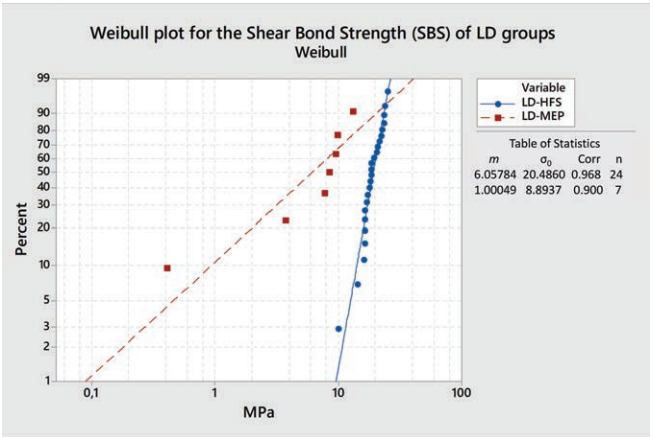

Figure 2. *Weibull plot for SBS of lithium disilicate (LD).*

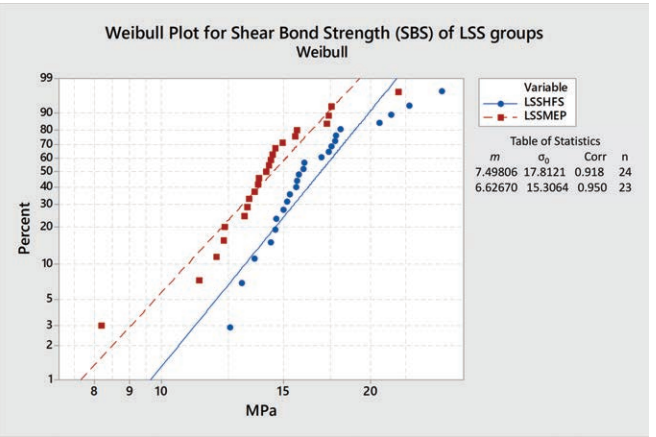

Figure 3. *Weibull plot for SBS of lithium silicate (LSS).*

Table 4: *Mean Roughness (R_a) Values (μm) with Standard Deviations for All Experimental Groups*[a]

Ceramic	Surface Treatment	Group Name	Mean Roughness (μm)
Lithium Disilicate (e.max CAD)	Control	C	0.29±0.05 B
	HF+Silane	LD-HFS	0.62±0.03 A
	MEP	LD-MEP	0.47± 0.10 B
Lithium Silicate (Celtra Duo)	Control	C	0.14± 0.02 B
	HF+Silane	LSC-HFS	1.33± 0.08 A
	MEP	LSC-MEP	0.39± 0.08 B
Lithium Silicate (Suprinity)	Control	C	0.24± 0.03 B
	HF+Silane	LSS-HFS	0.40± 0.02 A
	MEP	LSS MEP	0.16± 0.01 B

Abbreviations: LD: lithium disilicate; LSC: lithium silicate (Celtra Duo); LSS: lithium silicate (Suprinity).
[a]Different uppercase letters show statistical differences among groups of each ceramic.

the LSS and LSC groups, MEP also presented the greatest number of adhesive failures (Score A). After SBS, for the groups LSS-HFS (87.5%) and LSC-HFS (87.5%), the main failure type was Mixed 2 in the ceramic–resin cement interface + cohesive in ceramic (Score M2) (Table 2).

DISCUSSION

Many factors are involved in the clinical longevity of glass ceramic restorations,[31] such as the characteristics of the material used (composition, processing, and

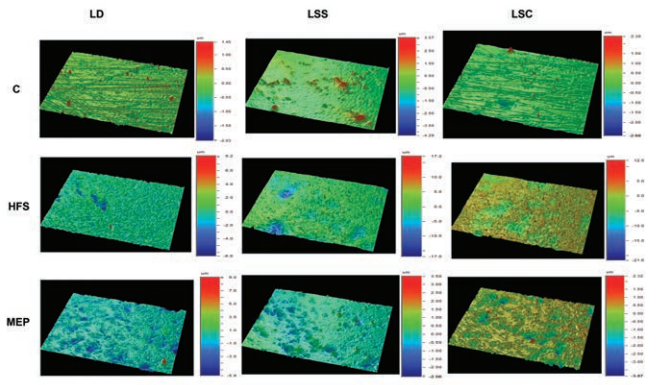

Figure 5. *3D Micrographs of surface topography (magnification 20×) of the specimens subjected to different surface treatment. LD: Lithium Disilicate; LSS: Vita Suprinity; LSC: Celtra Duo; C: Control (no treatment); HFS: Hydrofluoric acid + Silane; MEP: Monobond Etch & Prime/Ivoclar.*

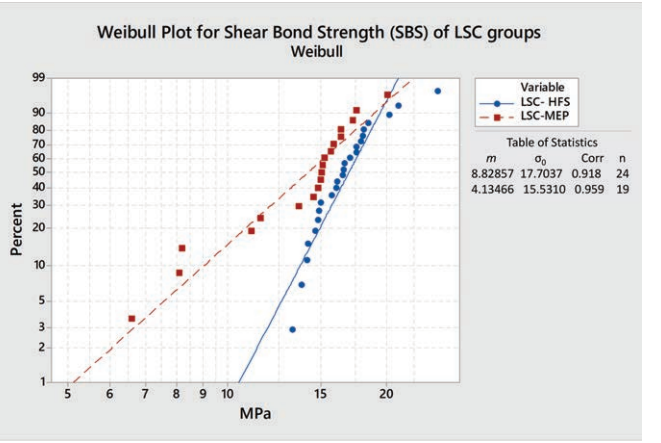

Figure 4. *Weibull plot for SBS of lithium silicate (LSC).*

thickness),[7,32] characteristics of the preparation and remaining substrate,[27,33] and the cementation protocol implemented,[31,34] which plays a key role in long-term adhesion. Thereby, the objective of this study was to evaluate the effect of different types of surface treatments on the adhesion of glass ceramics of *LD* and LS to resin cement. All the specimens in this study were subjected

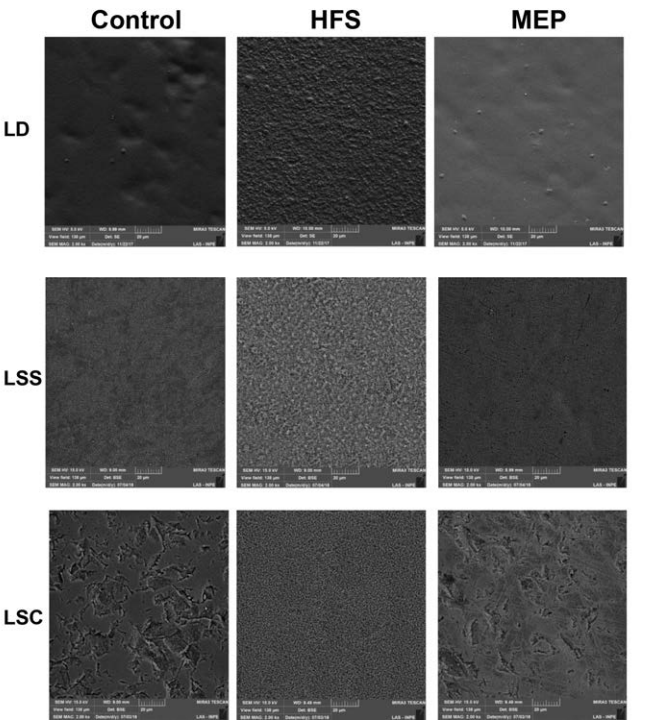

Figure 6. *Micrographs of SEM at 2500× magnification representing surface treatment groups. LD: Lithium Disilicate; LSS: Vita Suprinity; LSC: Celtra Duo; C: Control (no treatment); HFS: Hydrofluoric acid + Silane; MEP: Monobond Etch & Prime/Ivoclar.*

to thermocycling for 10,000 cycles, which simulates clinical conditions equivalent to 1 year of clinical use.[35]

The first hypothesis that the type of surface treatment would not influence the bond strength, regardless of the type of ceramic, was not accepted. In this study, different SBS values were found according to each material. Three glass ceramics were tested, and, despite the microstructural differences between these materials, the SBS results demonstrated that the HFS showed significantly higher adhesion values. Several studies have also shown that conditioning with HF followed by the application of silane was the best surface treatment for these glass ceramics.[5,7,13,14,22,36-39] HF etching causes micro-morphological changes and dissolves the glassy matrix creating micropores, where the resin cement can penetrate and provide micromechanical retention.[38,40] In addition, the application of a silane coupling agent to ceramic pretreated with HF provides a covalent chemical bond, which is one of the main factors of the efficient adhesion between glass ceramics and resin cements.[27] This treatment protocol offers the opportunity to improve micromechanical retention, and increase physical interactions and wettability with resin cement, which explains why it is the most suitable surface treatment.[41] According to some authors, this protocol, when balanced, prevents damage and weakening of the ceramic material, and dentists must follow the manufacturer's instructions for each ceramic, avoiding harmful effects of the material.[16,22]

Another surface treatment also investigated in this study was MEP. This treatment showed a significantly lower SBS for all ceramics investigated when compared to HF and higher SBS than SB and SC groups. Some authors report that MEP is less effective in glass ceramics than HF,[27,29,42] since this adhesive system contains ammonium polyfluoride—a milder acid with a lighter etching pattern that partially and homogeneously dissolves the glassy matrix[27]—generating a smoother surface alteration with less surface roughness and consequently lower adhesion values.[17,25,27,29,43-47] To compensate the pattern of conditioning, there has been suggested an active and prolonged application of MEP,[17,25] which leads to an increase in surface roughness, improving the interaction of resinous monomers (phosphoric acid methacrylate ester) with the ceramic surface and increasing the exposure of LD crystals with this acid.[25] Nevertheless, other authors state that the main MEP adhesion mechanism is chemical and, despite lower SBS in comparison to the HF etching, it seems to be a viable option, providing good adhesion values,[29] especially due to the simplification of procedures and in clinical situations of thin ceramic restorations.[28]

The second hypothesis that the surface treatment would influence the surface roughness regardless of the type of ceramic was not accepted. In this study, the HFS groups presented roughness higher than the MEP and control groups for all ceramics. Surface roughness is an important aspect that describes the effectiveness of pretreatment procedures on adhesion.[22,48] The literature shows that LS and LD materials have very similar microstructures. While LD presents small, needle-shaped crystals embedded in a glassy matrix, LS has slightly larger crystals with a more elongated, rounded, and rod-shaped appearance.[2,6] With regard to the HF group, the conditioning process selectively removes the glassy matrix, exposing the crystalline structure and generating a greater surface energy.[5,34] The SEM analysis revealed an irregular surface, with microporosity. Strasser and others[22] and Ramos and others[49] endorse that surfaces of glass ceramics etched with HF have strong and homogeneous corrosion patterns, resulting in a porous surface that favors adhesion. As a rule, roughness caused by MEP was slighter than the other treatments and statistically similar to the control groups. The SEM images of the MEP group specimens revealed less evident grooves; and the profilometry analysis proved that this treatment caused little increase in surface roughness (Figure 5). Strasser and others[22] confirm these small changes and suggest that this may also occur, because the primer present in the MEP itself caused the coating of surface irregularities and decreased R_a. Other authors also corroborate these findings.[27] Despite the lower performance compared to HF for LD ceramics, MEP also proved to be effective for LD, LSS, and LSC, presenting a high (m) and characteristic strength (σ_0) similar to HF, which reinforces the reliability of this adhesive interface, suggesting it as an option for surface treatment of glass ceramics, although further investigations should be carried out to complement these findings.

The failure analysis demonstrated different patterns of failures among treatments, with adhesive failures predominant in all surfaces of LD. For LSC and LSS, adhesive failures were more prevalent in group MEP. Whereas for HFS on the LSC and LSS ceramics, most of the failures were mixed (adhesive on the ceramic–cement interface and ceramic cohesive). Della and Northeast[50] claim that mixed failures can often occur due to the nonhomogeneous distribution of the SBS test, which generates stresses in the base materials. Contrastingly, the current study found this failure pattern more frequently only in groups with HFS. According to Baratto and others[31] and Mokhtarpour and others,[39] the presence of cohesive failures in ceramic

demonstrate that the strength of the substrate and cement are equal to that of the adhesion area, which indicates a more effective surface treatment. In concern to MEP, as mentioned above, the adhesive failures can be related to the little mechanical retention that this material generates,[27] which was also confirmed in our results of profilometry and SEM.

In the present study, a large number of pretest failures were also observed during thermocycling for all the ceramics, but it most clearly affected the LD groups with MEP, which may justify the smaller mean values of SBS and greater standard deviation for these treatments. According to Cadernas and others,[17] MEP, when applied to LD with the recommended time, causes a greater number of adhesive failures. El-Damanhoury and others[27] indicated that the application of MEP on LD produces a low surface roughness, which may have contributed to this failure profile. Other studies did not find significant differences between HFS and MEP[43,51]; but no aging of the specimens was performed. Besides the roughness, thermocycling contributes to reducing adhesion to resin cement.[29,52] The temperature difference causes saturation of the cement, with a greater hydrolytic degradation of the adhesive interface,[53] which may also have contributed to the large number of pretest failures.

Further studies evaluating surface treatments of LS and LD ceramics, especially with MEP and HF etching at different application times, on the bond strength and flexural strength, must be carried out. Controlled and randomized clinical trials are also needed to assess the long-term behavior of these materials and to establish an ideal surface treatment for each ceramic material.

CONCLUSIONS

Based on the work presented, it seems reasonable to conclude the following:

- HF followed of silanization promote higher values of bonding strength for LD and LS ceramics.
- HFS promoted higher roughness than MEP for all the ceramics.

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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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Effects of Charcoal Toothpaste on the Surface Roughness, Color Stability, and Marginal Staining of Resin Composites

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Clinical Relevance

Charcoal toothpastes cause roughness in resin composites similar to control toothpastes; however, some types of toothpastes can change the color and cause marginal staining of the resin composite restorations.

SUMMARY

Objective: This study was designed to evaluate the effects of charcoal toothpaste on the surface roughness, color stability, and marginal staining of resin composite restorations.

Methods: A total of 100 bovine incisors was collected. The crowns were sectioned and randomly divided into 10 groups (n=10) according to two study factors: toothpaste groups and nanoparticle resin composite groups. Five toothpastes—Bianco Pro Clinical (Bianco Oral Care, Uberlândia, MG, Brazil) - Control group; Bianco Carbon (Bianco Oral Care); NAT, Natural Suavetex Carvão Ativado (Suavetex, Uberlândia, MG, Brazil); Nano Action Black Be Emotion (Polishop, Jundiaí,

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SP, Brazil); and BIW, Black is White (Curaprox, Curaden AG, Kriens, Switzerland)—and two resin composites—Z350XT (Filtek Z350XT, 3M Oral Care) and Vittra (Vittra APS FGM, Joinville, SC, Brazil)—were used. Circular cavities with a diameter of 4 mm and a depth of 1 mm were prepared on the buccal face of the tooth crowns and restored with resin composites. The specimens were subjected to three months of simulated toothbrushing. The surface roughness (right angle [Ra], in micrometers [μm]) of the resin composites was measured before and after toothbrushing in five areas per specimen. The resin composite color and luminosity changes (ΔE and ΔL, respectively) were measured using reflectance spectroscopy (Vita EasyShade). Macro photographs were taken before and after toothbrushing to qualitatively analyze the marginal staining (MSt) of the resin composite restorations. Scanning electron microscopy (SEM) was performed before and after the simulated toothbrushing. Ra data were analyzed using two-way analysis of variance with repeated measures and the Tukey HSD test; MSt was analyzed using Kruskal-Wallis and Dunn tests (α=0.05), and the resin composite color change was analyzed using the clinically unacceptable level of ΔE > 3.3.

Results: Simulated brushing increased Ra irrespective of the resin composite or toothpaste used. No significant differences were found in Ra between the control group and all groups on which the charcoal toothpastes were tested. A clinically unacceptable level of resin composite color change (ΔE>3.3) was found after the use of most charcoal toothpastes. Use of Bianco Carbon resulted in marginal staining similar to that of the control group and was lower than that of the other charcoal toothpastes. Vittra brushed with black toothpaste showed the highest marginal staining.

Conclusion: Use of charcoal toothpaste resulted in Ra values of resin composites similar to those found with conventional toothpastes. Charcoal toothpaste generally resulted in clinical resin composite color changes (ΔE). All charcoal toothpastes, except Bianco Carbon, caused marginal staining of the resin composite restorations.

INTRODUCTION

Resin composites are the first choice for direct restorations in daily practice.[1] They are commonly used as substitutes for enamel and dentin[2] for diastema closure procedures, dental fractures, and direct veneers.[3] However, resin composite restorations are prone to staining, changing color, and wearing out due to many intrinsic and extrinsic factors,[4] such as photoinitiator system type, resin matrix degradation, insufficient irradiation time, and low irradiance of the light-curing unit used for polymerization.[5] Additionally, the oxidation of monomers or catalysts, exposure to thermal, mechanical, and chemical challenges in the oral environment, [6,7] and absorption of extrinsic stains can contribute to these alterations of resin composites.[8] Shrinkage stress is another relevant side effect that can be generated during light-curing of resin composites,[9] These stresses are linked to the creation or propagation of enamel cracks, which can lead to esthetic problems such as stained cracks.[10]

The loss of gloss and darkening in resin composite restorations creates negative esthetics.[11] Surface roughness is the major contributor to the extrinsic discoloration of resin composite restorations,[12] and this roughness is related to the organic matrix, inorganic filler composition, finishing and polishing procedures, and challenging processes that occur in the oral environment.[13,14] A high surface roughness can increase biofilm accumulation, mineral loss, topography alteration, and altered light reflectance from the enamel.[15] On resin composites, increased roughness can lead to staining or discoloration of the body and margins of restorations or, in more severe cases, even cause gingivitis, caries, and recurrent caries.[16]

Recently, charcoal-based toothpaste has been developed and commercialized for oral hygiene; it is considered fashionable toothpaste.[17] Charcoal-based products for dental hygiene can be produced in various formulations, such as powder form or even coal ashes.[18-20] The manufacturers of charcoal-based toothpastes claim that they have stain-removal and whitening effects, and these purported esthetic effects are used in promotions to customers. However, there is still a lack of evidence to support such claims for these products.[21] Instead, the opposite effect may occur, such as marginal staining of resin composite restorations and laminate veneers.[17] This might be an important drawback because marginal staining is often erroneously used as a criterion for the replacement of indirect and direct resin composite restorations.[9]

No scientific evidence is available to support the benefits of the charcoal-based toothpastes that are currently marketed.[21] Thus, it is clinically important to evaluate the effects of brushing teeth using different charcoal-based types of toothpaste on resin composite restoration surfaces. To the best of our knowledge, no

other study has verified these effects for resin composite surfaces. Therefore, this study was aimed to evaluate the effects of charcoal-based toothpaste on the surface roughness, color stability, and marginal staining of two nano-filled resin composites. The null hypothesis was that toothbrushing with charcoal toothpaste would not affect the surface roughness or cause color changes or staining of the margins of resin composite restorations.

METHODS AND MATERIALS

One hundred bovine incisors of similar shapes and colors were collected for use as substitutes for human teeth.[22,23] The specimens were stored in distilled water at 37°C before preparation and between all procedures. After prophylaxis, the roots of the teeth were removed using a high-speed water-cooled diamond disc (American Burrs, Palhoça, SC, Brazil). The crowns were embedded in epoxy resin (Buehler, Lake Bluff, IL, USA), and the buccal surface was finished with 600-grit sandpaper (3M, Sumaré, SP, Brazil) to obtain a parallel surface for cavity preparation. The teeth received circular cavities with a diameter of 4 mm and a depth of 1 mm, performed by inserting the entire head of a wheel diamond bur at a high speed (No 3053, KG Sorensen, Cotia, São Paulo, Brazil). The burs were replaced after 10 cavity preparations. A restorative procedure was performed by selective etching of the enamel with 37% phosphoric acid (Condac 37, FGM,) for 30 seconds. The cavities were washed using a water spray for 30 seconds and excess water was removed with absorbent paper. A self-etching adhesive (Ambar Universal APS, FGM) was applied in two layers onto the enamel and dentin surfaces with a microbrush (Cavibrush, FGM), followed by a light jet of air for 10 seconds to facilitate the evaporation of the solvent and light-curing for 10 s with an LED light-curing unit (LCU; Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein) at 1400 mW/cm², checked using a MARC Resin Calibrator (BlueLight, Halifax, Canada). The specimens were randomly divided into 10 groups (n=10); half of the specimens were restored with two increments of the nano-filled resin composite Filtek Z350 XT (A2E shade, 3M Oral Care, St Paul, MN, USA), and the other half were restored with the nano-filled resin composite Vittra APS (EA2 shade, FGM) and light-cured for 20 seconds for each increment. Descriptions of the resin composites are listed in Table 1. After the restorative procedure, the specimens were submitted to a finishing procedure using 600, 800, 1000, and 1200-grit sandpaper (3M, Sumaré), followed by polishing with 6-μm, 3-μm, 1-μm, and 1/4-μm grit diamond polishing pastes with the respective polishing cloths (Arotec, Cotia, SP, Brazil) for 2 minutes with each paper by a trained operator at the same rotation speed as in the metallographic polishing machine (Arotec). After each polishing step, the specimens were ultrasonically cleaned (Thornton, Vinhedo, SP, Brazil) in deionized water for 10 minutes to remove debris.

The surface roughness (Ra, μm) was analyzed before and after the toothbrushing cycles using a profilometer (SJ-301, Mitutoyo, Kanagawa, Japan). Five measurements were performed on the resin composite surface for each specimen at different positions using a cutoff length of 0.25 mm, speed of 0.25 mm/s, and length of 0.8 mm. Measurements were taken perpendicular to the direction of brushing. The Ra value for each specimen represented the mean Ra of five measurements.[24]

Blind measurements with a reflectance spectrophotometer (Vita EasyShade Advance 4.0, Vident, Brea, CA, USA) were used to evaluate surface color changes (ΔE) and luminosity changes (ΔL) of the resin composite restorations due to brushing

Table 1. *Resin Composites Used in this Study*

Resin Composites	Type	Shade	Monomers	Filler Type	Filler Volume (%)	Filler Weight (%)	Manufacturer	Batch Number
Filtek Z350XT	Nanoparticle	A2E	bis-GMA, UDMA, TEGDMA, bis-EMA	Silica, zirconia, aggregated zirconia/ silica clusters	63.3	78.5	3M Oral Care, St Paul, MN, USA	1901600177
Vittra APS	Nanoparticle	EA2	Methacrylate monomers mixture	Silica, zirconia	52-60	72-82	FGM, Joinville, SC, Brazil	051216

Abbreviations: bis-GMA, bisphenol A-glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; bis-EMA, bisphenol A diglycidyl methacrylate ethoxylated.

with charcoal toothpaste. The device was calibrated before the measurement of each specimen, and the color parameters were recorded before (baseline) and immediately after the toothbrushing cycles. Three measurements of the center of the resin composite restoration were performed for each specimen in the same position, and the mean of the three readings was calculated. ΔE and ΔL were chosen for analyzing the effects of any color changes. Tooth color was analyzed based on ΔL, Δa, Δb, and ΔE coordinates from the CIE $L^*a^*b^*$ color system, in which L^* values represent luminosity (a value of 100 corresponds to perfect white, while 0 indicates black); a^* indicates red (positive values) and green colors (negative values); b^* represents yellow (positive values) and blue (negative values).[25,26] The color change (ΔE) was determined using the following formula[27,28]: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. Three intervals were used to classify the color changes of the resin composite restoration: $\Delta E < 1.0$, imperceptible to the human eye; $1.0 < \Delta E < 3.3$, discernible by a skilled person and clinically acceptable; and $\Delta E \geq 3.3$, easily observed and clinically unacceptable.[26,29]

Marginal staining (MSt) was evaluated qualitatively by analyzing macro photographs taken before and immediately after the toothbrushing cycles. Photographs were taken by one operator using a digital single-lens reflex (DSLR) camera (Canon T5, Canon, Ota, Tokyo, Japan) with a macro lens (100 mm, Canon) and a macro ring flash (YN-14EX, Shenzhen Yongnuo, Futian District, Shenzhen, China). The same focal distance and photo parameters were used for all photographs. The photographs were saved and randomized using codes with letters and numbers for blind identification of the photographs. The photographs were analyzed by three trained operators. The resin composite restoration image was divided into four quadrants for analysis. The operators evaluated the MSt, and the sum of the stained quadrants was classified as Score I: 0 quadrants stained, II: 1 quadrant stained, III: 2 quadrants stained, IV: 3 quadrants stained, and V: 4 quadrants stained. Each evaluation was performed independently to avoid any influences of the other operators. During the evaluation, the resin composites or toothpastes evaluated with each specimen were unknown to the operators. In case of disagreements regarding score punctuation, the lowest rating was recorded.

The toothpastes selected for this study were a conventional toothpaste without charcoal, Bianco Pro Clinical, BPC (Bianco Oral Care) as control group and four charcoal toothpastes—Bianco Carbon, BCA (Bianco Oral Care), Natural Suavetex Carvão Ativado, NAT (Suavetex), Nano Action Black Be Emotion, NAB (Polishop), and black, white, BIW (Curaprox). Information about the toothpastes used is listed in Table 2. The specimens, embedded in polystyrene resin cylinders, were assembled on a matrix attached to a toothbrushing machine (Odeme Dental Research, Luzerna, SC, Brazil) with the resin composite surface restorations facing up. A mixture of toothpaste and artificial saliva (ratio 2:1, 8 g/4 mL by specimen)[30,31] was dispensed onto the matrix to cover the surface of the specimen. Heads of soft-bristle toothbrushes (Colgate Pro Cuidado, Colgate-Palmolive Co., New York, NY, USA) were cut and attached to the device. Specimens were subjected to 21,960 cycles,[31] simulating three months of toothbrushing, with a vertical loading of 200 g over the toothbrush heads and at a controlled temperature (25°C±1°C). A linear motion was performed over the surface of the specimens, as shown in Figure 1. After each specimen cycle, the toothbrush and toothpaste mixture were replaced, and

Table 2. *Toothpastes Used in this Study*

Toothpastes	Code	Main Components	Manufacturer
Bianco Pro Clinical (Control)	BPC	Tricalcium phosphate 3%	Bianco Oral Care, Uberlândia, MG, Brazil
Bianco Carbon	BCA	Tricalcium phosphate 3%, charcoal powder	Bianco Oral Care, Uberlândia, MG, Brazil
Natural Suavetex com Carvão Ativado	NAT	Charcoal powder, bambusa vulgaris extract, punica granatum extract, salvia sclarea extract	Suavetex, Uberlândia, MG, Brazil
Nano Action Black Be Emotion	NAB	Charcoal powder, cocos nucifera oil, sodium monofluorophosphate, 1192 ppm fluoride	Polishop, Jundiaí, SP, Brazil
Black is White	BIW	Hydroxyapatite, activated carbon, 1450 ppm fluoride, enzymes, 15000 ppm nano-hydroxyapatite, Prestige Sparkling Blue	Curaprox, Curaden AG, Kriens, Switzerland

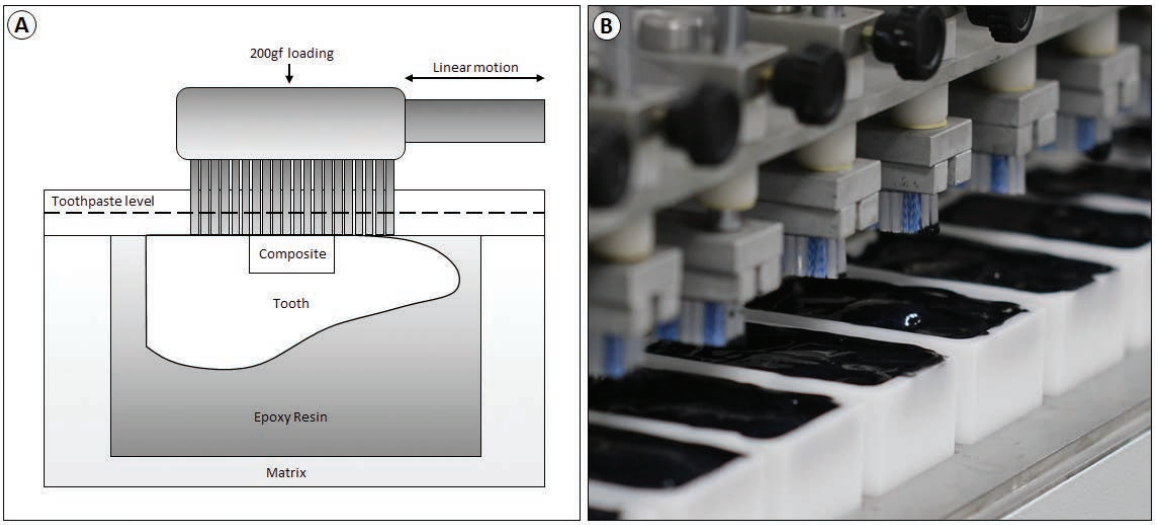

Figure 1. *Toothbrushing methodology. (A): Diagram of the toothbrushing method; (B): Specimens on the toothbrush machine.*

the brushing machine was completely cleaned using distilled water. After the brushing cycles, the specimens were washed with distilled water for 2 minutes, and the final photographs were taken. Color evaluation and Ra measurements were performed again using the same parameters. Representative specimens of each group were fixed on stubs and analyzed using scanning electron microscopy (Tescan Company, Brno, Czech Republic) with a 1000× magnification pre-brush to visualize the shape, quantity, and size of the filler content of the resin composites and a 100× magnification post-brush to verify the differences in roughness on the resin composite surface.

The Ra data (μm) were tested for normal distributions (Shapiro-Wilk) and equality of variances (Levene test), followed by parametric statistical tests using two-way repeated measures analysis of variance (ANOVA), followed by Tukey test. MSt was analyzed using the Mann-Whitney, Kruskal-Wallis, and Dunn's tests (α=0.05). The resin composite color change was analyzed qualitatively for the presence of a clinically unacceptable level ($\Delta E>3.3$)[26,29] and positive or negative values of ΔL.

RESULTS

The mean and standard deviations of the surface roughness (Ra, μm) before and after brushing are shown in Figure 2 and Table 3. Two-way ANOVA of repeated measurements demonstrated a significant influence of the resin composite type ($p<0.001$) and

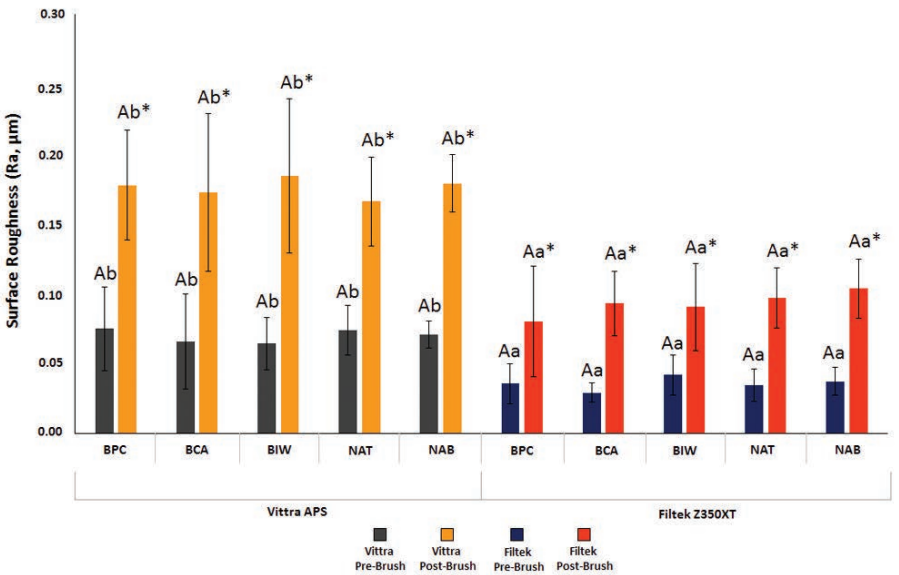

Figure 2. *Mean and standard deviation values of surface roughness before and after brushing. Different letters indicate significant differences (p<0.05); uppercase letters are used for comparing toothpastes; lowercase letters are used for comparing composite resins at each moment; and * is used for comparing pre- and postbrushing data. BPC, Bianco Pro Clinical (Bianco Oral Care) - control group; BCA, Bianco Carbon (Bianco Oral Care); NAT, Natural Suavetex Carvão Ativado (Suavetex); NAB, Nano Action Black Be Emotion (Polishop); BIW, Black is White (Curaprox).*

Table 3. *Mean and Standard Deviation Values for the Surface Roughness (Ra - μm) of Resin Composite Restorations Before and After Toothbrushing*

Toothpastes	Vittra APS (n=10)[a]		Filtek Z350XT (n=10)[a]	
	Prebrush	Postbrush	Prebrush	Postbrush
Bianco Pro Clinical	0.08 (0.03) Ab	0.18 (0.04) Ab[b]	0.04 (0.01) Aa	0.08 (0.04) Aa[b]
Bianco Carbon	0.07 (0.03) Ab	0.17 (0.06) Ab[b]	0.03 (0.01) Aa	0.09 (0.02) Aa[b]
Curaprox Black is White	0.06 (0.02) Ab	0.18 (0.06) Ab[b]	0.04 (0.01) Aa	0.09 (0.03) Aa[b]
Natural Suavetex	0.07 (0.02) Ab	0.17 (0.03) Ab[b]	0.03 (0.01) Aa	0.10 (0.02) Aa[b]
Nano Action Black Be Emotion	0.07 (0.01) Ab	0.18 (0.02) Ab[b]	0.04 (0.01) Aa	0.10 (0.02) Aa[b]

[a]*Different letters indicate significant differences (*p*<0.05). Uppercase letters are used for comparing toothpastes, lowercase letters are used for comparing composite resins at each moment*

[b]*For comparing pre- and postbrushing data.*

toothpaste type ($p<0.001$). All groups showed increased Ra after the toothbrushing cycles regardless of the toothpaste or resin composite used. Z350XT had lower Ra values before and after brushing than Vittra. Comparison of the BPC toothpaste control groups with all four charcoal toothpaste groups tested showed no significant difference in Ra values ($p>0.160$).

The color change (ΔE) and luminosity change (ΔL) results are shown in Figures 3 and 4, respectively. All resin composite restorations brushed with charcoal toothpaste presented clinically unacceptable color changes (ΔE>3.3), except for the combination of Z350/BCA (Figure 3). In the ΔL analysis, more visible alterations in luminosity were observed when charcoal toothpaste was used. Brushing with conventional BPC toothpaste caused no significant color or luminosity alterations.

The marginal staining results of the resin composite restorations are shown in Table 4, and representative images of all groups are shown in Figure 5. The Mann-Whitney test showed a significant difference only for the BIW toothpaste, with Vittra ($p=0.008$) showing the highest MSt level. The Kruskal-Wallis test showed no significant difference between BPC and BCA for both resin composites. However, the Dunn test showed a significant difference between BCA and BPC with BIW, NAT, and NAB, which exhibited different levels of MSt, indicating darkening of the margins of the restoration.

SEM images of all groups are shown in Figure 6. Z350XT presented lower irregularities compared with Vittra, irrespective of the toothpaste used. The control group, BPC, resulted in lower irregularities on the resin composite surfaces than the charcoal toothpastes tested (Figure 6). SEM images of Z350XT showed a more homogeneous distribution and smaller filler particle sizes than Vittra, which presented a more heterogeneous distribution with larger filler particles among the inorganic fillers (Figure 6A, 6B).

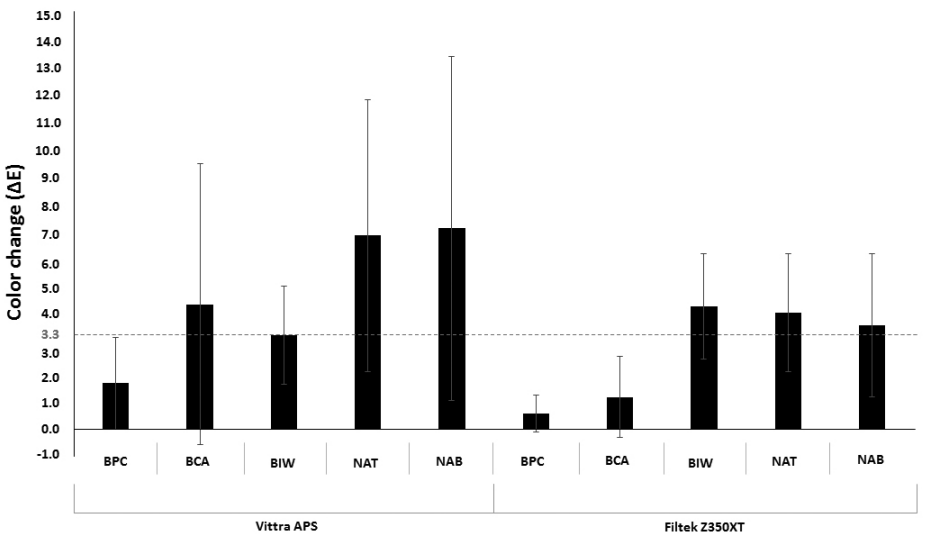

Figure 3. *Mean and standard deviation values of resin composite color changes (ΔE). The red dotted line (ΔE>3.3) indicates clinically unacceptable color change values. BPC, Bianco Pro Clinical (Bianco Oral Care) - control group; BCA, Bianco Carbon (Bianco Oral Care); NAT, Natural Suavetex Carvão Ativado (Suavetex); NAB, Nano Action Black Be Emotion (Polishop); BIW, Black is White (Curaprox).*

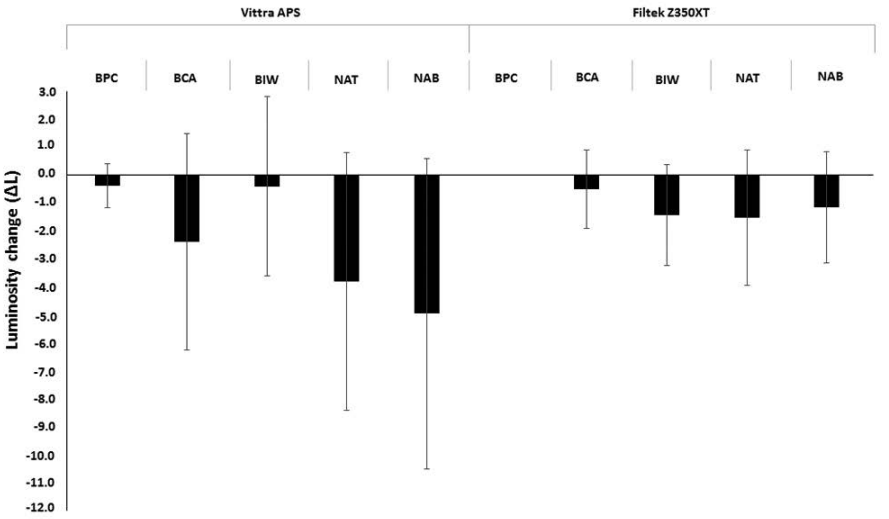

Figure 4. *Mean and standard deviation values of resin composite luminosity changes (ΔL).*

DISCUSSION

This *in vitro* study evaluated the effects of charcoal toothpaste brushing on the surface roughness, color stability, and marginal staining of resin composite restorations. According to the results of this study, charcoal toothpastes affected the surface roughness of the resin composites compared with the control toothpaste, stained the margins of the resin composite restorations, and produced color changes on the resin composite, requiring the null hypotheses to be rejected. Z350XT showed lower Ra values before and after the toothbrushing cycles than the Vittra specimens. This might be attributed to the smaller and more homogenous filler particles of Z350XT, as shown in Figure 7. This characteristic facilitates better polishing and a smoother resin composite surface[32] and may also result in more esthetic restorations. Although both resin composites tested were nanoparticulated resin composites, according to the manufacturers, Z350XT presented 20-nm silica fillers and 4-11-nm zirconia fillers, while Vittra presented 100-200-nm silica-zirconia fillers. Thus, Vittra contained a larger and more heterogeneous distribution of the filler elements, which accords with the Ra values obtained in this study.

The Ra values of both resin composite restorations increased after toothbrushing, regardless of the toothpaste used. Soft-bristle toothbrushes were used in this study.[33-35] The soft-bristle toothbrush under 200 g loading using conventional toothpaste (control group, BPC) caused a small increase in the Ra of the

Table 4. *Marginal Staining of Resin Composite Restorations Evaluated by a Qualitative Analysis of Stained Quadrants After Toothbrushing*

Toothpastes	Vittra APS[a]						Filtek Z350XT[a]					
	I	II	III	IV	V		I	II	III	IV	V	
Bianco Pro Clinical (control)	10	0	0	0	0	Aa	10	0	0	0	0	Aa
Bianco Carbon	10	0	0	0	0	Aa	10	0	0	0	0	Aa
Black is White	1	2	2	3	2	Bb	6	3	0	1	0	Aab
Natural Suavetex Carvão Ativado	4	4	2	0	0	Aab	3	0	2	5	0	Ab
Nano Action Black Be Emotion	2	4	3	1	0	Ab	3	3	3	0	1	Aab

Abbreviations: I, 0 quadrants stained; II, 1 quadrant stained; III, 2 quadrants stained; IV, 3 quadrants stained; V, all margins stained.
[a]Uppercase letters are used to analyze the difference between columns (each toothpaste for both composites). Lowercase letters are used to analyze the difference between rows (composite for all toothpastes).

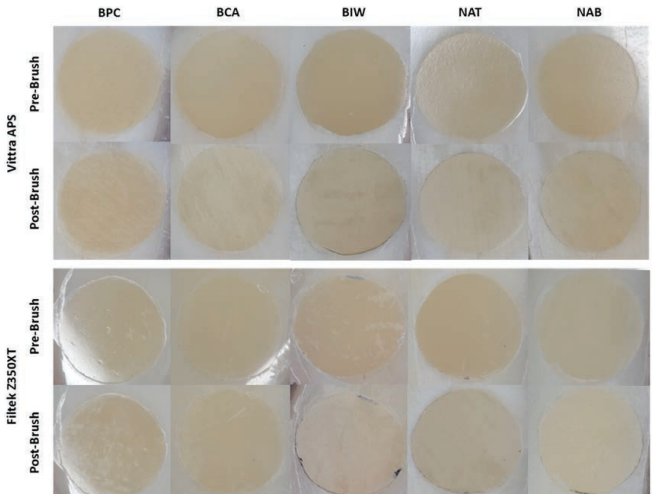

Figure 5. *Marginal staining of representative specimens of each resin composite restoration group.*

resin composite restorations, similar to the charcoal toothpastes tested. Follow-up and possible new finishing and polishing of the resin composite restorations are strongly recommended. Even the control toothpaste contains abrasive materials such as silica and hydrated silica,[36] which may increase the roughness of the resin composite surface. Increasing surface roughness can contribute to faster colonization by and maturation of biofilms, increasing the possibility of resin composite degradation and the risk for caries and periodontal inflammation.[37] An increase in Ra values equal or superior to 0.2 μm leads to greater biofilm retention, and when Ra is higher than 0.3 μm, biofilm retention may be detected by patients' lips and tongues, causing discomfort.[37-39] Vittra and Z350XT restorations brushed with all toothpastes reached Ra values below the threshold of 0.2 μm, as shown in Figure 2.

The color changes in the resin composite restorations (ΔE) were higher for the specimens brushed with charcoal toothpastes, reaching clinically unacceptable values (ΔE>3.3). This might be explained by the fact that some monomers, such as TEGDMA, are vulnerable to water sorption resulting in a higher level of staining caused by absorption of toothpaste components.[40,41] Charcoal particles and dark and gray pigments present in charcoal toothpastes are impregnated into the resin composite surface, changing the color. The dark pigments incorporated into the resin composites with increasing Ra caused darkening of the resin composites, as confirmed by negative ΔL values, which were more visible and significant in the charcoal toothpaste groups. The ΔE and ΔL values were higher in the Vittra groups, which might be due to the higher Ra of this resin composite, as higher surface roughness tends to increase staining susceptibility.[42-44]

The risk for marginal staining of resin composite restorations is a frequently asked question by patients, especially those that have esthetic restorations, who are considering the use of charcoal toothpastes. Except for BCA, all charcoal toothpastes presented MSt at different levels. The fact that BCA had no significant MSt might be due to the lower quantity of charcoal particles and lighter pigments contained, which resulted in the appearance of a gray rather than black color, as seen with the other tested charcoal toothpastes. The bonding agent used in this study was a self-etching adhesive containing the functional monomer 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). Selective enamel etching with 37% phosphoric acid for 30 seconds preceded the application of the adhesive system. The bonding strategy used and the performance of this adhesive system result in stable dentin bonds with results comparable to those of gold standard materials, particularly when applied in the self-etch mode.[45,46] Therefore, this is not considered a factor biased toward marginal staining. Although not the aim of the study, the authors were able to visualize pigmentation in enamel microcracks in some specimens. This might be another important concern, as enamel microcracks are not easy to treat, and, when severely stained, they can lead to the necessity of restorative intervention.

In other *in vitro* studies with charcoal toothpastes, the surface roughness of the enamel was evaluated, verifying the loss of minerals on the enamel surface caused by the abrasive properties of the toothpaste.[21,36] However, this study focused on effects on the resin composites, and all groups brushed with charcoal toothpastes showed similar roughness of the resin composite as the group with the control toothpaste. This might be because the susceptibility of enamel to toothbrush abrasion is higher than that of the restorative materials.[47] This study had some limitations, including a lack of complete information about the toothpaste compositions, such as the percentage of each component or whether there was a component that was not listed; this drawback is similar to that in a previous study.[41] Although this *in vitro* study tried to replicate general conditions that occur in the mouth, other conditions that can enhance surface roughness and marginal staining were not replicated. An acidic diet, brushing force, salivary conditions, and amount of toothpaste used by each patient are variations that can be replicated in clinical studies. Second, even though the toothbrushes used in this study were considered soft, this study did not test the effect of different bristle types; therefore, the similar surface roughness found for all groups could also be related to the toothbrush. Further studies may

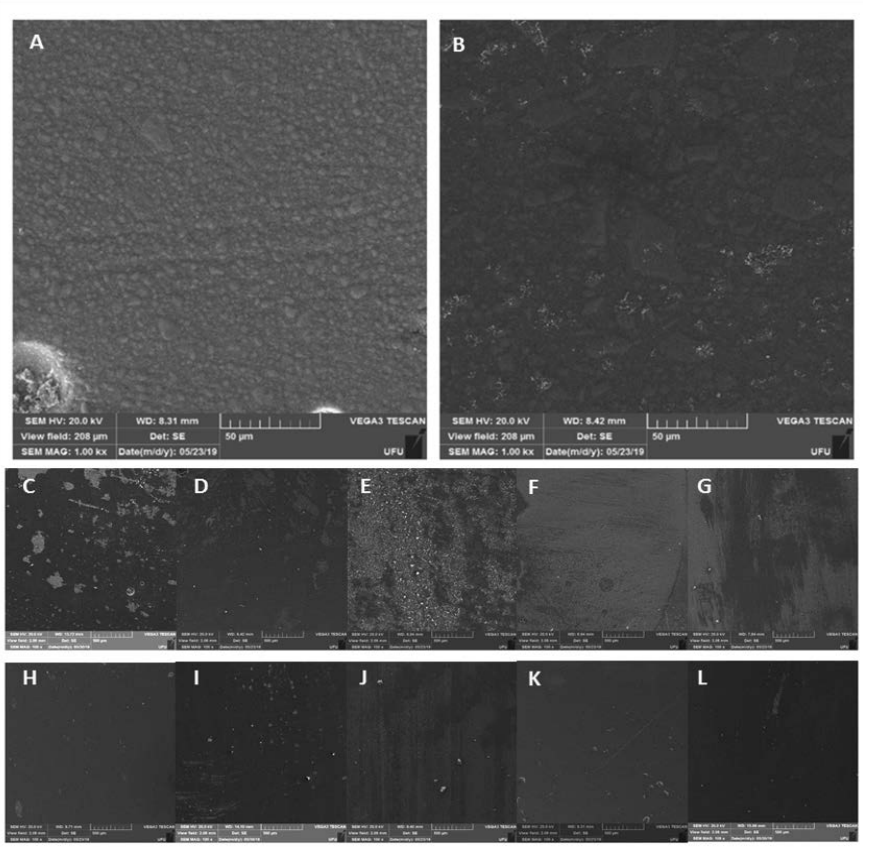

Figure 6. *Scanning electron microscopy images of resin composites before brushing (A-B: 1000x magnification) and after toothbrushing (C-L: 100x magnification). (A): Z350XT; (B): Vittra (1000X magnification); (C): Vittra/Bianco Pro Clinical; (D): Vittra/Bianco Carbon; (E): Vittra/Black is White; (F): Vittra/Natural; (G): Z350XT/Nano Action Black; (H): Z350XT/Bianco Pro Clinical; (I): Z350XT/Bianco Carbon; (J) Z350XT/Black is White; (K): Z350XT/Natural; (L): Z350XT/Nano Action Black (100X magnification).*

be conducted to assess the percentage and size of the toothpaste component particles and whether new finishing and polishing procedures can be performed to remove the marginal staining or reestablish the color of the resin composite restorations. In addition, studies focusing on different toothbrush bristles (hard, soft, and extra soft), staining cracks, or enamel microcracks should be conducted. However, the clinical relevance and timeliness of this study provoke a new line of thinking about the effects of charcoal toothpaste use on resin composite restorations.

CONCLUSIONS

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

1. The Ra value results of the charcoal toothpastes were similar to those of the conventional toothpaste.
2. The charcoal toothpastes caused changes in the resin composite color, generally at a clinically unacceptable level ($\Delta E>3.3$) and tended to darken the restorations ($\Delta L<0$).
3. The charcoal toothpastes, except for BCA, caused dark marginal staining of the resin composite restorations.

Conflict of Interest

The authors have no financial interest in any of the companies or products mentioned in this article.

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Masking Ability of the Combined Application of Opaquers and Resin Composite on Discolored Backgrounds

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Clinical Relevance

The combined application of opaquer and resin composite provides effective masking of mildly and intermediately discolored backgrounds, and contributes to less tooth reduction, thus preserving dental tissues. Alternative combinations should be applied to mask severely discolored backgrounds.

SUMMARY

The aim of this study was to evaluate the masking ability of a combined application of opaquers and resin composite over discolored backgrounds: A3, A3.5, C2, C3, and C4.

The groups were divided according to the opaquer brand, the number of opaquer coats (one or two), and the thickness of the resin composite layer (0.5 or 1.0 mm). The color measurements were made by a reflectance spectrophotometer (SP60, EX-Rite). The color difference between the opaquer + resin composite + background and a reference background was calculated using the CIEDE2000 formula. ANOVA and Tukey's *post hoc* test (α=0.05) were used to analyze the ΔE_{00} mean values. A bivariate analysis was used to determine the association between dependent and independent variables. The masking

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ability was rated by the ΔE_{00} visual thresholds of acceptability and perceptibility (Excellent Match: $\Delta E_{00} \leq 0.8$; Acceptable Match: $0.8 < \Delta E_{00} \leq 1.8$; Moderately Unacceptable Mismatch: $1.8 < \Delta E_{00} \leq 3.6$; Clearly Unacceptable Mismatch: $3.6 < \Delta E_{00} \leq 5.4$; Extremely Unacceptable Mismatch: $\Delta E_{00} > 5.4$).

The mean ΔE_{00} values ranged from 0.5 to 5.52. Masking ability was affected by the opaquer brand, thickness of the resin composite layer, and background shades. Most of the combinations that achieved either excellent or acceptable masking ability were obtained with combinations composed of one or two coats of opaquer and a 1.0-mm-thick resin composite layer for all backgrounds except C4. Acceptable results were also obtained for combinations with 0.5-mm-thick resin composite over C2, A3, and A3.5 backgrounds.

INTRODUCTION

Tooth discoloration in the anterior zone is a challenging clinical situation, especially when a single element is affected.[1] The proximity and contrast of this element with the adjacent teeth lead to a significant color mismatch.[2] Dental bleaching is a conservative technique that requires minimal intervention; therefore, it should be the first treatment choice for discoloration.[3] However, in some cases, discoloration is unresponsive to dental bleaching, or the esthetic outcome is not what is ideally expected.[3]

The resin composite layering technique is considered an option for masking discolored backgrounds. The combination of different shades and translucencies may give the final restoration a natural aspect, and also prevent transmission of the underlying dark color of the tooth surface or cavity floor.[4,5] However, direct resin composites have inherent limitations regarding opacification ability.[6] Depending on the severity of the discoloration, an opaque-shade resin composite layer that is at least 1.0-mm thick is needed to mask the underlying tooth structure.[7] Taking this into account, cavity preparation with tissue reduction is often required to provide thicker resin composite layers. Despite the variety of available approaches, masking is often not achieved without aggressive tooth preparation.[8-10]

Less invasive treatments performed in line with the minimal intervention approach should be preferred in the case of chromatic challenges.[1] Opaquers are fluid resins with high opacity agents, developed to be used in association with restorative materials to facilitate the masking of underlying structures[11, 12] and promote less tissue reduction.[7] Case reports show positive results for the combined applications of opaquers and resin composites.[10,12,13] However, the technical variations, application possibilities, and masking effectiveness of different degrees of discoloration have not yet been completely elucidated. A wide range of opaquers is commercially available. The ideal situation would be to have shades matching all of the resin composite systems. However, most of the commercial brands provide only one or two shade options, generally white opaque or universal opaque.[14] Therefore, it is important to investigate the masking ability of opaquers with different characteristics, shades, and opacification abilities, and determine the minimum thickness that will mask the discolored backgrounds.

The investigation of the processes involved in the masking ability of the combined application of opaquers and resin composites could improve esthetic outcomes and contribute to preserving dental structures by providing conservative dental preparations. Therefore, the purpose of this study was to evaluate the masking ability of different opaquer + resin composite combinations over simulated discolored backgrounds. The tested hypotheses consider that the masking ability of the combined application of opaquers and resin composites would be affected by the color of the backgrounds, by the brand of the opaquers, by the number of coatings of the opaquers, and by the resin composite thickness.

METHODS AND MATERIALS

Experimental Design

This laboratory study evaluated the masking ability of opaquers and resin composites placed over simulated dental backgrounds of different degrees of discoloration. The brand, composition, shade, and batch number of each material are presented in Table 1. The groups were divided according to the commercial brand of the opaquers, number of coatings of the opaquers (one or two coats), and thickness of the resin composite layer (0.5 or 1 mm). The experimental design and group divisions are presented in Figure 1.

Sample Preparation

Opaquer Coatings—A pilot study was conducted to determine the thickness of the opaquer coats. A thin layer of each opaquer was applied to a polyester sheet with a brush. After the opaquer was light-cured, the thickness of the opaquer + polyester sheet was measured with a digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Caliper, Takatsu-ku, Kawasaki, Kanagawa,

Table 1: *Materials, Manufacturers, Composition, Shade, and Batch Number*

Opaquer	Manufacturer	Composition	Shade	Batch Number
Empress Direct Opaque	Ivoclar Vivadent, Schaan, Liechtenstein	Dimethacrylates, barium glass, ytterbium trifluoride, Ba-Al fluorosilicate glass and mixed spheroidal oxides, catalysts, stabilizers, and pigments	Opaque	X16379
Opak	Angelus, Londrina, PR, Brazil	Bisphenol A diglycidyl methacrylate, urethane dimethacrylate, catalysts, stabilizers, pigments	A3	50458
Natural Flow Opaque	Nova DFL, Rio de Janeiro, RJ, Brazil	Bisphenol A diglycidyl methacrylate, dimethacrylate resins, boron-aluminum glass silicate, synthetic silica and pigments.	Opaque	18080524
Creative Color Opaquer	Cosmedent, Chicago, IL, USA	7,7,9-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diaza-hexadecan-1,16-diol dimethacrylate, bisphenol a diglycidyl methacrylate; 1,4 butanediol dimethacrylate	A3	184218
Resin Composite	**Manufacturer**	**Composition**	**Shade**	**Batch Number**
Z350 XT	3M ESPE, St Paul, MN, USA	Bisphenol A diglycidyl methacrylate, urethane dimethacrylate, triethylene glycol dimethacrylate, bisphenol hydoxyethyl methacrylate, polyethylene gycol dimethacrylate, BHT, silicate, zircônia	A1B	1911600460

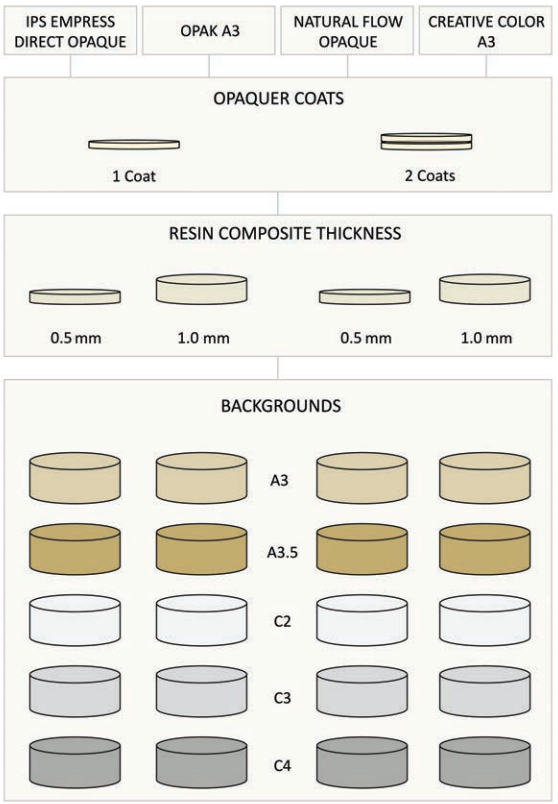

Figure 1. *Schematic representation of the experimental design and group division.*

Japan) and subtracted from the thickness of the polyester sheet. The same process was repeated in determining the thickness of two coats of each opaquer. All opaquer brands were measured five times for each option (one or two coatings), in triplicate, by the same operator, using a standardized procedure. The mean thickness values were used to determine the thickness of the opaquer coatings used in the present study.

One trained operator produced the samples. One drop of the opaquer was dispensed and pressed between two glass plates. Two polyester strips were placed between the opaquer and the glass plates for isolation purposes, and to prevent fracture of the sample after polymerization. A 0.75-kgf load was applied for 2 minutes to achieve disc films standardized at 30-μm thick and 11 mm in diameter. The upper and lower surfaces were light-cured for 40 seconds with a light-emitting diode (LED; Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) having 1000 mW/cm^2 irradiance.[15] The two-coat samples were obtained by pairing two opaquer coats using glycerin as a coupling medium between each coat.

Resin Composite Layers—The resin composite samples were made with an 11.0-mm diameter and 0.5-mm- or 1.0-mm-thick cylindrical metallic device. The resin composite was placed in one increment and light-cured for 40 seconds, on both sides, using an LED (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) having 1000

mW/cm² irradiance. Prior to polymerization, the upper surface of the samples was covered with a polyester strip and a glass plate with 1 kgf static load.[7]

Discolored Backgrounds and Reference Background—Opaque shade ceramic discs, 11.0 mm in diameter and 2.0-mm thick,[7,16,17] were used to simulate discolored dental backgrounds. The backgrounds were fabricated from feldspathic porcelain, dentin opacity, and VM13—shades A3, A3.5, C2, C3, and C4 (Vita Zahnfabrik, Bad Säckingen, Germany).

An A1 body-shade resin composite (Filtek Z350XT, 3M ESPE, St. Paul, MN, USA) disc, 11.0 mm in diameter and 4.0-mm thick,[9] was used as a reference to calculate the color difference for every combination tested. This resin composite disc was produced with the same resin composite shade used previously, simulating a tooth with no discoloration and representing the color objective to be achieved by the masking techniques.

Color Measurement—The color of the samples was measured using a calibrated reflection spectrophotometer (SP60 - EX Rite, Grand Rapids, MI, USA) over a white background. The resin composite layer (0.5-mm or 1.0-mm thick) and opaquer coatings (one or two coats) were combined to simulate various restorative masking options. All measurements were performed using glycerin as a coupling medium between all layers, and between the samples and the simulated backgrounds.

The total color differences were calculated using the L*, a*, and b* values of the resin composite specimens placed over each colored background (A3, A3.5, C2, C3, and C4), and the L*, a*, and b* values of the A1 body shade resin composite, using the CIEDE2000 color difference formula:

$$\Delta E_{00} = \left[\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T\left(\frac{\Delta C'}{k_C S_C}\right)\left(\frac{\Delta H'}{k_H S_H}\right)\right]^{\frac{1}{2}},$$

where $\Delta L'$, $\Delta C'$, and $\Delta H'$ refer to lightness, chroma, and hue differences among the color measurements, respectively, and k_L, k_C, and k_H are the parametric factors for the influence made by the conditions and the illumination. R_T (rotation function) accounts for the interaction of hue and chroma differences in the blue region. S_L, S_C, and S_H are the weighting functions for the color difference adjustment, considering the location variation of L*, a*, and b* coordinates.[7,18] Metric discontinuities due to mean hue computation and hue-difference computation were taken into account to calculate the ΔE_{00}.[19]

The interpretation of masking ability effectiveness was based on visual thresholds of acceptability and perceptibility, and on ratings described by Paravina and others.[20] The ΔE_{00} threshold values and interpretation ratings are presented in Table 2. The color shifts resulting from applying different opaquers and resin composite combinations were analyzed by the differences in CIEDE2000 lightness, chroma, and hue values.[21] The CIEDE2000 lightness (ΔL_{00}), chroma (ΔC_{00}), and hue (ΔH_{00}) color differences were defined as[22]

$$\Delta L_{00} = \frac{\Delta L'}{k_L S_L}; \Delta C_{00} = \frac{\Delta C'}{k_C S_C}; \Delta H_{00} = \frac{\Delta H'}{k_H S_H}.$$

Statistical Analyses

The mean ΔE_{00} values were assessed by analysis of variance (one-way ANOVA) and Tukey's *post hoc* test (α=0.05). A bivariate analysis was used to determine the association between the dependent (masking ability) and independent variables (opaquer manufacturer, opaquer coat, resin layer, and background shades), using the chi-square test followed by the residual adjustment test. The significance level adopted was 5% (α=0.05). Statistical analysis was performed using an SPSS software program (SPSS Statistics 23.0.0, IBM Armonk, Chicago, IL, USA).

RESULTS

The one-way analysis of variance showed significant differences among the groups (p<0.001) for all background shades. Table 3 presents the mean and standard deviation values of ΔE_{00} for each opaque + resin composite combination and background (A3, A3.5, C2, C3, C4). The lower ΔE_{00} values are associated with increased masking ability. The combinations with a 1.0-mm-thick resin composite layer presented lower mean ΔE_{00} values, regardless of the commercial brand or number of coatings of the opaquers. This pattern was observed for the majority of the backgrounds.

Table 2. *Interpretation of Color Differences Between Different Dental Materials and Structures Through 50%:50% Perceptibility (PT) and Acceptability Thresholds (AT)*[20]

Threshold	Rating and Interpretation[a]	ΔE_{00}
≤PT	(5) Excellent match	≤0.8
>PT, ≤AT	(4) Acceptable match	>0.8, ≤1.8
>AT, ≤AT x 2	(3) Mismatch type [a]	>1.8, ≤3.6
>AT x 2, ≤AT x 3	(2) Mismatch type [b]	>3.6, ≤5.4
>AT x 3	(1) Mismatch type [c]	>5.4

[a] *Mismatch types: [a], moderately unacceptable; [b], clearly unacceptable; [c], extremely unacceptable.*

Table 3. *Mean and Standard Deviation Values of ΔE_{00} for Each Tested Combination and Background*[a]

Background		A3	A3.5	C2	C3	C4
Opaquers	**Combination**					
E	a	2.57 (0.13) bFG	2.56 (0.30) bGH	1.79 (0.45) aCDEF	2.11 (0.29) abB	4.16 (0.14) cD
	b	1.36 (0.48) aABCD	1.48 (0.56) aCDE	0.94 (0.54) aABC	1.28 (0.63) aA	2.45 (0.23) bAB
	c	2.64 (0.13) abG	2.77 (0.13) bH	2.18 (0.53) aF	2.48 (0.06) abBC	3.77 (0.15) cD
	d	1.77 (0.25) bBCDE	1.96 (0.33) bDEFG	1.72 (0.45) bBCDEF	1.01 (0.31) aA	2.14 (0.37) bA
O	a	1.97 (0.09) abDEFG	1.85 (0.20) aDEF	2.11 (0.07) bF	3.05 (0.07) cC	5.52 (0.05) dF
	b	1.15 (0.24) bcABC	0.65 (0.06) aA	1.01 (0.15) bABCD	1.41 (0.16) cA	3.15 (0.11) dC
	c	1.96 (0.08) aDEFG	2.02 (0.23) aEFG	2.37 (0.20) bF	2.69 (0.06) cBC	4.09 (0.19) dD
	d	0.81 (0.10) aA	0.82 (0.15) aAB	1.18 (0.09) bABCDE	1.23 (0.20) bA	2.16 (0.10) cA
N	a	2.19 (0.50) aEFG	2.06 (0.20) aEFG	1.95 (0.67) aEF	2.54 (0.22) aBC	4.79 (0.29) bE
	b	1.02 (0.44) abAB	1.01 (0.26) abABC	0.50 (0.40) aA	1.31 (0.55) bA	2.72 (0.14) cB
	c	2.44 (0.22) bEFG	2.32 (0.16) abFGH	1.99 (0.31) aEF	2.44 (0.26) bBC	4.58 (0.14) cE
	d	1.23 (0.62) aABCD	1.37 (0.43) aBCD	0.99 (0.44) aABC	1.32 (0.50) aA	2.72 (0.30) bB
C	a	1.83 (0.34) bCDEF	1.37 (0.30) aBCD	2.14 (0.23) bcF	2.48 (0.14) cBC	4.73 (0.05) dE
	b	0.68 (0.30) aA	0.63 (0.20) aA	0.90 (0.40) abAB	1.23 (0.13) bA	2.86 (0.10) cBC
	c	1.66 (0.47) aBCDE	1.81 (0.18) aDEF	1.87 (0.13) abDEF	2.32 (0.17) bB	4.15 (0.13) cD
	d	0.70 (0.44) aA	0.74 (0.20) aA	0.88 (0.46) aAB	1.25 (0.23) aA	2.45 (0.10) bAB

Abbreviations: E, Empress Direct Opaquer; O, Opak; N, Natural Flow; C, Creative Color; a, one opaquer coat + 0.5-mm resin composite layer; b, one opaquer coat + 1.0-mm resin composite layer; c, two opaquer coats + 0.5-mm resin composite layer; d, two opaquer coats + 1.0-mm resin composite layer.

[a] *Different lowercase letters in the same line indicate statistically significant differences. Different uppercase letters in the same column indicate statistically significant differences. Standard deviation values inside the parentheses.*

In an overall analysis, the comparison between the different colored backgrounds showed significantly higher mean ΔE_{00} values for C4 background, for all multilayering combinations. The comparison between multilayering combinations showed significantly lower mean ΔE_{00} values for the combinations with 1 or 2 opaquer coats combined with 1.0-mm resin composite (Table 3).

The association values (%) between dependent and independent variables are shown in Table 4. There was

a significant association between masking ability and opaquer brand (χ^2=9.92; p=0.019), and between resin composite layer (χ^2=134.02; p<0.001) and background shade (χ^2=78.80; p<0.001). The Creative Color Opaquer (Cosmedent) was significantly associated with acceptable masking ability, whereas Empress Direct Opaque (Ivoclar Vivadent) was significantly associated with unacceptable masking. A 1.0-mm resin composite layer was significantly associated with acceptable masking ability, and a 0.5-mm resin composite layer was significantly associated with unacceptable masking ability. Background shades A3, A3.5, and C2 were significantly associated with acceptable masking capacity, whereas C4 was associated with unacceptable masking ability.

Figure 2 presents the mean ΔE_{00} values for each group and the respective visual thresholds of perceptibility and acceptability.[20] Excellent matches ($\Delta E_{00} \leq 0.8$) were observed for combinations of one or two opaquer coats + a 1.0-mm resin composite layer associated with A3, A3.5, and C2 backgrounds. The acceptable matches for the C3 background were 1.0-mm resin composite layer combinations, regardless of the number of coatings. Acceptable matches were obtained for combinations with 0.5-mm-thick resin composite over C2, A3, and A3.5 backgrounds. The majority of opaquer + resin composite combinations associated with the C4 background presented clearly unacceptable mismatch threshold values, and no masking ability was detected.

Figure 3 shows the ΔL_{00}, ΔE_{00}, and ΔH_{00} shifts for clearly unacceptable opaque + resin composite combinations over the C4 background. The ΔE_{00} color shifts were mostly influenced by ΔC_{00} for combinations associated with the A3, C2, C3, and C4 backgrounds. Overall, the combinations were just slightly affected by ΔH_{00} and ΔL_{00}, except for Opak combinations associated with the A3 and A3.5 backgrounds, and Empress combinations associated with the A3, A3.5, and C2 backgrounds, respectively.

DISCUSSION

The present study evaluated the masking ability of the combined application of four opaquers—in one or two coats—and one resin composite in two thicknesses—0.5 mm and 1.0 mm—over different background shades. The combined application of opaquer and resin composite

Table 4. *Association Values (%) Between Masking Ability and Opaquer Manufacturer, Opaquer Coats, Resin Composite Layer Thickness, and Background Shades*

	Masking Ability[a]		
Opaquers + Resin Composite	**Acceptable - ΔE_{00}≤1.8 n (%)**	**Unacceptable - ΔE_{00}>1.8 n (%)**	***p*-value**
Opaquers			
Creative Color	**56 (31.6%)**	44 (19.7%)	0.019
Empress	34 (19.2%)	**66 (29.6%)**	
Natural Flow	43 (24.3%)	57 (25.6%)	
Opaque	44 (24.9%)	56 (25.1%)	
Opaquer Coats			
1	93 (52.5%)	107 (48.0%)	0.365
2	84 (47.5%)	116 (52.0%)	
Resin Composite Thickness			
0.5 mm	31(17.5%)	**169 (75.8%)**	<0.001
1.0 mm	**146 (82.5%)**	54 (24.2%)	
Background Shades			
A3	**45 (25.4%)**	35 (15.7%)	<0.001
A3.5	**45 (25.4%)**	35 (15.7%)	
C2	**49 (27.7%)**	31 (13.9%)	
C3	37 (20.9%)	43 (19.3%)	
C4	1 (0.6%)	**79 (35.4%)**	

[a]*Percentual values (%) are in the parentheses. Absolute residuals in bold are those that exceed +/- 2.*

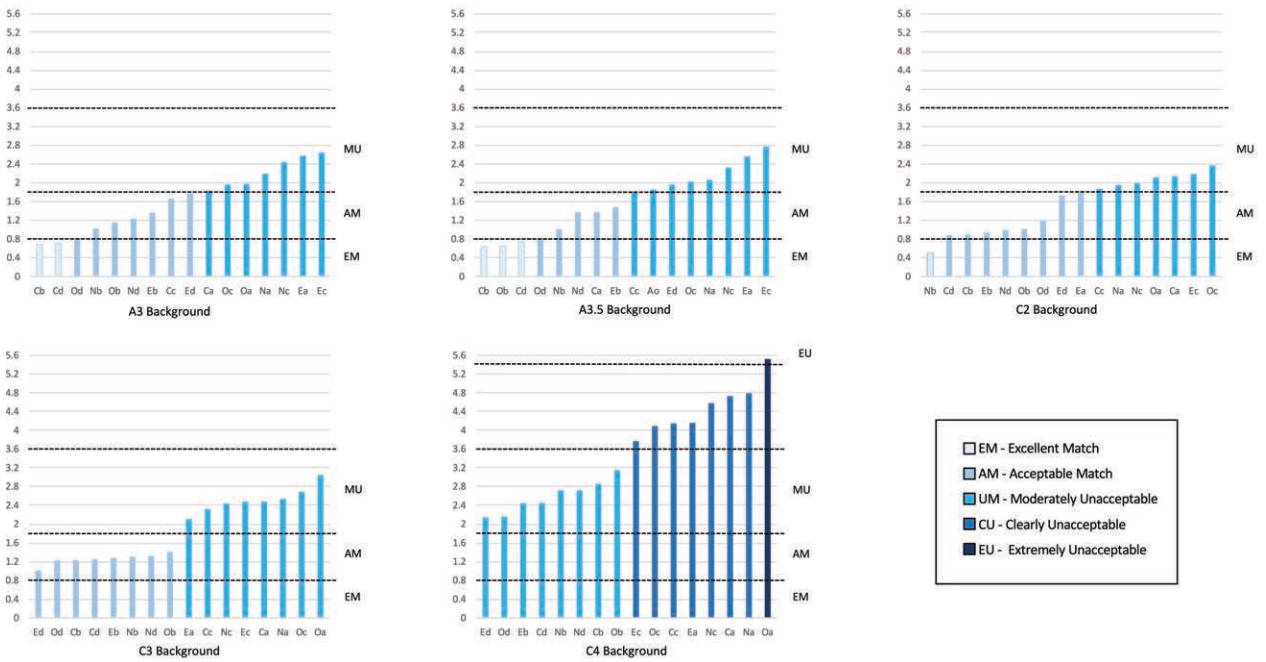

Figure 2. *Mean ΔE_{00} values for A3, A3.5, C2, C3, and C4 backgrounds and threshold interpretation ratings for each combination. Abbreviations: E, Empress Direct Opaquer; O, Opak; N, Natural Flow; C, Creative Color; a, one opaquer coat + 0.5-mm resin composite layer; b, one opaquer coat + 1.0 mm resin composite layer; c, two opaquer coats + 0.5-mm resin composite layer; d, two opaquer coats + 1.0-mm resin composite layer; EM, excellent match; AM, aceptable match; UM, moderately unacceptable mismatch; CU, clearly unacceptable mismatch; EU, extremely unacceptable mismatch.*

achieved effective masking ability over the majority of the backgrounds. The tested hypothesis was partially accepted because the masking ability was influenced by the opaquer brands, resin composite thickness, and background shades, whereas the number of opaquer coats did not significantly affect the masking ability.

According to the manufacturers' instructions, the opaquers should be applied with a fine brush in thin coats. Clinically, the opaquers are applied over the discolored background, with no set pattern.[23] Depending on the inherent characteristics of the opaquer, such as opacity/translucency, viscosity, color, and the relation between the severity of the discolored background versus the color objective to be achieved, an additional coating may be applied to increase the thickness of the opaquer layer, and to achieve greater

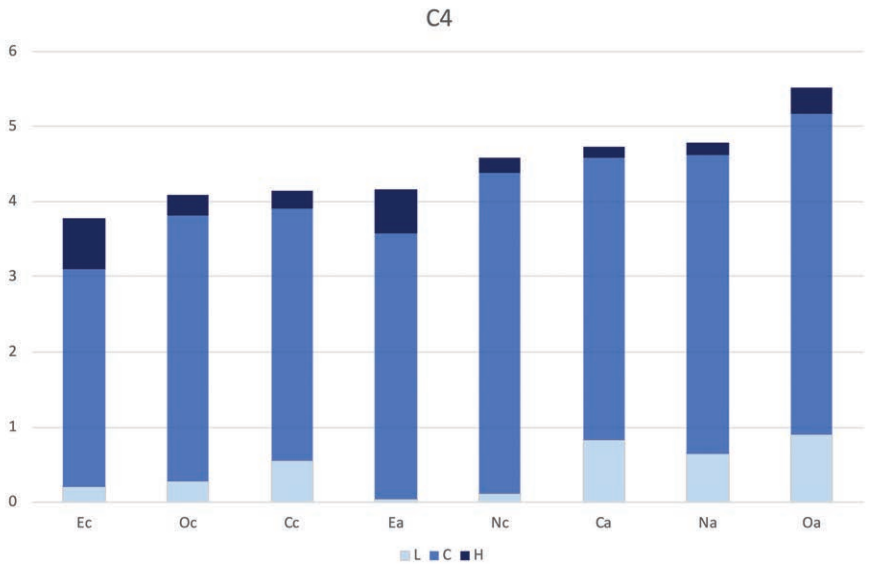

Figure 3. *CIEDE2000 (ΔE_{00}) color shifts for clearly unacceptable combinations associated with C4 discolored background. The influence of the lightness, chroma, and hue differences in the total color shifts is shown. Abbreviations: E, Empress Direct Opaquer; O, Opak; N, Natural Flow; C, Creative Color; a, one opaquer coat + 0.5-mm resin composite layer; c, two opaquer coats + 0.5-mm resin composite layer; L, ΔL_{00} CIEDE2000 lightness difference; C, ΔC_{00} CIEDE2000 chroma difference; H, ΔH_{00} CIEDE2000 hue difference.*

coverage of the background. The shade and the choice of the opaquer manufacturer may be selected by the operator, whereas the thickness cannot be completely controlled,[24] making it difficult to obtain the exact measurement of the coating thickness. The thickness of the opaquer coatings used in the present study was determined by a pilot study because the authors could not find any published data regarding the thickness of the opaquer coat to use as a reference.

Dental discolorations may be caused by different factors, such as pulp hemorrhage, pulp necrosis, pulp canal calcification, endodontic materials,[25] tetracycline-containing medicines, and exposure to food and beverage pigments and tobacco smoke.[26] Depending on the etiological factor that caused the color alteration, a wide variety of shade and discoloration intensities may be observed.[25,26] In the present study, five background shades were selected in order to simulate different conditions and degrees of masking difficulty found in clinical practice. The C2 background simulates a mild discoloration, A3 and A3.5 an intermediate discoloration, and C3 and C4 a severe discoloration, thus representing low, medium, and high masking difficulty.[27,28]

Resin composites have limited masking ability, owing to their inherent optical properties.[1] Their masking ability is affected by the translucency and thickness of the layers, as well as the degree of discoloration of the underlying tooth structures.[4,5,7-9,29-31] Darker backgrounds are more difficult to mask. In these situations, opaque shades and thicker resin composite layers are recommended to achieve improved masking ability.[4,7,16,32]

The concept of minimally invasive dentistry has driven esthetic treatments to adopt a more conservative approach, designed to preserve tooth structure.[33,34] However, conservative preparations imply a reduced thickness of the composite layer, hence a greater influence of the background on the final color of the restoration.[4,5,7,9,30,32] Opaquers may contribute to preserving dental structures because their high opacification ability allows them to be used in very thin coats.[1]

This study was undertaken to simulate esthetic treatment solutions in line with the minimally invasive concept, by testing the combined application of opaquers and resin composite layers with reduced thicknesses. The four opaquers tested are basically composed of highly pigmented resinous materials containing metal oxides that are responsible for their opacification, characteristic tint, and saturation.[1] The opaquers were selected among the commercially available brands. Opaquers with different characteristics within the commercial brands available were selected to represent a wide range of opacification possibilities. The shade selection was determined based on the options available for each product. Empress Direct Opaque (Ivoclar Vivadent) and Natural Flow Opaque (Nova DFL) have only one universal opaque shade option. Opak (Angelus) has two shade options (B0.5 and A3) and Creative Color (Cosmedent) has a wide variety of shades. Shade A3 was selected for both Opak (Angelus) and Creative Color (Cosmedent) to standardize the hue and chroma, because it was the only shade shared by the two products.

Color measurements were performed using glycerin as a coupling medium between the resin composite and the opaquer and the porcelain background, to enhance the optical contact between each layer of the specimens. Glycerin was used ultimately to simulate the oral environment[35] to prevent undesirable effects of air on optical properties[36] and to minimize the light refraction that occurs when a light beam crosses materials with different refractive indices.[37] It is recommended that the refractive index of the coupling agent and the tested materials be the same.[37,38] Glycerin, porcelain,[37] and resin composites[39-41] have similar refractive indices (n=1.5). However, it could be assumed that the opaquers would present a higher refractive index (n>1.5), owing to their higher opacity.[41] The possible difference in the refraction indices could be considered a limitation of the present study, and should be taken into account when interpreting the present results and applying them in clinical practice.

The effectiveness of the masking ability was visually interpreted according to the perceptibility and acceptability thresholds for ΔE_{00}.[20] In an overall analysis, an excellent match was achieved for a small number of combinations (8%). The feature that these combinations held in common was the thickness of the resin composite layer. The combinations that yielded excellent match were obtained with a 1.0-mm-thick layer of resin composite, regardless of the number of coatings of the opaquers. In the present study, the masking ability was negatively affected when the thickness of the resin composite layer was reduced to 0.5 mm. These findings are in line with previous research that has reported an improvement in masking ability when the thickness of the resin composite layer is increased.[4,5,7,8,16,29,30,32,42] Acceptable matches with a mean ΔE_{00} ranging from $0.8 > \Delta E_{00} \leq 1.8$ were also found, mainly for combinations of 1.0-mm-thick resin composite layers for all backgrounds except C4. Moderately unacceptable matches were observed in all backgrounds, and were generally associated with 0.5-mm resin composite layers. However, in some situations, acceptable results were observed for 0.5-mm-thick resin composites combined with both Creative Color

and Empress Direct opaquers over C2, A3, and A3.5 backgrounds. This confirms that opaquer coatings may improve the masking of discolored backgrounds with 1.0-mm and 0.5-mm-thick resin composite layers, and thus contribute to minimizing tooth reduction.

Previous studies showed effective masking with a 1.5-mm-thick dentin shade resin composite.[7,16] However, it is important to understand that additional space is required for resin composite layering to achieve a natural appearance in the final restoration.[43] The body shade resin composite used in the present study is considered a universal resin composite, with an intermediate translucency that is lower than the enamel shade and higher than the dentin shade.[9] The present study demonstrated that the combined application of opaquers with a less opaque universal resin composite may be achieved, thus reducing the space required for layering.

The tested backgrounds that simulated discolored tooth structures differed in regard to hue, chroma, and brightness. According to the VITA Lumin Classical Shade Guide manufacturer, the following sequence was observed when arranged in descending order of brightness (value): C2 > A3 > A3.5 > C3 > C4.[44] The C2 background was the most favorable color match, obtained from the combination of one coat of Natural Flow opaquer + 1.0-mm-thick resin composite layer, with a mean ΔE_{00} value of 0.5. In contrast, the C4 background had the highest chromatic discrepancy, obtained from the combination of one coat of Opak opaquer + 0.5-mm-thick resin composite layer, with a mean ΔE_{00} value of 5.52. These findings may be attributed to the brightness of the C2 and C4 backgrounds, since they represent the highest and the lowest brightness values, respectively, among the tested backgrounds.[44] Clearly unacceptable and extremely unacceptable mismatches were observed only for the C4 background, corroborating previous studies indicating that darker backgrounds with lower values are more difficult to mask.[4,5,7-9,16,30,32]

The relative visual ascending order of translucency among the tested opaquers considers Opak < Empress Direct Opaque < Creative Color ≤ Natural Flow (Figure 4). The most translucent opaquers were as effective as the least in masking most of the backgrounds. In contrast, not even the least translucent opaquers were able to achieve acceptable matching values for the C4 background.

The ability to mask different backgrounds is a complex mechanism that involves light absorption and scattering.[4] The metal oxides present in the composition of the opaquers increase the light that is reflected toward the observer, thus improving the ability to mask the color of the underlying background.[1,4] However, opaquers with high opacity do not always provide the best results. Excessive opacity may negatively affect the final color of the restoration, especially over mild discolorations and conservative preparations, leading to lifeless and unnatural results.[14] This is confirmed by the positive results achieved with both the Creative Color and the Empress Direct opaquers combined with a 0.5-mm-thick resin composite over C2, A3, and A3.5 backgrounds. Both opaquers tested presented effective masking with reduced thickness of the resin composite, but did not present the highest opacity visually.

The clinical significance of the results points out that not only do the opaquers differ in masking ability, but the masking ability is influenced by the background color and thickness of the composite layer. In general, the application of one or two opaquer coats combined with a 1.0-mm-thick layer of body shade resin composite is recommended for covering discolored backgrounds, and providing restorations with an acceptable match. However, in cases of mild and intermediate background discolorations (C2, A3, A3.5 shades), the application of one or two opaquer coats combined with a 0.5-mm-thick layer of body shade resin composite may also provide adequate masking of the background color. In order to mask darker substrates, alternative combinations with thicker layers of dentin shade resin composites should be applied.

The combined application of opaquer and resin composite is a less invasive option for masking discolored backgrounds. However, there are few studies that have addressed the combined effect of these materials.[10,13,45] The majority of the findings regarding the use of opaquers have been reported in the form of case reports; hence, this topic has not been thoroughly researched. The understanding of the optical behavior of each opaquer is essential to obtain the high-quality masking of discolored backgrounds. Future studies with different combinations of resin shades, stratification techniques, and thicker opaquer coatings are recommended to

Figure 4. *Photographic demonstration of the relative ascending order of translucency among the tested opaquers: Opak < Empress Direct Opaque < Creative Color ≤ Natural Flow.*

solve the difficulties regarding the masking of severely discolored backgrounds. To date, this study was able to clarify some important issues regarding the combined application of opaquers and resin composites, and also contribute to the understanding of the behavior of these materials of great, but underinvestigated, potential.

CONCLUSIONS

The masking ability of a combined application of opaquer and resin composite was affected by the opaquer brand, resin composite thickness, and background shade. Most of the results that achieved either excellent or acceptable masking ability were obtained with combinations composed of one or two coats of opaquer and a 1.0-mm-thick resin composite layer. Acceptable masking ability was obtained for combinations with 0.5-mm-thick resin composite over C2, A3, and A3.5 backgrounds, and with 1.0-mm-thick opaquer-resin combinations over all backgrounds except C4.

Conflict of Interest

The authors have no financial interest in any of the companies or products mentioned in this article.

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Departments

Errata

Operative Dentistry apologizes for the errors in the following manuscripts.

AD Loguercio, LJC Vargas, MW Favoreto, HF Andrade, CP F Borges, A Dávila-Sánchez, A Reis, CP Mora; Effects of Microabrasion Prior to In-office Bleaching on Hydrogen Peroxide Permeability, Color Change, and Enamel Morphology. *Oper Dent* 1 November 2021 **46(6)** 661-668. doi: https://doi.org/10.2341/20-179-L

There are errors in the author order and contact list. The correct author order and author affiliations list should read (corrections are underlined):

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Additionally, the legend in Table 3 should read:

*Identical uppercase or lowercase letters in each column indicate statistically similar means (one-way ANOVA and Tukey test, α=0.05).

D Kaisarly, M ElGezawi, R Haridy, A Elembaby, A Aldegheishem, R Alsheikh, KS Almulhim; Reliability of Class II Bulk-fill Composite Restorations With and Without Veneering: A Two-year Randomized Clinical Control Study. *Oper Dent* 1 September 2021 **46(5)** 491-504. doi: https://doi.org/10.2341/19-290-C

There are errors in the author order and contact list and corresponding author information. The correct author order, author affiliations list, and corresponding author information should read (corrections are underlined):

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BM Moran, PK Ziegelmann, SB Berger, A Burey, T de Paris Matos, E Fernández, AD Loguercio, A Reis; Evaluation of Tooth Sensitivity of In-office Bleaching with Different Light Activation Sources: A Systematic Review and a Network Meta-analysis. *Oper Dent* 1 September 2021 **46(5)** E199–E223. doi: https://doi.org/10.2341/20-127-L

There are errors in the author names and contact list, in the Summary, and in the Results. The correct author spelling and author affiliations list should read (corrections are underlined):

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In the Methods paragraph of the Summary:

The sentence, "A comprehensive search was performed in PubMed, Bridge Base Online (BBO), Latin American and Caribbean Health Sciences Literature database (LILACS), Cochrane Library, Scopus, Web of Science, and grey literature without date and language restrictions on April 23, 2017 (updated on September 26, 2019)."

Should read (correction is underlined):

"A comprehensive search was performed in PubMed, Bibliografia Brasileira de Odontologia (BBO), Latin American and Caribbean Health Sciences Literature database (LILACS), Cochrane Library, Scopus, Web of Science, and grey literature without date and language restrictions on April 23, 2017 (updated on September 26, 2019)."

In the Study Selection paragraph in the Results section:

The sentence, "After title screening, 227 studies remained, and this number was reduced to 32 full texts that were assessed for eligibility (Figure 1)."

Should read (correction underlined):

"After title screening, 228 studies remained, and this number was reduced to 32 full texts that were assessed for eligibility (Figure 1)."

Online Only Articles

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Carious and Eroded Substrates and the Bonding of Adhesive Systems: A Systematic Review and Meta-analysis

TT Fröhlich • BD Ilha • FZM Soares • RO Rocha

Caries-altered enamel and dentin of primary and permanent teeth impair bonding anderoded permanent dentin jeopardizes adhesion.

http://doi.org/10.2341/20-277-LIT

Evaluation of Cleaning Methods on Lithium Disilicate Glass Ceramic Surfaces After Organic Contamination

J Fagan-Junior • J Vesselovcz-Junior • J Puppin-Rontani • L Correr-Sobrinho • KMS Freitas • TC Robertson • RR Pacheco • NIP Pini • D Sundfeld

Air–water spray, 35% phosphoric acid, 70% alcohol, and Ivoclean are effective cleaning methods for removing saliva from a previously etched and silanized lithium disilicate glass ceramic. When contaminated with human blood, only Ivoclean cleaning paste was able to restore the initial bond strength.

http://doi.org/10.2341/20-176-L

Effect of Dentin Moisture in Posterior Restorations Performed with Universal Adhesive: A Randomized Clinical Trial

AS Castro • BM Maran • MF Gutierrez • K Chemin ML Mendez-Bauer • JP Bermúdez • A Reis • AD Loguercio

Dentin moisture seems not to be important for the postoperative sensitivity or clinical performance of posterior bulk-fill composite restorations, when a universal adhesive was applied.

http://doi.org/10.2341/20-215-C

Influence of Manual and Ultrasonic Scaling on Surface Roughness of Four Different Base Materials Used to Elevate Proximal Dentin–Cementum Gingival Margins: An *In Vitro* Study

HS Ismail • AI Ali • F Garcia-Godoy

In terms of surface roughness, resin-based composite could be recommended for gingival margin elevation of subgingival proximal cavities rather than glass ionomer-based restorative materials. Whenever noninvasive periodontal treatment is required for such restored cavities, hand scaling may be preferable rather than the ultrasonic method.

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Carious and Eroded Substrates and the Bonding of Adhesive Systems: A Systematic Review and Meta-analysis

TT Fröhlich • BD Ilha • FZM Soares • RO Rocha

Clinical Relevance

Caries-altered enamel and dentin of primary and permanent teeth impair bonding and eroded permanent dentin jeopardizes adhesion.

SUMMARY

Objective: To evaluate the influence of caries- and erosion-altered substrates (enamel and dentin) on the bond strength of adhesive systems to permanent and primary teeth through a systematic review and meta-analysis.

Methods: This review was conducted according to Preferred Reporting Items for Systematic Reviews and Meta-analyses (PRISMA). Laboratory studies reporting the bond strength of adhesive systems to caries- or erosion- altered substrates compared to sound enamel or dentin (control) were identified in the electronic databases (Medline/Pubmed, Scopus, and Lilacs). Two authors independently selected studies and extracted relevant data. Meta-analysis was performed considering the bond strength values as the outcome, and using a random-effects model, at a significance level of $\alpha = 0.05$. The quality of the studies (risk of bias) and heterogeneity among studies (Cochran and I2 tests) were assessed.

Results: Out of 1254 articles identified, 122 studies met all inclusion criteria, while 114 were included in the meta-analyses. The bond strength to sound enamel and to sound dentin were higher than to demineralized enamel and caries-affected dentin ($p<0.01$), respectively, both in permanent and primary teeth. Erosion impaired the bonding only to permanent tooth dentin ($p<0.01$). Bond strength to eroded enamel was not affected in permanent ($p=0.87$) or primary teeth ($p=0.49$). After aging, dentin bond strength was affected by carious and eroded challenges ($p<0.01$).

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Conclusion: The *in vitro* evidence suggests that bonding to dentin and enamel is jeopardized by demineralization associated with caries. Eroded dentin experiences decreased bonding only in permanent teeth. Bonding to enamel is not affected by erosion.

INTRODUCTION

Almost all adhesive system evaluations are performed using sound enamel and dentin.[1-3] However, bonding solely to sound substrates is unusual in clinical practice, as caries- or erosion-altered enamel and dentin are often present in clinical situations after caries excavation,[4-6] using minimally invasive dentistry concepts or due to the increased prevalence of dental erosion.[7,8]

Carious and erosive wear processes can significantly modify the enamel and dentin characteristics, reducing the mineral content and increasing the porosity on both substrates.[9-11] Caries demineralized enamel presents widened intercrystalline spaces and larger pore volumes than sound enamel.[12,13] On dentin, caries and erosive challenges induce modifications of the collagen structure,[14,15] content, and distribution of noncollagenous protein[15] and increased moisture.[16] Although they are different processes and cause different changes in enamel and dentin, it is expected that all chemical and physical modifications resulting from the carious and erosive processes on enamel and dentin may affect the adhesion to these substrates. In general, lower bond strength has been verified when caries- or erosion-altered substrates are compared to sound substrates,[17-27] especially in dentin. However, similar[28-33] or even superior[34-37] results are also found in these altered substrates. Differences in cariogenic and erosive challenge aggressiveness, laboratory protocols, and the adhesive systems tested may be associated with these divergent results.

Given the uncertainties and controversies on the impact of caries- or erosion-altered substrates on bond strength, this systematic review and meta-analysis aimed to evaluate the influence of substrate conditions (caries-altered and eroded) on immediate and long-term bonding performance of adhesive systems to enamel and dentin of primary and permanent teeth. The hypothesis tested was that the condition of the substrate—caries-altered or eroded—would negatively influence the bond strength values of enamel and dentin.

METHODS AND MATERIALS

Protocol

This systematic review was conducted according to the guidelines of the PRISMA statement (Preferred Reporting Items for Systematic Reviews and Meta-analyses).[38] The research question was as follows: Does the substrate condition (caries-altered or eroded) influence the bond strength of adhesive systems?

Information Sources and Search Strategy

The electronic databases PubMed (MEDLINE), Scopus, and LILACS were searched to identify studies through May 2020 that could be considered, with no limits on language or publication year. The search in the MEDLINE database via PubMed was performed using specific medical subjects headings (MeSH) and keywords as follows: ((((((((((((((((((((((((Dental Caries[MeSH Terms]) OR dental caries) OR caries) OR caries affected dentin*) OR caries-affected dentin*) OR carious-affected dentin) OR caries infected dentin*) OR demineralized dentin*) OR carious dentin*) OR demineralized enamel) OR demineralization) OR Tooth demineralization[MeSH Terms]) OR enamel caries) OR tooth demineralization) OR artificial caries) OR natural caries) OR Tooth Erosion[MeSH Terms]) OR tooth erosion) OR eroded enamel) OR eroded dentin*) OR dental erosion) OR erosion) AND ((((((((((((((((((((Adhesives[MeSH Terms]) OR adhesive*) OR adhesion) OR adhesive system*) OR Dental Bonding[MeSH Terms]) OR dental bonding) OR Dentin-Bonding Agents[MeSH Terms]) OR dentin bonding agent*) OR total-etch adhesive*) OR total-etch adhesive system*) OR total-etch) OR total-etching) OR conventional adhesive) OR etch-and-rinse adhesive*) OR self-etch adhesive*) OR self-etch adhesive system*) OR self-etch*) OR self-etching primer*) OR all-in-one adhesive*) OR one-bottle adhesive*) OR universal adhesive*)) AND (((((((((Enamel[MeSH Terms]) OR enamel) OR Dentin[MeSH Terms]) OR dentin*) OR sound) OR sound enamel*) OR normal dentin*) OR normal enamel OR noncarious dentin*)) AND (((((((((bond strength) OR microtensile) OR micro shear) OR tensile) OR Tensile Strength[MeSH Terms]) OR tensile strength) OR shear) OR shear strength) OR Shear Strength[MeSH Terms]). For Scopus and Lilacs, the keywords related to the search strategy were: Carious; Eroded; and Adhesive and Bond strength.

Selection, Inclusion, and Exclusion Criteria

Two independent review authors (TTF and BDI) with an excellent agreement (Kappa, κ=0.97) assessed the titles and abstracts of the potential articles, and selected considering the eligibility criteria: *In vitro* studies comparing the bond strength of adhesive systems between sound and altered substrates (caries-altered or eroded). The eligible papers were full text,

and studies that assessed root surface dentin, different application protocols of restorative, bond strength to brackets, or did not present immediate bond strength results were not included. The agreement between the authors, considering the exclusion criteria was substantial (κ=0.95). The reference lists of all included studies were manually screened to retrieve all relevant papers. Any disagreement regarding the eligibility was solved through discussion and consensus by a third reviewer (ROR).

Data Extraction

Two authors (TTF and BDI) performed the data extraction using a standardized form in Microsoft Office Excel 2016 (Microsoft Corporation, Redmond, WA, USA). For each paper, the following were systematically extracted: publication year, first author's country, type of teeth used, sample size, substrate (enamel or dentin), substrate condition (caries altered or eroded), type of lesion (natural or artificial), restorative material, adhesive system, mechanical test, storage time, and bond strength (mean values in MPa and standard deviations).

For studies that did not report the numerical bond strength values or that presented the results in graphs or figures, corresponding authors were contacted by e-mail (at least twice). If no information was provided, the study was not included in the meta-analysis.

Risk of Bias Assessment of Individual Studies

The risk of bias was based on and adapted from a previous study.[39] It was evaluated according to the article's description of the following parameters for quality assessment: teeth randomization, description of sample size calculation, specimens with similar cross-sectional area, failure mode evaluation, application according to the manufacturers' instructions, a single operator during the specimen preparation, and blinded operator to experimental condition during the tests. If the authors reported the parameter, the article received a "Yes"; if it was impossible to find the information, the article received a "No." Articles that reported 1-3 items were classified as having a high risk of bias, 4-5 items as medium risk of bias, and 6-7 items as low risk of bias.[40] Risk of bias was assessed considering the substrate condition—carious and eroded—separately.

Data Analysis

Pooled-effect estimates were obtained by comparing the immediate bond strength means (only 24-hour data) from sound and caries-altered enamel and dentin, separately, as well as considering the subgroups—permanent and primary teeth. The same analyses were performed to compare the sound and eroded substrates. Overall meta-analysis was performed considering eroded enamel and dentin, separately. Moreover, meta-analyses were performed for evaluating the influence of the aging (water storage) on bond performance for studies that had a storage time or aging group for at least 6 months for each substrate condition—caries-altered and eroded. For studies that evaluated more than one adhesive system, the bond strength means and standard deviations were combined to one mean and standard deviation for each substrate condition according to a predefined formula.[41] For studies that included water storage and thermocycling groups, only water storage data were considered.

All analyses were conducted using Review Manager (RevMan version 5.4 software, Cochrane Collaboration, Copenhagen, DENMARK, 2020) with a random-effect method. A p-value ≤ 0.05 was considered statistically significant. Statistical heterogeneity among studies was considered using the Cochran Q test and inconsistency I^2 test (>50% indicated high heterogeneity).[41]

RESULTS

Search and Selection

A flowchart summarizing the selection process for studies according to the PRISMA statement[39] is shown in Figure 1. A total of 1691 potentially eligible studies were found. After the removal of duplicates, 1254 records were examined by the titles and abstracts. From these, 1100 studies were excluded for not evaluating bond strength or comparing substrate conditions; three studies were excluded for assessing the bond strength of orthodontic brackets, five studies for considering root dentin as substrate, eleven studies for not evaluating an adhesive system, and three for reporting data similar to previously published studies. Also, four studies were excluded for not evaluating the immediate bond strength, and seven studies were not possible to obtain the full-text version after contact with the authors. One study was identified in references of included studies.[42] Therefore, 122 studies were included in the systematic review. Nevertheless, seven[43-49] of them did not present numerical data for mean and standard deviation even after contacting the authors by e-mail, and one study[50] did not inform clearly the number of teeth used and were not included in the meta-analysis. Thereby, 114 studies were included in the meta-analysis.

Characteristics of the Included Studies

Table 1 and Table 2 show the descriptive data of the included studies, separately by caries-altered and eroded substrate condition, respectively. Studies were

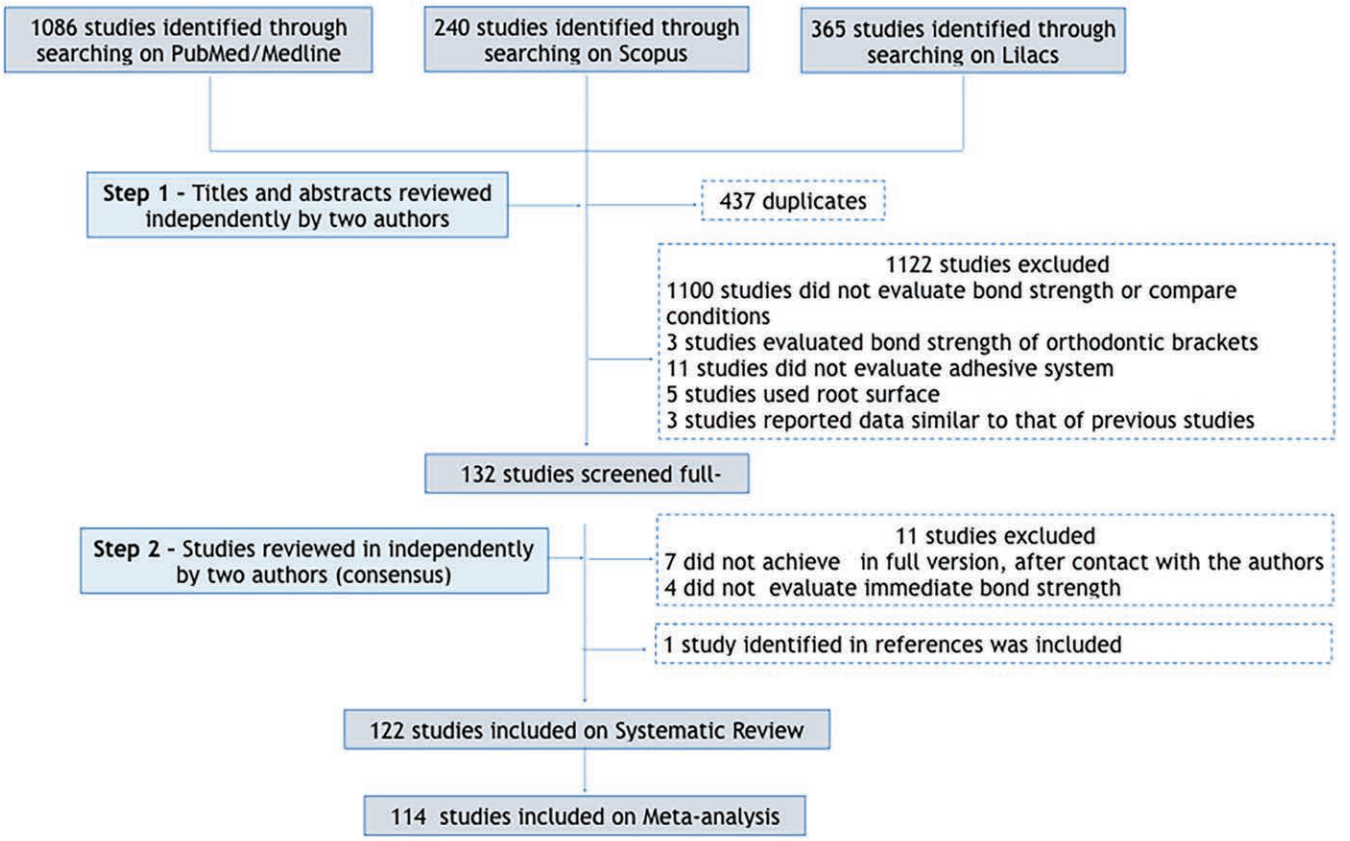

Figure 1. *Flowchart diagram of study selection according to PRISMA statement.*

published between 1994 and 2020. For the caries-altered condition, 96 studies were included, of which 93 were published papers[a] and three theses [53,101,102]; most of them (89 studies, 92.7%) evaluating the dentin substrate. Regarding the type of tooth, most of the studies evaluated permanent molars (65 studies, 67.7%), 16 studies used primary molars (16.7%), three studies (3.1%) evaluated both primary and permanent teeth, and 12 studies (12.5%) used bovine incisors. 52 studies used natural caries, 43 artificial lesions, and one study evaluated both types of lesions.[102] For the eroded condition, 27 studies (25 published articles[b] and two thesis[130,133]) were included, from which 20 evaluated dentin substrate, four evaluated enamel, [21,32,37,131] and three evaluated both substrates. [34,120,136] Among these studies, permanent molars were the most common teeth (59.2%), nine studies used bovine teeth, and two studies evaluated primary teeth.[120,136] All studies used artificial eroded lesions. Most of the primary studies only assessed immediate bond strength results after 24 hours of storage—12 studies considering caries-altered substrates and 11 studies considering eroded substrates evaluated the bond strength after aging (water storage). Microtensile was the most common bond strength test (82 studies, 67.2%), followed by the microshear bonding test (20 studies, 16.4%). Adhesive systems representative for all categories commercially available were considered in the included studies.

[a] *References 4-6,11,14,15,17,19,20,22,24,26,33,35,36,42-48,50-110*
[b] *References 18,21,23,28,29,32,34,37,49,120-129,131,132,134-137*

Meta-analyses

Eighty-nine studies were included in the meta-analysis comparing the adhesive bonding of caries-altered *versus* sound substrate, seven studies considering enamel bond strength[5,19,22,25,96,112,115] and 82 dentin bond strength. Caries-altered enamel and dentin were considered separately; and, for both the substrates, the comparison group was subgrouped into permanent and primary teeth. Meta-analysis comparing eroded *versus* sound substrate included 22 studies that assessed dentin substrate and seven studies that assessed enamel substrate.[21,32,34,37,120,131,136] Besides a subgroup meta-analysis for the eroded substrate on permanent and primary teeth was performed. A separate analysis was performed evaluating the bond strength after aging. Any study that evaluated the bond strength to demineralized or eroded enamel after six or 12 months of water storage

Table 1: *Characteristics of the Studies of Caries-altered Substrate Condition*

Study	Country	N[a]	Adhesive System	Bond Strength Test[b]	Substrate	Type of Lesion	Type of Teeth	Storage Time
Aggarwal & others[4]	India	10	Adper Scotchbond Multi-Purpose	μTBS	Dentin	Natural	Permanent molars	24 hours
Alves & others[17]	Brazil	6	Adper Single Bond 2, Adper SE Plus, Adper Easy One	μTBS	Dentin	Artificial	Primary molars	24 hours
Antoniazzi & others[19]	Brazil	6	Clearfil SE Bond, Scotchbond Universal	μSBS	Enamel	Artificial	Primary molars	24 hours
Arrais & others[6]	Brazil	9	Adper Single Bond 2, Clearfil SE Bond	μTBS	Dentin	Artificial	Permanent molars	24 hours
Bahari & others[51]	Iran	8	Adper Single Bond 2, Clearfil SE Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Barbosa-Martins & others[52]	Brazil	6	Adper Single Bond, Clearfil SE Bond	μTBS	Dentin	Artificial	Permanent molars	24 hours
Barbosa-Martins & others[20]	Brazil	6	Adper Single Bond 2	μTBS	Dentin	Artificial	Permanent molars	24 hours
Calvo[53]	Brazil	15	Adper Scotchbond Multi-Purpose	μSBS	Dentin	Artificial	Primary molars	24 hours and 6 months
Ceballos & others[54]	Brazil	4	Prime & Bond NT, Scotchbond 1, Clearfil SE Bond, Prompt-L-Pop	μTBS	Dentin	Natural	Permanent molars	24 hours
Cecchin & others[55]	Brazil	10	Single Bond	μTBS	Dentin	Natural	Primary molars	24 hours
Costa & others[33]	Brazil	8	Adper Single Bond 2	μTBS	Dentin	Artificial	Permanent molars	24 hours, 6 and 12 months
De Melo & others[43]	Brazil	8	All Bond SE	μTBS	Dentin	Artificial	Permanent molars	24 hours
Deshmukh & others[56]	India	15	[c]	SBS	Dentin	Natural	Primary molars	24 hours
Doi & others[57]	Japan	5	Clearfil SE Bond, Mac-Bond II, UniFil Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Doi & others[58]	Japan	5	Clearfil SE Bond	μTBS	Dentin	Artificial	Bovine incisors	24 hours
Doozandeh & others[59]	Iran	10	Adper Single Bond 2	SBS	Dentin	Natural	Permanent molars	24 hours
Ehudin & others[60]	USA	30	Allbond, Tenure/SB2, Scotchbond 2	TBS	Dentin	Artificial	Permanent molars	24 hours

Table 1: *Characteristics of the Studies of Caries-altered Substrate Condition (cont.)*

Study	Country	N[a]	Adhesive System	Bond Strength Test[b]	Substrate	Type of Lesion	Type of Teeth	Storage Time
Ekambaram & others[61]	China	12	[c]	μTBS	Dentin	Natural	Permanent molars	24 hours and 12 months
Ergücü & others[30]	Turkey	4	AdheSE, Adper Scotchbond, Multi-Purpose	μTBS	Dentin	Natural	Permanent molars	24 hours
Erhardt & others[62]	Brazil	6	Excite, Prime & Bond NT	μTBS	Dentin	Artificial	Bovine incisors	24 hours
Erhardt & others[63]	Brazil	10	Adper Scotchbond 1	μTBS	Dentin	Natural	Permanent molars	24 hours
Erhardt & others[64]	Brazil	48	Adper Scotchbond 1, Clearfil Protect Bond, AdheSE	μTBS	Dentin	Natural	Permanent molars	24 hours and 6 months
Erhardt & others[65]	Brazil	6	Adper Single Bond 2, Clearfil SE Bond	μTBS	Dentin	Artificial	Bovine incisors	24 hours
Ersin & others[66]	Turkey	6	Prime&Bond NT	μTBS	Dentin	Natural	Primary molars	24 hours
Farias de Lacerda & others[22]	Brazil	12	Clearfil S3 Bond, Single Bond Universal	μTBS	Enamel	Artificial	Bovine incisors	24 hours
Follak & others[67]	Brazil	7	Scotchbond Universal Adhesive, All-Bond Universal, Prime & Bond Elect, Adper Single Bond 2, Clearfil SE Bond	μTBS	Dentin	Artificial	Bovine incisors	24 hours and 6 months
Follak & others[68]	Brazil	7	Scotchbond Universal Adhesive, All-Bond Universal, Prime & Bond Elect, Adper Single Bond 2, Clearfil SE Bond	μTBS	Dentin	Artificial	Bovine incisors	24 hours
Giacomini & others[69]	Brazil	10	Adper Single Bond Universal	μTBS	Dentin	Artificial	Permanent molars	24 hours and 6 months
Giriyappa & Chandra[44]	India	8	Prime & Bond NT, Clearfil Liner Bond 2V, All Bond 2	SBS	Dentin	Natural	Permanent molars	24 hours
Hass & others[24]	Brazil	5	ScotchBond Universal, Futura Bond U, Prime & Bond Elect	μTBS	Dentin	Natural	Permanent molars	24 hours

Table 1: *Characteristics of the Studies of Caries-altered Substrate Condition (cont.)*

Study	Country	N[a]	Adhesive System	Bond Strength Test[b]	Substrate	Type of Lesion	Type of Teeth	Storage Time
Hosoya & others[31]	Japan	5, 11[d]	Clearfil SE Bond	μTBS	Dentin	Natural	Primary molars	24 hours
Huang & others[45]	China	c	Adper Single Bond 2	μTBS	Dentin	Natural	Permanent molars	24 hours
Itota & others[70]	Japan	10	Clearfil SE Bond, Unifil Bond, Mac-Bond II	TBS	Dentin	Artificial	Bovine incisors	24 hours
Khoroushi & others[71]	Iran	12	Clearfil SE Bond	SBS	Dentin	Natural	Permanent molars	24 hours
Kimochi & others[72]	Japan	7	Unifil Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Komori & others[46]	Brazil	10	Scotchbond Multi Purpose, Adper Single Bond 2	μTBS	Dentin	Natural	Permanent molars	24 hours and 6 months
Koyuturk & others[73]	Turkey	14	Prompt-L-Pop, AQ Bond, Clearfil SE Bond, Optibond Solo Plus, One-Step Plus/ Tyrian SPE	SBS	Dentin	Natural	Permanent molars	24 hours
Koyuturk & others[74]	Turkey	20	Clearfil S3, Xeno V	μTBS	Dentin	Natural	Primary molars	24 hours
Krithi & others[75]	India	15	Adper Single Bond 2/ Plus Clearfil SE Bond	μSBS	Dentin	Artificial	Permanent molars	24 hours
Kucukyilmaz & others[76]	Turkey	8	Clearfil SE Bond	μTBS	Dentin	Artificial	Permanent molars	24 hours
Kunawarote & others[77]	Japan	10	Clearfil SE Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Leal & others[78]	Brazil	3	Clearfil S3 Bond Plus	μTBS	Dentin	Artificial	Permanent molars	24 hours and 6 months
Lenzi & others[79]	Brazil	5	Adper Single Bond 2	μTBS	Dentin	Artificial	Primary and Permanent molars	24 hours
Lenzi & others[80]	Brazil	6	Clearfil SE Bond, Adper Single Bond 2	μTBS	Dentin	Artificial	Primary molars	24 hours and 12 months
Lenzi & others[81]	Brazil	6	Clearfil SE Bond, Adper Single Bond 2	μTBS	Dentin	Artificial	Primary molars	24 hours and 12 months

Table 1: *Characteristics of the Studies of Caries-altered Substrate Condition (cont.)*

Study	Country	N[a]	Adhesive System	Bond Strength Test[b]	Substrate	Type of Lesion	Type of Teeth	Storage Time
Lenzi & others[32]	Brazil	5	Clearfil SE Bond, Scotchbond Universal, Adper Single Bond Plus	μTBS	Dentin	Artificial	Primary molars	24 hours and 12 months
Lima & others[47]	Brazil	3	Prime and Bond Elect Universal	μTBS	Dentin	Artificial	Permanent molars	24 hours
Lopes & others[83]	Brazil	10	Single Bond	SBS	Dentin	Artificial	Permanent molars	24 hours
Macedo & others[66]	USA	8	Adper Single Bond 2, One Step Plus	μTBS	Dentin	Artificial	Permanent molars	24 hours
Marquezan & others[85]	Brazil	5	Adper Single Bond 2	μTBS	Dentin	Artificial	Primary molars	24 hours
Maske & others[86]	Brazil	10	Clearfil SE Bond	SBS	Dentin	Artificial	Bovine incisors	24 hours
Mobarak[87]	Egypt	20	Clearfil SE Bond, Clearfil DC Bond, Bond Force, AdheSE One, Adper Prompt-L-Pop	μSBS	Dentin	Natural	Permanent molars	24 hours
Mobarak & El-Badrawi[88]	Egypt	10	Clearfil S3 Bond Plus, G-aenial Bond, Single Bond Universal	μSBS	Enamel	Artificial	Permanent molars	24 hours
Mobarak & others[25]	Egypt	20	Clearfil SE Bond	μSBS	Dentin	Natural	Permanent molars	24 hours and 2 years
Nakajima & others[15]	Japan	10	All Bond 2, Scotchbond Multi-Purpose, Clearfil Liner Bond 2	μTBS	Dentin	Natural	Permanent molars	24 hours
Nakajima & others[89]	Japan	5	Scotchbond Multi-Purpose[d]	TBS	Dentin	Natural	Permanent molars	24 hours
Nakajima & others[90]	Japan	4	Clearfil Liner Bond 2, MacBond II	μTBS	Dentin	Artificial	Permanent molars	24 hours
Nakajima & others[91]	Japan	6	One-Step Single Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Nakajima & others[26]	Japan	11	Clearfil Protect Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Nakornchai & others[35]	Thailand	10	Clearfil SE Bond, Single Bond	μTBS	Dentin	Natural	Primary molars	24 hours
Neves & others[92]	Belgium	5	Clearfil SE Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Nicoloso & others[93]	Brazil	6	Scotchbond Universal, Clearfil SE Bond, Adper Single Bond 2	μTBS	Dentin	Artificial	Permanent molars	24 hours

Table 1: *Characteristics of the Studies of Caries-altered Substrate Condition (cont.)*

Study	Country	N[a]	Adhesive System	Bond Strength Test[b]	Substrate	Type of Lesion	Type of Teeth	Storage Time
Oliveira & others[94]	Brazil	10	Adper Easy One	μTBS	Dentin	Artificial	Permanent molars	24 hours
Omar & others[95]	Egypt	10	Scotchbond Multi-Purpose, Clearfil SE Bond, Xeno IV	μTBS	Dentin	Natural	Permanent molars	24 hours
Ortiz-Ruiz & others[96]	Spain	20	Futurabond M+	SBS	Enamel	Artificial	Bovine incisors	24 hours
Paranhos & others[97]	Brazil	5	Clearfil SE Bond, Single Bond	μTBS	Dentin	Artificial	Permanent molars	24 hours
Perdigão & others[98]	Portugal	10	All Bond 2, Amalgambond Plus, Prisma Universal Bond 3, Scotchbond Multi-Purpose	SBS	Dentin	Artificial	Permanent molars	24 hours
Pereira & others[99]	USA	5	Single Bond, Adper Prompt L Pop	μTBS	Dentin	Natural	Permanent molars	24 hours
Pires & others[5]	Brazil	7	Single Bond Universal, Adper Single Bond 2, Clearfil SE Bond	μSBS	Enamel	Natural	Permanent molars	24 hours
Piva & others[100]	Brazil	5	Prime & Bond NT, Clearfil SE Bond	μSBS	Dentin	Natural	Permanent molars	24 hours
Sanabe[101]	Brazil	4	Adper Scothbond Multi Purpose, Adper Single Bond 2, Clearfil SE Bond, Adper Prompt L-Pop	μTBS	Dentin	Artificial	Permanent molars	24 hours
Silva[102]	Brazil	12	ScotchBond Universal	μTBS	Dentin	Artificial Natural	Permanent molars	24 hours
Scheffel & others[48]	Brazil	4	Prime & Bond NT	μTBS	Dentin	Artificial	Primary and Permanent molars	24 hours
Schmidlin & others[103]	Switzerland	10	[c]	SBS	Dentin	Artificial	Bovine incisors	24 hours
Scholtanus & others[104]	Netherlands	5	Adper Scotchbond 1 XT, Clearfil S3 Bond, Clearfil SE Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Sengün & others[105]	Turkey	12	Prime Bond, One Coat Bond, Clearfil SE Bond, Etch & Prime 3.0, Solid Bond	SBS	Dentin	Natural	Permanent molars	24 hours

Table 1: *Characteristics of the Studies of Caries-altered Substrate Condition (cont.)*

Study	Country	N[a]	Adhesive System	Bond Strength Test[b]	Substrate	Type of Lesion	Type of Teeth	Storage Time
Sengün & others[106]	Turkey	15	Optibond Solo Plus	SBS	Dentin	Natural	Permanent molars	24 hours
Shibata & others[107]	Brazil	6	Clearfil MegaBond, MTB-200, G-Bond Plus, Adper Easy Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Singh & others[108]	India	10	Single Bond	TBS	Dentin	Natural	Permanent molars	24 hours
Sonoda & others[109]	Japan	5	Prime & Bond NT [c]	μTBS	Dentin	Natural	Permanent molars	24 hours
Tachibana & others[110]	Brazil	10	Clearfil SE Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Taniguchi & others[111]	Japan	12	Clearfil Protect Bond, Bond Force	μTBS	Dentin	Natural	Permanent molars	24 hours
Tedesco & others[42]	Brazil	6	Adper Single Bond 2 Adper ES Plus	μTBS	Dentin	Artificial	Primary molars	24 hours and 2 years
Tedesco & others[112]	Brazil	5	Adper Single Bond, Clearfil SE Bond	μSBS	Enamel	Artificial	Primary and permanent molars	24 hours
Toledano & others[113]	Spain	6, 9[e]	Single Bond, Clearfil SE Bond, FL-Bond II	μTBS	Dentin	Natural	Permanent molars	24 hours
Tosun & others[36]	Turkey	15	PQI, Optibond Solo Plus	μSBS	Dentin	Natural	Primary molars	24 hours
Wang & others[14]	China	5	All Bond 2, Prime & Bond NT, Clearfil SE Bond, Xeno III	μTBS	Dentin	Natural	Bovine incisors	24 hours
Wei & others[114]	Japan	10	Clearfil SE Bond, Clearfil Tri-S Bond, Single Bond	μSBS	Dentin	Natural	Permanent molars	24 hours
Wiegand & others[115]	Switzerland	10	Heliobond	SBS	Enamel	Artificial	Bovine incisors	24 hours
Xuan & others[116]	China	7	Adper Single Bond 2, Clearfil SE Bond, Clearfil S3 Bond, iBond GI	μTBS	Dentin	Natural	Permanent molars	24 hours
Yazici & others[117]	Turkey	6	Clearfil SE Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Yoshiyama & others[27]	Japan	6	FluroBond, Single Bond	μTBS	Dentin	Natural	Permanent molars	24 hours
Yoshiyama & others[11]	Japan	7	Single Bond[d]	μTBS	Dentin	Natural	Permanent molars	24 hours

Table 1: *Characteristics of the Studies of Caries-altered Substrate Condition (cont.)*

Study	Country	N[a]	Adhesive System	Bond Strength Test[b]	Substrate	Type of Lesion	Type of Teeth	Storage Time
Zanchi & others[118]	Brazil	15	Clearfil SE Bond, Adper Single Bond 2	μTBS	Dentin	Natural	Permanent molars	24 hours
Zanchi & others[119]	Brazil	5	Single Bond 2, Prime & bond NT	μTBS	Dentin	Natural	Permanent molars	24 hours
Zawaideh & others[50]	Jordan	[b]	Single Bond	μSBS	Dentin	Artificial	Primary molars	24 hours

Abbreviations: SBS, shear bond strength, TBS, tensile bond strength, μSBS, microshear bond strength, μTBS, microtensile bond strength
[a] Number of teeth per group.
[b] Not specified clearly by authors.
[c] Commercial name is not specified clearly by authors or used an experimental adhesive.
[d] Different number of teeth according groups: 5 teeth for sound substrate and 11 teeth for caries-affected substrate.
[e] Different number of teeth according groups: 6 teeth for sound substrate and 9 teeth for caries-affected substrate.

were included so that comparisons could be made only for dentin substrate, including 11 studies for carious dentin and 11 studies for eroded dentin.

Caries-altered vs Sound Substrate

Figures 2 and 3 show the results for the meta-analyses considering caries-altered enamel and dentin, respectively. The overall meta-analyses showed that the bond strength was significantly impaired by both the caries-altered enamel (Z= 3.23, p=0.001) and dentin (Z=12.93, p<0.0001). Significant heterogeneity for overall dentin (p<0.0001, I^2=91%) and enamel (p<0.0001, I^2=92%) were observed. Likewise, a statistically significant difference was found favoring sound enamel compared to caries-altered enamel in permanent (Z=2.68, p<0.01) and primary teeth (Z=5.81, p<0.01). Caries-altered dentin also impaired the bond strength both in permanent (Z=12.41, p<0.01) and primary teeth (Z=4.36, p<0.01). The data were heterogeneous for subgroup meta-analysis, except for the primary enamel subgroup (p=0.76, I^2=0%). The analysis considering the long-term bond strength (Figure 4) was only possible for dentin and also showed a statistically significant difference, favoring the sound substrate (Z=6.30, p<0.01). High heterogeneity was observed (p<0.01,I^2= 86%).

Eroded vs Sound Substrate

Figures 5 and 6 show the results for the meta-analyses considering eroded enamel and dentin, respectively. No significant difference was found for sound and eroded enamel in overall meta-analysis (Z=0.16, p=0.87), or considering permanent (Z=0.51, p=0.61) and primary enamel subgroups (Z=0.69, p=0.49). The data were heterogeneous for all analyses (overall: p<0.01, I^2=87%; permanent enamel: p<0.01, I^2 =89% and primary enamel analysis: p<0.01, I^2=83%). The overall meta-analysis showed that the bond strength was significantly impaired by the eroded dentin (Z= 4.53, p<0.01). When the type of teeth was considered separately, significant difference also was found for sound and eroded permanent dentin subgroup meta-analysis (Z=4.64, p<0.01), while primary dentin was not significantly impaired for the eroded condition (Z=0.66, p=0.51). The data were heterogeneous for overall (p<0.01, I^2=91%) and subgroup analysis (p<0.01; I^2=90% and I^2=74%). The bond strength to eroded dentin remains significantly impaired after aging (Z=4.17, p<0.01) (Figure 7). The data was heterogeneous (p<0.01, I^2=86%).

Assessment of Risk of Bias and Quality of Evidence of the Included Studies

Most of the included studies for caries-altered (Table 3) and eroded (Table 4) conditions presented a medium risk of bias. The parameters that most often received "No" were: the description of sample size calculation, a single operator during the specimen preparation, and the blinded operator to experimental condition during the tests.

DISCUSSION

This review is the first to summarize data from laboratory literature on the bonding performance of adhesive systems applied to both caries-altered and eroded enamel and dentin—the two most clinically significant substrates. The meta-analyses showed that caries-altered and eroded dentin jeopardized the immediate and long-term bonding of adhesive systems. In contrast, only caries-altered enamel negatively influenced the adhesive bond strength. Thus, the hypothesis tested in this review that the condition of the

Table 2: *Characteristics of the Studies of Eroded Substrate Condition*

Study	Country	N[a]	Adhesive System	Bond Strength Test	Substrate	Type of Lesion	Type of Teeth	Storages Times
Amsler & others[18]	Switzerland	15	Clearfil SE Bond, Scotchbond Universal	SBS	Dentin	Artificial	Permanent molars	24 hours and 12 months
Assunção & others[120]	Brazil	12	Adper Single Bond 2, Single Bond Universal, OptiBond FL, Bonde Force	μSBS	Dentin and Enamel	Artificial	Primary molars	24 hours
Augusto & others[28]	Brazil	5	Futurabond M+	μTBS	Dentin	Artificial	Bovine incisors	24 hours
Casas-Apayco & others[21]	Brazil	8	Adper Single Bond 2	μTBS	Enamel	Artificial	Bovine incisors	24 hours
Cersosimo & others[121]	Brazil	10	Clearfil SE Bond	μSBS	Dentin	Artificial	Permanent molars	24 hours
Costa & others[122]	Brazil	6	Clearfil SE Bond	μTBS	Dentin	Artificial	Permanent molars	24 hours and 6 months
Cruz & others[123]	Brazil	10	Adper Single Bond 2, Clearfil SE Bond, Adper Easy One	μSBS	Dentin	Artificial	Bovine incisors	24 hours and 6 months
Cruz & others[29]	Brazil	6	Adper Single Bond 2	μSBS	Dentin	Artificial	Bovine incisors	24 hours
Deari & others[124]	Switzerland	6	OptiBond FL	μTBS	Dentin	Artificial	Permanent molars	24 hours
Ding & others[125]	Korea	7	Adper Single Bond 2	μTBS	Dentin	Artificial	Permanent molars	24 hours
Flury & others[126]	Switzerland	20	Clearfil SE Bond	μTBS	Dentin	Artificial	Permanent molars	24 hours
Flury & others[127]	Switzerland	16	Adper Scotchbond 1XT, OptiBond FL	SBS	Dentin	Artificial	Permanent molars	24 hours and 12 months
Forgerini & others[128]	Brazil	8	Scothbond Universal, Adper Single Bond Plus, Clearfil SE Bond	μSBS	Dentin	Artificial	Bovine incisors	24 hours and 6 months
Francisconi-dos-Rios & others[23]	Brazil	7	Adper Single Bond 2	μTBS	Dentin	Artificial	Permanent molars	24 hours, 6 and 12 months
Frattes & others[34]	Brazil	11	Scothbond Universal	μTBS	Dentin and Enamel	Artificial	Bovine incisors	24 hours
Giacomini & others[129]	Brazil	10	Adper Single Bond Universal	μTBS	Dentin	Artificial	Permanent molars	24 hours and 6 months

Table 2: *Characteristics of the Studies of Eroded Substrate Condition (cont.)*

Study	Country	N[a]	Adhesive System	Bond Strength Test	Substrate	Type of Lesion	Type of Teeth	Storages Times
Landmayer[130]	Brazil	7	Adper Single Bond 2	μTBS	Dentin	Artificial	Permanent molars	24 hours
Lenzi & others[131]	Brazil	12	Adper Single Bond 2	μSBS	Enamel	Artificial	Bovine incisors	24 hours
Machado & others[132]	Brazil	10	Adper Single Bond 2	μSBS	Dentin	Artificial	Permanent molars	1 and 6 months
Rigolizzo[133]	Brazil	10	Adper Single Bond 2, Clearfil SE Bond	μTBS	Dentin	Artificial	Permanent molars	24 hours
Siqueira & others[134]	Brazil	7	Adper Single Bond 2, Scotchbond Universal	μTBS	Dentin	Artificial	Permanent molars	24 hours and 3 years
Siqueira & others[135]	Brazil	5	All-Bond Universal, Ambar Universal, Clearfil Universal, FuturaBond U, One Coat 7 Universal, Peak Universal Bond, Prime & Bond Elect, Scotchbond Universal, Tetric n-bond Universal, Xeno Select Universal	μTBS	Dentin	Artificial	Permanent molars	24 hours
Tedesco & others[136]	Brazil	10	Adper Single Bond 2	μSBS	Dentin and Enamel	Artificial	Primary molars	24 hours and 12 months
Wang & others[32]	Brazil	26	Adper Single Bond 2	μTBS	Enamel	Artificial	Bovine incisors	24 hours
Yakubi & others[37]	Japan	10	All-Bond Universal, Adhese Universal	SBS	Enamel	Artificial	Bovine incisors	24 hours
Zimmerli & others[137]	Belgium	4	OptiBond FL, Clearfil SE Bond	μTBS	Dentin	Artificial	Permanent molars	24 hours and 12 months
Zumstein & others[49]	Switzerland	23	Clearfil SE Bond, Scotchbond Universal	μTBS	Dentin	Artificial	Permanent molars	24 hours and 12 months

Abbreviations: SBS, shear bond strength; μSBS, microshear bond strength; μTBS, microtensile bond strength
[a] *Number of teeth per group.*

substrate—caries-altered or eroded—would negatively influence the bond strength values of enamel and dentin, and has not been fully rejected.

This systematic review and meta-analysis found a statistically significant higher bond strength value to sound compared to caries-altered dentin. Similar to the results of our study, a previous systematic review[138] also found that carious and sound dentin differed in bonding values. The decrease in bond strength in caries-altered dentin could be a consequence of irregular and defective hybrid layer formation, with inadequate monomer infiltration into the altered

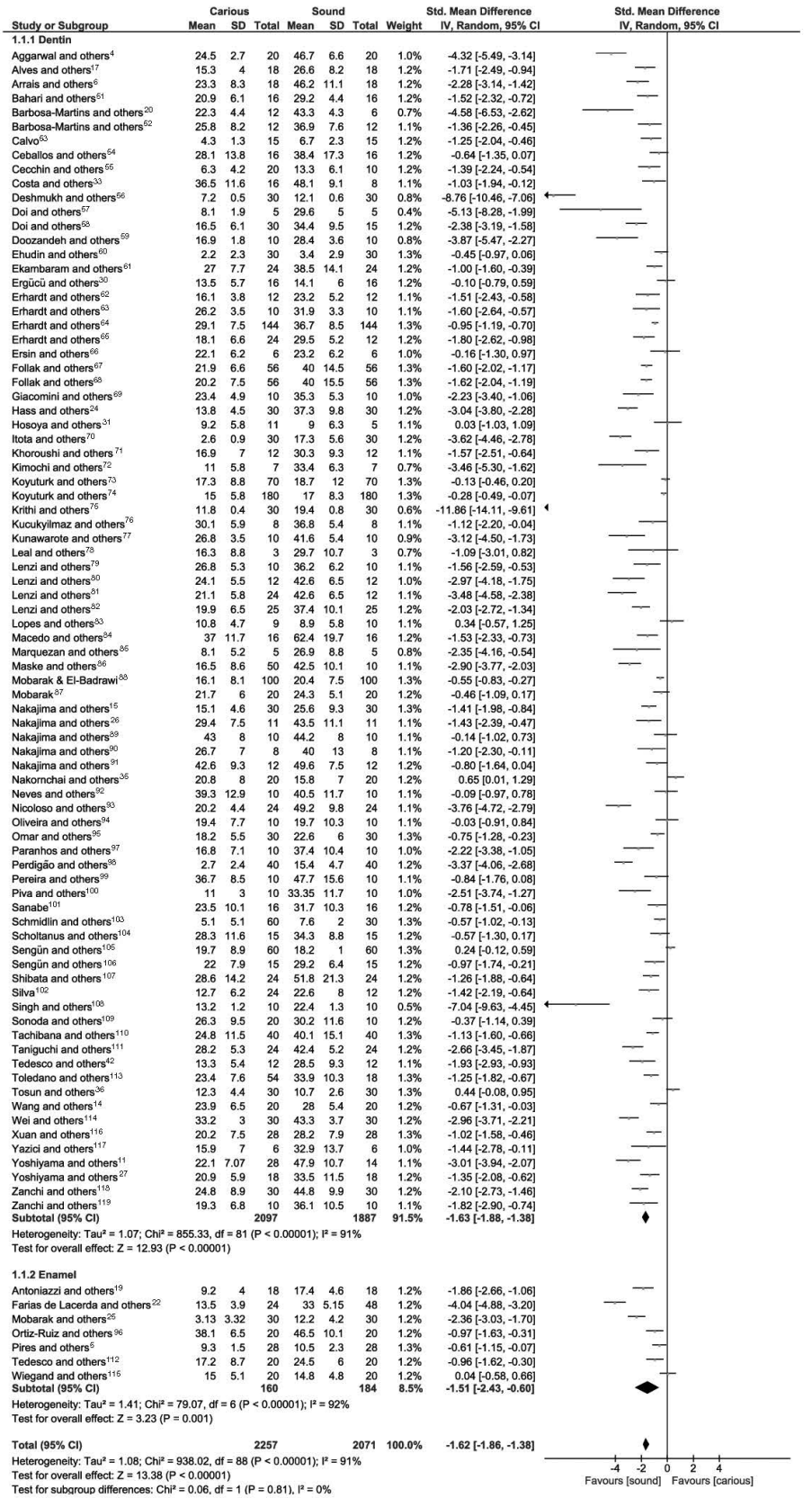

Figure 2. *Forest plot of the included studies concerning caries-altered enamel substrate.*

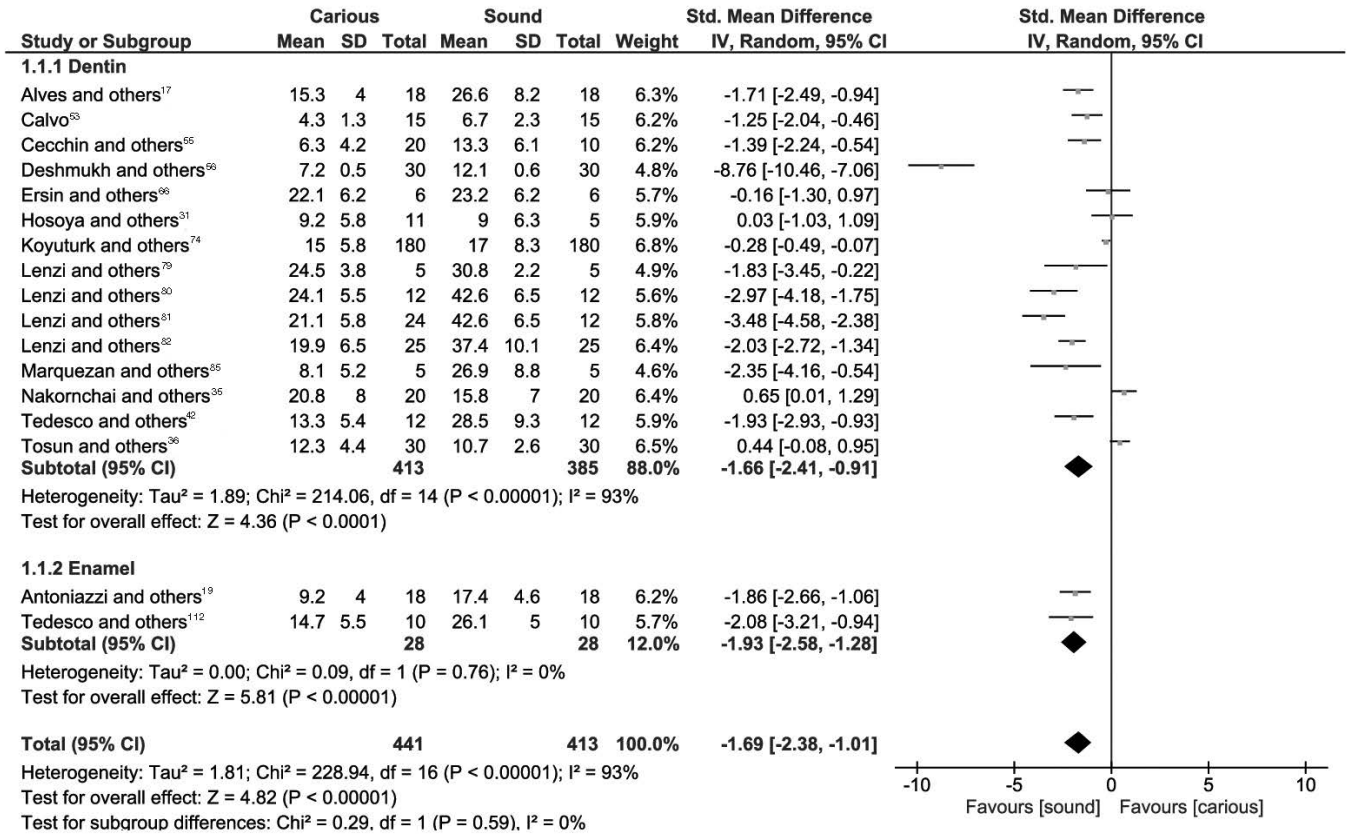

Figure 3. *Forest plot of the included studies concerning caries-altered dentin substrates.*

substrate.[6] Carious dentin is less mineralized,[6,114] more irregular, and porous than sound dentin.[45] In natural lesions, carious dentin also presents acid-resistant mineralized precipitates within the tubules,[45,84] making the substrate more impermeable to water.[11]

A significant number of studies were included in our meta-analysis, compared to Isolan and others[138] that included only 40 studies. Besides that, studies using not only permanent but also primary teeth were included. Considering the chemical and morphological differences[139,140] between primary and permanent teeth as bonding substrates,[141] a subgroup meta-analysis was also performed considering primary and permanent teeth separately. Higher bond strength was found for

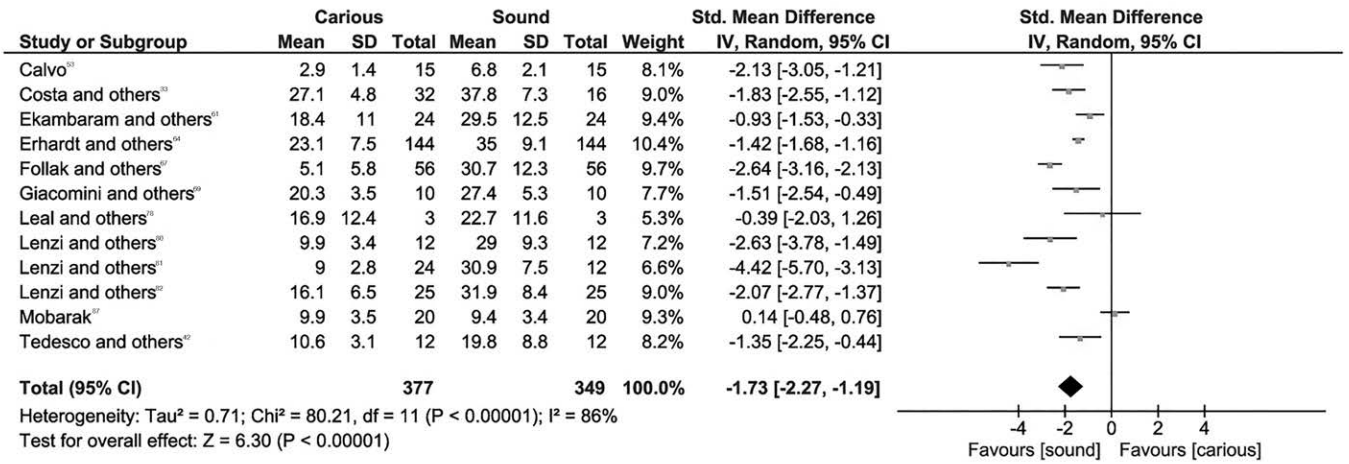

Figure 4. *Forest plot of the included studies concerning caries-affected dentin after aging.*

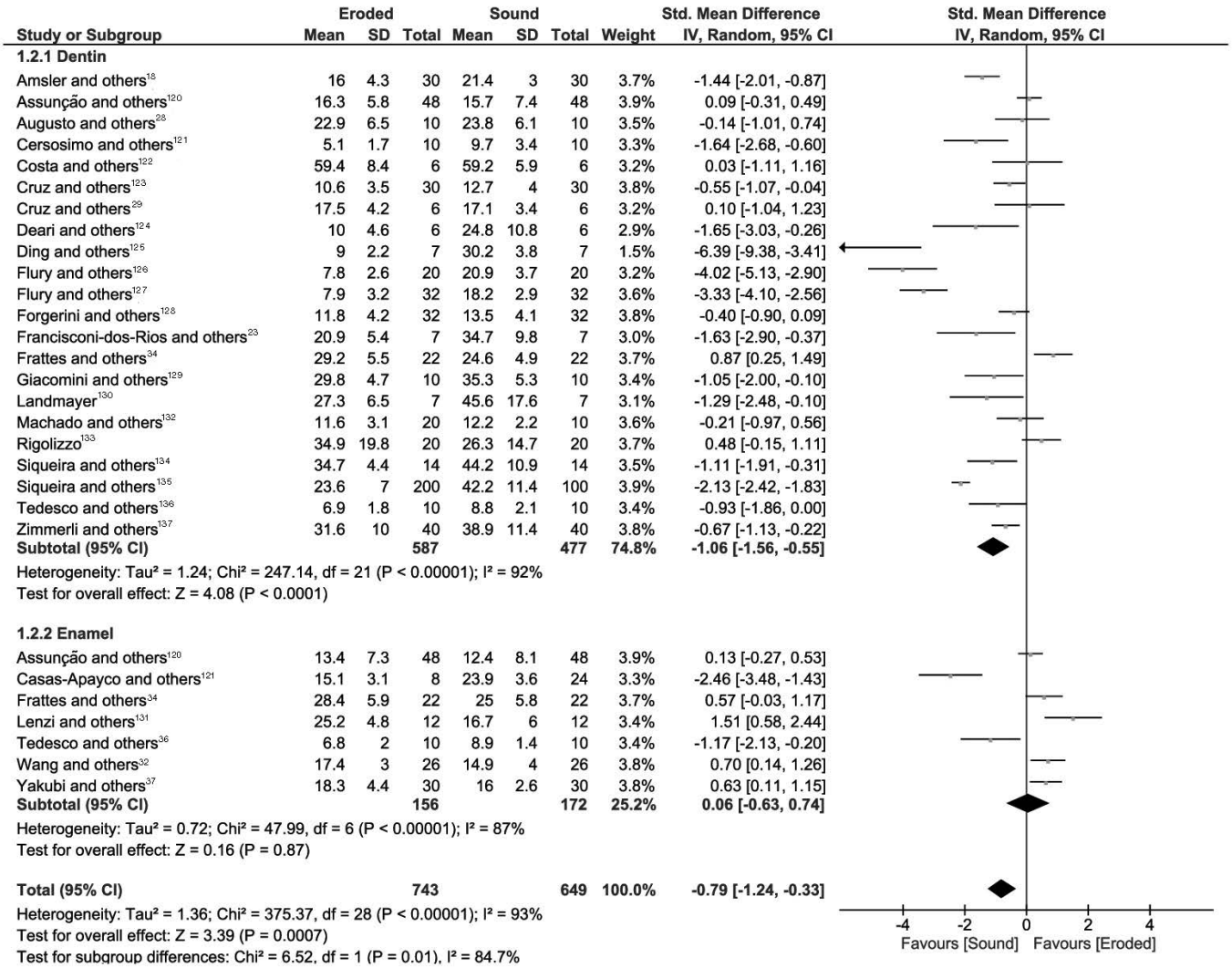

Figure 5. *Forest plot of the included studies concerning eroded enamel substrate.*

sound compared to caries-altered enamel and dentin, of both permanent and primary teeth. Primary teeth present lower mineral content of in peritubular and intertubular dentin[140] and higher tubular density[140] that favors the formation of thicker hybrid layers[142] and lower bond strength values than permanent dentin.[141]

Data on the bond strength of caries-altered enamel had not yet been summarized in the literature. Although enamel is considered a reliable substrate that produces stable and strong adhesion, this systematic review showed that caries-altered enamel compromises the immediate bonding in permanent and primary teeth. This finding may be attributed to the lower content of minerals, higher porosity, and enlargement of the intercrystalline spaces in demineralized enamel,[12,13] which may lead to an unsatisfactory etching pattern and infiltration of monomers, resulting in reduced bond strength.[112] It is essential to consider that, in bond strength studies, the tooth surface is abraded to obtain flat surfaces. Thus, the outermost layer of the enamel, usually aprismatic,[139] is eliminated, even in primary molars.

The interest in the performance of adhesive systems on eroded substrates is more recent; the studies included in this review were published in the last 10 years; thus, a considerably smaller number of studies comparing sound and eroded substrates were included. The findings of this systematic review show that immediate bond strength to eroded dentin is notoriously critical. Eroded dentin has a thicker layer of exposed collagen that may not be adequately infiltrated by resin monomers, which may explain the lower bond strength values obtained.[126,135,137] Differently, this is not observed when only primary teeth were

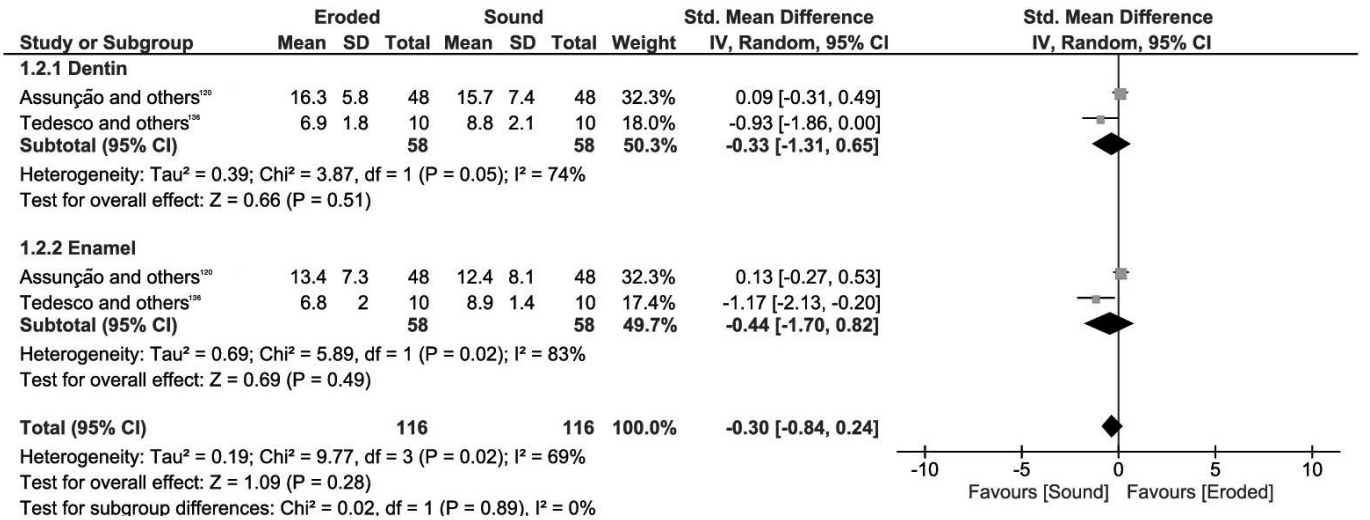

Figure 6. *Forest plot of the included studies concerning eroded dentin substrate.*

considered. However, it is important to note that only two studies[120,136] could be included in this analysis and more investigations are required to confirm this finding. Conversely, an opposite trend was observed in eroded enamel, as similar bond strength values to sound enamel were found, independently of type of teeth—primary or permanent. This is because the erosive effect occurs differently in enamel and dentin. The erosive challenge increases the porosity of enamel[10] and may promote a stronger interlocking of adhesive resin to enamel, explaining the noninterference of bond strength values when compared to the sound substrate. In our systematic review, separate meta-analyses were performed considering the effect of aging on bond strength to caries-affected and eroded dentin. The results allow us to confirm that both substrate conditions continue to impair the bond strength after aging. The characteristics of altered substrates, as less mineral content, higher water content, and the activity of matrix metalloproteinases,[18] may cause an accelerated bond degradation in caries-affected and eroded dentin.[137] It was not possible to perform the meta-analysis considering long-term bond strength to enamel because only one study presented after-storage data for the eroded condition.[58] Likewise, no study presented long-term data considering demineralized enamel, and no study presented after-storage data for demineralized enamel. Therefore, long-term studies are needed to determine the effect of substrate conditions in bond strength of enamel after aging.

High heterogeneity was found, even in subgroup meta-analyses. The high heterogeneity found may be influenced by the significant variables among the studies, mainly regarding the number of adhesive systems tested and the bond strength test. Several factors were variable in the primary studies. Human

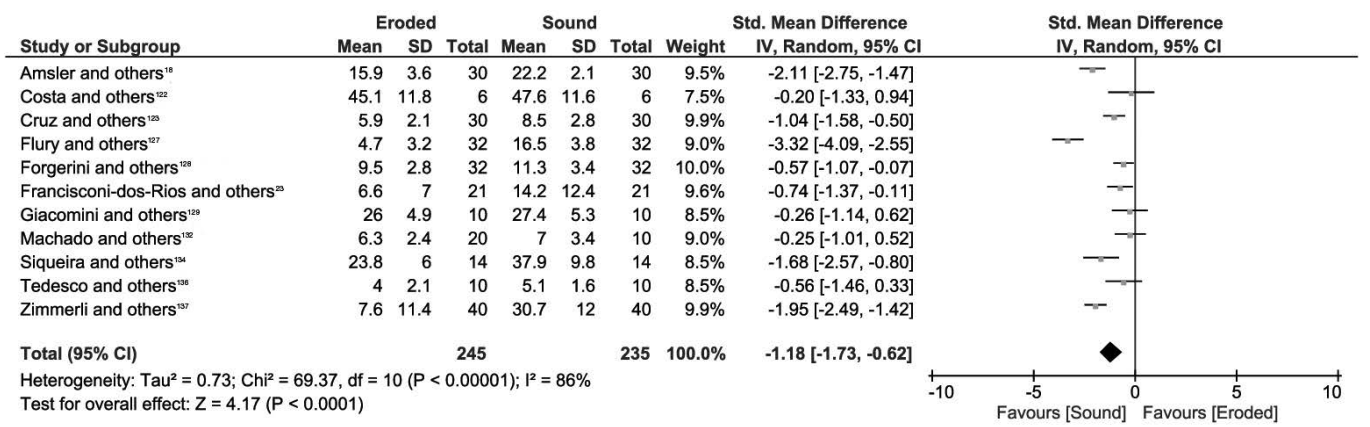

Figure 7. *Forest plot of the included studies concerning eroded dentin after aging.*

Table 3: *Risk of Bias Assessment for Studies of Caries-altered Substrate Condition*

Studies	RS	SSC	SNTG	SSCS	FME	MI	SO	BO	OR
Aggarwal & others[4]	No	No	Yes	Yes	No	Yes	No	No	High
Alves & others[17]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Antoniazzi & others[19]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Arrais & others[6]	Yes	No	Yes	Yes	No	Yes	No	No	Moderate
Bahari & others[51]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Barbosa-Martins & others[52]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Barbosa-Martins & others,[20]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Calvo[53]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Ceballos & others[54]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Cecchin & others[55]	Yes	No	Yes	Yes	No	Yes	Yes	No	Moderate
Costa & others[33]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
De Melo & others[43]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Deshmukh & others[56]	No	No	Yes	Yes	Yes	No	No	No	High
Doi & others[57]	No	No	Yes	Yes	Yes	No	No	No	High
Doi & others[58]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Doozandeh & others[59]	Yes	No	Yes	Yes	Yes	No	No	No	Moderate
Ehudin & others[60]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Ekambaram & others[61]	No	No	Yes	Yes	Yes	No	No	No	High
Ergücü & others[30]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Erhardt & others[62]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Erhardt & others[63]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Erhardt & others[64]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Erhardt & others[65]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate

Table 3: *Risk of Bias Assessment for Studies of Caries-altered Substrate Condition (cont.)*

Studies	RS	SSC	SNTG	SSCS	FME	MI	SO	BO	OR
Ersin & others[66]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Farias de Lacerda & others[22]	No	No	Yes	Yes	Yes	No	No	No	High
Follak & others[67]	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Low
Follak & others[68]	Yes	No	Yes	Yes	Yes	Yes	Yes	Yes	Low
Giacomini & others[69]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Giriyappa & Chandra[44]	Yes	No	Yes	Yes	No	Yes	No	No	Moderate
Hass & others[24]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Moderate
Hosoya & others[31]	No	No	No	Yes	Yes	Yes	No	No	High
Huang & others[45]	No	No	[a]	Yes	No	Yes	Yes	No	High
Itota & others[70]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Khoroushi & others[71]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Kimochi & others[72]	No	No	Yes	Yes	Yes	No	No	No	High
Komori & others[46]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Koyuturk & others[73]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Koyuturk & others[74]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Krithi & others[75]	Yes	No	Yes	Yes	Yes	No	No	No	Moderate
Kucukyilmaz & others[76]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Kunawarote & others[77]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Leal & others[78]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Lenzi & others[79]	No	No	Yes	Yes	Yes	No	No	No	High
Lenzi & others[80]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Lenzi & others[81]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Lenzi & others[82]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Lima & others[47]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate

Table 3: *Risk of Bias Assessment for Studies of Caries-altered Substrate Condition (cont.)*

Studies	RS	SSC	SNTG	SSCS	FME	MI	SO	BO	OR
Lopes & others[83]	Yes	No	Yes	Yes	Yes	Yes	No	Yes	Low
Macedo & others[84]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Marquezan & others[85]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Maske & others[86]	No	No	Yes	Yes	Yes	No	No	No	High
Mobarak[87]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Mobarak & El-Badrawi[88]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Mobarak & others[25]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Nakajima & others[15]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Nakajima & others[89]	Yes	No	Yes	Yes	No	Yes	No	No	Moderate
Nakajima & others[90]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Nakajima & others[91]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Nakajima & others[26]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Nakornchai & others[35]	Yes	No	Yes	Yes	No	Yes	No	No	Moderate
Neves & others[92]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Nicoloso & others[93]	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	Low
Oliveira & others[94]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Omar & others[95]	Yes	No	Yes	Yes	No	Yes	No	No	Moderate
Ortiz-Ruiz & others[96]	Yes	No	Yes	Yes	Yes	No	No	No	Moderate
Paranhos & others[97]	Yes	No	Yes	Yes	Yes	No	No	No	Moderate
Perdigão & others[99]	Yes	No	Yes	Yes	Yes	No	No	No	Moderate
Pereira & others[99]	No	No	Yes	Yes	No	Yes	No	No	High
Pires & others[5]	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Low
Piva & others[100]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Sanabde[101]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Silva[102]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Scheffel & others[48]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate

Table 3: *Risk of Bias Assessment for Studies of Caries-altered Substrate Condition (cont.)*

Studies	RS	SSC	SNTG	SSCS	FME	MI	SO	BO	OR
Schmidlin & others[103]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Scholtanus & others[104]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Sengün & others[105]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Sengün & others[106]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Shibata & others[107]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Singh & others[108]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Sonoda & others[109]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Tachibana & others[110]	Yes	No	Yes	Yes	No	No	No	No	High
Taniguchi & others[111]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Tedesco & others[42]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Tedesco & others[112]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Toledano & others[113]	Yes	No	No	Yes	Yes	Yes	No	No	Moderate
Tosun & others[36]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Wang & others[14]	Yes	No	Yes	Yes	No	Yes	No	No	Moderate
Wei & others[114]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Wiegand & others[115]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Xuan & others[116]	Yes	No	Yes	Yes	No	Yes	No	No	Moderate
Yazici & others[117]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Yoshiyama & others[27]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Yoshiyama & others[11]	No	No	Yes	Yes	No	No	No	No	High
Zanchi & others[118]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Zanchi & others[119]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Zawaideh & others[50]	No	No	No	Yes	Yes	No	No	No	High

Abbreviations: RS, random sequence; SSC, sample size calculation; SNTG, same number of teeth per group; SSCS, speciment with similar cross-section; FME, failure mode evaluation; MI, manufacturer's instructions; SO, single operator; BO, blinded operator; OR, overall rating.

[a]*Not specified clearly by authors.*

Table 4: *Risk of Bias Assessment for Studies of Eroded Substrate Condition*

Studies	RS	SSC	SNTG	SSCS	FME	MI	SO	BO	OR
Amsler & others[18]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Assunção & others[120]	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	Low
Augusto & others[28]	Yes	Yes	Yes	Yes	Yes	Yes	No	No	Low
Casas-Apayco & others[21]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Cersosimo & others[121]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Costa & others[122]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Cruz & others[122]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Cruz & others[29]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Deari & others[125]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Ding & others[125]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Flury & others[126]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Flury & others[127]	No	No	Yes	Yes	Yes	No	No	No	High
Forgerini & others[128]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Francisconi-dos-Rios & others[23]	Yes	No	Yes	Yes	Yes	No	No	No	Moderate
Frattes & others[34]	Yes	No	Yes	Yes	Yes	No	No	No	Moderate
Giacomini & others[129]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Landmayer[130]	Yes	No	Yes	Yes	Yes	Yes	No	Yes	Low
Lenzi & others[131]	Yes	No	Yes	Yes	No	Yes	No	No	Moderate
Machado & others[132]	Yes	No	Yes	Yes	Yes	No	No	No	Moderate
Rigolizz[133]	No	No	Yes	Yes	Yes	Yes	No	No	Low
Siqueira & others[134]	Yes	No	Yes	Yes	Yes	Yes	Yes	No	Low
Siqueira & others[135]	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	Low
Tedesco, & others[136]	Yes	No	Yes	Yes	Yes	Yes	No	No	Moderate
Wang & others[32]	Yes	No	Yes	Yes	Yes	No	No	No	Moderate
Yakubi & others[37]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Zimmerli & others[137]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate
Zumstein & others[49]	No	No	Yes	Yes	Yes	Yes	No	No	Moderate

and bovine teeth were used in the primary studies; but, this should not be considered as a significant factor, as previous studies have already shown the similarity between them in studies of bond strength.[40] Likewise, several adhesive systems of different categories were used, but, as in previous systematic reviews of *in vitro* studies[141,143] in the present review, the manufacturers or adhesive categories were not considered separately in subgroup analysis. Besides, most of the studies had a medium risk of bias. Although there is a guideline[144] for conducting and reporting *in vitro* studies on dental materials, it does not seem to be widely used and moderate or high risk of bias, as well as the high heterogeneity in systematic reviews of laboratory studies seems to be usual.[39,40] It is also worth considering the incomplete description or even the lack of important information regarding the study parameters, contributing to the studies' heterogeneity. Although laboratory data should not be translated directly to the clinical situation, bond strength tests are useful to provide data on a specific parameter, as the influence of the substrate condition, ranking the adhesive systems according to bonding data, and ultimately, an initial indication of bonding performance.[145] Thus, future studies with high-quality design are needed to draw a more reliable conclusion about the effect of the substrate condition on bond strength. Besides that, all studies that evaluated the bond strength to eroded enamel and dentin used artificial models to create this substrate, as *in vitro* or *in situ* models are valid to create an eroded substrate.[146] Considering caries-altered substrates, although *in vitro* and *in situ* models are the most commonly employed methods in cariology research,[147] some studies used natural lesions as dentin substrate. Although the characteristics of natural or artificially created carious substrates may vary, different results are not to be expected from those found in the present study. Similarly, different erosive solutions were used in primary studies, as erosive cola-based drinks and citric acid solution, although the similar effect of these solutions could be considered.

CONCLUSIONS

Based on the current study findings, the substrate condition influences the bond strength of adhesive systems. Caries-altered enamel and dentin of permanent and primary teeth impair bonding, while erosion only decreases the bond strength of adhesive systems to permanent dentin. Alternatives to improve adhesion to caries-altered and eroded substrates should be investigated.

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Conflict of Interest

The authors certify that they have no commercial or associative interest that represents a conflict of interest in connection with the manuscript. 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. The authors alone are responsible for the content and writing of this paper.

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Evaluation of Cleaning Methods on Lithium Disilicate Glass Ceramic Surfaces After Organic Contamination

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Clinical Relevance

Air–water spray, 35% phosphoric acid, 70% alcohol, and Ivoclean are effective cleaning methods for removing saliva from a previously etched and silanized lithium disilicate glass ceramic. When contaminated with human blood, only Ivoclean cleaning paste was able to restore the initial bond strength.

SUMMARY

The purposes of this study were to 1) evaluate the effectiveness of different cleaning methods from a previously etched and silanized lithium disilicate glass ceramic (EMX) surface after contact with organic fluids (saliva or human blood) and 2) assess the effect of applying a new silane layer after the cleaning methods on the microshear bond strength

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(mSBS) of resin cement to EMX. EMX discs were etched with 5% hydrofluoric acid (HF) and properly silanized. Three control groups were created (n=10): control (without contamination), saliva positive, and human blood positive. Later, after new contaminations, the samples were distributed into four groups according to the cleaning method (n=20): air–water spray (AWS), 35% phosphoric acid, 70% alcohol, or Ivoclean cleaning paste. After the cleaning methods, subgroups were submitted to a new silane layer application, or not (n=10). All samples received a thin layer of a bonding agent and, subsequently, three light-cured resin cement cylinders were prepared on each EMX surface for the mSBS test. This test was performed on a universal testing machine at a vertical speed of 1 mm/minute until rupture. Contaminated and cleaned silanized EMX surfaces were assessed by scanning electron microscopy (SEM) (n=1). The noncontaminated control group showed an average mSBS of 18.7 MPa, and the positive saliva and human blood control groups yielded a 34% and 42% reduction in bond strength, respectively, compared to the uncontaminated control ($p<0.05$). For saliva-contaminated surfaces, all cleaning methods were effective and not different from one another or the control group ($p>0.05$). However, for human blood contamination, only Ivoclean cleaning paste was effective in restoring μSBS to uncontaminated control group levels ($p>0.05$). SEM images showed a clean surface (ie, with no contaminant residues) after the cleaning methods, regardless of the organic contaminant type. All the assessed cleaning methods were effective in removing saliva from the silanized EMX surface; however, only Ivoclean was able to restore the adhesion quality when the silanized EMX surface was contaminated with human blood.

INTRODUCTION

Glass ceramics are widely used in dentistry as a restorative material for esthetic and morphological reconstruction due to their biocompatibility, ability to mimic optical characteristics of enamel and dentin, and adequate chemical stability.[1-5] The clinical success (ie, strong adhesion) of glass ceramic restorations is highly dependent on the adhesive bonding–interaction between dental tissues, resin cement, and glass ceramic.[6]

For proper bonding to resin cements, glass ceramics are previously etched with hydrofluoric acid (HF) that dissolves the glassy phase, thereby promoting surface roughness. This increases the surface area and surface energy for micromechanical interlocking to resin cements.[7-11] Thereafter, a silane coupling agent is applied to yield chemical bonding between silica present in the glass ceramics and methacrylate groups of the resin cements.[12-15] HF etching followed by silane coupling agent is deemed as the most adopted intaglio surface protocol for adequate bonding to glass ceramics. This technique is essential for long-lasting glass ceramic restorations.[14,16]

During try-in procedures (internal and proximal fit adaptation, and esthetic assessments) of the ceramic on the prepared tooth, the intaglio glass ceramic surface treatment may become contaminated with saliva or human blood.[17-21] Saliva or human blood contamination may take place as a result of 1) the impossibility for rubber dam isolation, 2) marginal gingival bleeding from unsatisfactory provisional restoration finishing/polishing/adaptation, 3) marginal gingival inflammation related to gingivitis, and 4) oversight of previous tooth prophylaxis. Both saliva and human blood organic contaminants have a negative influence on the bond strength between resin cements and glass ceramics.[17,18,22,23]

Several methods (air/water spray, ethanol, phosphoric acid, and plasma) have been suggested to clean the contaminated ceramic surface prior to bonding procedures with certain degrees of success.[18,19,21,23,24] Recently, a commercial product was designed to effectively clean ceramic surfaces after saliva contamination and has since been confirmed.[20,22,25] Most of the laboratory studies evaluated the proposed cleaning methods before silane application;[18,22,23,25] however, higher contact angles were reported after silane application on glass ceramics as a result of a hydrophobic surface.[26,27] As such, it can be assumed that the cleaning methods for organic contaminants would perform better after silane application, and thereby properly restore the bonding strength to glass ceramics.

Therefore, the purpose of this laboratory study was to evaluate the efficacy of several cleaning methods on previously etched and silanized lithium disilicate glass ceramic after saliva or human blood contamination on the microshear bond strength (μSBS) to resin cement. The effect of a new silane layer application after the cleaning methods was also assessed. The tested hypotheses were: (1) the cleaning methods will restore the bond strength; (2) the cleaning methods will remove organic contaminants from silanized ceramic surfaces; and (3) silane reapplication after the cleaning methods will improve the bond strength.

METHODS AND MATERIALS

Ceramic Specimens

Two hundred and one discs (10-mm diameter x 3-mm thick) of a lithium disilicate reinforced glass ceramic (IPS e.max Press - shade LTA2, Ivoclar Vivadent, Schaan, Liechtenstein) (EMX) were fabricated according to the manufacturer's instructions.[7] The EMX samples were placed in a horizontal position and embedded in acrylic resin using polyvinyl siloxane (PVS) molds (20-mm diameter x 20-mm height). To obtain a flat, polished, and homogeneous surface, the samples were submitted to sequential polishing using silicon carbide abrasive papers (#400 and #800, Norton SA, São Paulo, SP, Brazil) in a water-cooled automatic polisher (Metaserv 250, Buehler, Lake Buff, IL, USA). Thereafter, all EMX specimens were cleaned in an ultrasonic bath for 10 minutes and dried using oil-free compressed air. The materials used in this study are described in Table 1.

The EMX surfaces were etched with 5% HF (Condac Porcelain, FGM, Joinville, SC, Brazil) for 20 seconds, rinsed using oil-free air–water spray (AWS) for 30 seconds, and air dried for 30 seconds. A silane coupling agent (Monobond N, Ivoclar Vivadent) was actively applied to the etched EMX surface with a disposable microbrush for 15 seconds, left to react for 60 seconds, and air-dried until all solvents were eliminated.

Ceramic Surface Contamination

Thirty etched/silanized EMX samples were randomly assigned into three control groups (n=10): no contamination (control), saliva positive control (SPC), and human blood positive control (BPC) (Figure 1). Organic components in SPC and BPC conditions were not removed prior to bond strength testing. One hundred and sixty etched/silanized EMX samples were randomly distributed into two groups according to the organic contaminant: saliva (SA) or human blood (HB). Subgroups were created according to the adopted cleaning method (n=20): AWS, 35% phosphoric acid (PPA) (UltraEtch, Ultradent Inc, South Jordan, UT, USA), 70% liquid alcohol (70A) (Prolink, Guapiaçu, SP, Brazil), and a commercial cleaning paste (Ivoclean, Ivoclar Vivadent) (IVO). Following the cleaning methods, half of the specimens (n=10) were subjected to a new silane layer re-application, as previously described (Figure 2).

Control Groups

For the control group (no contamination), a thin layer of a bonding agent (Scotchbond MultiPurpose Bond - "Step-3", 3M Oral Care, St Paul, MN, USA) was applied onto the etched/silanized EMX surface and light cured for 20 seconds using a polywave LED light curing unit (Bluephase N, Ivoclar Vivadent) at 1200 mW/cm^2,

Table 1: *Materials Used in This Study*

Material	Brand Name (Manufacturer)	Composition
Lithium disilicate glass ceramic	IPS e.max Press (Ivoclar Vivadent)	SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, other oxides and ceramic pigments
Porcelain etchant	Condac Porcelana 5% (FGM Produtos Odontológicos)	5% hydrofluoric acid (HF)
Silane coupling agent	Monobond N (Ivoclar Vivadent)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate
Phosphoric acid	Ultra-Etch (Ultradent Inc)	35% phosphoric acid, glycol, cobalt aluminate blue spinel
Alcohol	Álcool 70 Prolink (Prolink Indústria Química)	70% alcohol solution
Commercial cleaning paste	Ivoclean (Ivoclar Vivadent)	Sodium hydroxide, ZrO_2, water, polyethylene glycol, pigments
Bonding agent (adhesive)	Scotchbond MP (3M Oral Care)	Bisphenol A diglycidyl dimethacrylate (BisGMA), 2-hydroxyethyl methacrylate (HEMA), amines, photoinitiator
Light-cured resin cement	Variolink Esthetic (Ivoclar Vivadent)	Urethane dimethacrylate (UDMA) and methacrylate monomers, ytterbium trifluoride and spheroid mixed oxide, initiators, stabilizers, pigments

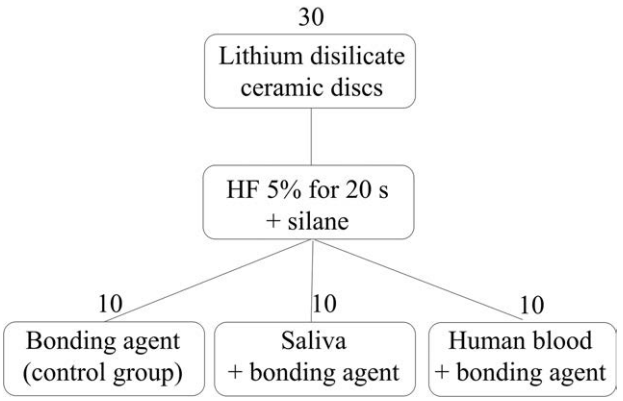

Figure 1. *Distribution of the control groups.*

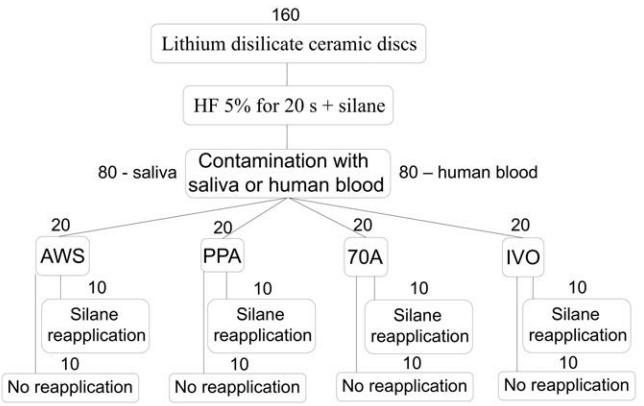

Figure 2. *Distribution of the cleaning methods and silane reapplication groups.*

with the curing tip positioned as close as possible to the EMX surface (<0.5 mm). For the SA and HB groups, after silane application, SA or HB were dropped on the EMX surface and let to react for 60 seconds. Next, an air blast was applied to remove any excess. A thin layer of the bonding agent was applied and light cured as described above. Thereafter, the EMX samples were prepared for mSBS testing. SA and HB were collected from a healthy donor, who did not eat or drink 2 hours prior to the collection procedure. In the SA groups, 1 mL of unstimulated human saliva was applied to the EMX surface using a graduated sterile pipette and left to react for 60 seconds. For the HB groups, one drop of human blood was collected from the fingertip (previously decontaminated with 70% alcohol) with 20 gauge lancets (Roche, Mannheim, Germany). The blood was then applied to the silanized EMX surface and allowed to react for 60 seconds.

Cleaning Methods and Silane Reapplication Groups

The following cleaning methods were applied after SA or HB (n=20) (Figure 2): an oil-free AWS was applied on the silanized and contaminated EMX surface for 20 seconds and air dried; 35% PPA was actively applied onto the silanized and contaminated EMX surface with a disposable microbrush for 20 seconds, followed by an oil-free AWS for 20 seconds and air dried; 70A was actively applied onto the silanized and contaminated EMX surface for 20 seconds with a disposable microbrush, followed by a oil-free AWS for 20 seconds and air dried; Ivoclean (IVO) cleaning paste (Ivoclar Vivadent) was actively applied onto the silanized and contaminated EMX surface for 20 seconds with a disposable microbrush. Subsequently, an AWS was applied for 20 seconds and then air dried.

Half of the silanized EMX surfaces received a fresh silane layer after the cleaning methods (n=10) (Figure 2). Next, a thin layer of the bonding agent was applied onto all EMX surfaces and light cured for 20 seconds using the polywave LED light curing unit, with the curing tip positioned as close as possible to the EMX surface (<0.5 mm).

Microshear Bond Strength Test (μSBS)

The microshear bond strength (μSBS) methodology has been previously described.[7,10] Round, 1-mm thick elastomer molds (Oranwash L, Zhermack, Italy) containing three cylinder-shaped orifices (∅=1 mm) were made and positioned onto the EMX ceramic surfaces for the bonding area. The orifices were filled with a light-cured resin cement (Variolink Esthetic-Shade Neutral, Ivoclar Vivadent), and Mylar strip and glass slab were placed over the top. A vertical load of 250 g was applied for 2 minutes to standardize the height of the resin cement cylinders. Next, the load and glass slab were removed, and the resin cement was light-cured for 40 seconds using the polywave LED light curing unit with the curing tip in close contact with the Mylar strip. All specimens were stored in deionized water at 37°C for 24 hours. After storage time, the elastomer mold was carefully sectioned with a #11 scalpel blade and removed. Cylinders that presented any flaws or defects were discarded. Three cylinders were fabricated on each ceramic disc (30 cylinders for each group).

A thin steel wire with a diameter of 0.2 mm was looped around each cylinder and aligned with the bonding interface for μSBS assessment. The μSBS test was performed using a universal testing machine (EMIC DL 500; Emic, São José dos Pinhais, PR, Brazil) with a 100 N load cell at a crosshead speed of 1.0 mm/minute until failure. The bond failure areas were

classified into four modes: adhesive (mode 1); cohesive within resin cement (mode 2); cohesive within ceramic (mode 3); and mixed, involving resin cement, adhesive and/or cohesive within the ceramic (mode 4).

Scanning Electron Microscopy (SEM) Evaluation

To observe the surface morphology of the silanized and contaminated surfaces before and after the cleaning methods, one specimen of each evaluated group (n=1) was prepared. After EMX surface etching with 5% HF, silanization, contamination with SA or HB, and cleaning protocols, the EMX samples were mounted on aluminum stubs and sputter coated with gold (Balzers - SCD 050, Balzers Union AG, Fürstentum, Liechtenstein) for 120 seconds at 40 mA. EMX surfaces were then examined by the same operator using SEM (JSM 5600 LV, JEOL, Tokyo, Japan) with 2000× magnification at 15 kV.

Statistical Analysis

Ten EMX samples were tested for each group, and the mean value of the three resin cement cylinders was considered the mean mSBS (MPa) value for each sample. Shapiro–Wilk analysis was performed to verify data normality. The mSBS data from control groups were subjected to one-way ANOVA (surface contaminants) and Tukey post-hoc test (α=0.05). The comparison among the different cleaning methods was submitted to a one-way ANOVA and multiple comparisons were performed using Tukey post-hoc test (α=0.05). Evaluation of the effect of silane reapplication was performed using an independent *t*-test (α=0.05).

RESULTS

Microshear Bond Strength Test (μSBS)

According to Table 2, when organic contaminants (saliva or human blood) were left on the silanized EMX surface, μSBS values decreased compared to the uncontaminated control group (p<0.05). Considering the saliva contamination, the cleaning methods were not different from one another when considering μSBS, all of which were effective in restoring the bond strength provided by the control group that was not contaminated (p>0.05) (Table 2).

For the groups contaminated with human blood, IVO was not different than AWS and PPA methods (p>0.05). IVO removed more organic compounds than 70A (p<0.05) and was the only the cleaning method able to restore the bond strength with values that did not differ from the uncontaminated control group (Table 2).

The reapplication of silane after contamination with saliva and AWS decreased bond strength values (p<0.05) (Table 3). When human blood contamination was subjected to PPA and 70A, the reapplication of the silane also decreased the bond strength values (p<0.05). For the other groups, silane reapplication did not result in higher bond strength values, regardless of the contaminant or cleaning method performed (p>0.05).

There was no effect of the organic contaminants, cleaning methods, or silane reapplication on the distribution of failure patterns (p>0.05). Bond failure occurred due to adhesive failure in 96.9%, cohesive failure in 2.6%, and mixed failure in 0.5% of cases involving resin cement. There were no cohesive failures in EMX.

SEM Evaluation

The images resulting from SEM analysis are presented in Figures 3 through 6. The uncontaminated surface etched with 5% HF depicted the glassy matrix removal and exposure of lithium disilicate crystals (Figure 3). Organic contaminants were found on the silanized EMX surface when not submitted to any cleaning method (Figure 4). The cleaning methods were able to remove organic contaminants (Figure 5 – saliva; Figure 6 – human blood). When exposed to human blood, organic contaminant was found on the silanized ceramic surface, except when subjected to IVO.

Table 2. *Means of μSBS (SD) of the Cleaning Methods Compared to Control Groups*[a]

Groups	μSBS (MPa)	
	Saliva Contamination	Human Blood Contamination
AWS	16.6 (5.7) A	15.2 (5.2) BC
PPA	15.5 (6.1) A	15.3 (4.5) BC
70A	15.8 (5.9) A	13.9 (5.9) C
IVO	16.0 (5.8) A	16.8 (5.2) AB
Contaminated control group	12.3 (4.1) B	10.8 (3.8) D
Uncontaminated control group	18.7 (4.9) A	

Abbreviations: AWS, Air–water spray; PPA, 35% Phosphoric acid; 70A, 70% Alcohol; IVO, Ivoclean.

[a] *Letters within a column indicate statistical difference among groups (p<0.05).*

Table 3: *Group Mean μSBS (SD) of Silane Reapplication Following Different Cleaning Methods for Human Saliva/Blood Removal from EMX Surfaces*[a]

Groups/ Cleaning Methods	μSBS (MPa)			
	Saliva Contamination		Human Blood Contamination	
	+	–	+	–
AWS	15.0 (5.5) B	18.1 (5.6) A	15.3 (5.0) A	15.2 (5.5) A
PPA	16.4 (6.5) A	14.6 (5.7) A	14.0 (4.5) B	16.6 (4.2) A
70A	16.1 (5.4) A	15.4 (6.4) A	12.5 (5.6) B	15.3 (4.6) A
IVO	16.8 (5.9) A	15.2 (5.6) A	17.4 (6.2) A	16.2 (3.9) A

Abbreviations: AWS, Air–water spray; PPA, 35% phosphoric acid; 70A, 70% Alcohol; and IVO, Ivoclean.

[a] *Letters within a column indicate statistical difference among groups (p<0.05).*

DISCUSSION

This laboratory study aimed to evaluate the efficacy of different cleaning methods to remove saliva or blood from previously etched and silanized lithium disilicate reinforced glass ceramic and the influence on bond strength. The first two tested hypotheses were accepted, since the cleaning methods restored the bond strength and the cleaning methods removed the organic contaminants from the silanized ceramic surface; however, the third was rejected, since silane reapplication after cleaning methods did not improve the bond strength.

When the silanized glass ceramic was contaminated with saliva, there was a reduction of 35% in the bond strength compared to the noncontaminated control group (Table 2). This may lead to early debonding of glass ceramic restorations. Other *in vitro* studies reported similar detrimental bond strength results.[17,18,22,23,28-32] Saliva is a very dilute fluid and consists mainly of water (99.4%) with a small percentage of solids (0.6%). Solids are made up of macromolecules (ie, proteins, glycoprotein sugars, enzymes, and mucins), inorganic particles (ie, calcium, sodium, and chloride), and organic particles (ie, urea, amino acids, fatty acids, and free glucose). Additionally, microorganisms, food residues, white blood, and epithelial cells are present in saliva.[21,28,34,35] Salivary components are able to adsorb the intaglio silanized ceramic surface (Figure 4A), creating a thin and invisible residual organic film. This film significantly hinders proper micromechanical–chemical interaction between EMX surface and resin cement, and may also impair the polymerization of said luting composite resin.

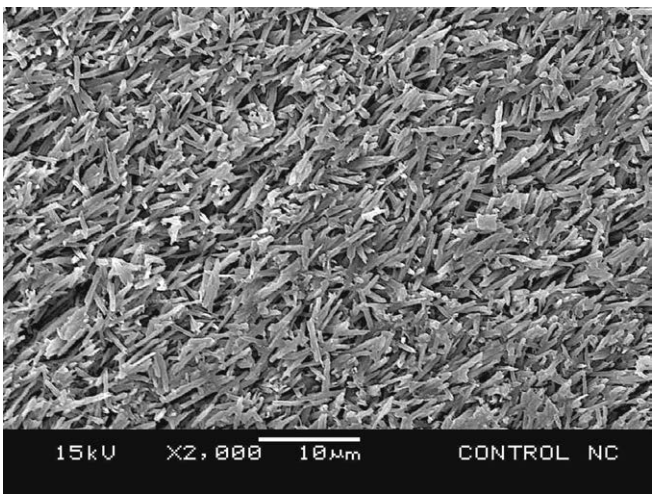

Figure 3. *Representative scanning electron microscopy (SEM) image (2000× magnification) of the control group (uncontaminated EMX surface) after etching with 5% hydrofluoric acid for 20 seconds.*

Laboratory studies have evaluated different cleaning methods on solely etched lithium disilicate glass ceramic with HF after saliva contamination,[18,22,25,29,31,32,35] but no consensus was reached. On the other hand, in the present study, all the proposed cleaning methods applied on a silanized lithium disilicate glass ceramic successfully restored the initial bond strength after contamination with saliva (Table 2). As seen in previous studies, higher contact angles were found in the group that had received HF conditioning followed by silane application, turning the glass ceramic surfaces from hydrophilic into hydrophobic, thereby reducing the material's surface energy.[26,27] As the silanized EMX repels water-based contaminants, it is easier to remove salivary film from the EMX-etched surface. Several laboratory studies[17,19,31,32] reported that silanization prior to saliva contamination showed a "hydrophobic protective effect" on the etched glass ceramics (ethanol

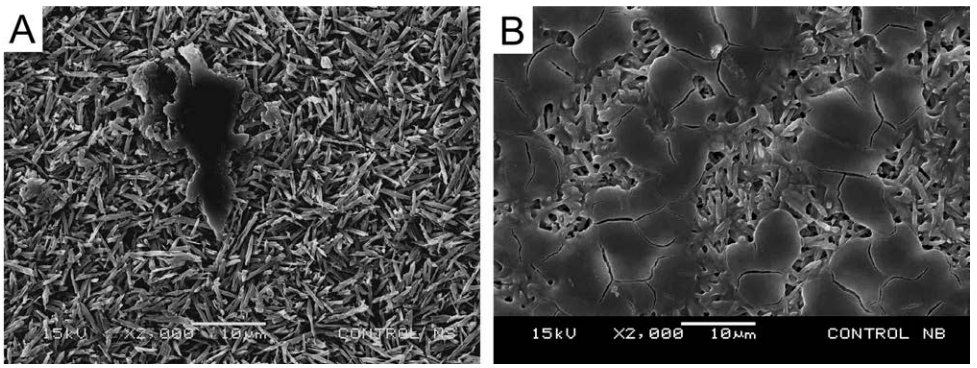

Figure 4. *Representative SEM images (2000× magnification) of the positive control groups (A, saliva; B, human blood) after etching the EMX surface with 5% hydrofluoric acid (HF) for 20 seconds and further silane application.*

application after rinsing with water, water rinsing only, 37% phosphoric acid or 80% ethanol, and experimental cleaning paste containing zirconium oxide and sodium hydroxide).[19,32] The proposed cleaning methods were also effective at restoring the bond strength, corroborating the results of the present study.[32]

The HB groups presented a reduction of 42% in bond strength compared to the uncontaminated control group (Table 2). This result is in agreement with other laboratory studies that have shown that human blood (consisting of several types of cells—ie, leukocytes, erythrocytes, and platelets—immersed in plasma)[36,38] contamination causes a large decrease in adhesive strength between resin increments during a resin restoration,[34] and between resin cement and dentin.[39-42] Phark and others[38] verified through X-ray photoelectron spectroscopy that contamination by saliva or blood left a complex organic and inorganic layer (thickness that did not exceed 10 nm) over microporosities of a modified zirconia. This was also observed in the present SEM images (Figure 4). This "dirt" layer may be responsible for the reduction in the bond strength values of the group contaminated with blood (Table 2). Both SA and HB impaired adequate micromechanical interaction between resin cement–EMX and the adequate chemical interaction between silane and the adhesive–resin cement.

To the best of our knowledge, there are no laboratory studies evaluating different cleaning methods on silanized lithium disilicate glass ceramic surface contaminated with human blood. SEM images (Figure 4) depicted that blood contamination forms a film much more complex than saliva, making it almost impossible to visualize the lithium disilicate crystals. The augmented barrier associated with blood contamination is due to the difference in the type and quantity of organic and inorganic elements. Even after the application of silane, the human blood may

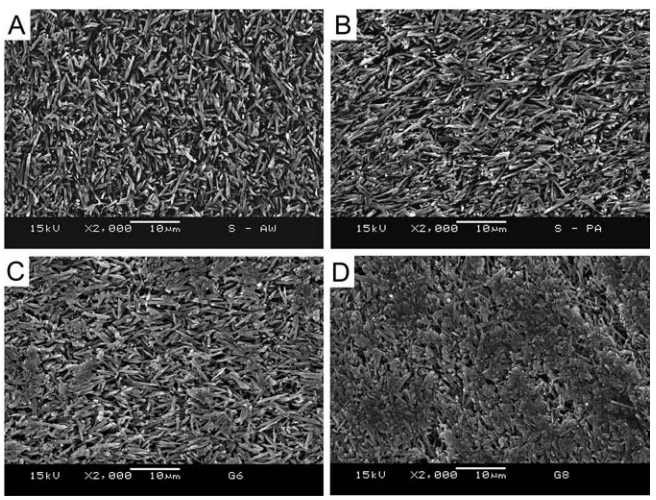

Figure 5. *Representative scanning electron microscopy (SEM) images (2000× magnification) of the contaminated EMX surface with saliva and later subjected to the cleaning methods: A, air–water spray (AWS); B, 35% phosphoric acid; C, 70% liquid alcohol (70A); and D, Ivoclean.*

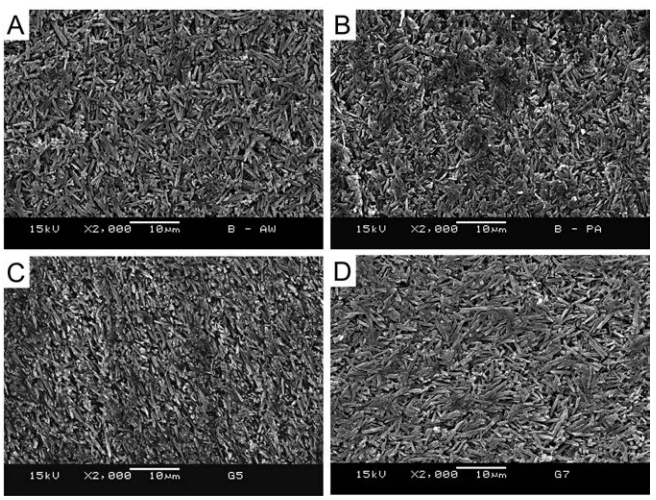

Figure 6. *Representative scanning electron microscopy (SEM) images (2000× magnification) of the contaminated EMX surface with human blood and later subjected to the cleaning methods: A, air–water spray (AWS); B, 35% phosphoric acid; C, 70% liquid alcohol (70A); and D, Ivoclean.*

have bonded strongly to the silanized EMX surface, making it difficult to remove (blood has less water than saliva, and plasma is more viscous than water, making its removal more difficult.). Despite the fact that AWS, PPA, and 70A yielded a cleaner EMX surface (Figure 6), they improved the bond strength up to 45% compared to the silanized EMX surface HB-contaminated. However, this improvement in bond strength was not to the point of values comparable with the noncontaminated control group (Table 2). It was observed that IVO—a hypersaturated solution of zirconium oxide and sodium hydroxide particles—was the only method capable of restoring the bond strength values comparable with the noncontaminated control group. This may suggest that IVO is able to dissolve the human blood constituent proteins, and subsequent rinsing can remove it from the silanized EMX surface.

According to the manufacturer's instructions, a fresh silane layer should be applied after cleaning with Ivoclean. In the present study, reapplying silane after each cleaning method did not yield higher bond strength and in some cases decreased it (Table 3). These results are in disagreement with other laboratory studies,[17,19] which reported that re-silanizing after decontamination protocols positively influenced bond strength values. Despite having no deleterious effect, Nikolaus and others[19] state that multiple or very-thick silane layers may have a negative effect on the bond strength, as it can lead to a cohesive failure.[43] The negative effect of a fresh silane layer on bond strength may be due to 1) the fresh silane layer would not have new Si-OH sites to react with the ceramic surface and form siloxane bonds, since they have already reacted within the first silane layer; 2) inadequate solvent removal after application of the second layer, which may alter the properties of resin-based materials; and 3) the methacrylate groups of the fresh silane (2nd layer) may react with the methacrylate groups of the first silane layer. Thus, the chemical interaction of silane with methacrylate groups of the bonding agent—resin cement may be affected.

The cleaning methods and products of the present study were chosen because they are easily found in dental offices. It is desirable to have a contaminant-free intaglio ceramic surface prior to adhesive cementation. However, if contamination occurs, it is preferable that it occurs after it has been previously etched and silanized. Dentists should exercise caution when checking the fit of the glass ceramic on the prepared tooth, since friction might cause damage to the etched/silanized surface. To avoid any damage, the impression/scanning of the prepared tooth and the fabrication of the glass ceramics must be carried out respecting the dental materials properties and, thus, avoiding/minimizing misadaptations. In the present study, a fresh layer of silane was not applied after contamination with saliva or human blood in positive control groups (where contaminants were not removed from the silanized EMX surface before μSBS testing) showing contaminants should be removed from the ceramic surface, as they impair the bonding procedure leading to reduction in bond strength. Future studies should address the effect of hydrolytic, mechanical, and thermal aging on the bond strength after cleaning methods and fresh application of silane.

CONCLUSIONS

Within the limitations of the present study, it can be concluded that: 1) Contamination with saliva or human blood impairs adherence to silanized lithium disilicate glass ceramic; 2) all cleaning methods (AWS, 35% phosphoric acid, 70% ethanol, and Ivoclean cleaning paste) demonstrated effectiveness in removing saliva contamination of silanized lithium disilicate glass ceramic; however, when contaminated with blood, only Ivoclean cleaning paste was effective at restoring the initial bond strength to silanized lithium disilicate glass ceramic; and 3) application of a fresh silane layer after the cleaning methods of the silanized lithium disilicate glass ceramic did not yield statistically different results from groups that were not resilanized. In some groups, there was a reduction in bond strength values after the application of a new silane layer.

Regulatory Statement

This study was conducted in accordance with all the provisions of the human subjects oversight committee guidelines and policies. The local Ethics Committee in Research approved the present laboratory research: approval #10271919.7.0000.5220.

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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Effect of Dentin Moisture in Posterior Restorations Performed with Universal Adhesive: A Randomized Clinical Trial

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Clinical Relevance

Dentin moisture seems not to be important for the postoperative sensitivity or clinical performance of posterior bulk-fill composite restorations, when a universal adhesive was applied.

SUMMARY

Objectives: This double-blind, randomized clinical trial evaluated the influence of dentin moisture on postoperative sensitivity (POS), as well as, on clinical performance in posterior bulk-fill composite restorations, using a universal adhesive, until 12 months after clinical service.

Methods and Materials: In accordance with a split-mouth design, 45 patients received posterior restorations, restored with a bulk-fill resin

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composite (Filtek Bulk Fill, 3M Oral Care) and a universal adhesive used in etch-and-rinse mode (SBU; Single Bond Universal Adhesive), which were applied on dry or moist dentin, with a cavity depth of at least 3 mm. Three operators placed 90 Class I/Class II restorations. Patients were evaluated for spontaneous and stimulated POS in the baseline, and after 48 hours, 7 days, and at 6 and 12 months. In addition, secondary parameters (marginal discoloration, marginal adaptation, fracture, and recurrent caries) were evaluated by World Dental Federation (FDI) criteria after 7 days and at 6 and 12 months. Statistical analyzes were performed using the Chi-square, Fisher exact, Friedman, Kruskall–Wallis, and Mann–Whitney tests (α=0.05).

Results: No significant spontaneous and stimulated POS was observed when SBU was applied in dry and moist dentin (p>0.05). A significant and higher risk of spontaneous POS (20.0%; 95%CI 10.9-33.82 for dry dentin and 22.22%; 95%CI 12.54-36.27 for moist dentin) occurred up to 48 hours after restoration placement for the dry and moist dentin groups (p<0.02). However, the POS intensity was mild up to 48 hours with no significant difference between dry and moist dentin groups (p>0.79). When secondary parameters were evaluated, no significant differences between the groups were observed.

Conclusion: Dentin moisture did not influence POS in posterior bulk-fill composite restorations when associated with a universal adhesive applied in etch-and-rinse mode.

INTRODUCTION

Direct resin composite restorations in posterior teeth have increased worldwide,[1] either due to the prohibitions related to the use of mercury-based materials such as amalgam[2] or due to the increased aesthetic needs of the population.[3] In this sense, a recent literature review showed that composite resin restorations are considered as the material of choice in dental schools around the world for restoring occlusal and occluso-proximal cavities in permanent teeth.[4]

Unfortunately, several clinical studies indicated that reported postoperative sensitivity (POS) after posterior resin composite restorations remains a challenge in dentistry.[5,6] The POS is related to many factors, such as the cavity preparation procedure, adhesive approach, type of resin composite used, and placement technique.[7-11]

However, one of the most important factors related to the POS is the anecdotal clinical perception that use of phosphoric acid on dentin (when etch-and-rinse adhesives are applied) significantly increase the POS.[12] After etching and rinsing the dentin, the removal of the smear layer and the opening of the dentinal tubules increases the dentin permeability and their hydraulic conductance.[13] After the adhesive system application, if the resin monomers did not correctly infiltrate in the demineralized dentin, voids occurred in the hybrid layer. Several studies showed that voids frequently occurred when the dentin was kept dry after phosphoric acid etching.[14,15] These unfilled spaces may allow dentin fluid movement, especially under external stimuli. This, in turn, sensitizes the nerve endings in the dentin tubules, and it may cause POS.[16]

The wet-bonding technique is a very simple technique to improve adhesive infiltration.[16] In this technique, if the dentin demineralized matrix is kept fully hydrated by the clinician during the adhesive procedure, it will not cause a collapse of collagen fibrils, and free space will be available for resin infiltration.[14,15] Due to the intrinsically wet nature of dentin, it is necessary to use ethanol- or acetone-based adhesives.[15,17] Therefore, in the last three decades, wet-bonding has been the most popular technique to maintain an adequate degree of moisture for an etch-and-rinse adhesive.[18]

However, the popularity of wet-bonding techniques changed with the emergence of a new generation of adhesives called universal or multimode adhesives.[19,20] These adhesives are single-bottle adhesive systems similar to self-etch adhesives but include several acidic functional monomers, including 10-methacryloyloxydecyl dihydrogen phosphate (MDP) the most known among them. Functional monomers promote chemical bonding between the enamel and dentin and the indirect materials, such as glass ceramics, zirconia and metals, following a manufacturing of one product for application in different clinical situations.[21,22]

To guarantee that MDP provides stable and durable interfaces, all universal adhesives must contain water, because water is essential for ionizing the acidic functional monomers that make self-etching possible.[17,23] Although, the exact amount of water content of the universal adhesives was not disclosed by the manufacturers, several studies have already claimed that universal adhesives contain approximately 10–25 wt% of water.[24-27]

Due to the self-capacity of water to reexpand the air-dried and collapsed collagen mesh, for adhesive resin infiltration,[13] keeping the dentin dry or moist after the phosphoric acid application does not make a

difference in the universal adhesive's bonding quality, which was observed in several recently published *in vitro* studies.[25-28] Furthermore, recent clinical studies in noncarious cervical lesions have shown that universal adhesive systems are less sensitive to dry and moist dentin, because no significant differences in terms of clinical performance (retention, marginal adaptation, or discoloration) were observed when MDP-based universal adhesives were evaluated through 3 years of follow-up.[29-33] However, all previously published clinical trials were performed on noncarious cervical lesions. Unfortunately, there is a huge regional variability of permeability, cavity format, and dentin moisture in the dentin of posterior restorations compared to the dentin walls of noncarious cervical lesions.[34] Therefore, it is very important to evaluate the effect of degree of dentin moisture (dry or moist) and the subsequent effect on the clinical performance of an MDP-based universal adhesive in posterior resin composite restorations.

Thus, this double-blind, randomized clinical trial evaluated the influence of dentin moisture on spontaneous and stimulated POS in posterior resin composite restorations using a universal adhesive applied in etch-and-rinse mode, after 48 hours, 7 days, and 6 and 12 months. In addition, the marginal discoloration, marginal adaptation, fracture, and recurrence of caries were evaluated by World Dental Federation (FDI) criteria after 6 and 12 months. The null hypotheses were: (1) dentin moisture does not influence the spontaneous and stimulated POS evaluated at different times (48 hours, 7 days, and 6 and 12 months) when compared to a universal adhesive applied in etch-and-rinse mode on dry dentin. (2) Dentin moisture does not influence the other evaluated clinical parameters (marginal staining, fracture, marginal adaptation, and the recurrence of caries) at different times (6 and 12 months) when compared to a universal adhesive applied in etch-and-rinse mode and used on dry dentin.

METHODS AND MATERIALS

Ethics Approval and Protocol Registration

The State University of Ponta Grossa Ethics Committee on Involving Human Subjects reviewed and approved the protocol and consent form for this study (protocol 1.752.848). Written informed consent was obtained from all participants prior to starting the treatment. The experimental design followed the Consolidated Standards of Reporting Trials (CONSORT) statements.[35] This was a randomized, double-blind clinical trial, registered in the Clinical Trials Registry. The restorations were placed in the clinics of the State University of Ponta Grossa from October 2017 to December 2018. We informed all participants about the nature and the objectives of the study, but they were not aware of what tooth received the specific treatments under evaluation.

Participant Recruitment

Patients were recruited as they sought treatment in the clinics of the State University of Ponta Grossa School of Dentistry. Those who qualified for the study were recruited in the order in which they reported for the screening session, thus forming a convenience sample. Participants were recruited through written advertisements placed on the university's walls.

Sample Size

The sample size calculation was based on the absolute risk of spontaneous POS in posterior resin composite restorations. According to the literature, the risk of POS was approximately 30% in deep and large restorations.[7,9-11] Using an α of 0.05, a power of 80%, and a two-sided test, the minimal sample size was 45 restorations in each group (considering 20% loss) to detect a 20% difference between groups with the adhesive in dry dentin.

Eligibility Criteria

Two pretrained dentists examined 63 participants to check if the subjects met the inclusion and exclusion criteria (Figure 1). The evaluations were performed using an intraoral mirror, an explorer, and a periodontal probe. Participants had to be in good general health, at least 18 years old, and present at least 20 teeth under occlusion and at least two carious lesions and/or indication of replacement restorations (fracture, secondary caries, and temporary restoration) in different hemiarches with depths $\geq$ 3 mm, which were diagnosed using an interproximal radiograph. As much as possible, we always tried to select participants with two cavities in the same hemiarch, the same cavity type, and the same number of cavity surfaces to be restored.

Participants with dental prostheses, extremely poor oral hygiene, severe or chronic periodontitis, severe bruxism, parafunctional habits, continuous use of medication that may alter the perception of pain (analgesic, anti-inflammatory, etc.), and patients undergoing bleaching treatments or who were pregnant were excluded. Based on preestablished criteria, we selected 45 subjects who volunteered for this study (Figure 1).

Randomization Sequence Generation, Allocation Concealment, and Blinding

A staff member not involved in the research protocol performed the randomization process within subjects through http://www.sealedenvelope.com. Details of the allocated group were recorded on cards contained in sequentially numbered and sealed opaque envelopes. A staff member who was not involved in any of the clinical trial phases prepared these. The allocation assignment was revealed by opening the envelope on the day of the restorative procedure to guarantee the concealment of the random sequence and to prevent selection bias. The operator who implemented the interventions was not blinded to the procedure. However, the participants and the examiners were blinded to the group assignment.

Baseline Characteristics of the Selected Teeth and Calibration Procedure

The same three trained and calibrated dentists involved in the selection of participants carried out the restorative procedures. The features of the posterior restorations were evaluated prior to the placement of the restorations. Features, such as the presence of antagonist and attrition facets were observed and recorded. Patients were assessed for their risk of caries, and parafunctional habits, such as bruxism, for each patient were estimated by means of clinical and sociodemographic information, taking in account the incipient caries lesions and a history of caries and parafunctional habits.

Spontaneous preoperative sensitivity was evaluated prior to examination as well as the different preoperative sensitivity stimuli (air, cold, heat, vertical, and horizontal touch). To measure the sensitivity by air, air-drying was applied for 10 seconds from a dental syringe placed 2 cm from the surface of the tooth; the percussion sensitivity was measured with percussive load applied vertically on the occlusal aspect of the tooth and horizontally (vestibular area) on the buccal aspect of the tooth with the blunt end of a mouth mirror handle, as well as in the contralateral tooth; cold stimulation was conducted through the application of a swab with Endo Ice (Maquira, Maringá, PR, Brazil) applied to the vestibular face in the cervical region of the restored tooth; and heat stimulation was applied to the tooth surface with a gutta-percha stick (Dentsply, Sirona, Charlotte, NC, USA).[36]

Spontaneous preoperative sensitivity was evaluated through the intensity of tooth sensitivity measurement through the Visual Analogue Scale (VAS) and the NRS (Numerical Rating Scale). The VAS scale consists of a 10-cm linear scale with the words "no pain" at one end and "unbearable pain" on the other. The NRS consists of five verbal points with the 0 meaning "no pain" and 4 meaning "severe pain".

For the calibration procedure step, the study director placed one restoration for each group to identify all the steps involved in the protocol. Then, three operators placed another four restorations for each group under the supervision of the study director in a clinical setting. Any discrepancies of the restorative protocol were identified and discussed with the operator prior to starting the study. At this point, the operators were considered trained to perform the restorative procedures. The calibrated operators restored all teeth under the supervision of the study director.

Interventions: Restorative Procedure

The interventions were standardized by a detailed protocol, which is briefly summarized below. A preliminary dental prophylaxis of the tooth surface was performed with pumice and water in a rubber cup, with the aim of removing the salivary pellicle and any remaining dental plaque, followed by rinsing and drying. Using a shade guide, the proper shade of the resin composite was determined. Local anesthesia was applied with a 3% mepivacaine solution (Mepisv, Nova DFL, Rio de Janeiro, RJ, Brazil), and all restorations were placed under rubber dam isolation. The operators did not prepare any additional retention or bevel in the cavities.

All subjects received a minimum of two restorations, one from each experimental group, in different cavities previously selected according to the inclusion criteria. The cavity dimensions in millimeters (height, width, and depth) and the cavity geometry were also recorded. The cavity design was performed using a spherical diamond bur (#1013-1017; KG Sorensen, Barueri, SP, Brazil) mounted in a high-speed handpiece with an air–water spray. It was only applied for the removal of defective restorations or for the elimination of carious tissues (caries-infected dentin). No liner or base was used. For restoration of class II cavities, a sectional matrix system (Palodent, Dentsply Sirona) was preferentially used. However, circumferential matrix systems were used when a good adaptation could not be obtained with the sectional matrix system.

Then, an application of 34% phosphoric acid (Scotchbond Universal Etchant, 3M Oral Care, St. Paul, MN, USA) was conducted for 15 seconds in dentin/enamel, followed by rinsing with a dental syringe for 10 seconds. Afterward, in the groups assigned for dry dentin, all dentin surfaces were dried for 10 seconds at a distance of 2 cm between the tip of the air syringe and the dentin surface. At the end, the dentin surface was

Table 1: *Adhesive System and Resin Composite: Composition and Application Mode*

Adhesive System and Resin Composite	Composition/Batch Number[a]	Application in Etch-and-Rinse Mode		
Single Bond Universal Adhesive (3M Oral Care, St Paul, MN, USA)	1. Scotchbond Universal Etchant (643399): 34% phosphoric acid 2. Adhesive (691954): Methacryloyloxydecyl dihydrogen phosphate, phosphate monomer, dimethacrylate resins, hydroxyethyl methacrylate, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, silane, camphorquinone	Apply Etchant for 15 seconds. Rinse for 10 seconds.	Dry dentin: Air dry (10 seconds) to remove excess of water and keep dentin completely dry Wet dentin: Air dry (2-4 seconds) to remove only excess of water and keep dentin visible moist	Apply the adhesive for 20 seconds with vigorous agitation. Gently stream of air for 5 seconds. Light-cure for 10 seconds (1000 mW/cm^2)
Filtek Bulk Fill Posterior Restorative (3M Oral Care) Shade A2 and A3	Resin Matrix: AUDMA (urethane aromatic dimethacrylate)/ UDMA/1,12-dodecane-DMA (12-dodecane dimethacrylate) (N68566) Fillers: Combination of a non-agglomerated/ non-aggregated 20 nm silica filler, a non-agglomerated/ non-aggregated 4 to 11 nm zirconia filler, an aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles) and a ytterbium trifluoride filler consisting of agglomerate 100 nm particles; 76.5 wt%, 58.4 vol%. Photoinitiator: Camphorquinone	Insert in the cavity bulk increases of up to 4-5 mm in thickness, and light-cure each area of the surface of the restoration with 1000 mW/cm^2 for 30 seconds.		

[a] *According to the manufacturer's instructions.*

completely dry, without any signs of moisture. In the groups assigned for moisture dentin, only the excess water in the dentin surface was removed through air-drying for 2-4 seconds at a distance of 2 cm between the tip of the air syringe and the dentin surface. At the end, the entire dentin surface was shiny, because moisture was visible (Table 1).[29-31]

The Single Bond Universal Adhesive (SBU; 3M Oral Care, also known as Scotchbond Universal in some countries) was shaken, and a small drop was put in a microbrush (Cavibrush, FGM, Joinville, SC, Brazil). Then, the microbrush was rubbed onto the surface of the dentin under manual pressure, followed by thinning with gentle air-drying for 5 seconds. At the end, the entire surface was light cured (Radii Cal, SDI, Victoria, Australia) for 10 seconds (1000 mW/cm^2; Table 1). The resin composite Bulk Fill (3M Oral Care) was used in a single increment and photoactivated for 30 seconds (1000 mW/cm^2; Radii Cal, SDI, Victoria, Australia). After finishing the restorations, the occlusal adjustment was carried out, and followed by finishing and a final polishing with fine-grained diamond tips FF (KG Sorensen, Barueri, SP, Brazil) and polishing with rubber bowls (Astropol, Ivoclar Vivadent, Schaan, Liechtenstein).

Examination After Restorative Procedure

Spontaneous POS was the primary clinical outcome analyzed, and it was assessed at 48 hours, 7 days, and 6 and 12 months, using the VAS and NRS, as

Table 2: *World Dental Federation (FDI) Criteria Used for Clinical Evaluation (Hickel and others)*[37,38]

	Functional Properties				
	1. Fracture	**2. Marginal Adaptation**	**3. Contact Point/ Food Impact**	**4. Radiographic Exam**	**5. Patient View**
1. Clinically very good	Restoration retained, no fractures/ cracks	Harmonious outline, no gaps, no discoloration	Normal contact point (floss or 25 µm)	No pathology, harmonious transition between restoration/ tooth	Entirely satisfied
2. Clinically good (after correction very good	Small hairline crack	Marginal gap (50 µm) or small marginal fracture removable by polishing	Slightly too strong but no disadvantage	Acceptable cement excess present or positive/negative step present at margin <150 µm	Satisfied
3.Clinically sufficient / satisfactory (minor shortcomings with no adverse effects but not adjustable without damage to the tooth)	Two or + larger hairline cracks and/ or chipping (not affecting the marginal integrity)	Gap < 150 µm not removable or several small enamel or dentin fractures	Slightly too weak, no indication of damage to tooth, gingivae or periodontal structures	Marginal gap < 200 µm; negative steps visible with no adverse effects. Noticed or poor radiopacity of filling material	Minor criticism due to aesthetic shortcomings; some lack of chewing comfort or; Time consuming procedure and/or similar; No adverse clinical effects
4. Clinically unsatisfactory (repair for prophylactic reasons)	Chipping fractures which damage marginal quality; bulk fractures with or without partial loss (- than ½ of the restoration)	Gap > 250 µm or dentin/ base exposed; chip fracture damaging margins or notable enamel or dentin wall fracture	Too weak (100 µm metal blade can pass) and possible damage (food impaction). Repair possible	Marginal gap >250 µm; cement excess accessible but not removable or; negative steps >250 µm and repairable	Desire for improvement (reshaping of anatomic form or refurbishing etc.)
5. Clinically poor (replacement necessary)	Partial or complete loss of restoration	Filling is loose but in situ	Too weak and/ or clear damage (food impaction) and/or pain/ gingivitis)	Secondary caries, large gaps; apical pathology or; Fracture/loss of restoration or tooth	Completely dissatisfied and/oral adverse effects including pain
Acceptable or not acceptable (n, % and reasons			Functional criteria		

previously described. The stimulated POS was also evaluated (secondary outcomes) at 7 days, 6 months, and 12 months. At each time, the restoration was evaluated for sensitivity caused by air application, vertical and horizontal percussion, and cold and heat stimulation, as described in the initial evaluation. The

Table 2: *World Dental Federation (FDI) Criteria Used for Clinical Evaluation (Hickel and others) (cont.)*[37,38]

	Esthetic Properties		Biological Properties	
	6. Marginal Staining	**7. Color Stability and Translucency**	**8. Postoperative (Hyper-) Sensitivity**	**9. Recurrence of Caries**
1. Clinically very good	Good color match No difference in shade and translucency	No marginal staining	No hypersensitivity	No secondary or primary caries
2. Clinically good (after correction very good	Minor deviations	Minor marginal staining (under dry conditions) is present	Low hypersensitivity for a limited period of time	Very small and localized demineralization. No operative treatment required
3.Clinically sufficient / satisfactory (minor shortcomings with no adverse effects but not adjustable without damage to the tooth)	Clear deviation but acceptable. Does not affect aesthetics: (more opaque; translucent; dark or bright)	Moderate marginal or surface staining not noticeable from a speaking distance	Premature/slightly more intense or delayed/ weak sensitivity; no subjective complaints, no treatment needed	Larger areas of demineralization, but only preventive measures necessary (dentin not exposed)
4. Clinically unsatisfactory (repair for prophylactic reasons)	Localized - clinically unsatisfactory but can be corrected by repair (too opaque; translucent; dark or bright)	Localized marginal staining is present and not removable by polishing. The aesthetic properties of the dentition are affected.	Premature/ very intense; extremely delayed/weak with subjective complaint or negative Sensitivity Intervention necessary but not replacement	Caries with cavitation (localized and accessible and can be repaired
5. Clinically poor (replacement necessary)	Unacceptable, replacement necessary	Generalized/ profound marginal discoloration is present. Replacement is necessary	Very intense, acute pulpitis or non-vital. Endodontic treatment is necessary	Deep secondary caries or exposed dentin
Acceptable or not acceptable (n, % and reasons	Aesthetic criteria		Biological criteria	

final values of spontaneous POS were divided into two categories: percentage of patients who reported POS at least once during treatment (absolute risk) and overall POS intensity over 48 hours, 7 days, 6 months, and 12 months. Furthermore, in the 6- and 12-month return visits, the clinical outcomes, such as marginal staining, fracture, marginal adaptation, and recurrence of caries, were evaluated using the World Dental Federation (FDI)[37, 38] criteria (Table 2).

Statistical Analysis

The statistician was blinded to the type of study groups, and the statistical analyses followed the intention-to-treat protocol according to CONSORT suggestions.[35] Participants who experienced at least one event of POS in each evaluation time (48 hours, 7 days, and 6 and 12 months) were considered as have POS. The risk of spontaneous and stimulus (air, cold, heat, horizontal, and vertical percussion) POS between the groups in each time were compared using the Chi-square test and

Fisher exact test. The risk of spontaneous POS among different times for each group were compared with the Friedman repeated measures analysis of variance by rank.

The intensities of spontaneous POS in each group for different evaluation times (48 hours and 7 days) were evaluated using the Friedman repeated measures analysis of variance by rank and Mann–Whitney tests (VAS) and one-way repeated measures, and Tukey test (NRS). The intensity of spontaneous POS in each time for both the groups was evaluated using the Mann–Whitney test (VAS) and *t*-test for dependent variables (NRS).

Additionally, the risks of POS according to cavity characteristics were compared using the Chi-square test. Statistical analyses for each item and overall parameter (FDI criteria) were performed. The differences in the ratings of the two groups and each group at baseline, after 6 months and after 12 months were tested with the Wilcoxon rank sum test repeated measures analysis of variance by rank (α=0.05). In all statistical tests, the alpha was set at 5% (Statistica for Windows 7.0, StatSoft Inc, Tulsa, OK, USA).

Characteristics of the Participants and Cavities

No modifications were performed in the experimental protocols, and they were implemented exactly as planned. Twenty-seven women and 18 men participated in this study. The mean age of the participants was 30.0 ± 8.20 years. Ninety restorations were placed, 45 for each group. The restorations were distributed into class I (75) and class II (15) cavities (Table 3). The homogeneity of cavity characteristics between the study groups can be seen in Table 3. Seven participants did not attend the 6 and 12 months recall, because they moved to another city (Figure 1).

RESULTS

POS Evaluation

A higher risk and intensity of spontaneous POS for both groups occurred up to 48 hours after restoration placement, with statistically significant differences for other evaluation times (Table 4, p<0.02; Table 5, p>0.01) However, no statistically significant difference was found for the risk and intensity of spontaneous POS in each period when dry and moist dentins were compared (Tables 4 and 5; p>0.58). It is noteworthy that, in a 1-week evaluation period, the intensity of spontaneous POS was considered mild when measured through the VAS and NRS scales (Table 5).

After 1 week, 6 months, and 12 months, a few participants reported experiencing stimulus POS, with

Table 3: *Characteristics of the Research Subjects, Dental Arches and Cavities Per Group*

Characteristics of Research Subjects		
Gender Distribution	**Number of Subjects**	
Male	18	
Female	27	
Age Distribution (years)		
20-29	29	
30-39	10	
40-49	4	
>49	2	
Characteristics of Dental Arches and Cavities	**Number of Restorations**	
Presence of Antagonist	**Dry Dentin**	**Moist Dentin**
Yes	44	45
No	1	0
Attrition Facet		
Yes	3	3
No	42	42
Arch Distribution		
Maxillary	19	20
Mandibular	26	25
Cavity Depth		
3 mm	16	14
4 mm	21	21
>4 mm	8	10
Black Classification		
I	37	38
II	8	7
Number of Restored Surfaces		
1	35	38
2	10	7
3	0	0
4	0	0
Reasons for Restoration		
Marginal fracture	1	0
Esthetic reasons	18	17
Marginal discoloration	0	0
Bulk fracture	7	8
Primary/Secondary caries lesion	19	20

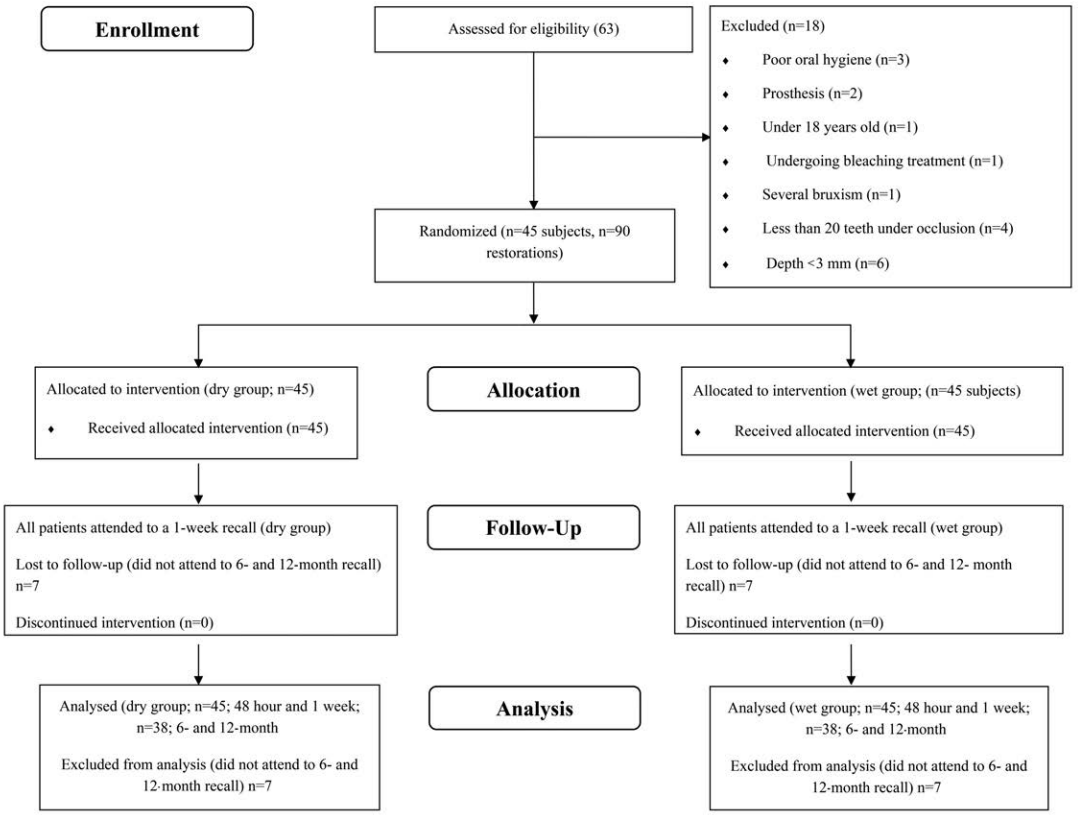

Figure 1. *Participant flow diagram in the different phases of the study design. Np, number of participants; Nr, number of restorations.*

no statistically significant difference when dry and moist dentin were compared (Table 6; $p>0.59$). However, no participants needed to take oral medication to reduce POS. When the cavities' characteristics were evaluated, the type of cavity, the number of surfaces, and the cavity depth did not show any significant differences (Table 7; $p>0.58$).

Other Clinical Parameters

Fourteen restorations showed small marginal discrepancies after the 12-month recall, with no statistical difference between the dry and moist dentin groups (Table 8; $p=1.0$). Five restorations showed some marginal fractures after the 12-month recall, with no statistical difference between the groups (Table

Table 4: *Number of Patients with Spontaneous POS/Total During 12 Months of Follow-up, as well as the Absolute Risk of POS*

Time Assessment		Dry Dentin[a]		Moist Dentin		*p*-value[b]
		Number of Patients with POS/Total	Absolute Risk (95%CI)	Number of Patients with POS/Total	Absolute Risk (95%CI)	
Preoperative	Baseline	1/45	2.22(0.39-11.57) A	3/45	6.67 (2.29-17.86) a	0.6
Postoperative	Up to 48 hours	9/45	20(10.9-33.82) B	10/45	22.22 (12.54-36.27) b	1.0
	7 days	3/45	6.67(2.29-17.86) A	2/45	4.44 (1.23-14.83) a	1.0
	6 months	2/38	5.26(1.46-17.29) A	0/38	0.00 (0.00-9.18) a	1.0
	12 months	2/38	5.26(1.46-17.29) A	1/38	0.00 (0.47-13.49) a	1.0

[a]*Different uppercase (dry dentin) and lowercase letters (moist dentin) indicate significant differences among time assessment (Friedman test;* p<*0.05).*
[b]*Chi-square or Fisher exact test (*p<*0.05).*

Table 5: *Intensity of Spontaneous POS Experienced by Patients During 7 Days of Follow-Up*

Time Assessment	Visual Analogue Scale[a]		*p*-value[c]	Numerical Rate Scale[b]		*p*-value[c]
	Dry Dentin	Moist Dentin		Dry Dentin	Moist Dentin	
Up to 48h	5 (4.7) B	3.5 (2-5) B	0.62	5.2 (2.9) b	3.8 (2.17) b	0.54
7 days	1 (1.2) A	1 (1-2) A	1.0	1.1 (1.1) a	1.2 (1.3) a	1.0

[a]*Mean and standard deviation; different lowercase letters indicate significant differences among time assessment (1-way repeated measures ANOVA and Tukey test;* p*<0.05).*

[b]*Median and interquartile range; different uppercase letters indicate significant differences among time assessment (Friedman test and Mann–Whitney test;* p*<0.05).*

[c]*Chi-square or Fisher exact test (*p*<0.05).*

8; p=0.72). Five restorations showed some marginal discolorations after the 12-month recall. Once again, no statistical difference between dry and wet dentin groups was observed (Table 8; p=0.45). No restorations had recurrent caries at the 12-month recall (Table 8; p=1.0).

DISCUSSION

The present randomized clinical trial evaluated POS, as well as the clinical performance of posterior bulk-fill resin composite restorations, associated with a universal adhesive applied in the etch-and-rinse mode in dry and moist dentin. The results of the present study showed that keeping the demineralizing dentin dry or moist did not significantly increase the spontaneous and stimulated POS in resin composite posterior restorations, leading us to accept the first null hypothesis. To the extent of the authors' knowledge, this is the first study that evaluated the effect of dentin moisture on the clinical performance of resin composite in posterior restorations using a universal adhesive.

Several *in vitro* studies have shown that it is necessary to keep the dentin moist to achieve a proper adhesive infiltration in the demineralized dentin and, consequently, allow adequate sealing and high immediate bond strength values.[13-16] On the other hand, low bond strength values were achieved when adhesive systems were applied in dry dentin, mainly because there was shrinkage of collagen fibrils after the drying procedure.[13-16]

Table 6: *Number of Patients who Experienced Provoked Pre- and Postoperative/Total to Different Stimulus in the Baseline and 7 Days Follow-Up*

Time Assessment/Stimulus		Dry Dentin		Moist Dentin		*p*-value[a]
		Number of Patients with POS/Total	Absolute Risk	Number of Patients with POS/Total	Absolute Risk	
Preoperative	Air	1/45	2.22 (0.30-11.57)	2/45	4.44 (1.23-14.83)	1.0
	Cold	25/45	55.56 (4118-6906)	26/45	57.78 (43.3-71.03)	0.83
	Heat	4/45	8.89 (3.51-20.73)	5/45	11.11 (4.34-23.5)	0.97
	Horizontal percussion	2/45	4.44 (1.23-14.83)	1/45	2.22 (0.30-11.57)	1.0
	Vertical percussion	3/45	6.67 (2.29-17.86)	4/45	8.89 (3.51-20.73)	0.69
Postoperative (7 days)	Air	2/45	4.44 (1.23-14.83)	1/45	2.22 (0.30-11.57)	1.0
	Cold	8/45	17.78 (9.29-31.33)	10/45	22.22 (12.54-36.27)	0.59
	Heat	2/45	4.44 (1.2-14.83)	1/45	2.22 (0.30-11.57)	1.0
	Horizontal percussion	1/45	2.22 (0.30-11.57)	2/45	4.44 (1.23-14.83)	1.0
	Vertical percussion	2/45	4.44 (1.23-14.83)	2/45	4.44 (1.23-14.83)	1.0

[a]*Chi-square or Fisher exact test (*p*<0.05).*

Table 7: *Number of Patients (%) who Experienced Spontaneous Postoperative Sensitivity up to 48 Hours Follow-up According to the Characteristics of Dental Arches and Cavities*

Characteristics	Number of Sensitive Teeth (%)		*p*-value[a]
	No	Yes	
Cavity Depth			
3 mm	25 (83.3)	5 (16.6)	0.58
More of 3 mm	46 (76.6)	14 (23.4)	
Black Cavity			
Class I	59 (78.6)	16 (21.4)	1.0
Class II	12 (80)	3 (20)	
Number of Restored Surfaces			
1 or 2 faces	71 (78.8)	19 (21.11)	1.0
3 or 4 faces	0	0	

[a]*Chi-square test and Fisher exact test.*

However, universal adhesives seemed to have a different behavior when applied in dry and moist dentin.[25,27,28,39,40] Universal adhesives can be used as a self-etch system, and the addition of water in their composition is important, because it ionizes the acidic groups, allowing the formation of hydronium ions, which etch hydroxyapatite.[41] The water content of the universal adhesives is strongly related to the pH, because the water is essential for ionizing the acidic functional monomers, thus making self-etching possible.[17,41]

Table 8: Number of Evaluated Restorations for Dry and Moist Dentin Classified According to the World Dental Federation (FDI) Criteria (Hickel and others)[37,38]

FDI Criteria	Score[a]	Baseline		6 Months		12 Months	
		Dry	Wet	Dry	Wet	Dry	Wet
Marginal Adaptation	VG	45	45	36	38	32	30
	GO	—	—	1	—	4	6
	SS	—	—	1	—	2	2
	UN/PO	—	—	—	—	—	—
Marginal Staining	VG	45	45	32	36	35	36
	GO	—	—	5	1	3	2
	SS	—	—	1	1	—	—
	UN/PO	—	—	—	—	—	—
Fractures	VG	45	45	37	36	36	35
	GO	—	—	1	2	2	3
	SS	—	—	—	—	—	—
	UN/PO	—	—	—	—	—	—
Recurrence of Caries	VG	45	45	38	38	38	38
	GO	—	—	—	—	—	—
	SS	—	—	—	—	—	—
	UN/PO	—	—	—	—	—	—

[a]*VG, clinically very good; GO, clinically good; SS, clinically sufficient/satisfactory; UN, clinically unsatisfactory; PO, clinically poor.*

According to the manufacturer, SBU contains approximately 10% of water.[42] Perdigão and others[39] were the first to evaluate the effect of dry and moist dentin on the performance of SBU. The authors showed that the ultramorphology evaluation of the adhesive–dentin interface observed similar hybrid layer formation when SBU was applied in dry or moist dentin. The authors speculated that the water contained in SBU may be able to plasticize the collapsed collagen network, allowing for re-expansion and reopening of the interfibrillar spaces for the infiltration of resin monomers.[43] These results were recently confirmed through several studies.[25,27,28] For instance, Choi and others[25] and Tsujimoto and others[28] showed that the immediate bond strength and bond fatigue strength of SBU did not show any significant difference when dentin was kept dry or moist.[25,28]

In addition, a second component of SBU, the presence of polyalkenoic acid copolymer, could be partially responsible for the similar clinical results observed in the present study. Actually, according to Sezinando and others[44], the presence of polyalkenoic acid copolymer in the SBU showed better immediate and 6-month bond strength results, when compared to an experimental SBU without this component. However, according to the manufacturer, the use of polyalkenoic acid copolymer provides a better moisture stability.[45,46] Therefore, we hypothesized that, due to the presence of polyalkenoic acid copolymer in the SBU, this adhesive is less sensitive to moisture variations, when dry or moist dentin conditions were simulated,[45,46] as occured in the present study. Future clinical studies need to be done to confirm this hypothesis.

All these characteristics of SBU help to explain the similarity of immediate POS, as well as POS after several times of evaluation, when the universal adhesive was applied in the dry or moist dentin. However, it's important to mention that the spontaneous and stimulus POS was very low after 1 week, as previously demonstrated in recent clinical studies that evaluated the same commercial brand.[47,48] These results agree with a recent published meta-analysis of clinical studies,[12] indicating that POS generated immediately after placement of a restoration appears to be the result of trauma produced by restorative procedures, but usually this problem disappears after 1 week.[10,47]

The percentage of POS in the present study was higher to that compared to a nonrandomized clinical study run by Guggenberger and others.[49] However, that data was only published as an abstract, which prevents us from evaluating the methodology and the underlying risk of bias of the study. Several important technical details (the type of cavity restored, resin composite, rubber dam use, finishing, and polishing procedure, etc) and study features (randomization, allocation concealment, blinding, outcome measurement, management of missing data, publication of the study protocol, etc) are not available for evaluation. All these characteristics are likely responsible for the differences between the present results and the results reported in that abstract.

Usually, the POS measured by randomized and independent clinical trials [10,47,48] are higher than the percentage of POS measured by the studies conducted by manufacturers. The risk of POS sensitivity of bulk-fill composites, when associated to universal adhesives in randomized clinical trials, are quite variable in the literature. For instance, Tardem and others[47] and Yazici and others[50] showed lower rates of POS (2%-4%) than the present study. On the other side, the results of the present study are similar to Costa and others[10] and Afifi and others,[51] as they reported risk rates of 19% and 26%, respectively. Several methodological differences could explain these different results. Reis and others,[12] in a systematic review of POS in posterior restorations, observe a great variation among the way researchers assess the POS. This fact makes difficult the comparison between results of difference randomized clinical trials, and efforts need to be done to standardize the measurement of POS in posterior restorations.

It is worth mentioning that the enamel was also kept wet in the moist dentin group. In the past, dentists were taught to dry enamel vigorously after rinsing off the acid etchant in order to check for an adequately etched aspect of enamel.[36] This was not a concern when a universal adhesive was used, because, even with dry or moist enamel, some studies showed that there are not differences in terms of immediate and long-term bond strength as well as bond fatigue strength with the enamel.[27,28] Actually, no significant differences in terms of marginal discrepancies were observed at the 12-month follow-up when enamel or dentin margins were kept dry or moist, as well as other parameters, leading the authors to partially accept the second null hypothesis.

Furthermore, no significant difference was observed when dry and moist dentin were compared after 6 and 12 months of clinical evaluation, when other clinical parameters (fracture and recurrence of caries) were compared, leading the authors to partially accept the second null hypothesis. The present and previous studies indicate that the use of a bulk-fill resin composite could be considered an interesting alternative to restore posterior teeth. Unfortunately, the results of the present study are difficult to compare with the previous literature, because this is the first study to evaluate the effect of dentin moisture on the

clinical performance of a universal adhesive associated with a bulk-fill composite. However, some studies showed similar clinical performance when evaluating SBU applied in moist dentin in posterior restorations in comparison with the present ones.[47,48]

In general, the results of the present study in posterior resin composite restorations are similar to clinical studies of noncarious cervical lesions when SBU was evaluated.[29-31] In these studies, no significant clinical differences were observed when universal adhesive systems were applied in the etch-and-rinse mode in dry and moist dentin for 3 years after follow-up.[29-31] Despite all clinical differences, the results of the present study are in agreement with previous ones showing excellent clinical performances of SBU when posterior restorations, as performed in the present study, are compared with noncarious cervical restorations in terms of morphological and physiological differences.[34,50]

Regarding the characteristics of the cavities, it was possible to show that the risk of spontaneous POS was not correlated with the complexity of the restoration (class I or II and the number of restored surfaces), which was previously observed in several studies.[7,9,51] Although it is expected that more extensive cavities (cavities with more surfaces involved) showed more POS when compared with more simple cavities, there is not a consensus in the literature,[7,9,51] because a fewer number of restorations has been evaluated. Related to the cavity depth, the same controversial results were found.[47,52] Future systematic reviews of clinical studies in posterior restorations need to be evaluate the effect of these variables (number of restored surfaces and cavity depth) to confirm the hypothesis.

There are some limitations in the present clinical study. Only short-term (6- and 12-month) follow-up results were described. Future long-term clinical evaluation needs to be done to confirm the effect of dry or moist dentin on other clinical parameters (marginal adaptation, marginal discoloration, fracture, and recurrence of caries, among others). In this study, only a universal adhesive was evaluated. Unfortunately, as each universal adhesive contains a specific composition, these results could not be extrapolated for all universal adhesives, specifically those with less water in their compositions.[25,27,28] Similarly, some studies showed that, if an over-wet dentin was simulated, the adhesive performance of SBU was affected.[26,53] However, the authors believe that the results of the present study will encourage researchers to investigate the same concept for other universal adhesive systems, and then a body of evidence will be produced around the concept of wet/dry dentin bonding for universal adhesives. Therefore, to increase the external validity of the concept herein demonstrated for other adhesive systems, other randomized clinical trials are recommended.

CONCLUSIONS

The moisture of dentin did not influence POS or the clinical performance in posterior bulk-fill composite restorations when associated with an MDP-containing universal adhesive applied in etch-and-rinse mode.

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Regulatory Statement

The local Ethics Committee on Involving Human Subjects reviewed and approved the protocol and consent form for this study (State University of Ponta Grossa). The approval code issued for this study is 1.752.848. This was a randomized, double-blind clinical trial, registered in the Clinical Trials Registry (REBEC) under identification number RBR-83CD7J.

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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Influence of Manual and Ultrasonic Scaling on Surface Roughness of Four Different Base Materials Used to Elevate Proximal Dentin–Cementum Gingival Margins: An *In Vitro* Study

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Clinical Relevance

In terms of surface roughness, resin-based composite could be recommended for gingival margin elevation of subgingival proximal cavities rather than glass ionomer-based restorative materials. Whenever noninvasive periodontal treatment is required for such restored cavities, hand scaling may be preferable rather than the ultrasonic method.

SUMMARY

Aim: To evaluate and compare the effects of both manual and ultrasonic scaling on surface roughness of four different base materials, used for elevating dentin/cementum gingival margins of proximal cavities.

Methods and Materials: Eighty human upper molars with compound Class II mesial cavities, with gingival margins 1 mm below the cemento–enamel junction (CEJ), were divided into four different groups according to the type of the base material used; resin-modified glass ionomer (RMGI), glass hybrid (HV-GIC), flowable bulk-fill resin composite (Bulk Flow) and bioactive ionic resin (Activa). This was followed by completing the restorations with the same resin composite. All materials were used according to the manufacturers' instructions. All

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groups were further subdivided into two subgroups according to the scaling technique: manual (hand) or ultrasonic. All restorative and scaling procedures were performed after fixation of specimens with acrylic beside neighboring teeth to simulate natural contact. The mean surface roughness (Ra, μm) of all specimens was measured quantitatively and qualitatively by a three-dimensional (3D) surface analyzer system at two stages; (1) after thermal cycling for 5000 cycles without scaling and (2) after scaling. Data were statistically analyzed using analysis of variance (ANOVA), Tukey post hoc tests, and paired sample *t*-tests (at α=0.05).

Results: For baseline readings, the Bulk Flow group had the lowest R_a values, while HV-GIC group had the highest. RMGI and Activa groups had no statistical significant difference between their R_a values (p>0.05). For post scaling readings, hand scaling had significantly lower R_a values than ultrasonic scaling in all the material groups (p<0.05), except in the Bulk Flow group, where both scaling methods were not significantly different from each other (p>0.05)

Conclusion: Bulk Flow had the smoothest surfaces when cured against a matrix band compared with the other tested base materials. When hand and ultrasonic scaling methods were compared, the latter technique had more detrimental effect on the surface texture of the four tested base materials.

INTRODUCTION

Large posterior defects with proximal caries extending below the cemento–enamel junction (CEJ) and cavity margins located beneath the gingival tissues represent a very common clinical situation.[1] Beside the challenges encountered during restoration of such defects, deep cervical margins are critical areas that may cause gingival irritation and periodontal pockets,[1,2] especially if restorations are overhung or rough.[3]

The only structure that has a biological reaction after the invasion of the biological width is the connective tissue attachment, which is very selective about surfaces to be attached to; it needs cementum on one side and bone on the other. By contrast, epithelial attachment is not specific; it is capable of attaching to enamel, cementum, and restorative material, as long as the surface is hard, smooth, and clean.[4] *In vivo* studies showed a positive correlation between the surface roughness and the rate of supra and subgingival plaque accumulation that may lead to periodontal inflammation and an increased pathogenic bacterial colonization.[5,6] These previous findings highlighted the importance of smooth restorations placed below the gingiva.

The health of periodontal tissue should be maintained using the least invasive approaches.[7] Noninvasive periodontal treatment procedures, including scaling and root planning, are considered as the first line for management of the periodontal conditions.[8] Different methods of scaling and root planning can control the gingival inflammation and bleeding index.[9] In addition, a previous systematic review showed that subgingival mechanical debridement increased the mean attachment gain of gingival tissues.[10] These noninvasive periodontal procedures are of a particular importance in subgingival Class II and V cavities.[11] On the other hand, it was reported that all scaling methods have a negative effect on the surface smoothness of both the root and restorative materials.[12-14] Thus, although periodontal benefits are obtained after scaling procedures, there is still the risk of increasing the surface roughness after these procedures, affecting the long-term success of the treatment.[15]

Proximal cavities with cervical margins below the gingiva are usually restored using either an open-sandwich technique or cervical margin relocation concepts,[16,17] where direct restorations are used as a base for elevating the proximal cavity margin from an intracrevicular to a supragingival position and then completing the rest of the cavity with either direct or indirect options.[17] Different base restorative materials were investigated in the literature for gingival margin elevation in such situations, including different modifications of glass ionomers and resin composites.[18,19]

Deep proximal cervical restorative surfaces are inadvertently subjected to different scaling procedures. Thus, the influence of such procedures on surface roughness of these restorative surfaces should be of interest. There is ample data regarding the effect of different scaling techniques on soft tissues, root surfaces, and even on restorative materials placed in Class V cavities. However, there are insufficient studies reporting the effect of scaling techniques on deep proximal cervical restorative surfaces. Therefore, this study evaluated and compared the effect of both manual and ultrasonic scaling on surface roughness of four different base materials, three of them were glass ionomer-based and one was resin composite-based, used for elevating dentin–cementum gingival margins of the proximal cavities. The research hypotheses were: (1) there is no difference in surface roughness between different base materials; (2) there is no effect on roughness of the four base materials following either manual or ultrasonic scaling.

METHODS AND MATERIALS

Materials

Four commercially available restorative materials were tested in the current study. Resin-modified glass ionomer (Fuji II LC) (RMGI), glass hybrid (EQUIA Forte) (HV-GIC), flowable bulk-fill resin composite (Tetric N-Flow Bulk Fill) (Bulk flow), and bioactive ionic resin (ACTIVA BioACTIVE RESTORATIVE) (Activa). The detailed description of the materials is presented in Table 1.

Cavity Preparation

Eighty sound human upper molars recently extracted due to periodontal disease were included in this study; they had approximately similar dimensions, and were examined with stereomicroscopy to confirm that they were caries and crack free, then cleaned of soft tissue and calculus deposits with ultrasonic scaler, and stored in 0.1% thymol solution until used.

Compound Class II cavities with standardized dimensions were prepared on the mesial surfaces of all teeth using cylindrical, medium-grit diamond burs (K881 012, öko DENT, Germany) under copious water coolant with a high speed handpiece (W&H, RC-90RM, Austria). A pencil was used to mark the outline before preparation. The cavity dimensions were: occlusal: 3 mm buccolingual width, 3 mm depth; box: 1 mm below the CEJ, 1.5 mm mesiodistal dimension at the cervical floor, and 4 mm bucco-lingual width.[20] The margins were not beveled with slightly rounded line angels. A new bur was used after every five preparations. The dimensions were verified using a graduated periodontal probe.[20] After preparation, cavities were examined for any defects. Buccal and palatal walls of the proximal boxes of all teeth were marked with pencil 1.5 mm above the CEJ (to mark the level of the base material) (Figure 1A,B).

Tooth Fixation

Following cavity preparation, each tooth was fixed with mesial and distal acrylic neighboring teeth (Banna, Alexandria, Egypt) using condensation silicone impression material (Silaxil putty, Lascod, Italy). The teeth were fixed in a way to simulate natural contact and correct occluso-gingival level of the three teeth.

Table 1: *Materials Used in the Study*

Base Material	Type	Manufacturer	Composition	Filler Particle Size	Lot Number
Fuji II LC	Resin-modified glass ionomer	GC Corporation, Tokyo, Japan	Powder: 95% strontium fluoroalumino silicate glass Liquid: polyacrylic acid (20%-25%), 2-hydroxyl ethyl methacrylate bicarbonate (1%-5%), proprietary ingredient (5%-15%)	4.5 μm	1904231
EQUIA Forte	Conventional highly viscous glass ionomer	GC Corporation, Tokyo, Japan	Powder: 95% strontium fluoroalumino-silicate glass (including highly reactive small particles), polyacrylic acid powder Liquid: 5% polyacrylic acid, polycarboxylic acid, tartaric acid	25 μm + 4 μm	1808163
Tetric N flow bulk fill	Flowable bulk-fill resin composite	Ivoclar Vivadent, NY, USA	Bis-GMA, UDMA, TEGDMA, Ivocerin, Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide (filler loading: 68.2 wt%)	5 μm	Y35353
Activa Bioactive Restorative	Bioactive resin matrix and bioactive glass fillers	Pulpdent; Watertown, MA, USA	Powder: diurethane dimethacrylate, bis (2-(methacryloyloxy) ethyl) Phosphate, barium glass, ionomer glass, sodium fluoride, colorants Liquid: polyacrylic acid/maleic acid copolymer (filler loading: 56 wt%)	4 μm to submicron	190619

Abbreviations: Bis-GMA, Bisphenol A-glycidyl methacrylate; UDMA, Urethane dimethacrylate; TEGDMA, Triethylene glycol dimethacrylate.

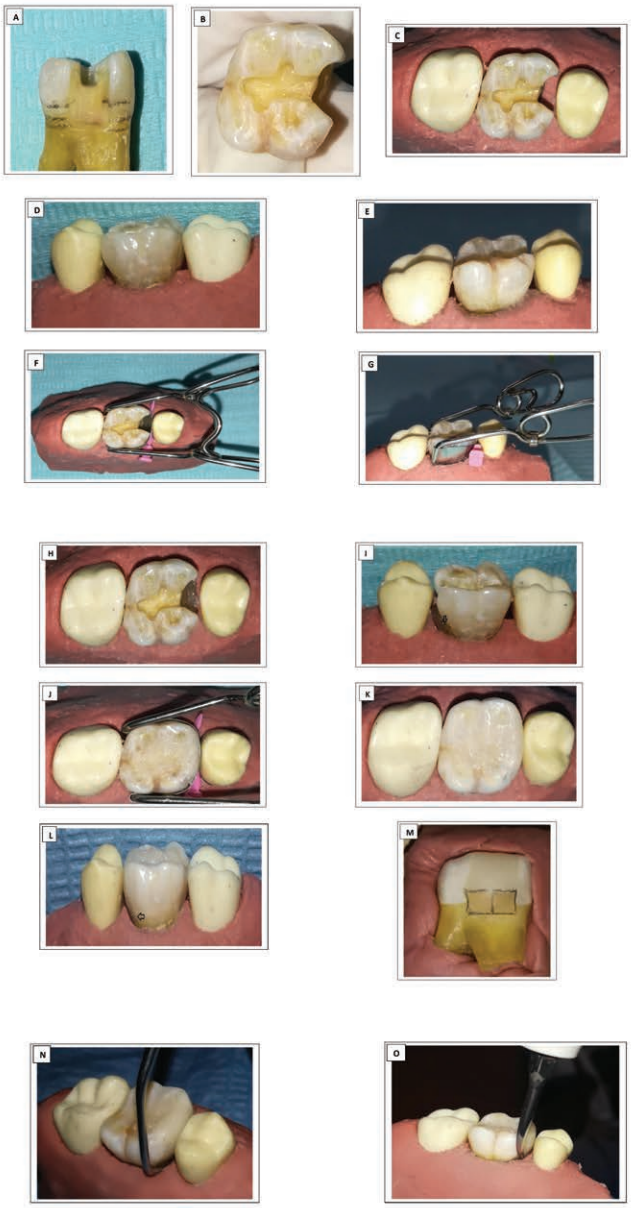

Figure 1. *Methodology steps for a representative specimen (Bulk Flow). A: Proximal cavity part outline and dimensions, the three lines mark 1.5 mm above CEJ, CEJ, 1 mm below CEJ. B: Occlusal cavity part outline and dimensions. C: Occlusal view for fixing the teeth in silicone impression material (putty consistency). D,E: Buccal and palatal views for fixation. F,G: Occlusal and buccal views for the Saddle matrix band and diamond wedge in place. H,I: Occlusal and buccal views after base placement, black arrow indicates the base material level (just below the contact). J,K: Occlusal views for the final restoration before and after finishing and polishing. L: Buccal view for the final restoration (black arrow indicates the level of base material/ junction between base material and overlying composite). M: Specimen in the 2nd silicone block for roughness evaluation, the margins of the base material were marked and divided into two halves—buccal and palatal. N: Gracey curette (no. 11/12) in place. O: Ultrasonic tip was placed at zero angulation while the end of the tip 1 mm below gingival simulation.*

The impression material was shaped using a dental wax carver (Lecron wax carver, Accurate Manufacturing, Pakistan) to simulate the correct contour and level of the gingiva around the teeth, especially parts simulating buccal and palatal gingival papilla. The impression material was kept at the level of buccal and palatal CEJ of the three teeth, then was left to set for 5 minutes before trimming of the excess using number #11 surgical blades (Tianda Medical Instruments Co, Huaian, China) (Figure 1C,D,E). Teeth were randomly assigned into four different groups, 20 molars each, according to the base material used. Each group's teeth were numbered from 1 to 20, with a specific color for each material group on the distal surface.

Restorative Procedures

After preparation and fixation procedures, cavities were washed with water and dried. For RMGI and HV-GIC groups, the gingival margins of the cavities were conditioned as recommended by the manufacturer with dentin conditioner (GC Co, Tokyo, Japan) for 20 seconds, followed by rinsing and drying. Occlusal and proximal enamel margins of all the cavities were selectively etched with 37% phosphoric acid (N-Etch, Ivoclar Vivadent, NY, USA) for 15 seconds, rinsed with water for the same time, excess water was blotted without desiccation. For Bulk Flow and Activa groups, a universal adhesive (Tetric N-Bond Universal, Ivoclar Vivadent, NY, USA) was applied before base placement on all cavity surfaces, air dried and light cured as recommended by the manufacturers' instructions with a LED curing light (Elipar Deep Cure, 3M Oral Care, St Paul, MN, USA) operating at 1000 mW/cm^2, and checked periodically after every 5 samples with a radiometer (Radiometer 100, Demetron Research Corp, Danbury, CT, USA).

Large-sized saddle contoured metal matrix bands with an enlarged subgingival ledge (N 1.313-0.035 mm, Tor VM, Moscow, Russia) with Small Spring clip (N 1.003 Tor VM, Moscow, Russia) were applied around each cavity while making sure that the end of the band was beyond the gingival margin of the cavity. Then, a suitable-sized plastic wedge (Diamond Wedges, Bioclear, Tacoma, WA, USA) was applied in the gingival embrasure between each tooth and neighboring premolar, ensuring intimate adaptation between the gingival margin of the cavity and internal surface of the band (Figure 1F,G). After that, each group was restored up to 1.5 mm above the CEJ using the group specific base material in a bulk technique (Figure 1H,I). All base materials were mixed, dispensed, and cured (RMGI, Activa and Bulk Flow groups) according to the manufacturers' instructions. For RMGI and HV-

GIC groups, the universal adhesive was applied after base placement with the same technique mentioned earlier. The remaining cavity was restored with a nanohybrid resin composite material (Tetric N-Ceram, Ivoclar Vivadent) that was inserted in the cavity in 2-mm horizontal increments using a plastic instrument until the cavity was completely filled.[21] Each increment was cured from the occlusal surface for 20 seconds. Additional curing for 40 seconds was performed from the proximal surface after removal of the wedge and matrix band (Figure 1J).

All specimens were stored in distilled water at 37°C for 24 hours in an incubator (BTC, Model: BT1020, Egypt) prior to the finishing and polishing procedures.[22] Finishing and polishing of the occlusal surfaces was performed with Al_2O_3 discs (Tor VM, Moscow, Russia) using a low-speed handpiece (Strong 204, Daegu, South Korea) under water cooling; proximal surfaces were kept without finishing and polishing to simulate clinical situations (Figure 1K,L). All procedures were performed by a single operator using magnification (4× loupes, Amtech, Wenzhou, China).

Thermocycling

After restoration, each specimen was removed from the rubber base block. All specimens (n=80) were thermocycled for a total number of 5000 cycles (SD Mechatronik thermocycler, Germany), which represents approximately 6 months of clinical service before scaling.[23] The specimens were alternated between 5°C and 55°C ± 2°C, according to ISO 11405 (International Standards Organization) recommendations, continuously checking for water temperature, with a dwell time of 15 seconds and a transfer time of 5 seconds.[24] Afterwards, all the specimens were carefully evaluated under an optical microscope to check for cracks. Finally, each specimen was fixed in a second rubber base block where the mesial surface to be evaluated was facing upwards to facilitate roughness evaluation (Figure 1M).

Pre-instrumentation Roughness Reading

The area of the base material (2.5 mm × 4 mm) was marked with pencil, and then divided into buccal and palatal haves. Each half was assessed quantitatively and qualitatively for surface roughness using a 3D surface analyzer system. Each half was photographed using a USB digital microscope with a built-in camera (U500× Capture Digital Microscope, Guangdong, China) connected with an IBM compatible personal computer using a fixed magnification of 120× and a resolution of 1280×1024 pixels per image. The digital microscope images were cropped to 350×400 pixels using Microsoft Office picture manager to specify and standardize the area of roughness measurement. The cropped images were analyzed using WSxM software (Ver 5 develop 4.1, Nanotec, Electronica, SL, Spain) where a 3D image of the surface profile was created.[25,26] Three 3D images with an area of 10 μm × 10 μm were collected at different sites for each half; then the average of the three readings were recorded as the surface roughness (R_a, μm) value for either buccal side or palatal side to serve as the prescaling baseline controls.[12]

Scaling

After initial roughness evaluation, each group was further subdivided into 2 subgroups (n=10) according to the scaling technique; (1) hand scaling subgroup, or (2) ultrasonic scaling subgroup, and returned back to the rubber base mold with the same proximal and occlusal relation with the two neighboring teeth used for restoration.

For hand scaling subgroups, a Gracey curette (no 11/12; Goldman Products Inc, Wauconda, IL, USA) was used. Each buccal/palatal side received 10 apical to coronal consecutive strokes parallel to the long axis of the tooth using medium force,[12,27] each side of the curette was used with either buccal or lingual side scaling, and when the interproximal side was changed, the side of the curette was replaced. Curettes were sharpened using a ceramic sharpening stone (SST-C3, Osung, Seoul, South Korea) after every tooth. A sharp, new curette was used for each group (Figure 1N).

For ultrasonic scaling subgroups, a piezoelectric ultrasonic scaling device (Intelligence PS-25, Rolence Enterprise Inc., Taoyuan, Taiwan) with one type of subgingival fine-diameter ultrasonic tip was used (P1, Woodpecker, Guangxi, China).[28] The machine was operated according to the manufacturer's instructions under profuse water irrigation for cooling of the scaler tip at medium power settings, standard lateral force, and a frequency of 29 kHz for all specimens.[13,14] The side of the scaler tips were placed in the mesial interproximal areas from both buccal and palatal sides (between each specimen and premolar) 1-mm below the gingival simulation with zero angulation in relation to the base material.[11,29] Each side received 10 apical to coronal consecutive strokes.[29] A new scaler tip was used for each group (Figure 1O).

One experienced periodontist instrumented all the specimens who was blind to the restoration step. Following instrumentation, all the specimens were removed from the rubber base mold, thoroughly rinsed with water for 10 seconds, and then cleaned in an ultrasonic cleaner for 3 minutes. Finally, specimens

were placed back in the second rubber base block for roughness evaluation.

Postinstrumentation Roughness Evaluations

The same areas of base materials were evaluated for the second time, as previously described in the preinstrumentation roughness evaluation.

Statistical Analysis

Sample Size Calculation—The sample size for this study was calculated before conducting any work using G*Power program (G*Power Ver. 3.0.10, Kiel, Germany).[13] The total sample size of 64 teeth achieved 80% power (equal to type II error); type I error (α) was 0.05. Due to the new methodology proposed by our study, two more teeth were included in each subgroup to have a total sample size of 80 teeth.

Statistical Methods—The R_a values for buccal and palatal sides for both control and scaling subgroups of all base material groups were compared using independent sample *t*-tests; when there was no significant difference, the R_a value of each tooth with each scaling technique tested was calculated by obtaining the mean R_a value of the six readings combined, three from the buccal side and three from the palatal. All data were statistically analyzed using SPSS (SPSS version 20, IBM, Chicago, IL, USA). R_a values proved to be normally distributed after they were subjected to the Shapiro–Wilk test, and the homogeneity of variances was tested using Levene's test; so parametric tests were used to compare the study groups. One-way analysis of variance (ANOVA) was used to compare the control groups of the four materials; when significant differences were detected, a pairwise comparison was performed using Tukey post hoc tests (at α=0.05). The effect of scaling technique per each material group was evaluated using paired sample *t*-tests. Two-way ANOVA was used to determine the effect of study variables (base material type and scaling technique), and their interaction on surface roughness followed by Tukey post hoc test (at α=0.05).

RESULTS

One-way ANOVA revealed that baseline R_a readings for all the groups were statistically significant (p<0.05). The mean R_a values and standard deviations for baseline readings are presented in Table 2. Pairwise comparisons revealed that Bulk Flow had the lowest R_a values followed by RMGI and Activa, respectively; the latter two had no statistically significant difference between their R_a values. The highest R_a values were shown by HV-GIC.

Table 2: *Mean ± Standard Deviation (μm) of Ra Values of Baseline Readings of All Base Materials Evaluated*[a]

Base Material	Control
RMGI	0.162 ± 0.005 b
HV-GIC	0.194 ± 0.010 c
Bulk Flow	0.131 ± 0.010 a
Activa	0.165 ± 0.005 b

Abbreviations: RMGI, Resin modified glass ionomer; HV-GIC; Highly viscous glass ionomer cement; Bulk Flow, Flowable bulk fill resin composite; Activa, ACTIVA BioACTIVE Restorative.

[a]*Groups identified with the same lowercase letters are not significantly different (Tukey HSD;* p*<0.05).*

Paired sample *t*-test results (Table 3) revealed that, regardless of the base material used, both scaling methods adversely affected the surface smoothness in a significant way (p<0.05).

Two-way ANOVA showed that both study variables significantly affected the R_a values (p<0.05); the interaction between them were also significant (p<0.05). The mean R_a values and standard deviations for subgroups of all the base materials are presented in Table 4. Tukey honestly significant difference (HSD) multiple comparisons revealed that manual scaling had significantly lower R_a values than ultrasonic scaling in all groups (p<0.05), except in the bulk flow group, where the scaling methods were not significantly different (p>0.05). RMGI, Bulk Flow, and Activa hand scaling subgroups had the lowest R_a values; on the other hand, HV-GIC ultrasonic subgroup had the roughest surfaces. Representative 3D and histogram images for the four base materials are shown in Figure 2.

DISCUSSION

Previous clinical and histological studies have linked the presence of subgingival cervical margins and the increase in bacterial plaque, gingival indices, and probing depth.[1,11] Smooth subgingival restorations are required to prevent jeopardizing the periodontal health in these critical areas.[30] Therefore, this study evaluated and compared the surface roughness of different restorative materials placed below the CEJ in clinically simulated subgingival restorations, in order to determine the smoothest base material to be used in restoring such defects.

The main objective of prevention and/or treatment of periodontitis includes periodic removal of plaque and calcified deposits from the teeth and restorations.[8] This procedure is usually accomplished by different scaling techniques that may accidently not only affect the dental tissues but also the restorative surfaces creating

Table 3: *Results of Comparing R_a Values of each Base Material Evaluated Before and After Each Scaling Method*

	Paired Differences					*t*	*df*	Sig. (2-Tailed)
	Mean	Std Deviation	Std Error Mean Lower	95% Confidence Interval of the Difference				
				Lower	Upper			
RMGI control 1 – RMGI hand	0.016	0.007	0.002	0.022	0.011	6.926	9	$p<0.001$
RMGI control 2 – RMGI ultrasonic	0.049	0.011	0.003	0.057	0.041	14.215	9	$p<0.001$
HV-GIC control 1 – HV-GIC hand	0.033	0.009	0.002	0.039	0.026	11.587	9	$p<0.001$
HV-GIC control 2 – HV-GIC ultrasonic	0.058	0.007	0.002	0.063	0.053	26.371	9	$p<0.001$
Bulk Flow control 1 – Bulk Flow hand	0.056	0.008	0.002	0.062	0.049	20.452	9	$p<0.001$
Bulk Flow control 2 – Bulk Flow ultrasonic	0.041	0.008	0.001	0.048	0.035	15.233	9	$p<0.001$
Activa control 1 – Activa hand	0.017	0.005	0.001	0.021	0.013	9.173	9	$p<0.001$
Activa control 2 – Activa ultrasonic	0.028	0.008	0.002	0.034	0.023	11.271	9	$p<0.001$

Abbreviations: RMGI, Resin-modified glass ionomer; HV-GIC; Highly viscous glass ionomer cement; Bulk Flow, Flowable bulk-fill resin composite; Activa, Activa Bioactive Restorative.

roughness that may lead to unfavorable periodontal consequences.[11] Thus, this study also evaluated the effect of scaling techniques on roughness of restorations placed below the CEJ, to determine the most suitable scaling technique in proximal subgingival restored areas.

The selection of the four tested base materials was based on both open-sandwich technique and cervical margin relocation concepts.[16,17] The open-sandwich technique includes using conventional glass ionomer cement (GIC) for elevation of the gingival margin. High clinical failure rates have been reported with this material.[31] Thus, modifications using RMGI and HV-GIC have been introduced to be used with this technique with acceptable long-term outcomes.[18,32] Recent studies argue that glass ionomer with its hydrophilic nature, flexibility, and chemical bonding could be a more suitable option for bonding to deep, moist dentin–cementum margins.[33,34] That is why RMGI and HV-GIC were included in this study as base materials.

On the other hand, cervical margin relocation includes using a flowable resin composite base to lift the proximal gingival margin. Kielbassa and others[31] reported how promising this technique is in their systematic review. Bulk-fill resin composites have been developed with different chemical compositions to reduce polymerization shrinkage stress. In addition, they can be placed in layers up to 4 mm in thickness and cured in one single step.[19] Thus, they can be quickly applied and save chair time, especially when used for deep and large cavities.[35] Previous studies found higher bond strength for flowable bulk fill compared

Table 4: *Mean ± Standard Deviation of R_a Values (μm) of All Base Materials Evaluated with Different Scaling Methods*[a]

Scaling Method / Material	Hand	Ultrasonic
RMGI	0.177 ± 0.006 a	0.214 ± 0.007 c
HV-GIC	0.227 ± 0.007 d	0.253 ± 0.007 e
Bulk Flow	0.179 ± 0.004 a	0.180 ± 0.002 a
Activa	0.184 ± 0.006 a	0.194 ± 0.005 b

Abbreviations: RMGI, Resin-modified glass ionomer; HV-GIC; Highly viscous glass ionomer cement; Bulk Flow, Flowable bulk-fill resin composite; Activa, Activa Bioactive Restorative.

[a] *Groups identified with the same lowercase letters are not significantly different (Tukey HSD; $p<0.05$).*

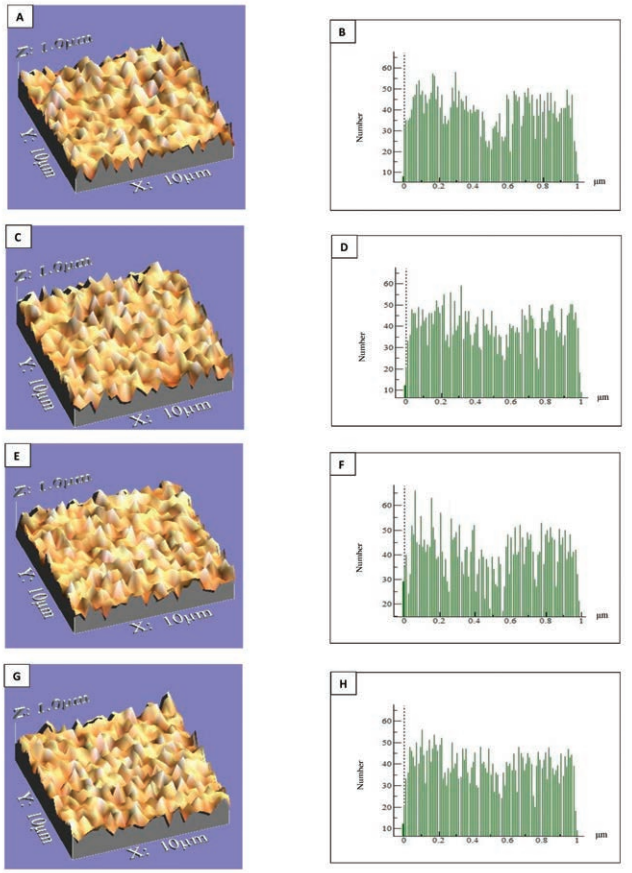

Figure 2: *Representative 3D and histogram images for the four base materials. For 3D images, spikes at the borders are artifacts. For histogram images, the Y axis represents the number of repetitions for each reading (µm), the Ra value was recorded as the mean of the five readings with the highest repetitions. A,B: Representative 3D and histogram images for hand scaling subgroup in RMGI group. C,D: Ultrasonic in HV-GIC. E,F: Control in Bulk Flow. G,H: Hand in Activa.*

to nanofilled layered composites when bonded to proximal dentin–cementum margins;[20,36] moreover, others reported promising marginal adaptation of flowable bulk fill with such margins.[37] That is why a flowable bulk-fill material was tested in this study.

Bioactive restorative materials are a relatively new category that react to pH changes in the mouth by providing calcium, phosphate, and fluoride ions to maintain the chemical integrity of the tooth structure. ACTIVA BioACTIVE restorative is one in this category.[38] Benetti and others[39] stated that this material has a promising mechanical behavior, as it showed comparable flexural strength and fracture toughness to flowable and bulk-fill resin composites.[40,41] On the other hand, the material's compromised hardness suggests the importance of using an abrasive-resistant resin composite as coverage.[42] That is why it was used in this study as a base material.

This study included two different scaling methods: hand and ultrasonic. Scaling and root planning by curettes (hand) are the most frequently used procedures for removing of subgingival calculus and treatment of periodontal diseases, due to their low cost and effectiveness in reducing the clinical signs of inflammation and the levels of pathogens.[43] Despite different developments in scaling techniques, hand instrumentation is still considered the gold standard.[44] Ultrasonic scaling is considered to be less strenuous for the operator and more comfortable for patients than hand scaling, and can be performed in considerably less time.[44] Thus, it has become increasingly popular for subgingival debridement.[28]

Upper molars were used in this study, as their buccal surface is the second most predominant site for supragingival calculus deposition after the lingual surface of the six lower anterior teeth.[45] Supragingival calculus creates an environment more conducive to subgingival plaque accumulation,[46] thus, a significant association between supra- and subgingival calculus was found,[46] indicating the importance of subgingival scaling and root planning in upper molars area.

After cavity preparation, the teeth were fixed with neighboring teeth using condensation silicone impression material (putty consistency). This material was selected based on several fixation trials with other materials like wax, stone, and impression compound. These trials failed due to both distortion and brittleness that prevented good fixation of teeth or due to excess stiffness that prevented placement of matrix bands and wedges for restoration.

Contoured sectional bands and wedges were used during restoration to create correct clinically simulated contact areas, gingival embrasures, and proximal surface contour.[47] The cavities were filled with base materials up to 1.5 mm above the CEJ that nearly corresponded to the level just below the proximal contact, which was then built with overlying resin composite with the rest of the cavity. Interproximal wear usually affects the proximal contact tightness, and consequently the contact should be built with hard and strong restorative materials,[48] like the overlying resin composite used in this study. Five thousand cycles of thermocycling was performed to simulate 6-months of clinical service before scaling procedures.[23] This was chosen based on the frequent 6-month scaling and prophylactic polishing interval performed by most of the dentists.[49]

In this study, evaluation of surface roughness was performed both quantitatively and qualitatively in a nondestructive method, to allow detailed visualization for the surface without contact.[50] This would accurately

allow further recording of roughness values for the same specimens after scaling.

All scaling procedures were carried out by one periodontist to eliminate interoperator variability and minimize variations in stroke length, force, and pressure applied during instrumentation. A piezoelectric ultrasonic unit was chosen in this study, as its oscillation pattern produces movement that is primarily linear in direction, in contrast to magnetostrictive devices with their circular motion.[28] This linear motion provides more efficient calculus removal with less damage to the surface being scaled.[51] A number of *in vitro* studies demonstrated that working parameters such as power settings and tip angulation can determine the amount of damage for the root or restoration surface.[28,51] The device was operated at medium power settings as a previous review mentioned that increasing the power settings from medium to high can lead to high surface roughness and alterations.[52] Narrow probe-shaped tips were used in this study, as it was reported that they are less aggressive to root dentin than wide probe-shaped tips.[28] In piezoelectric units, tip angulation has the most important effect on surface alteration depth of the scaled surfaces.[52] The least surface damage was obtained when the tip was used at zero angulation,[52] as was used in the current study.

Although there was no significant difference between R_a values for buccal and palatal sides for either control or subgroups of all material groups, it is worth mentioning that roughness values from the palatal side were higher than the buccal side in most of the specimens. This could be explained by the wider gingival embrasure on the palatal side than buccal embrasure,[53] making either curette or ultrasonic tip touch a wider surface palatally, thus creating more roughness.

The results of this study showed that R_a baseline readings differed among the four base materials; Bulk Flow had the smoothest surfaces, while HV-GIC had the roughest, therefore, the first null hypothesis was rejected. The literature has already shown that curing of resin-based materials against a matrix band can produce a relatively smooth surface compared with any finishing and polishing procedures.[54,55] This smooth surface is related to the resin-rich layer that is accumulated against the band.[55] A previous study explained that the compression applied through the matrix band on the surface of the resin-based materials can probably cause the filler particles to slide in the organic matrix, so that smaller particles, with lower density, appeared more and closer to the top in relation to the larger ones.[56] This may indicate that filler particle shape and size can have a minor role in affecting the surface roughness when curing the resin-based materials against the matrix band. HV-GIC has no resin methacrylate content; its composition is mainly formed of 90%-95% strontium fluoroalumino silicate glass (FAS) (25 μm),[57] which mostly protruded on the surface when the material was left to set against the matrix band, thus having the highest roughness values among the base materials. The amount of resin in the RMGI used in this study was 1%-5% [2-hydroxyethyl methacrylate (HEMA)], most of its composition also contains FAS;[57] this heterogeneous and biphasic chemical composition can explain why it has higher roughness values than Bulk Flow.

Activa contains 42 wt% organic resin, while Bulk Flow contains 28 wt%. The organic resin of Activa is called an "ionic resin," which has a more hydrophilic nature compared to Bulk Flow and contains a small amount of water. This aqueous ionic resin may cause the material to be more susceptible to matrix degradation after thermal cycling, as was previously reported.[58] Yilmaz and others[59] showed that following thermal cycling, the hydrophilicity of the material allowed water to penetrate more easily, leading to matrix degradation, exposing the underlying filler particles and increasing its roughness.

The results of the current study showed that regardless of the scaling technique used, the roughness values increased significantly compared with baseline readings, which means the second null hypothesis was rejected. This is in accordance with previous studies that examined the scaled surfaces under scanning electron microscope (SEM) and found valleys, cracking at the filler–matrix interface, filler dislodgment, and even removal of a whole layer of the root surface or restoration,[11,29,60] and reported that this surface alteration is an unavoidable complication of the scaling procedures.

The hand scaling technique had smoother surfaces than the ultrasonic technique for all the base materials in this study, except for Bulk Flow where both the scaling methods created comparable roughness values. This could be explained by the finding of a previous study showing that hand scaling instruments usually made greater contact area with the surface than ultrasonic tips;[29] a greater contact area could result in masking the roughness created by the instrument resulting in smooth surface. Mishra and others[61] noticed that hand scaling resulted in more surface flattening than ultrasonic scaling, and attributed the smooth surface they found after hand scaling to this surface loss. Moreover, hand scaling was reported to facilitate better tactile proprioception and controlled movement to the operator, resulting in a smoother surface.[62] A possible explanation for the increased R_a values for ultrasonic subgroups is the vibration effect of the ultrasonic

scaling that could potentially create more cracks and greater filler dislodgement.[13] Conversely to the current results, previous studies found that ultrasonic scaling produced smoother surfaces when compared to hand scaling.[63,64] The difference in results could be attributed to different experimental designs, including surfaces to be scaled, in the current study restorative materials and in the former studies root dentin, methods of roughness evaluation, in addition to different characteristics of the instruments like shape, size and material of the tips used, and different force and pressure of application due to operator variability.

Although the tested HV-GIC used in this study was reported to have promising wear resistance and microhardness results compared with other types of glass ionomers,[65,66] its roughness values increased significantly after both scaling techniques, especially after ultrasonic scaling. During scaling procedures, the weak polysalt matrix phases in HV-GIC, which usually press against the matrix band, were easily removed while the harder unreacted FAS glass particles protruded from the surface;[60] this may explain why this material had the highest R_a values among the other scaled base materials. In addition, Buldur and others[65] observed deep cracks, pits, and fissures after aging on the same material surface under SEM, even if the surfaces were varnished; so maybe ultrasonic scaling increased these already existing cracks and resulted in the highest roughness values.

For resin-based materials, the resin-rich layer that forms the smooth surface resulting from adaptation of the material against the matrix band during restoration is usually removed after any surface alteration,[67] like scaling procedures in this study. Consequently, inorganic fillers have an effect on surface roughness. The surface roughness values increased with the increase of the filler particle size.[11,13,14] RMGI, Activa, and Bulk Flow have nearly the same filler particle size (5 μm); this may explain the comparable R_a results after hand scaling. On the other hand, after ultrasonic scaling, both RMGI and Activa R_a values differed significantly. This may be explained by the results of previous studies showing significantly higher wear resistance with Activa compared to the RMGI used in this study;[66,68] the authors partially attributed the cause to the resilient resin matrix with energy-absorbing elastomeric components of Activa.[68] This higher wear resistance may lead to less degradation by ultrasonic scaling and subsequent lower inorganic filler exposure and roughness values.[11] Another possible reason was suggested by Garoushi and others,[68] as they found that the RMGI used in this study had a greater initial burst of fluoride release than Activa. Leaching of ions from filler particles of regular fluoride-releasing materials was attributed previously to filler–matrix debonding, because of a weakened filler. This leads to microcracks and higher degradation of the material[69]; these cracks may be aggravated by ultrasonic scaling.

Bulk Flow was the only material that showed no significant difference between scaling methods. The smooth surfaces of this material could be related to filler shape. The Bulk Flow used in this study has homogenous, rounded-shaped fillers compared with the irregular, heterogeneous-shaped fillers in other base materials. Marghalani and others[67] concluded that spherical-shaped fillers may allow more flow and stress relaxation of the material resulting in smooth surface compared to irregular ones that may develop stress concentration around them. Another possible reason is the form in which Bulk Flow is supplied. Bulk Flow is a single component material, whereas in the case of glass ionomers and Activa, powder has to be mixed with liquid or two pastes have to be mixed, respectively, therefore risking more air bubble incorporation and increased porosity.[66] These porosities may get enhanced after ultrasonic instrumentation leading to greater surface roughness. The Bulk Flow used in this study is not like conventional flowable composite with their lower filler amount; instead, the filler content reaches 68.2 wt%, leading to high wear resistance and less susceptibility to degradation by ultrasonic devices.[11]

The literature suggests that the critical threshold of roughness for patients was nearly 0.3 μm, and the threshold for biofilm accumulation was 0.2 μm[70]; so it could be inferred that, in the current study, none of the materials' roughness with either scaling technique would be perceptible by the patient, and that HV-GIC with both scaling techniques and RMGI when ultrasonically scaled would be at a risk for biofilm accumulation. Considering that their R_a values were just above the biofilm threshold, it is worth mentioning that Quirynen and others[71] found that variations around biofilm threshold had only a negligible impact on bacterial adhesion. In addition, a recent study showed that surface roughness had a minor role in the retention of a fully grown biofilm.[72]

It should be recognized that the present *in vitro* study has limitations; the scaling techniques were performed by one operator; consequently, the manual pressure exerted, even after specific training, cannot be considered replicable or standardized. In addition, only two scaling techniques were assessed among different other promising scaling methods. The methodology performed in the current study is new and intended to simulate the clinical situation, so further *in vitro* studies, including microbiological adhesion assessment and

even histological evaluation for the attachment of gingival epithelial cells to the tested base materials' surfaces, are needed. In addition, clinical studies are required to assess periodontal healing in critical dental areas, like below the proximal CEJ, after different scaling methods for debridement of microbial deposits on restorative materials placed in such areas.

CONCLUSIONS

Within the limitations of the present study, the following may be concluded:

1. In terms of surface roughness, the evaluated resin-based composite may be recommended to restore subgingival proximal margins rather than the tested glass ionomer-based restorative materials, especially when curing and setting of these base materials are done against a matrix band.
2. Both hand and ultrasonic scaling methods had a negative effect on the surface quality of the four tested base materials, so they should be performed with caution when used on restored subgingival proximal areas.
3. Considering changes in surface texture following the use of each scaling technique, the present study showed that hand scaling may be preferable to ultrasonic scaling for the tested base materials, especially for glass ionomer-based restorative materials.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the human subjects' oversight committee guidelines and policies of Faculty of Dentistry, Mansoura University Ethics Committee. The approval code issued for this study is A05121120.

Conflicts 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. The authors alone are responsible for the content and writing of this paper. Dr. Garcia-Godoy is a consultant for Pulpdent, manufacturer of Activa. This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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Volume 47 / Number 2
March/April 2022

www.jopdent.org
123-238

Clinical Technique/Case Report

123 A Clinical Presentation of the Direct Gold/Composite Sandwich Restoration
DB Henry

131 Three-year Follow-up of Conservative Direct Composite Veneers on Eroded Teeth
RQ Ramos • NF Coelho • GC Lopes

Clinical Research

138 Longevity of Direct Resin Composite Restorations in Maxillary Anterior Crown Fractures: A 4-year Clinical Evaluation
B Korkut • M Özcan

Literature Review

147 Color Change of Resin-based Composites After ***In Vitro*** Bleaching Protocols: A Systematic Review and Meta-analysis
ML Vidal • OE Pecho • K Collares • SBN Brandeburski • A Della Bona

Laboratory Research

163 Effect of the Sample Preparation and Light-curing Unit on the Microhardness and Degree of Conversion of Bulk-fill Resin-based Composite Restorations
SSL Braga • ACT Schettini • ELO Carvalho • CAK Shimokawa • RB Price • CJ Soares

173 Push-out Bond Strength of Two Fiber Posts in Composite Resin Using Different Types of Silanization
RM Novis • BLT Leon • FMG França • CP Turssi • RT Basting • FLB Amaral

183 Three-dimensional Quantification of Enamel Preservation in Tooth Preparation for Porcelain Laminate Veneers: A Fully Digital Workflow ***In Vitro*** Study
J Gao • L Jia • X Tan • H Yu

190 The Influence of Cement Removal Techniques on ***In Situ*** Bacterial Adhesion and Biodegradation at the Marginal Interface of Ceramic Laminates
OFF de Brito • JM Sandes • FCG de Lima • JB Oliveira Júnior • LC Alves • FA Brayner • GQ de Melo Monteiro

202 Surface Treatment and Cementation of Lithium Silicate Ceramics Containing ZrO_2
JD Martins • DMD Moura • CM Lima • RLA de Carvalho • FPP Leite • ROA Souza

214 Effects of Charcoal Toothpaste on the Surface Roughness, Color Stability, and Marginal Staining of Resin Composites
GF Bragança • PB Ferreira Soares • J Simeão Borges AB Fernandes Vilela • PC Santos • Filho CJ Soares

225 Masking Ability of the Combined Application of Opaquers and Resin Composite on Discolored Backgrounds
R Maronesi Lehr • B Gabardo Perez • B Gaidarji • A Dalmolin • L Brandão Durand

Departments

236 Errata

238 Online Only Articles

Online Only Articles

E52 Carious and Eroded Substrates and the Bonding of Adhesive Systems: A Systematic Review and Meta-analysis
TT Fröhlich • BD Ilha • FZM Soares • RO Rocha

E81 Evaluation of Cleaning Methods on Lithium Disilicate Glass Ceramic Surfaces After Organic Contamination
J Fagan–Junior • J Vesselovcz–Junior • J Puppin–Rontani • L Correr–Sobrinho • KMS Freitas • TC Robertson • RR Pacheco • NIP Pini • D Sundfeld

E91 Effect of Dentin Moisture in Posterior Restorations Performed with Universal Adhesive: A Randomized Clinical Trial
AS Castro • BM Maran • MF Gutierrez • K Chemin • ML Mendez–Bauer • JP Bermúdez • A Reis • AD Loguercio

E106 Influence of Manual and Ultrasonic Scaling on Surface Roughness of Four Different Base Materials Used to Elevate Proximal Dentin–Cementum Gingival Margins: An ***In Vitro*** Study
HS Ismail • AI Ali • F Garcia–Godoy