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CLINICAL TECH/ CASE REPORT

CLINICAL RESEARCH

LABORATORY RESEARCH

ONLINE ONLY

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Clinical Technique/Case Report

The Vented Crown: A Pictorial Case Report

B Small

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Clinical Relevance

This case report will describe and show in detail all the clinical and laboratory steps involved with the vented cast gold crown utilizing an external vent or escape channel.

SUMMARY

The venting of crowns has been shown to be one of the important factors in achieving the optimal marginal fit that can lead to improved longevity. This case report describes the technique and demonstrates the preparation, laboratory work, seating, and finishing of the vented cast gold crown. This technique can also be used on porcelain fused to metal crowns.

The venting of crowns has been shown to be one of the factors that can improve the fit and reduce the marginal gap of both cast and porcelain crowns.[1–4] The venting can be accomplished by utilizing an internal[5,6] or external[7] vent, also called escape channels. Tjan[8] compared the two venting methods and stated that "an internal escape channel, die spacing, or occlusal venting substantially enhances the complete seating of full cast crowns." Wilson and others[9] have described other factors that can affect the complete seating of full crowns, such as the viscosity of the cement used, passivity of fit, and die spacing. The following pictorial case report will show the details of the isolation, preparation, impression, laboratory work, seating, cementing, and finishing of a full cast gold crown utilizing the external venting technique with a cemented pin. Using this technique takes only an extra few minutes but can improve the longevity of any crown, assuming that attention to detail is practiced during each step.

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DOI: 10.2341/11-463-T

DESCRIPTION OF THE TECHNIQUE

A crown is prepared following the protocol described by Tucker,[10] including almost parallel walls, mesial and distal hollow grinds, and a knife-like margin placed with a flame-shaped 860-014. An impression is made using polyvinylsiloxane. The impression is poured using a die stone and a hinge articulator. The die is trimmed, and a silicon crown mold is used to help wax the occlusal surface. A small hole is placed in the wax-up large enough to allow a plastic burnout pin to fit into the hole. The crown along with the plastic pin are cast utilizing a type 2 cast gold with a ringless investment system. The casting is separated from the button, finished, and polished using sandpaper disks and powders. Following complete seating of the casting on the tooth and cessation of the zinc phosphate cement flowing from the vent hole, the pin being held onto a pin seater

Figure 1. *The mandibular right quadrant has been isolated using heavy dark rubber dam and a #26 retainer on the second molar and a sectioned #212 to help expose the mesial margins on the operating tooth.*

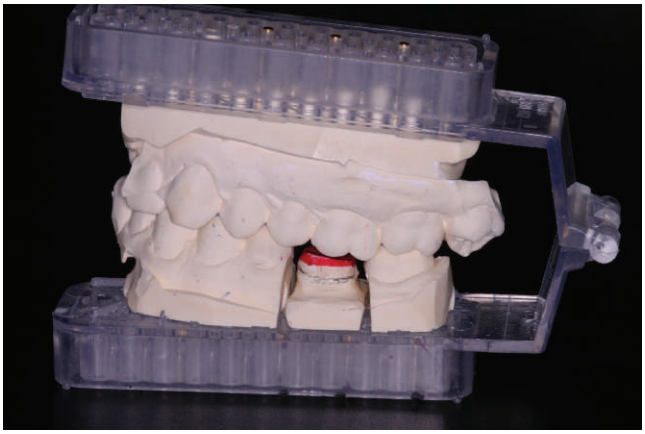

Figure 2. *Completed die model.*

Figure 3. *A thin layer of lining wax being placed on die using electric waxer.*

Figure 4. *Occlusal view of silicon mold on die.*

Figure 5. *Adding modeling wax prior to removal of occlusal mold.*

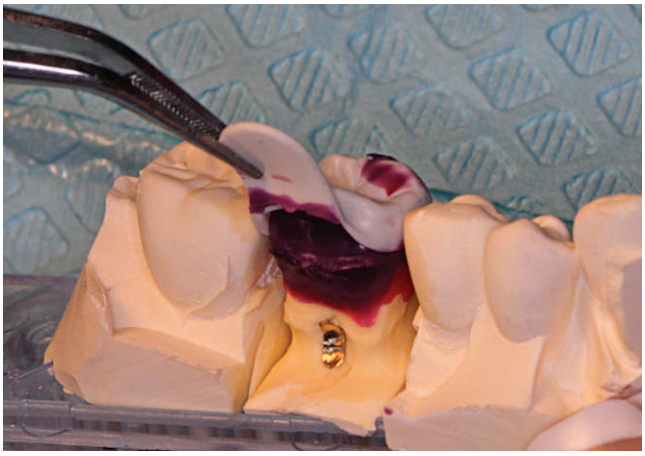

Figure 6. *Peeling mold carefully from die.*

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Figure 7. *Handpiece carefully making hole in wax pattern —a small twist drill can also be used for this step. It is important that the hole must be the size of the plastic burnout pins seen in Figure 25.*

with an adhesive dot is very carefully placed into the vent hole and tapped in using a gold foil mallet. Sometimes, if necessary, a small depression is made in the crown prep to allow the pin to completely seat into the crown without hitting tooth structure. The excess pin is then removed using a high-speed diamond and disks of the operator's choice. Finally, the casting is polished using sandpaper disks and aluminum oxide powders.

POTENTIAL PROBLEMS

There are only a couple of potential problems with this technique. The laboratory work must be very exact, and paying attention to every detail is extremely important. A second problem could be the handling of the pin. This can be a little difficult since it is so small. The pin seater used in this case with the adhesive dot facilitated the placement of the pin.

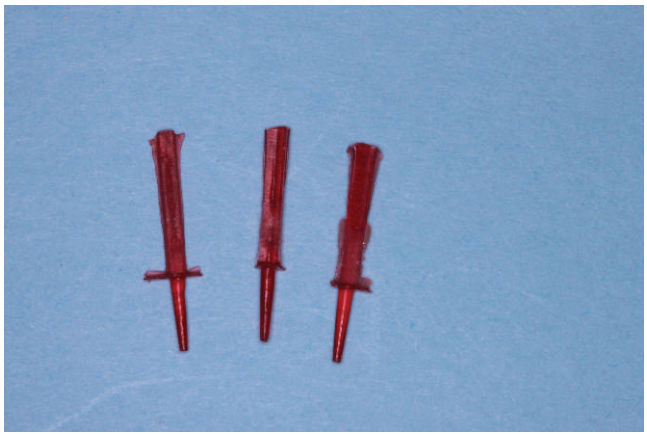

Figure 8. *Plastic burnout pins used for vent pin.*

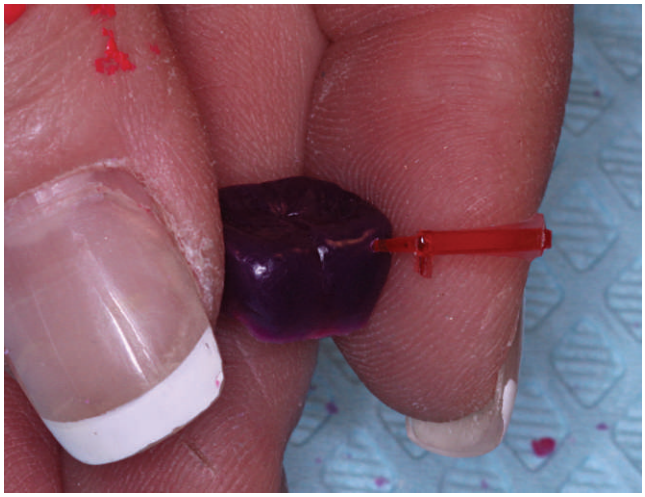

Figure 9. *View showing plastic burnout pin in hole created in wax. The mesio-buccal usually is the best place to place the escape channel since it is easily accessible for finishing.*

Figure 10. *Wax-up and plastic burnout pins on sprue former.*

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Figure 11. *Casting following pickling in bath of phosphoric acid water and urea (Pre-Vox, Ivoclar, Amherst, NY).*

SUMMARY OF ADVANTAGES AND DISADVANTAGES

Advantages: Better marginal fit of crown.

Disadvantages: None.

LIST OF MATERIALS USED

Impression material: Aquasil, Dentsply, Milford, DE

Plastic taper pins #700: Wilkerson Company, Post Falls, ID

Emery impression tray: Emery Dental, Salem, OR

Figure 12. *The completed vented crown with pin in place.*

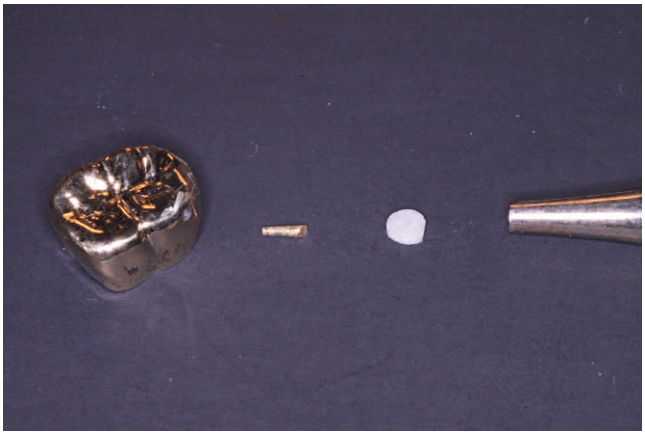

Figure 13. *The crown, pin, adhesive dot, and pin-placing instrument.*

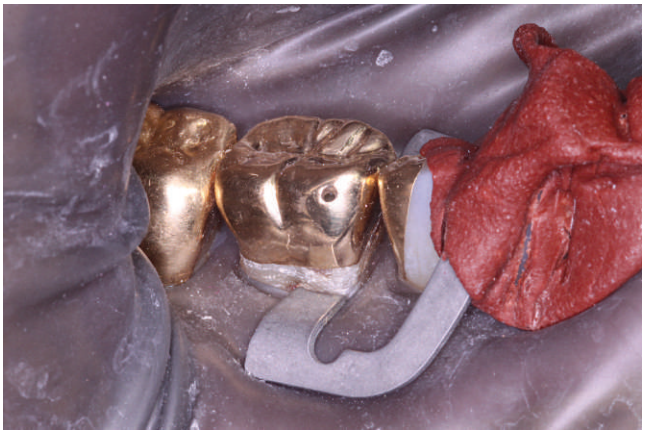

Figure 14. *First step at the seating visit is to try the crown in checking contacts at complete seating on tooth. Make sure that margins are exposed beyond the rubber dam.*

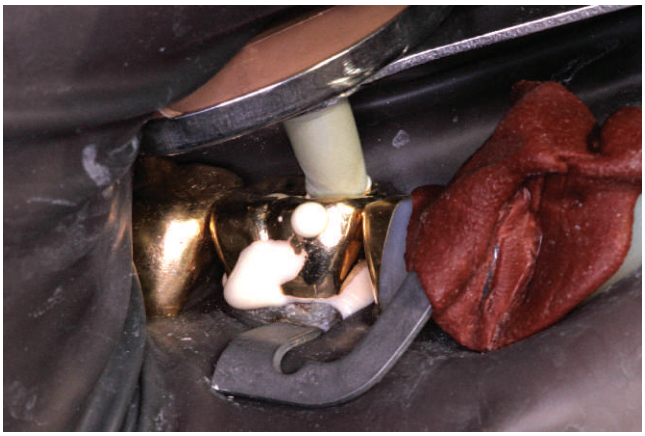

Figure 15. *Crown seated with cement extruding through vent on mesio-buccal. The instrument between the crown and the opposing arch is a Medarts seater (Pearson Dental Supply). When using this seater, make sure that the angle is correct and is allowing full seating.*

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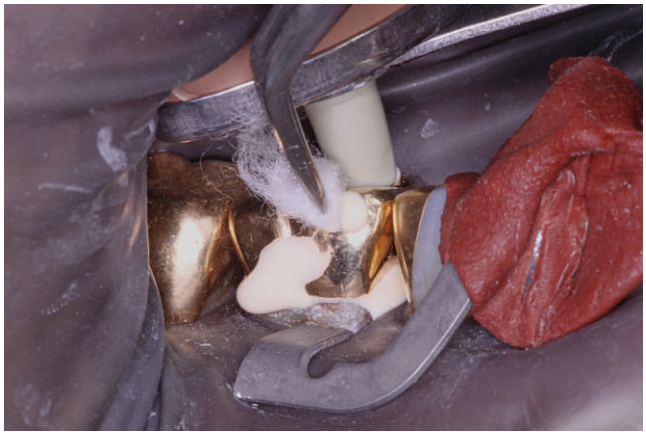

Figure 16. *Image showing the wiping off of the excess zinc phosphate cement.*

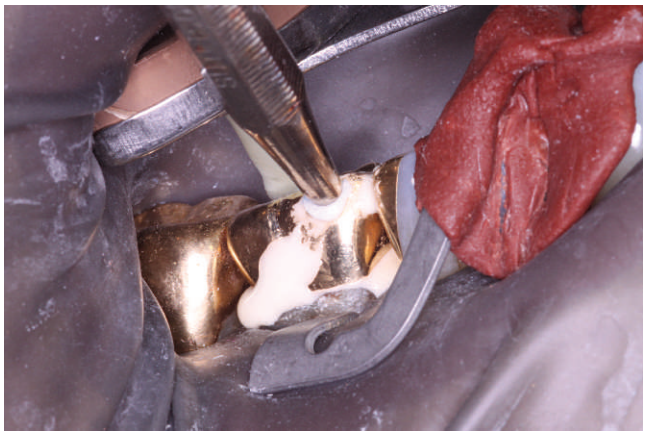

Figure 17. *Cast pin being placed using seating tool. This step can be done with a pair of cotton pliers, but sometimes the very small pin is difficult to hold.*

Figure 18. *A wood stick placed to help achieve complete seating. Note pin on place on the mesio-buccal.*

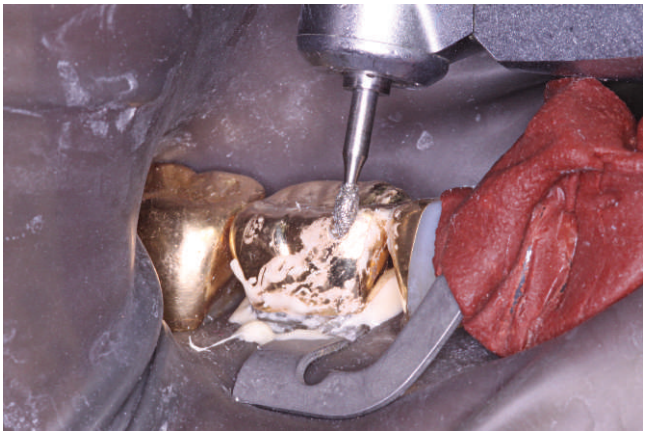

Figure 19. *The excess pin can be removed with a diamond or sandpaper disk and then polished with disks and powders. If the pin does not fully seat, sometimes a small dimple is placed in the tooth at the end on the vent channel to make room for the pin.*

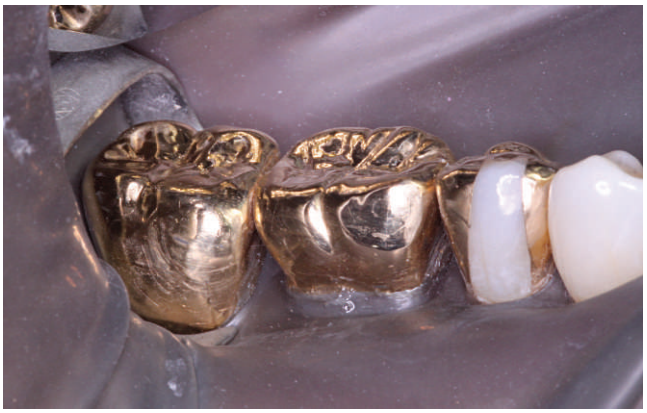

Figure 20. *Buccal view of completed crown.*

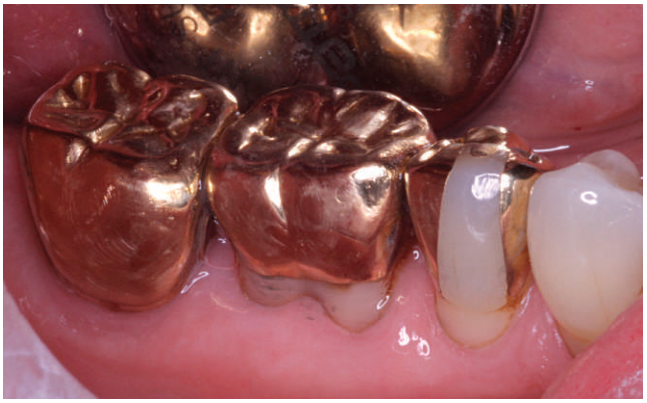

Figure 21. *Buccal view of completed crown one year postop.*

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Diamonds: Brasseler USA, Savannah, GA

Articulator: WOW, Premier Dental Products, Plymouth Meeting, PA

Dowel pins for articulator: Premier Dental Products

Die stone: Fuji Rock tan, GC America, Alsip, IL

Investment system: Starvest, Emdin International Corporation, Irwindale, CA

Biofit Morphology Occlusal Molds: Jensen Dental Solutions, New Haven, CT

Gold: JRVT, Jensen Industries, New Haven, CT

Leather mallet: James Gourley, DDS, Bainbridge Island, WA

Pin seater: Suter Dental Company, Chico, CA

Medarts seater: Pearson Dental, Sylmar, CA

Adhesive dots: Accudots, Hu Freidy, Chicago, IL

Sandpaper disks: EC Moore Company, Dearborn, MI

Aluminum oxide powders: Universal Photonics, 15- and 1-micron sizes, Hicksville, NY

Conflict of Interest

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(Accepted 5 July 2012)

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Class II Composite Restorations and Proximal Concavities: Clinical Implications and Management

M Patras • S Doukoudakis

Clinical Relevance

Proper configuration of the proximal surface of a Class II composite restoration is essential for the preservation of dental and periodontal tissues and subsequent long-term success. Adequately customized or designed wedges can assist in reproducing an imitation of natural form in the interproximal area and ensure sufficient contact tightness with the adjacent tooth.

SUMMARY

Clinical experience supports the notion that the restoration of MOD cavities may pose a challenge to the practitioner. Proper placement of precontoured matrices and commercial wedges help the clinician to establish an optimal emergence profile and sufficient contours. However, the presence of proximal concavities in premolars or molars can turn the reproduction of previous cervical architecture into an even more demanding task. Wedges with customized form or adequate design can precisely conform the matrix to the cavosurface area and prevent any gap formation. This article presents two different options that allow for successful and predictable reestablishing of anatomically correct contours and optimal proximal contacts in posterior teeth with proximal concavities.

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INTRODUCTION

Recent technological advances in adhesive dentistry along with the increasing patient demand for tooth-colored restorations have forced the routine use of contemporary resin composites for the restoration of carious lesions. However, especially in Class II composite restorations, among the most difficult challenges to clinicians is achieving perfect adaptation of resin composite to the margins and the internal walls of the cavity or the prevention of

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overhangs at the cavosurface margin. Unlike amalgam, composite resins cannot easily be condensed into all regions of the prepared cavity, which in turn affects the establishment of sufficient proximal contacts. In addition to that, the cervical proximal margins in Class II restorations are often considered to be the Achilles' heel, as dentin bonding is often less predictable.

Established United States Public Health Services (USPHS) criteria evaluate anatomic form and marginal characteristics of restorations, implying that marginal and internal adaptation are crucial for the longevity and good prognosis of resin composite restorations.[1–3] Moreover, it is well documented[4,5] that the formation of overhangs provokes food impaction, subsequent recurrent caries, and periodontal problems. Consequently, clinical experience supports the notion that the proper placement of the matrix and wedge are of paramount importance in order to achieve ideal form, function, and esthetics for the success of posterior resin composite restorations.[6]

In recent decades numerous developments have been made in the field of matrices and wedges that are used in posterior teeth. As resin composite is becoming the most frequently used restorative material, many of these products are specifically targeted for improved results with those restorations.[6] The constant search for the "perfect" system is ongoing, as the diversity of clinical cases seems to be endless. One of the most demanding clinical challenges is the restoration of concave proximal cervical areas, which are most commonly located in the mesial aspect of the upper first premolars and first lower molars as well as the distal side of the upper first molars.[7] These prominent concavities occupy an area located cervical to the mesial contact area and extend to the corresponding tooth depression (Figure 1).[7] When the cervical margin of a Class II restoration is located in the area of proximal root invaginations, tooth contour seems "incompatible" with the convex shape of most matrices, as the latter are commonly used for the restoration of convex-shaped proximal areas.

As concave-shaped matrices have not been developed for such distinct clinical situations, the dental clinician has to rely on the proper shape of the wedge. Dental wedges usually serve to compress the matrix to the remaining healthy tooth structure across the entire buccolingual length, apical to the gingival cavosurface line angle.[8] A review of the literature revealed limited references on that topic, suggesting molding the wedge with compound,[9] light-cured resin,[10] the utilization of elastic cords,[11] or the use of two wedges to seal the cavity.[12]

Figure 1. *Proximal concavity at the mesial aspect of an extracted upper first premolar.*

PURPOSE

The purpose of this article is to recommend two different options that will enable the clinician to solve one of the main aforementioned issues regarding Class II composite restorations, that is, the adequate sealing of the gingival cavosurface margin when a proximal concavity exists. The following two clinical cases will illustrate two ways of conforming the matrix to the proximal concavity of the tooth, thus preventing an overhang and securing ideal contours of the restoration.

DESCRIPTION OF TECHNIQUES

Case 1: Customization of Wooden Wedge

A 35-year-old female patient presented with a carious lesion on the mesial aspect of her first upper

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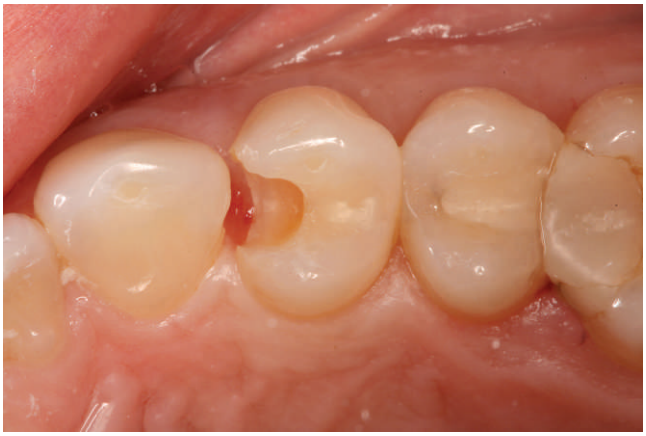

Figure 2. *Occlusal view of the cavity preparation. Note the existence of a proximal concavity.*

premolar. A conventional wooden wedge of adequate dimensions was placed during the preparation of the cavity (pre-wedging technique) in order to protect the soft tissues, separate the adjacent teeth, and ensure proper proximal contacts.[13] During the cavity preparation, special care was given so that the cavosurface margin finished on enamel, in order to minimize the microleakage (Figure 2). Upon completion, a proximal concavity made it impossible for the precontoured sectional matrix (Contact Matrix, Danville Materials, San Ramon, CA, USA) to maintain precise contact with the cavosurface margin. Therefore, a larger wedge (Kerr, Bioggio, Switzerland) than that needed was selected and customized with a #15 blade in a corresponding convex shape to accommodate the interproximal space (Figure 3).[8]

After the wedge was inserted in the proximal space, securing the matrix in place, proper marginal

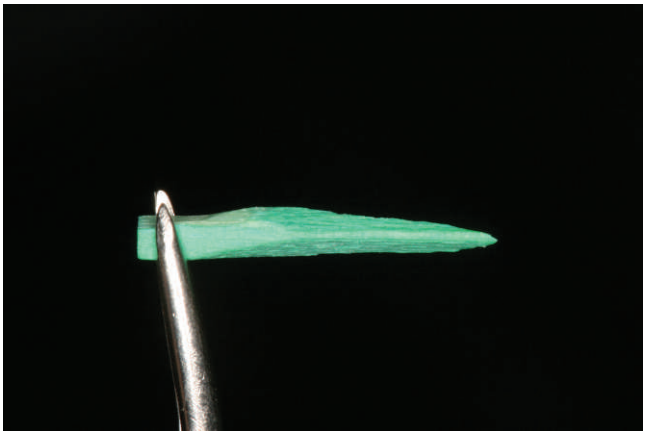

Figure 3. *A selected wooden wedge was properly contoured with the use of a surgical blade.*

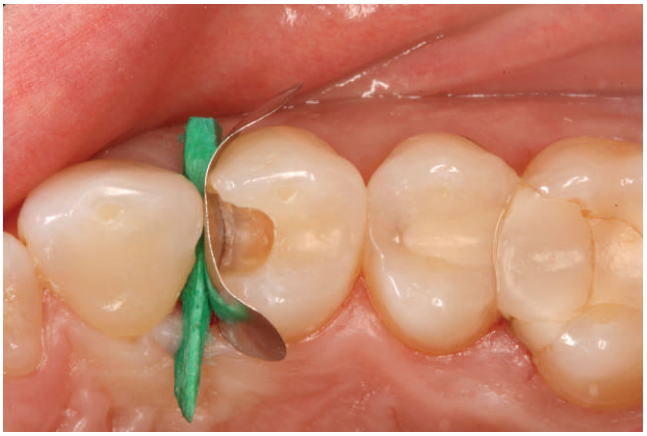

Figure 4. *The insertion of the customized wedge ensures adequate matrix adaptation.*

fit was verified (Figure 4).[8] A ring maintained their positions, held the matrix against the tooth surfaces, and added to the wedge's separation capacity.[6,14] Subsequent burnishing of the matrix provided adequate configuration and contact with the adjacent tooth.[6] The total etch technique was used and the composite (Tetric EvoCeram, Ivoclar Vivadent AG, Liechtenstein) was preheated, then applied by incremental layering and polymerized in a soft-start mode (Figure 5) in order to improve adaptation and reduce shrinkage at the cervical interface.[15,16]

Case 2: Plastic Wave-Wedge

A 40-year-old female patient presented to the clinic with a failing composite restoration that also had clinically unacceptable contours (Figure 6). After a rubber dam was placed the old restoration and secondary caries were removed and the outline form of the cavity preparation was assessed (Figure 7). Upon insertion of the conventional wooden wedge a

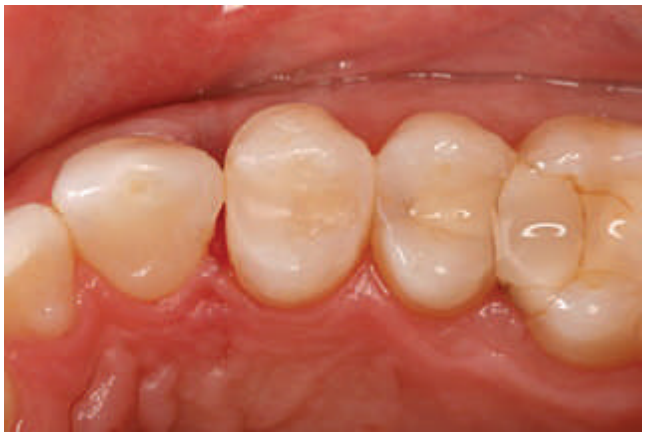

Figure 5. *Postoperative view of the final restoration.*

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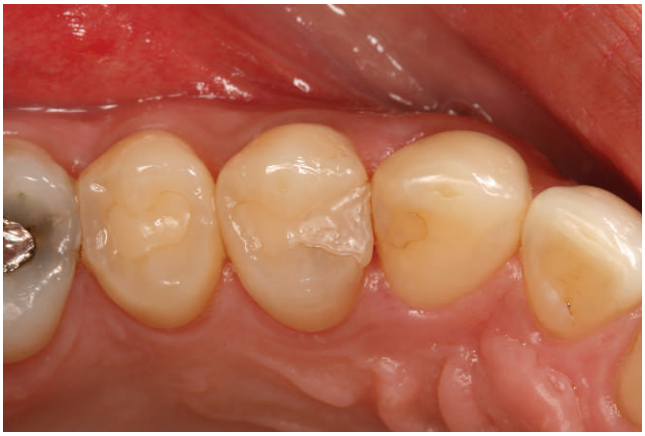

Figure 6. *Preoperative occlusal view of the existing restoration.*

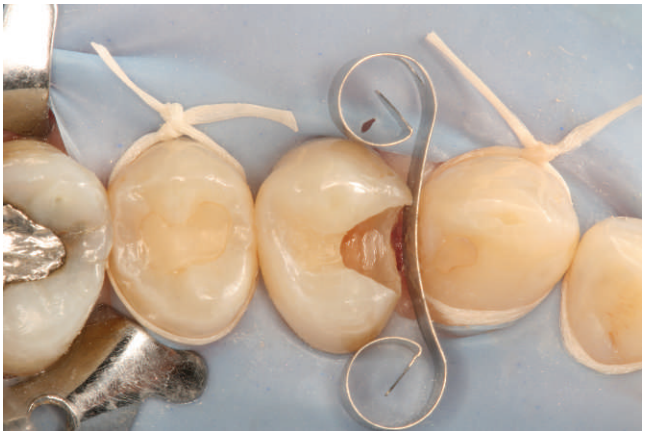

Figure 7. *After the removal of the restoration, a proximal concavity could be observed.*

misfit between the matrix and the cavosurface margin was verified (Figure 8). A plastic wedge (Wave-Wedge, Triodent Ltd, Katikati, New Zealand) (Figure 9) that has been lately introduced[17] was

Figure 8. *Placement of a conventional wooden wedge results in incomplete sealing of the cavosurface margin.*

Figure 9. *The Wave-Wedge (Triodent Ltd) in three different, color-coded sizes.*

selected in the case in order to aid in the adaptation of the matrix. This wedge is designed with a wave curvature that allows for an optimal adaptation to the anatomy of the cervix of the tooth. Furthermore, its elasticity enables the wedge to expand after entering the critical interproximal area. For that reason, the wedge was forced beyond its central concavity to ensure proper sealing (Figure 10), to prevent any gap formation in the cavosurface area, and to facilitate composite layering (Figure 11). All the subsequent steps were carried out as mentioned above, and a radiograph verified the acceptable contours at the mesial aspect of the restoration (Figure 12).

Potential Problems

The potential problems identified included the following: 1) difficulty in establishing sufficient contours of the wooden wedge (in the first case)

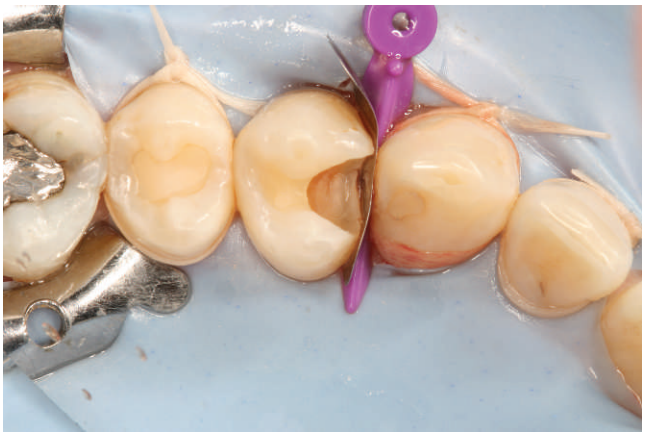

Figure 10. *The Wave-Wedge's design allows for adequate sealing.*

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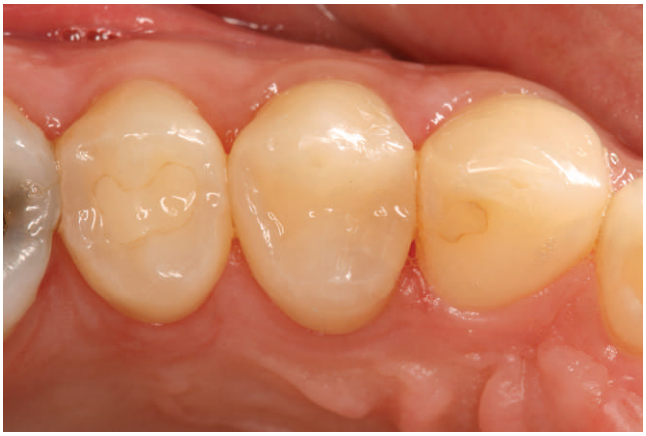

Figure 11. *Occlusal view of the completed restoration showing optimal contours and proximal contact with the adjacent tooth.*

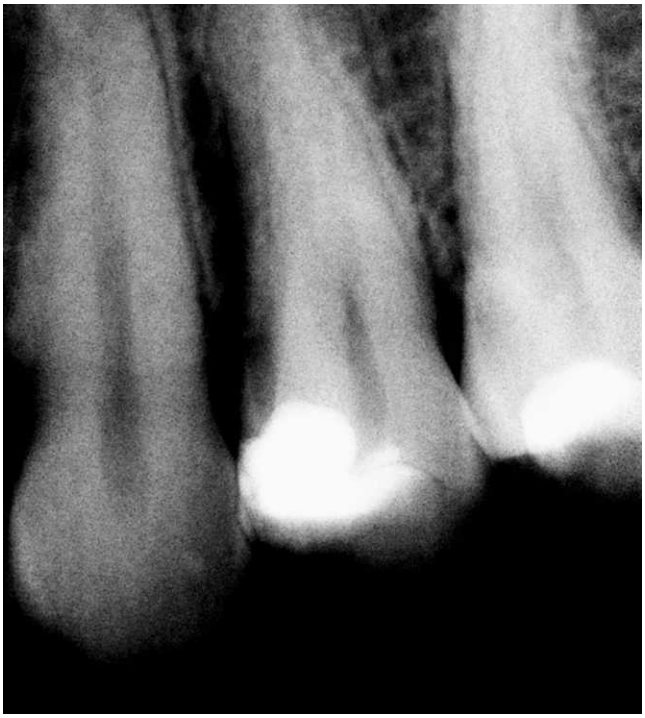

Figure 12. *The radiograph verifies the absence of an overhang at the cavosurface margin.*

and 2) potential displacement of the plastic wedge or inadequate tooth separation (in the second case)

DISCUSSION

Resin composites are considered to be the state-of-the-art materials with which to facilitate direct posterior restorations. However, the restoration of a MOD cavity is often a concern for the clinician, who has to overcome problems associated with adequate handling of interproximal areas as a result of constraints in clinical access.[6] Given the aforementioned inherent limitations, the presence of any proximal anatomical variations in premolars or molars can pose a challenge to the practitioner.[6] Proper reproduction of the proximal concavity is largely dependent on the shape and relation of the sectional matrix and corresponding wedge.[6,8]

Among the basic requirements, a dental wedge must be able to cause tooth separation, provide resistance against the matrix, and precisely conform it to the anatomical surfaces of the tooth to be restored. Loose fit of the matrix allows oral fluids to contaminate both the cavity and the restorative materials. Therefore, this improper adaptation compromises the longevity of the restoration and induces its potential failure in the future. Furthermore, any gap formations will develop overhangs and unacceptable contours of the tooth in the interproximal space, leading to plaque accumulation.[4,5] This is even more important as the finishing burs and strips cannot approximate concave gingival margins,[6] and the difficulty in gaining access can definitely endanger the integrity of the adjacent teeth and the periodontium.

As the restoration's success lies in meeting the fundamental prerequisites, the custom wedge modifications with a bur or blade[8] or the utilization of precontoured wedges can present viable alternatives to conventional wedges and enhance the clinician's armamentarium.

Summary of Advantages and Disadvantages

From a clinician's perspective, the two options illustrated above represent very effective ways of addressing such clinical situations, thus providing the practitioner with sufficient comfort at the same time.[6] Both wedge designs reflect the shape of the proximal concavities; thus, they may offer various advantages in an efficient and simplified manner. Their customized shape can easily be adapted to tooth contours, create a tight seal, and ensure matrix manipulation. In such a clinical situation the adequate transition from the concave cervical area to the convex shape of the contact area is mandatory. This individual reproduction of previous cervical architecture prevents any overhang formation and minimizes the need for lengthy finishing procedures.

In the authors' experience, the potential disadvantages of the present approaches could be the time needed for the techniques to be well adopted and

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distortion of the matrix during the insertion of the wedge, as well as the cost of the plastic wedges.

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

Minimal Invasive Treatment for Defective Restorations: Five-Year Results Using Sealants

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Clinical Relevance

Improvement of the marginal seal of restoration defects is a conservative approach that may improve their quality over time. Minimally invasive treatments of defective restorations showed similar outcome to replaced restorations after five years.

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SUMMARY

Replacement of dental restorations has been the traditional treatment for restorations that are defective. In this five-year randomized clinical trial, restorations with localized marginal defects were treated with sealants.

Thirty-two patients (mean age, 26.8 years) with 126 Class I and Class II restorations with defective margins (amalgam n=69 and resin-based composite n=57) were recruited. Treatment was seal with pit and fissure sealant on localized marginal defects (group A: n=43) and was compared with total restoration replacement (group B: n=40) and untreated restorations (group C: n=43) as negative and positive controls. Restorations were assessed by two examiners using the modified US Public Health Service criteria, observing five clinical parameters: marginal adaptation, roughness, marginal stain, teeth sensitivity, and secondary caries at baseline and at five years after treatment.

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At the five-year recall examination, 23 patients with 90 restorations (71.4% recall rate) were examined. A significant improvement was observed in the marginal adaptation of the restorations in group A compared with group B. None of the treated group showed trends to downgrade in any parameter. Tooth sensitivity and secondary caries showed a low frequency in all groups. No significant difference in marginal adaptation of the restorations was found between amalgam and resin-based composite restorations (p=0.191). This study demonstrated that marginal sealing of restorations is a minimally invasive treatment that may be used instead of the replacement of restorations with localized marginal defects.

INTRODUCTION

Dental restorations may demonstrate degradation in the intraoral environment over time, and the principal reasons for deterioration are marginal deficiencies, fracture, and wear, possibly leading to secondary caries and/or tooth sensitivity.[1–3] Traditionally, those failures have led to complete replacement of the restorations, including in the presence of minor imperfections. Restoration replacement represents a major concern in dental practice, reaching up to 60% of general dentistry interventions.[4]

In recent times, with more insight into cariology, tooth longevity, dental biology, and dental materials science, a minimally invasive philosophy has prevailed, and the advantages of repairing rather than replacing restorations have been progressively emphasized.[5–11]

Complete restoration replacement has the disadvantages of being time-consuming, leading to unnecessary removal of healthy tooth tissue, including in areas away from the localized defects. Unnecessary removal of sound tooth tissue may result in enlarged preparation and restoration size, which could alter the proposed treatment plan and possibly result in irreversible injuries to pulp tissues.[7,8,12–15]

During recent years, new strategies such as repair and refinishing or sealing of localized defects have shown an overall improvement in the clinical properties of defective restorations, thereby increasing their longevity through minimal intervention.[9,15–17] Whenever possible, repair of restorations can be more cost-effective and acceptable to patients than restoration replacement. Because it preserves tooth structure, it has the potential to allow patients to retain most of their teeth during their lifetime.[18] In addition, the clinical results of these combined studies have changed education in operative dentistry as repair of restorations is routinely taught in most dental schools.[10,19–21]

The aim of this randomized clinical trial was to assess sealed defects at the margins of Class I and Class II amalgam and resin-based composite (RBC) restorations and to follow-up the results after five years. The hypothesis to be tested was that after five years, sealing the defects at the margins would show similar performance as restorations that were replaced.

METHODS AND MATERIALS

Thirty-two patients (19 female and 13 male; mean age, 26.8 years) with 126 Class I (n=94) and Class II (n=32) amalgam (n=69) and RBC (n=57) restorations with defective margins participated in the study. The experimental treatment group was the application of a pit and fissure sealant on localized defects in the margins of restorations (group A: n=43). The comparison groups were total restoration replacement (group B: n=40) and untreated restorations (group C: n=43), serving as negative and positive controls. Restorations were assessed using the modified US Public Health Service criteria (Table 1) observing five parameters: marginal adaptation, roughness, marginal stain, tooth sensitivity, and secondary caries by two examiners (E.F., J.M.).

Inclusion Criteria

The inclusion criteria were: 1) patients with amalgam (Am) and RBC restorations with marginal deficiencies that were judged to be suitable for sealing, 2) older than 18 years of age, 3) having more than 20 teeth in their mouth, and 4) being able to sign the consent form. In addition, the restorations had to be in functional occlusion with an opposing natural tooth and have at least one proximal contact area with an adjacent tooth.

Exclusion Criteria

The exclusion criteria were: 1) contraindications for regular dental treatment based on their medical history, 2) special esthetic requirements that could not be solved by this alternative treatment, 3) xerostomia or taking medication that significantly decreased salivary flow, 4) high caries risk, or 5) psychiatric or physical diseases that interfered with oral hygiene.

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Table 1: *US Public Health Service/Ryge Clinical Criteria*[20]

Clinical Characteristic	Alpha	Bravo	Charlie
Marginal adaptation	Explorer does not catch when drawn across the restoration-tooth interface	Explorer falls into crevice or has one-way catch when drawn across the restoration-tooth interface	Dentin or base is exposed
Surface roughness	The surface of restoration has no surface defects	The surface of restoration has minimal surface defects	The surface of restoration has severe surface defects
Secondary caries	There is no clinical diagnosis of caries	N/A	Clinical diagnosis of caries
Marginal stain	There is no discoloration between the restorations and tooth	There is discoloration on less than half of the circumferential margin	There is discoloration on more than half of the circumferential margin
Teeth sensitivity	No sensitivity when an air syringe is activated for two seconds at a distance of half an inch from the restoration with the facial surface of the proximal tooth covered with gauze	Sensitivity is present when an air syringe is activated for two seconds at a distance of half an inch from the restoration with the facial surface of the proximal tooth covered with gauze and ceases when the stimulus is removed	Sensitivity is present when an air syringe is activated for two seconds at a distance of half an inch from the restoration with the facial surface of the proximal tooth covered with gauze and sensitivity does not cease when the stimulus is removed

Sample Size Determination and Randomization

Sample size was determined *a priori* using G*Power 2,[22] with an error probability of $\alpha=0.05$, effect size 0.3, and power ("β-1 error probability) of 0.80. The restorations with marginal defects (Bravo) were randomly assigned (performed by PASS software version 2004, Keysville, UT, USA) to one of three groups of treatment: A, sealing of margins (n=43); B, replacement (n=40); and C, untreated (n=43).

The Institutional Research Board and Ethical Board of the Dental School at the University of Chile approved the study (project PRI-ODO-0207). Only faculty members were allowed to provide the restorative treatment, and all patients signed inform consent forms and completed a registration form.

Caries Risk Assessment

A graphical computed program (Cariogram) was used to assess individual patients' caries risk; the program weighted the interaction between the following 10 caries-related factors: caries experience, related general disease, diet contents, diet frequency, plaque amount by Silness Löe Index, semiquantitative detection of mutans streptococci and lactobacilli in saliva by caries risk test (CRT) bacteria (Ivoclar, Vivadent AG, Schaan, Lichtenstein), fluoride program, amount of saliva stimulated secretion by CRT buffer (Vivadent), saliva buffer capacity, and clinical judgment. Patients were classified as high, intermediate, and low caries risk. In addition, the results also indicated where targeted actions to improve the situation would have the best effect.[23]

Restoration Assessment

The quality of the restorations was evaluated using the modified US Public Health System/Ryge criteria (Table 1).[24] Two examiners (J.M. and E.F.) assessed the restorations independently and by visual (mouth mirror number 5, Hu Friedy Mfg Co Inc, Chicago, IL, USA) and tactile examination using an explorer (N° 23 Hu Friedy) and indirectly by radiographic (Sirona Heliodent Vario, Charlotte, NC, USA) examination (Bite Wing, DF57, Kodak Dental System Healthcare, Rochester, NY, USA). All the restorations were examined at baseline and each year up to five years. The five parameters used in the examination were marginal adaptation, roughness, secondary caries, marginal stain, and tooth sensitivity (Table 1). If any difference was recorded between the two examiners and an agreement could not be reached, a third clinician (G.M.) was called to assist with the decision process. If the three clinicians did not reach an agreement, the lower score was recorded. All three clinicians participated in calibration exercises at the beginning and before the last examination took place, and the interexaminer reliability results were

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Kappa=0.74 at the baseline and Kappa=0.87 at the fifth year.

A change from Bravo to Alpha was considered an improvement, and a change from Alpha to Bravo was considered deterioration.

Treatment Groups

A. Sealing of margins: Defective areas were acid etched with 35% phosphoric acid for 15 seconds. A resin-based sealant (Clinpro Sealant, 3M ESPE) was applied over the defective area. The sealant was polymerized with a photocuring unit (Curing Light 2500, 3M ESPE) for 40 seconds. Rubber dam isolation was used for this procedure. All treatments were provided by the same clinician (G.M.).
B. Replacement group: The defective restoration was totally removed and replaced with either a new amalgam (Tytin, Kerr Corporation, Orange, CA, USA) or RBC restoration (Filtek Supreme, 3M ESPE). Rubber dam isolation was used for this procedure. All treatments were provided by the same clinician (J.E.).
C. Untreated group: The defective restorations did not receive any treatment.

Patients were recalled each year for five years for clinical evaluation by the same examiners, using the same criteria as used at baseline.

Failed restorations were removed from the study and treated according to their diagnosed needs.

Digital photographs and bitewing radiographs were taken for all the restorations before and after treatment and every year prior to the examination.

Statistical Analysis

Wilcoxon test was used to compare the preoperative and postoperative conditions at the fifth year, and the Kruskal-Wallis test and Mann-Whitney post hoc tests were used for comparisons among groups at the error probability of α=0.05 (SPSS version 15.0, SPSS Inc, Chicago, IL, USA).

RESULTS

Twenty-three patients (14 female, 9 male) with 90 amalgam (n=53, 43-Class I and 10-Class II), and resin based composite (n=37, 34-Class I and 3-Class II) restorations distributed in three groups (group A: n=37; group B: n=23; group C: n=30) were evaluated every year and up to five years. The study had an overall attrition rate of 28.6% (5.7% per year), with nine patients with 36 restorations who were unable to be contacted.

When comparing the baseline assessment of restorations with the results after five years (Figure 1), group A showed a statistically significant improvement in marginal adaptation (p=0.0001). No significant difference was found for tooth sensitivity and secondary caries. In contrast, a significant downgrade was observed for surface roughness and marginal staining (p=0.0001 and p=0.005, respectively).

The results for Group B (Figure 2) after 5 years showed a significant improvement (p=0.022) for marginal adaptation, with secondary caries being less prevalent (p=0.008). No significant differences could be seen for marginal stain, roughness, and sensitivity.

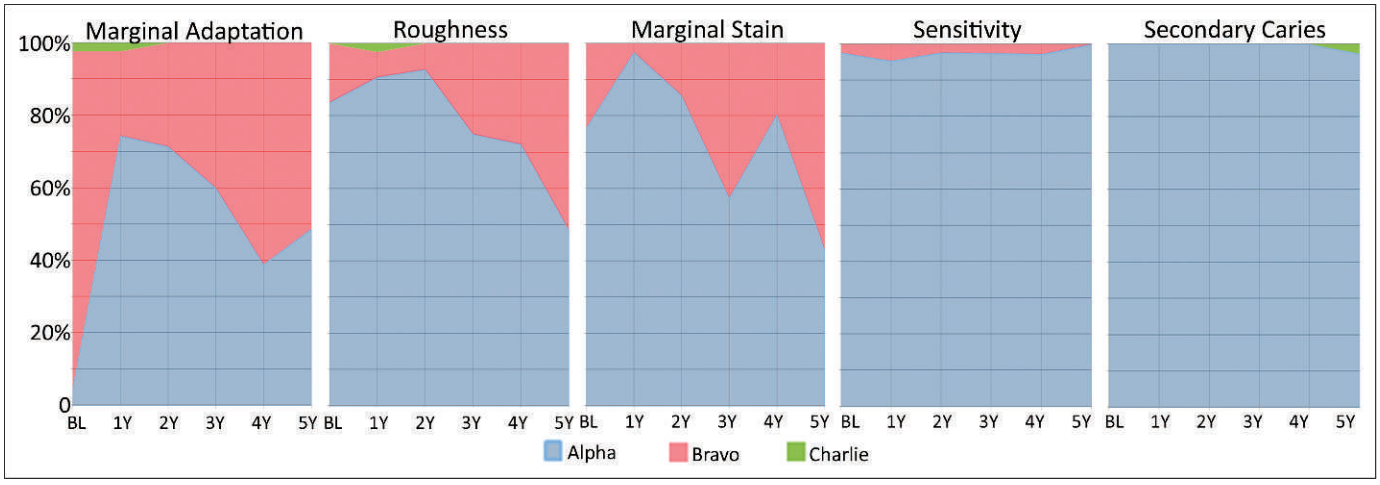

Figure 1. *Yearly summary results from group A according to the US Public Health Service/Ryge scores for each clinical parameter. BL = baseline; 1Y-5Y observation periods.*

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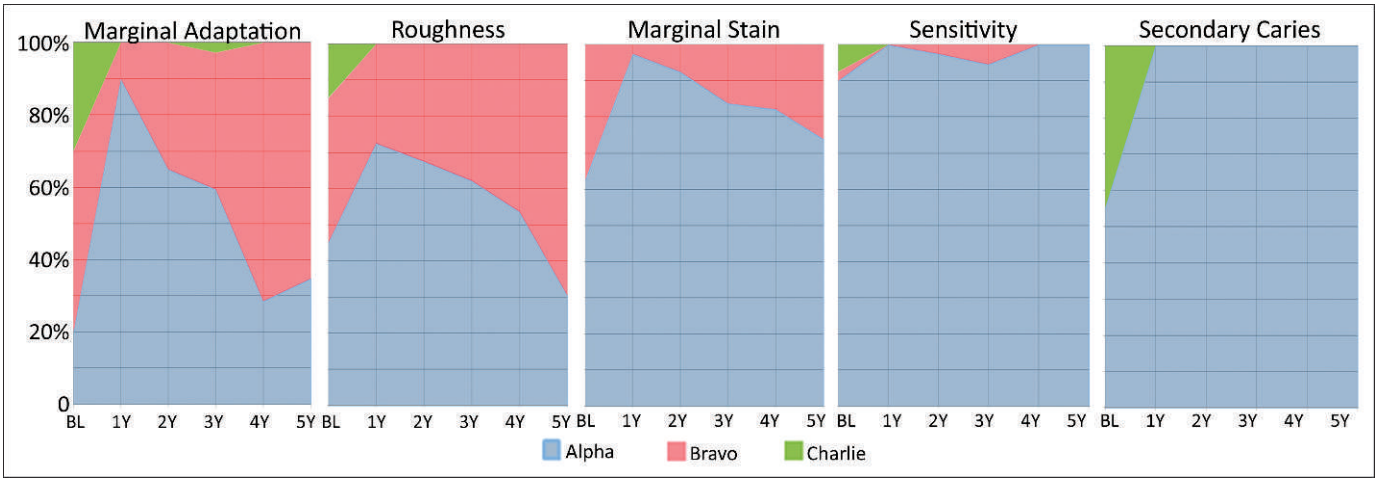

Figure 2. *Yearly summary results from group B according to the US Public Health Service/Ryge scores for each clinical parameter. BL = baseline; 1Y-5Y observation periods.*

Group C (Figure 3) presented a significant downgrade in marginal adaptation (p=0.02), roughness (p=0.001), and marginal stain (p=0.001), with no differences between the two restoration materials (p=0.130). No changes were observed for sensitivity and secondary caries after the fifth-year examination (p=1.00 for both).

No significant differences were observed between groups A and B (p=0.658) for all the clinical parameters observed (Figure 4). However, both groups showed significantly improved results for marginal adaptation when compared with group C (p=0.0001).

The comparison between groups in marginal staining showed a downgrade in group A and the same in group C (p=0.189). Group B compared with group A and C showed an upgrade in the period (p=0.001). No significant differences were observed between the three groups (p=0.073), but all groups showed downgrades. In the secondary caries parameter, the only group that showed changes was group B, with an upgrade statistically better than group A and C (p=0.000 and p=0.001; Figure 4).

No significant difference was found between amalgam and RBC restorations for any of the groups.

DISCUSSION

The management of composite or amalgam restoration with localized defects is a common challenge in clinical practice. Some restorations may certainly require replacement, while others may be given

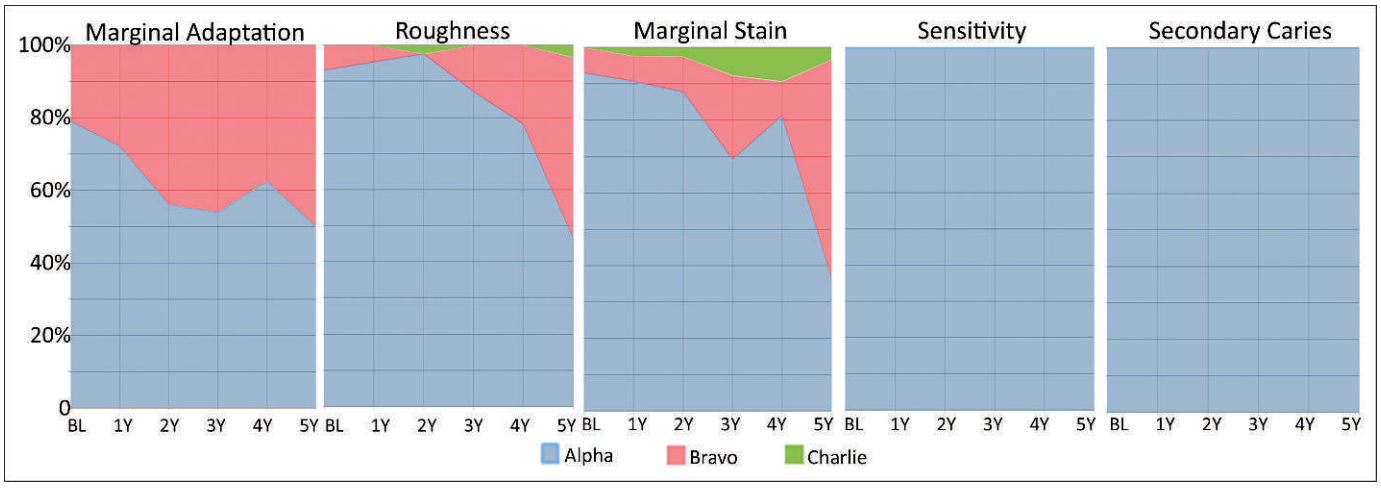

Figure 3. *Yearly summary results from group C according to the US Public Health Service/Ryge scores for each clinical parameter. BL = baseline; 1Y-5Y observation periods.*

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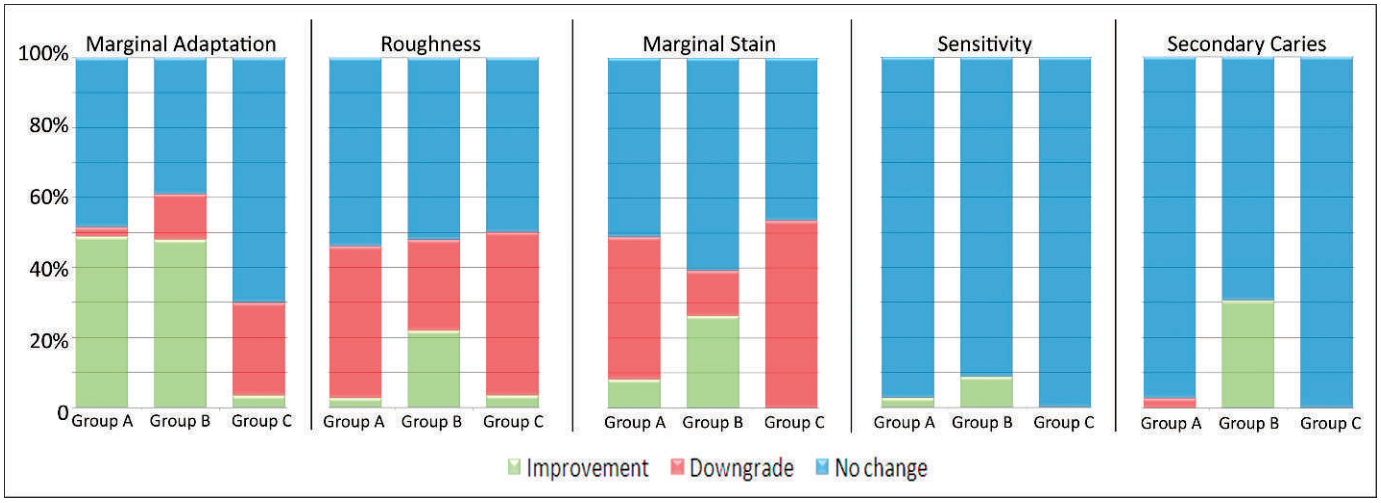

Figure 4. *Summary of the changes in US Public Health Service/Ryge scores between baseline and fifth-year evaluation (results expressed in percentages).*

extended longevity through the use of alternative procedures.[25,26]

A conservative approach to the management of defective restorations, if appropriate, has the potential to be less costly in terms of time and cost, less traumatic for patients, less likely to result in iatrogenic damage, possibly obviate the need for the use of local anesthesia and, more importantly, preserve tooth structure.[17,27] Furthermore, extended longevity of existing restorations may enhance patients' general health and satisfaction. Significant differences exist among dentists when deciding whether or not a restoration should be replaced.[28–33]

Although minimally invasive dentistry has been introduced in the dental curriculum, it has taken place only in recent years. Therefore, several dentists have not been trained on proper diagnosis and application of the minimally invasive procedures. McAndrew and others[34] concluded that it is possible to reduce examination time and provide convergence to a defined standard through a basic training program that can significantly influence the restoration replacement rate among general dental practitioners. In the dental school in Santiago, Chile, defined criteria are used by clinical students, and alternative methods to replacements are taught, including the repair of localized defects in restorations.

It is recognized that the chipping of margins of the restorations is an early sign of deterioration in clinical service, which tend to be restricted to a small part of the restoration, usually a short segment of the cavosurface margin.[35] Sealing with pit and fissure sealants, a minimally invasive procedure, will reduce the indication for replacement of the restoration.

The longevity of dental restorations mainly depends on the continuity of the interface between the restorative material and adjacent tooth tissue.[36] Some marginal defects may be sealed to increase the life of restorations.

The results of the present study showed that sealing defective margins had similar results to restoration replacement for marginal adaptation after five years. The benefit of this procedure is that it is prompt, minimally invasive to patients, and less involved than replacement for clinicians. In addition, if a sealant fails, it does not necessarily mean the presence of secondary caries, and therefore, the procedure could be repeated multiple times. In this study, only 5% of sealed restorations showed alpha value at baseline, increasing to 74% during the first year, followed by continuous margin deterioration during the next years, reaching 49% alpha value at the fifth year. The experimental and the control groups showed the same trend of downgrade of marginal adaptation over time. Amalgam and RBC restorations in marginal adaptation showed comparable annual failure rates, as shown by Manhart and others[1] in their prospective clinical studies. However, three other published studies reported better longevity of amalgam restorations compared with composite restorations.[37–39]

Despite the evident loss of restorative material at the margins of the existing restorations, observed by macroscopic clinical and photography detection, sealant was able to maintain the marginal integrity of both amalgam and RBC restorations, even though

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no chemical bond occurred between amalgam and pit and fissure sealant.

At baseline, the experimental group presented an alpha score of 77% for the marginal stain parameter. After one year, it increased to 98%, but the next years showed deterioration, achieving 43% at the fifth year. Similarly, the control groups showed the same direction of downgrade, possibly because of the cavity design defects or traumatic occlusal forces that may have been inherited restoration factors were not modified.

Roughness was a parameter that presented improvement only for the replacement group during the first year (from 45% to 73% alpha value). After that, it showed constant deterioration, similar to the other groups. Logically, the sealant treatment was limited to the areas marginal to the restoration, not including other parts of the restoration. Thus, to improve roughness and the margins of the restoration, it is recommended that, in addition to sealing the margins, the surface of the restorations be polished to reduce development of surface roughness, which could potentially increase the adherence of plaque and biofilm to teeth and restorations.[26,40]

Tooth sensitivity showed a slight but not significant improvement in restorations that were sealed when compared with those that were not in the first year. Sensitivity gradually disappeared (100% alpha), and at the fourth and the fifth years, teeth showed no sensitivity. However, no significant differences could be observed among groups, and the limited sensitivity that was present was probably related to other preexisting conditions such as dentin exposure areas or reversible pulpitis.

Restorations with marginal defects without visible evidence of soft dental tissues on the wall or base of the restoration should be monitored, repaired, or sealed instead of replaced.[36] Alternative treatments are specially indicated for the highly dental-motivated patient who presents a good standard of oral health and seeks care regularly,[35] as it is important to consistently check that the sealants are present and functional. In addition, as in any planned procedure, it is important that the patient is completely informed of the advantages and possible disadvantages of the treatment.[35]

The low attrition rates in the current study (5.5% per year) are probably related to the fact that patients have regularly attended the Dental School.

An uncontrolled event of this study was related with the restorations of group C that belonged to the control group and had been previously placed by different and nonstandardized clinicians. Despite this fact, all groups showed a similar trend of restoration downgrade during the observation period.

The application of pit and fissure sealant has been considered a good preventive agent for use against the development and progression of pit and fissure caries.[41] Sealants have also been used to successfully arrest occlusal caries lesions.[42] The present study shows an improvement in marginal adaptation of defective restorations sealed with pit and fissure sealants after five years when compared with restorations that were not treated. It also shows similar results to restorations that were replaced, therefore questioning the need for replacement when sealant is a viable option of treatment.

CONCLUSIONS

The application of a resin sealant at the margin of a defective restoration presented similar marginal adaptation results as restoration replacement after five years. The sealing of defective margins of Class I and II amalgam and RBC restorations is a viable alternative to the replacement of restorations. It increases the restoration longevity with minimal intervention, cost, and trauma to the adjacent tooth structures.

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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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The Clinical Effectiveness of Various Adhesive Systems: An 18-Month Evaluation

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Clinical Relevance

The clinical effectiveness of three different adhesive systems including a self-etching and two etch-and-rinse adhesives was acceptable in noncarious cervical lesions subsequent to 18-month evaluation.

SUMMARY

The aim of this clinical trial was to compare the clinical performance of three different adhesive systems over 18 months in noncarious cervical lesions (NCCLs). Thirty patients, with at least three noncarious cervical lesions, were enrolled in the study. One operator randomly restored a total of 90 lesions with resin composite (Herculite XRV). The restorations were bonded with either Optibond FL (OF), three-step total-etch; Optibond Solo Plus (OS), two-step total-etch; or Optibond All-In-One (OA), one step self-etch. The restorations were clinically evaluated at baseline and after six, 12, and 18 months using the eight United States Public Health Services criteria. Data were analyzed using Friedman and Wilcoxon signed ranks tests ($p<0.05$). After 18 months, the retention rate was (OF) 96.5%, (OS) 93.1%, and (OA) 89.7%. Differences among the three adhesive systems for evaluated criteria were not observed in comparison of the mean Alfa score percentages. There was a significant increase in marginal discoloration for (OA) adhesive after 18 months compared with baseline ($p=0.011$). Other restoration criteria had no statistically significant differences among the three adhesives ($p>0.05$). With the exception of marginal discoloration, the clinical effectiveness of three types of adhesive systems in NCCLs was acceptable after 18 months. However, using the one-step self-etch adhesive may lead to some marginal discolorations.

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INTRODUCTION

Noncarious cervical lesions were used as clinical models to evaluate the performance of adhesive systems because 1) they involve both enamel and dentin margins, 2) they present no macromechanical retention and require at least 50% bonding to dentin, 3) they are widely available and usually found in premolars and anterior teeth with good clinical access, 4) they have the worst long-term prognosis because of their mixed cavity margins and high stress buildup in the cervical area, and 5) preparation and restoration of these lesions are relatively easy, reducing practitioner variability.[1–3] In the restoration of these lesions, a variety of materials such as resin-based composites with diverse bonding characteristics have been used. An important factor in the success of the resin-based composite restorations is the properties of adhesive bonding agents. Resin-based adhesive systems can be classified as either etch-and-rinse systems or self-etch systems. The disadvantages of the etch-and-rinse systems are the technique sensitivity and the likely discrepancy between the extent of demineralization and monomer infiltration and subsequent degradation of these adhesives when they are exposed to the oral environment during passing time.[4–6] The key advantages of self-etch adhesives are their easy and fast application procedures.[7] This approach significantly reduces technique sensitivity. Infiltration of adhesive occurs simultaneously with the etch process; therefore, discrepancy between both processes is low and less time-consuming.[8–10] Knowing the success and longevity of various adhesives enables practitioners to choose the most appropriate material for clinical use. The information on bonding effectiveness of adhesives in laboratory conditions indicates that bond strength of the all-in-one systems to enamel and dentin are not as high as other adhesive systems.[11,12] However, the high success rate of one-step self-etch adhesives in recent clinical trials has been reported.[7,13,14] The proper test to evaluate the dental adhesive is its clinical performance under functional and natural situations.[15] Therefore, this study evaluated the 18-month clinical effectiveness of the one-step self-etch adhesive Optibond All-In-One, the two-step total-etch adhesive Optibond Solo Plus, and the three-step total-etch adhesive Optibond FL in noncarious cervical lesions.

METHODS AND MATERIALS

Patients and Lesions Selection

The participants in this study were 30 patients aged 20 to 50 years who had at least three noncarious, nonsclerotic cervical lesions. The selected teeth had healthy periodontium and contacted the opposing teeth with a normal occlusal relationship. No more than 50% of the lesion's cavosurface margins involved enamel. In the present study, the extension of noncarious cervical lesions in the selected teeth was limited to the buccal surface of teeth without extension into the proximal surfaces, and the teeth had no previous restoration or carious lesion in other surfaces. The depth of the cavities was not more than 2 mm as measured by a probe. In addition, the operating area could be isolated. Patients with severe medical complications, poor oral hygiene, extreme caries susceptibility, or heavy bruxism were excluded from the study. The proposal was approved by the Regional Medical Research Ethics Committee with the registration code of IRCT138709301509N1, and all patients signed a written consent form.

Material Selection

Ninety cervical lesions were restored either with Optibond FL (OF; Kerr Corporation, Orange, Calif, USA), Optibond Solo Plus (OS; Kerr Corporation), or Optibond All-In One (OA; Kerr Corporation). Composition and application procedures of the three adhesives are shown in Table 1. All lesions were restored with a universal microhybrid composite (Herculite XRV, Kerr Corporation).

Restorative Procedures

One experienced operator, who followed standard procedures, placed all restorations. The distribution of the materials and tooth locations were randomized (Table 2). For measuring tooth sensitivity, the teeth were prepared without local anesthesia injection. The cervical lesions were first cleaned using a rubber cup with pumice-water slurry to remove the dental plaque. The internal walls were lightly roughened with a diamond bur (Diatech Dental AG, Swiss Dental Instruments, CH-9435Heerbrugg). Isolation of the tooth was achieved by cotton rolls and retraction cords. Tooth preparation did not include retentive grooves or enamel bevels. No liners or bases were applied. The adhesive systems were applied according to the manufacturer's recommendations (Table 1). The composite resin Herculite HRV (shade A2) was placed in two increments from cervical to incisal and cured using an Optilux 500 light-curing unit (Demetron LC, Kerr Corporation) with a light output of 500 mW/cm^2. Each composite resin layer was polymerized for 20 seconds. After curing, finishing was accomplished using fine-grit diamond burs (Brasseler, Savannah, GA, USA) and

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Table 1: *Adhesives Used in the Study and Application Mode According to the Manufacturer's Instructions*

Adhesive	Composition (Batch Number)	Application Mode
Optibond FL (Kerr Corporation, Orange, CA, USA)	Etchant: 37.5% phosphoric acid Primer: HEMA, GPDM, PAMM, CQ, ethanol, water (3093079); adhesive: TEGDMA, UDMA, Bis-GMA, HEMA, GPDM, filler, CQ (3096500)	Etch with 37.5% phosphoric acid for 15 s, rinse for 15 s and dry for 5 s, apply primer with light brushing motion for 15 s, air-dry for 5 s, apply adhesive with light brushing motion for 15 s, air-dry for 3 s, and light cure for 20 s
Optibond Solo Plus (Kerr Corporation, Orange, CA, USA)	Etchant: 37.5% phosphoric acid; adhesive: Bis-GMA, HEMA, GDMA, GPDM, ethanol, CQ, ODMAB, BHT, fumed silicon dioxide, A174, barium aluminoborosilicate, Na2Si6F (31513)	Etch with 37.5% phosphoric acid for 15 s, rinse for 15 s and dry for 5 s, apply the adhesive and rub for 15 s, dry for 3 s, and light cure for 20 s
Optibond All-In-One (Kerr Corporation, Orange, CA, USA)	Uncured methacrylate ester, ethyl alcohol, water, acetone, monomers, inert mineral fillers, ytterbium fluoride, photoinitiators, accelerators, and stabilizers (3075076)	Shake the bottle for 10 s, apply the adhesive and rub for 20 s, repeat the procedure, air-dry lightly for 5 s, and light cure for 10 s

Abbreviations: A174, gamma-methacryloxypropyltrimethoxysilane; BHT, 2,6-Di-tert-butyl-4-methylphenol; Bis-GMA, bisphenol A glycidyl methacrylate; CQ, camphorquinone; GDMA, glycerol dimethacrylate; GPDM, glycerol phosphate dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; ODMAB, 2-(ethylhexyl)-4-(dimetylamino) benzoate; PAMM, phthalic acid monoethyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

the Sof-lex polishing disc system (Sof-Lex, 3M ESPE, Dental Products, St. Paul, MN, USA) under water cooling to obtain a smooth surface.

Clinical Evaluation Criteria

All restorations were evaluated using the United States Public Health Services (USPHS) criteria (Table 3). Evaluation criteria included color match, marginal discoloration and adaptation, recurrent caries, anatomic form, postoperative sensitivity, retention, and surface roughness. The restorations were examined at baseline (one week later) and six, 12, and 18 months by two calibrated evaluators who were blinded to the adhesive used per lesion and patient. When disagreement occurred during the evaluation, the final decision was made by consensus of both examiners. Tooth sensitivity was assessed by a visual analog scale by questioning the patients after a three-second air blast directed at the restoration site from a distance of 1 cm. After that, scores greater than 2 were accepted as the presence of tooth sensitivity. Tooth vitality and gingival response tests were recorded with a pulp tester and visual inspection and probing at the gingival margins, respectively. Digital color photographs were taken at each recall.

Statistical Analysis

The clinical outcome and durability of the three adhesive systems were compared and analyzed using

Table 2: *Distribution of the Adhesives Among Dental Arches and Postoperative Sensitivity*

Adhesive	Maxillary		Mandibular		Total	Postoperative sensitivity
	Anterior	Posterior	Anterior	Posterior		
Optibond FL	13	3	6	6	28	14
Optibond Solo Plus	18	2	6	6	32	17
Optibond All-In-One	14	4	7	5	30	16
Total	45	9	19	17	90	47

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Table 3: *Using the United States Public Health Services Criteria for Restoration Evaluation*

Criterion	Inspection Method	Score
Color match	Visual inspection with mirror at a distance of 45 cm	Alfa: No mismatch in room light in 3 to 4 s
		Bravo: Perceptible mismatch but clinically acceptable
		Charlie: Esthetically unacceptable (clinically unacceptable)
Marginal discoloration	Visual inspection with mirror at a distance of 45 cm	Alfa: No discoloration anywhere along the margins
		Bravo: Superficial staining (removable, usually localized)
		Charlie: Deep staining (not removable, generalized)
Caries formation	Visual inspection with explorer, mirror, and radiographs	Alfa: No evidence of caries
		Charlie: Evidence of caries along the margins of the restorations
Anatomic form	Visual inspection with explorer and mirror, if needed	Alfa: The restoration is continuous with existing anatomic form
		Bravo: Generalized wear but clinically acceptable (50% of margins are detectable, catches explorer going from material to tooth)
		Charlie: Wear beyond dentino-enamel junction (clinically unacceptable)
Marginal adaptation (marginal integrity)	Visual inspection with explorer and mirror, if needed	Alfa: Undetectable crevice along the margin
		Bravo: Detectable V-shaped defect in enamel only
		Charlie: Detectable V-shaped defect in dentino-enamel junction
Retention	Visual inspection with explorer and mirror	Alfa: Retained
		Bravo: Partially retained
		Charlie: Missing
Surface roughness	Visual inspection with explorer and mirror	Alfa: Restoration is as smooth as the adjacent tooth structure
		Bravo: Restoration is rougher than the adjacent tooth structure
		Charlie: Restoration is rougher than the adjacent tooth structure and contains pits and fissures
Postoperative sensitivity	Asking the patients	Alfa: None
		Charlie: Some

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Table 4: *United States Public Health Services Criteria Acquired at Each Recall for the Studied Parameters*

Parameters	Score	Baseline			6 Mo			12 Mo		
		Optibond FL	Optibond Solo Plus	Optibond All-In-One	Optibond FL	Optibond Solo Plus	Optibond All-In-One	Optibond FL	Optibond Solo Plus	Optibond All-In-One
Color match	A	30	30	30	28	26	25	28	25	24
	B	0	0	0	0	1	2	0	2	3
	C	0	0	0	0	0	0	0	0	0
Marginal discoloration	A	30	30	30	28	26	23	28	25	21
	B	0	0	0	0	1	4	0	2	5
	C	0	0	0	0	0	0	0	0	1
Marginal adaptation	A	30	30	30	27	27	27	27	27	27
	B	0	0	0	1	0	0	1	0	0
	C	0	0	0	0	0	0	0	0	0
Retention	A	30	30	30	28	27	26	28	27	26
	B	0	0	0	1	0	1	0	0	1
	C	0	0	0	0	2	2	1	2	2
Surface roughness	A	30	30	30	28	27	26	28	27	26
	B	0	0	0	0	0	1	0	0	1
	C	0	0	0	0	0	0	0	0	0

Abbreviations: A, Alfa, B: Bravo; C, Charlie.

the Friedman and Wilcoxon signed ranks tests. In this study, $p<0.05$ was considered statistically significant.

RESULTS

Noncarious cervical lesions were restored in 30 patients at baseline; only 29 patients (96.6%) could be evaluated at every recall during the 18-month period. The reason for dropout was traveling and moving of a participant. The USPHS criteria acquired for the changed parameters in three categories of adhesives after six, 12, and 18 months are shown in Table 4. Differences among the three categories of adhesive systems were not observed when comparing the mean Alfa score percentages ($p>0.05$). Retention rates after 18 months were 96.5% for OF, 93.1% for OS, and 89.7% for OA. The differences in retention rates were not statistically significant ($p>0.05$). There was a significant difference in marginal discoloration for OA adhesive after 18 months compared with baseline ($p=0.011$). Even though other restoration criteria had no statistically

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Table 4: Extended.

18 Mo		
Optibon FL	**Optibond Solo Plus**	**Optibond All-In-One**
28	25	24
0	2	3
0	0	0
28	25	20
0	2	6
0	0	1
27	27	27
1	0	0
0	0	0
28	27	26
0	0	1
1	2	2
28	27	26
0	0	1
0	0	0

significant differences among the three adhesives, the three-step total-etch adhesive was found to be superior to the other adhesives after 18 months ($p>0.05$). Gingival inflammation around the restorations was not observed at any recall time.

DISCUSSION

In this clinical trial, a three-step etch-and-rinse, a two-step etch-and-rinse, and a one-step self-etching adhesive from one manufacturer were compared for their clinical effectiveness. All patients received restorations composed of all three adhesives to minimize the influence of the oral environment. At the end of 18 months, the recall rate was 96.6%. Regarding the retention rate, there were no significant differences between the three adhesives. Based on the American Dental Association guidelines, an adhesive material must have a retention failure rate less than 10% at the 18-month recall, and this recall time is sufficient to show the presence of an acceptable seal in clinical tests.[16] In this study, at the end of 18 months, the failure rates were less than the defined border rates in all three adhesives. Optibond FL had the highest retention rate, followed by Optibond Solo Plus and Optibond All-In One. In the clinical study carried out by Reis and others,[17] a higher success rate was recorded for a three-step etch-and-rinse adhesive compared with a two-step etch-and-rinse adhesive, which was in agreement with the results of the present study. Failure rates of adhesives reported by van Dijken and Pallesen[18] were 7.7% in the one-step self-etch adhesive group and 5.6% in the two-step etch-and-rinse adhesive group, respectively. This finding is consistent with our findings that the retention rate of the all-in-one adhesive was lower than other adhesives.

Tooth flexure has been described as either a lateral or axial bending of a tooth during occlusal loading. This flexure produces the maximum strain in the cervical region, and the strain seems to be resolved in tension or compression within local regions, sometimes causing the loss of gingival enamel prisms or failure of bonded class V restorations in preparations with no retentive grooves, the same as tooth preparations in the current study.[19] Another etiological factor for noncarious cervical lesions is the mechanical and chemical wear. Abfraction, abrasion, and erosion are the three main causes of formation of noncarious cervical lesions.[20] Therefore, elimination of the etiological factors along with the restorative procedure is the key to success for treatment of these lesions. Moreover, in incomplete bonded restorations, this flexure may produce changes in fluid flow and microleakage, leading to sensitivity and pulpal inflammation.[13,14,21] In the present study, marginal discoloration was observed only as superficial discoloration (Bravo score) and mostly occurred in the OA group rather than in the OS or OF groups, which is in agreement with the findings of the study by Loguercio and others.[21] They concluded that the higher marginal discoloration in the one-step self-etch adhesives might be due to the inferior etching pattern of these systems. The pH values of OF, OS, and OA adhesives are 1.8, 2.1, and 2.5, respectively.[22] The less acidity of OA could explain the higher marginal discoloration values and

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lower retention rate of this adhesive. Some studies have demonstrated that pretreatment using 37% phosphoric acid can improve retention rates.[23,24] An excess or deficiency of the filling material may contribute to the occurrence of marginal staining. Therefore, it is important for the clinicians to follow the basic rules during adhesive materials placement. One explanation for marginal staining is the degree of conversion that does not occur completely in self-etch adhesives because of the existence of water and more hydrophilic monomers in their content.[25] The hydrophilicity, functionality, size of monomers, and filler content in adhesives affect the water sorption, solubility, crosslink density, and degree of conversion.[25,26] The OS adhesive containing glycerol dimethacrylate monomer and filler showed better clinical results in comparison with OA, indicating higher crosslink density along with an increased degree of conversion in the polymer network structure. In the OF adhesive, the presence of fillers and the use of a hydrophobic layer are the two major reasons for the higher performance compared with the other adhesives. Postoperative sensitivity has been attributed to several factors, such as operative trauma, desiccation, leakage, and other sources.[27–30] The ability of the adhesive layer to coat and bond to the tooth structure plays a key role in reducing sensitivity. In the present study, all three adhesives performed the same in this regard. In addition, Perdigão and others demonstrated that self-etch and total-etch adhesives did not differ with regard to postoperative sensitivity.[31] Other evaluation criteria including caries recurrence and marginal integrity were also rated satisfactorily in three adhesive groups. Further long-term clinical studies are required to confirm the results of the present clinical trial, and the evaluation of clinical performance of the one-step self-etch adhesives with various commercial brands are warranted in future investigations.

CONCLUSIONS

Within the limitations of the present study, it can be concluded that restoration of noncarious cervical lesions with the one-step self-etching adhesive can be an appropriate alternative to more complicated adhesives.

Conflict of Interest

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

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Effect of 10% and 15% Carbamide Peroxide on Fracture Toughness of Human Dentin *In Situ*

LE Tam • P Bahrami • O Oguienko
H Limeback

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Clinical Relevance

There was no significant decrease in mean dentin fracture toughness after 10% and 15% carbamide peroxide bleaching *in situ*. This provides some reassurance that dentin is not overtly weakened by the bleaching protocol used in this study.

SUMMARY

Purpose: **Although damage to the structural integrity of the tooth is not usually considered a significant problem associated with tooth bleaching, there have been some reported negative effects of bleaching on dental hard tissues *in vitro*. More studies are needed to determine whether the observed *in vitro* effects have practical clinical implications regarding tooth structural durability.**

Objectives: **This *in situ* study evaluated the effect of 10% and 15% carbamide peroxide (CP) dental bleach, applied using conventional whitening trays by participants at home, on the fracture toughness of dentin.**

Methods: **Ninety-one adult volunteers were recruited (n ≈ 30/group). Compact fracture toughness specimens (approximately 4.5 × 4.6 × 1.7 mm) were prepared from the coronal dentin of recently extracted human molars and gamma-radiated. One specimen was fitted into a prepared slot, adjacent to a maxillary premolar, within a custom-made bleaching tray that was made for each adult participant. The participants were instructed to wear the tray containing the dentin specimen with placebo, 10% CP, or 15% CP treatment gel overnight for 14 nights and to store it in artificial saliva when not in use. Pre-bleach and post-bleach tooth color and tooth sensitivity were also evaluated using ranked shade tab values and visual analogue scales (VASs), respectively.**

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Within 24–48 hours after the last bleach session, the dentin specimens were tested for fracture toughness using tensile loading at 10 mm/min. Analysis of variance, Kruskal-Wallis, χ^2, Tukey's, and Mann-Whitney U tests were used for statistical analysis. The level of significance was set at $p<0.05$ for all tests, except for the Mann-Whitney U tests, which used a Bonferroni correction for post hoc analyses of the nonparametric data ($p<0.017$).

Results: The placebo, 10% CP, and 15% CP groups contained 30, 31, and 30 participants, respectively. Mean fracture toughness (± standard deviation) for the placebo, 10% CP, and 15% CP groups were 2.3 ± 0.9, 2.2 ± 0.7, and 2.0 ± 0.5 MPa*m$^{1/2}$ respectively. There were no significant differences in mean fracture toughness results among the groups ($p=0.241$).

The tooth sensitivity VAS scores indicated a significantly greater incidence ($p=0.000$) and degree of tooth sensitivity ($p=0.049$ for VAS change and $p=0.003$ for max VAS) in the bleach groups than in the placebo group. The color change results showed generally greater color change in the bleach groups than in the placebo group ($p=0.008$ for shade guide determination and $p=0.000$ for colorimeter determination).

Conclusions: There were no significant differences in *in situ* dentin fracture toughness results among the groups. The results of this study provide some reassurance that dentin is not overtly weakened by the bleaching protocol used in this study. However, the lack of a statistically significant difference cannot be used to state that there is no effect of bleach on dentin fracture toughness.

INTRODUCTION

Tooth bleaching is a popular procedure that can be prone to overuse in an attempt to achieve a whiter tooth color, either by using a bleach concentration that is too high or by bleaching for a prolonged period of time. However, it is not known whether the active ingredients in tooth bleaching materials (hydrogen peroxide or carbamide peroxide [CP]) damage the structural integrity of the tooth. Thus, identifying the short- and long-term effects of tooth bleaching is a targeted research priority on the American Dental Association Research Agenda.[1] Although many studies have investigated the effects of tooth bleaching on enamel and dentin surface properties, such as hardness,[2–4] surface morphology,[5–7] bonding,[3,4,8–10] surface demineralization,[11,12] and abrasion/erosion,[13] relatively fewer studies have investigated the effects of tooth bleaching on enamel and dentin mechanical properties, such as strength or fracture toughness.[14,15]

It has been reported that the flexural strength and modulus of bovine dentin decreased after an *in vitro* direct daily application of carbamide peroxide.[16] Significant reductions in tensile and shear strengths of dentin were reported after an *in vitro* direct intracoronal bleach application of 30% hydrogen peroxide.[17] It has been shown in another *in vitro* study that the fracture toughness of dentin was significantly reduced by the indirect (through intact enamel) application of peroxide bleaching agents (this represents a simulation of clinical bleaching of teeth with full enamel coverage) and by a direct bleach application method to dentin, and that fracture toughness was further reduced with a longer application time period (8 weeks vs 2 weeks) and a higher (16% vs 10%) bleach concentration.[18] The clinical relevance of these *in vitro* studies is uncertain. The fracture resistance of endodontically treated teeth was reported to decrease after an internal and external bleaching procedure.[19] However, the observed decrease in tooth fracture resistance was attributed more to a bleach-induced decrease in bond strength rather than a bleach-induced effect on the tooth structure itself. In the clinical setting, where the estimated number of tooth bleachings performed on or by patients ranges in the millions, there have been no published reports of tooth fractures attributable to tooth bleaching procedures. *In situ* or *in vivo* studies are needed to determine whether the observed *in vitro* effects have practical clinical implications regarding tooth structural durability.

The objective of this study was to determine the effect of dental bleaches, applied in a conventional manner by patients, on the fracture toughness of dentin placed *in situ*. If a decrease in *in situ* dentin fracture toughness or surface hardness is found, the results of this study would provide valuable confirmation to the *in vitro* literature that suggests that tooth weakening may occur as a result of direct bleach treatment. The null hypothesis for this study was that bleaching has no effect on the fracture toughness of dentin *in situ*.

Tooth sensitivity and color changes were also evaluated in this study to confirm the bleaching effect for each patient.

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MATERIALS AND METHODS

Ethics approval for the collection of teeth and for this *in situ* study was obtained (University of Toronto Office of Research Ethics Protocol #21379 and #24941, respectively).

Human molars (extracted within 3 months of the experiment and stored in 1% chloramine solution) were collected to provide the dentin for testing. One dentin specimen was obtained from each tooth. Compact tension test specimens (Figure 1), based on an American Society for Testing and Materials standard specimen[20] and described previously,[18,21] were prepared from the dentin below the occlusal enamel initially using a water-cooled, low-speed diamond saw (Buehler Ltd, Lake Bluff, IL, USA), keeping the location and orientation of the dentin standardized. High-speed dental instrumentation was then used to form the rectangular block-shaped specimen with approximate dimensions 4.5×4.6×1.7 mm. A central notch was made with a 0.28 mm thick diamond disc (ThinFlex, Premier Products Co, Plymouth Meeting, PA, USA) and sharpened with a razor blade to act as a stress concentrator. A tungsten carbide drill bit (LA ¼ round, catalog 00400327, Brasseler, Savannah, GA, USA) was used to drill two cylindrical holes, approximately 0.8 mm in diameter, in each specimen to provide means of attachment for mounting. A micrometer (Digimatic Caliper, Mitutoyo Corporation, Kanagawa, Japan) was used to measure specimen dimensions (a, B, W, H, and N) to the nearest 0.01 mm. Equipment, instruments, and preparation materials were disinfected, sterilized, and/or disposed for each individual specimen's preparation following standard universal precautions infection-control procedures. Finally, the dentin specimens were stored in artificial saliva[22] in individual containers and sterilized using gamma-irradiation at 2.5 MRad for 1500 minutes (cobalt source [Co-60] from GammaCell-220, Atomic Energy of Canada Limited, Kanata, Canada).

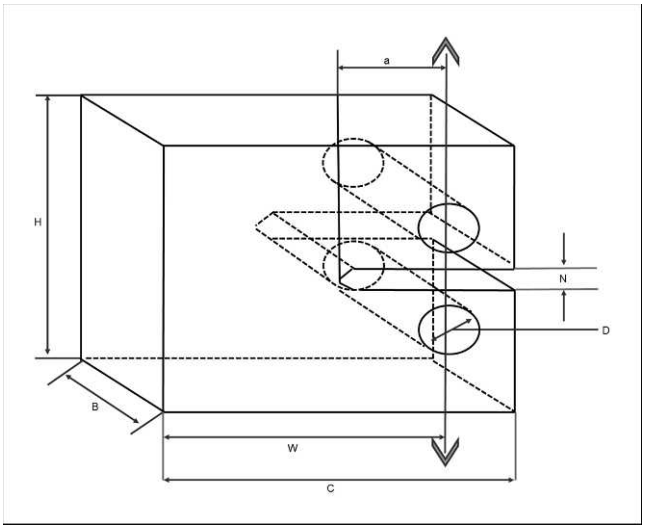

Figure 1. *Diagram of fracture toughness specimen. Total width (C) ≈ 4.6 mm. Net width (W) ≈ 3.75 mm. Height (H) ≈ 4.5 mm. Thickness (B) ≈ 1.7 mm. a/W ratio ≈ 0.45–0.55. Notch width (N) < 0.65W. Effective notch length (a) = 0.25W – 0.4W. Hole diameter (D) ≈ 0.8 mm. B/W ratio = 0.25–1.25. Large arrowheads indicate direction of tensile loading.*

The selected sample size (n=30/group) was based on a sample-size calculation of 27 using a Type 1 error alpha = 0.05, Type 2 error beta = 0.2, a high standard deviation (0.8 $MPa*m^{1/2}$) value compared with those generally obtained in previous *in vitro* dentin fracture toughness testing done by the author to reflect the higher variance seen *in situ* compared with *in vitro*, and a smallest difference of clinical interest value = 0.7 $MPa*m^{1/2}$. The criteria for acceptance into the study were as follows: adults (≥18 years old), willingness to participate in the study, a noncontributory medical history (not pregnant, not lactating), and a noncontributory dental history (ie, no xerostomia, no untreated carious lesions, not presently undergoing orthodontic treatment). Compensation of $20 was provided for each participant in the study. Participants who had recently (within 1 year) bleached their teeth were not included in the study. The one-year exclusion period was based on general current recommendations for frequency of tooth bleaching (based on need for re-bleaching after color regression).

The bleach materials included 10% or 15% CP (Opalescence or Opalescence 15% PF, Ultradent Products Inc, South Jordan, UT, USA). A placebo gel (Ultradent Products), without the active CP, was used as the control. The investigators gave participants the opportunity to join the placebo, 10% CP, or 15% CP groups, and recruitment continued until the approximate selected sample size was reached for each group.

Custom-made bleaching trays were constructed to deliver the bleach to the participant's teeth and to hold the dentin specimen. LC Block Out Resin (Ultradent Products) was applied to the facial surfaces of the incisors and premolars on the dental stone model to an approximate thickness of 0.5 mm. A composite resin block with the approximate dimensions of the dentin fracture toughness specimen was bonded onto the buccal surface of a maxillary premolar on each dental stone model to create a space for the actual dentin specimen in the bleaching tray. Vinyl tray material (Sof-Tray Regu-

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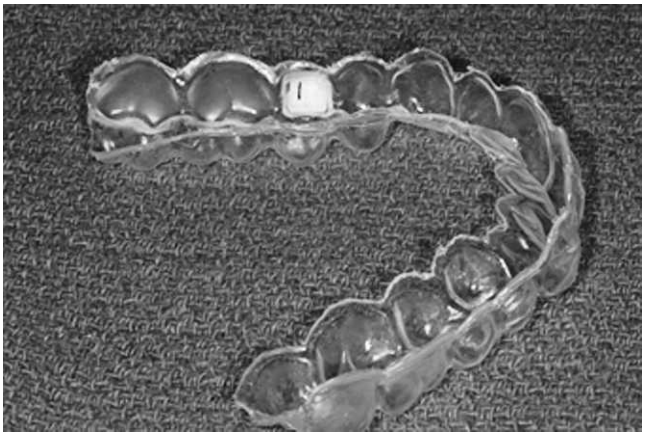

Figure 2. *The dentin fracture toughness specimen is shown sutured in place within the custom-made bleach tray.*

lar 0.35", Ultradent Products) was then vacuum-formed to the stone model and trimmed along the gingival margins. A randomly selected dentin fracture toughness specimen was inserted into the tray in the space created by the composite resin block and sutured to the tray (4-0 silk black-braided, Ethicon Inc, Somerville, NJ, USA) (Figure 2).

All participants received a demonstration and instructions concerning the proper use of the bleaching agents. To standardize participants' oral hygiene regimens, they were asked to not use dentifrices that contained whitening agents. Participants were instructed to wear the bleaching tray containing the dentin specimen overnight for 14 nights to simulate a typical at-home bleaching regimen. At the end of each daily bleach treatment, the participants rinsed the tray and dentin specimen with tap water to remove all external traces of bleach and stored the tray and dentin specimen in artificial saliva until the next bleach treatment. If the participants experienced tooth sensitivity, they were advised that they could choose to skip one or two days of bleaching. Participants were also advised that they were free to stop participating at any time during the study. Participants were asked to make a daily record of the following on a provided log form: (1) the number of hours of bleaching done and (2) the degree of tooth sensitivity experienced as shown on a 10-mm visual analogue scale (VAS) (Table 1).

Tooth sensitivity was also tested by before-bleaching and after-bleaching cold testing. The cold testing involved placing 0.3 mL of 7°C glycerine gel to the middle of the facial surface of the lateral incisor for 3 seconds. At the end of 3 seconds, the gel was wiped off, and the patient was asked to score the lateral incisor tooth sensitivity on a VAS. The daily tooth sensitivity and the cold test VASs were measured to the nearest millimeter to determine the change in tooth sensitivity score for the cold test after the study compared with before the study (VAS change = VAS score for cold test after the study – VAS score for cold test before the study), the maximum tooth sensitivity score during the study period (max VAS), and the percentage of participants who experienced no sensitivity (where VAS=0 for each treatment day).

Pre- and post-bleach tooth shades for one central incisor were recorded by visual matching and by using an Easy Shade colorimeter (Vident, Brea, CA, USA). Shade selection was carried out under similar clinical conditions for each participant in a neutral-colored room. The visual evaluation was made by

Table 1. *Form Participants Used to Log Daily Records of Number of Hours of Tray Wear and Degree of Tooth Sensitivity Based on a 10 mm Visual Analogue Scale (VAS)*

Date	Hours of Tray Wear	Tooth Sensitivity after indicated hours of tray wear: Please mark an X on the line to represent the maximum degree of tooth sensitivity experienced during the day. "A" represents no sensitivity and "B" represents extreme sensitivity.
		A tooth sensitivity B

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Table 2. *Rank Scores Assigned to Each Shade Tab According to Value*

	Darkest----------																			Lightest
	Classic Shade Guide[1]																Bleach Shade Guide[2]			
Shade Tab	C4	A4	C3	B4	A3.5	B3	D3	A3	D4	C2	C1	A2	D2	B2	A1	B1	040	030	020	010
Rank	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

[1] Vita Classic Shade Guide (Vident, Brea).
[2] *Bleach Shade Guide (Ivoclar Vivadent).*

comparing the shade tabs (Vita Classic shade guide, Vident, and Bleach Shade Guide, Ivoclar Vivadent, Amherst, NY, USA) with the middle third of the selected upper central incisor by the same investigator before and after treatment. There was no attempt to use or calibrate more than one investigator for pre- and post-bleach shade tab color assessment. A custom-made self-cure resin jig was used to ensure standardized positioning of the Easy Shade probe. The 16 shade tabs from the Vita Classic guide and 4 shade tabs from the Bleach Shade guide tabs were ranked according to value from 20 (highest value = 010) to 1 (lowest value = C4) (Table 2). The colorimeter shade results were limited to the 16 Vita Classic shade tabs only. Color change was determined by subtracting the pre-bleach shade from the post-bleach shade for each participant. Pre- and post-bleach digital photographs of the anterior teeth (in a fixed position) were also taken under standardized lighting conditions in a neutral-color room.

Within 24 hours after the last bleaching session, the dentin fracture toughness specimens and log forms were returned to the investigator, and the post-bleach tooth color and sensitivity assessments were done. Within 24 to 48 hours after the last bleaching session, the dentin specimens were mounted on an Instron universal testing machine (Model 4301, Instron Corp, Canton, MA, USA) for fracture toughness testing using a custom-designed mounting jig. Tensile loading was applied at a rate of 10 mm/minute until specimen fracture. The force recorded at fracture was used to calculate fracture toughness, K_{1C}.

The patient age, number of nights and hours of bleach or placebo treatment, fracture toughness results, and tooth sensitivity scores (VAS change and max VAS) were analyzed using analysis of variance ($p<0.05$). The Kruskal-Wallis ($p<0.05$) test was performed to analyze the color data, and χ^2 ($p<0.05$) were conducted to compare the gender and incidence of no tooth sensitivity (VAS = 0). Tukey's test ($p<0.05$) and Mann-Whitney U test with a Bonferroni correction ($p<0.017$) were used for post hoc analyses of the parametric and nonparametric data, respectively.

RESULTS

There were 30, 31, and 30 participants in the placebo, 10% CP, and 15% CP groups, respectively. The gender, mean age, and mean number of nights and hours of bleach treatment for each group are shown in Table 3. There were no significant differences among the groups for gender ($p=0.519$),

Table 3. *Gender and Age Distribution of Groups and Number of Nights and Hours of Placebo or Bleach Application (mean ± standard deviation)*[a]

	No.	Men	Women	Age, years	No. of Nights	No. of Hours
Placebo	30	14	16	29.4 ± 8.0^{y}	13.8 ± 0.7^{y}	85 ± 22^{y}
10% CP	31	10	21	27.2 ± 6.4^{y}	13.7 ± 1.8^{y}	$91 \pm 19^{y,z}$
15% CP	30	12	18	26.3 ± 6.0^{y}	14.1 ± 0.7^{y}	101 ± 17^{z}

[a] *Results denoted with the same superscript letters indicate no significant difference ($p>0.05$).*

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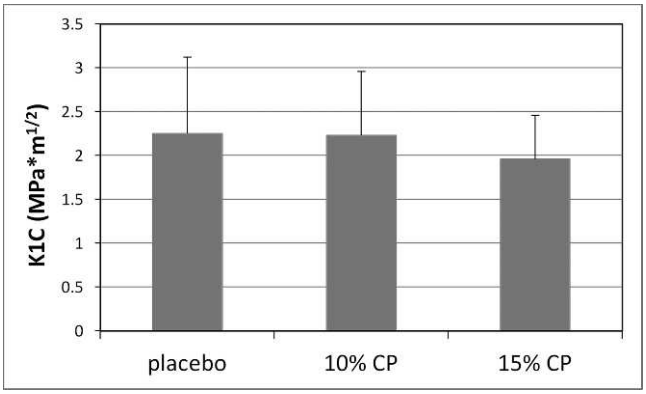

Figure 3. *Mean fracture toughness (K_{1C}) (± standard deviation) for the placebo, 10% CP, and 15% CP groups. There were no significant differences in fracture toughness results among the groups (*p*=0.241).*

age (p=0.216), and nights bleached (p=0.381). There was a significant difference in the number of hours bleached (p=0.007); the 15% CP bleach group wore the tray for a significantly greater number of hours than the placebo group.

The K_{1C} results are shown in Figure 3. Mean (± standard deviation) fracture toughness results for the placebo, 10% CP, and 15% CP groups were 2.3 ± 0.9, 2.2 ± 0.7, and 2.0 ± 0.5 MPa*$m^{1/2}$, respectively. There were no significant differences in fracture toughness results among the groups (p=0.241).

Tooth sensitivity data from the VAS scores are tabulated in Table 4. Compared with the placebo group, one or both of the bleach groups reported a significantly greater increase in the tooth sensitivity score for the cold test after the study compared with before the study (VAS change, p=0.049), a higher degree of maximum tooth sensitivity during the study period (max VAS, p=0.003), and a smaller percentage of sensitivity-free patients (VAS = 0, p=0.000).

Table 4. *Tooth Sensitivity Results*[a]

Group	VAS Change[b]	max VAS[c]	% VAS = 0[d]
Placebo	$0 + 8^{y}$	12 + 23	75
10% CP	$2 + 13^{y,z}$	$36 + 32^{y}$	17^{y}
15% CP	$7 + 13^{z}$	$31 + 28^{y}$	10^{y}

[a] *Results denoted with the same superscript letters indicate no significant difference (p>0.05 for VAS change and max VAS; p>0.017 for % VAS).*
[b] *Difference (mean + standard deviation) in VAS scores, in millimeters, for the cold test after the study compared with before the study (VAS change = VAS score for cold test after the study – VAS score for cold test before the study).*
[c] *Maximum tooth sensitivity VAS score (mean ± standard deviation) in millimeters, reported during the study period (max VAS).*
[d] *Percentage of participants who experienced no sensitivity (VAS = 0 for each treatment day).*

Tooth (central incisor) color data are shown in Table 5. There was no significant difference in initial (pre-bleach) tooth color among the groups when determined visually using the shade guides (p=0.175) or the Easy Shade colorimeter (p=0.182). There were significant differences in color change results when determined visually using the shade guides (p=0.008) or the Easy Shade colorimeter (p=0.000); color changes were generally greater in the bleach groups than in the placebo group. Although the color of the dentin specimens was not specifically measured, whitening of the dentin specimens in the bleach groups was evident.

DISCUSSION

This study investigated the effects of a placebo, 10% CP, and 15% CP treatment on dentin fracture toughness *in situ* and assessed tooth color change and tooth sensitivity *in vivo*. In contrast to the number of *in vitro* studies that have assessed the effect of tooth bleaching on enamel and dentin, the number of *in situ* studies is relatively small. A few *in situ* studies have measured the effects of tooth bleaching on enamel microhardness and have reported no significant differences.[23–26] The effect of tooth bleaching on the fracture toughness of dentin has not been studied *in situ*.

The participants and investigators were not blinded to the treatment used in this study. The lack of blinding was recognized as a potential source of bias, especially during shade assessment by the investigator and during recording of tooth sensitivity by the participant. However, it was decided to give participants the opportunity to choose their treatment group. This precluded the possibility of patient blinding. Furthermore, we considered that blinding throughout the experimental period would have been difficult because teeth generally do become noticeably whiter and more sensitive in the bleach groups. The main objective of this study was fracture toughness assessment, and the lack of blinding was not expected to have a significant effect on the eventual fracture toughness tests, in which the results are not subjective.

Fracture toughness is an intrinsic material property that measures the fracture resistance of the material. A small reduction in the fracture resistance of the tooth could have a great impact over the

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Table 5. *Tooth (Central Incisor) Color Data*[a]

Group	Pre-bleach Shade Rank (Visual)[b]	Color Change (Visual)[c]	Pre-bleach Shade Rank (Easy Shade)[b]	Color Change (Easy Shade)[c]
Placebo	14.7 ± 2.4^{y}	1.1 ± 2.0^{y}	12.5 ± 4.2^{y}	0.3 ± 0.9^{y}
10% CP	15.0 ± 3.2^{y}	2.8 ± 2.8^{z}	13.6 ± 3.5^{y}	$1.3 \pm 1.3^{y,z}$
15% CP	13.9 ± 2.8^{y}	3.2 ± 2.3^{z}	13.9 ± 2.3^{y}	1.7 ± 2.0^{z}

[a] *Results denoted with the same superscript letters indicate no significant difference ($p>0.017$).*
[b] *Pre-bleach shade ranks (mean ± standard deviation) as determined by the operator (visual) or the Easy Shade colorimeter (Vident) using ranked shade tabs (Vita Classic Shade Guide,Vident, Brea or Bleach Shade Guide, Ivoclar Vivadent). A higher shade rank represents a lighter shade.*
[c] *Color change results (mean ± standard deviation) were determined by subtracting the pre-bleach shade rank from the post-bleach shade rank for each participant.*

lifetime of the tooth as a result of fatigue and crack propagation. Therefore, it is important to characterize any changes to the structural integrity of dentin that could occur as a result of bleaching treatment. The previously reported *in vitro* reductions in dentin fracture toughness observed as a result of bleach treatment[18] were not confirmed by this *in situ* study. Factors that may have contributed to the different *in vitro* and *in situ* dentin fracture toughness results include saliva and bleach considerations.

The *in vitro* specimens were stored in 37°C artificial saliva during the entire study period,[18] while the *in situ* specimens were exposed to human saliva during bleach treatment and stored in room-temperature artificial saliva when not undergoing bleach treatment. Human saliva has a buffering action because of the bicarbonate and phosphate systems, and it contains inorganic electrolytes, such as calcium phosphorus and fluoride, as well as enzymes and bacteria. Smidt and others[27] stated that the buffering capacity and remineralization potential of saliva *in vivo* could overcome the detrimental effects of bleach on enamel microhardness and surface morphology observed *in vitro*. In an *in situ* study of enamel microhardness, it was concluded that saliva led to mineral reposition on bleached enamel surfaces and reestablishment of hardness values similar to those of non-bleached specimens.[24] It is possible that human saliva played a role in this *in situ* study by preventing or reversing a potential reduction in dentin fracture toughness caused by bleach application.

Although there would have been significant variation in the amount of bleach applied to the bleaching tray because of participant variability, overall the *in situ* dentin specimens were probably exposed to a smaller amount of bleach than the *in vitro* specimens. The *in vitro* dentin specimens were exposed to bleach on all of its surfaces.[18] The *in situ* dentin specimens in our study were exposed to direct bleach application primarily on one surface only. The bleach on the *in situ* dentin specimens was also subject to washout, salivary dilution, and salivary antioxidants. The reduced amount of bleach on the *in situ* dentin specimens may partially explain why there was no significant difference in dentin fracture toughness among the groups in this study. However, there was sufficient bleach in the tray to cause a bleach effect, as evidenced by the color change and tooth sensitivity results.

Dentin is chromatic and enamel is not. Dentin is therefore the target tissue for bleaching. Studies of direct applications of bleach to dentin are relevant because dentin can be exposed to direct bleach application in clinical situations. It is impossible to avoid direct contact of bleach to dentin when there is exposed dentin during a typical home bleaching treatment using a tray or strips. Two common clinical situations in which dentin is exposed are occlusal attrition or root recession. In those situations, one or two surfaces of dentin would be exposed to direct bleach application. This *in situ* study mimicked these clinical situations better than the previous *in vitro* study[18] by limiting the direct application of bleach primarily to one dentin surface.

As expected, the bleach groups showed significant increases in tooth sensitivity results and color change results compared with the placebo group, and there was a trend for greater color change and tooth sensitivity with the greater bleach concentration. The standard deviations for the tooth sensitivity and color change results were high, suggesting a wide range of potential bleach treatment results. The lack of significant difference in color change (Easy Shade) between the placebo and 10% CP group

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was probably because of the high standard deviation in the color change results.

The number of total bleaching hours for the 15% CP group was significantly greater than that for the placebo group. It is likely that the patients in the 15% CP group were more motivated to wear the bleaching tray for longer periods of time because they perceived that their teeth were becoming whiter. The greater number of bleach hours, in addition to the higher bleach concentration, for the 15% CP group may have further contributed to these participants' increased tooth sensitivity and color change results compared to the placebo group.

The design of this *in situ* study is more clinically relevant than the design of *in vitro* studies. The results of this study therefore could provide some reassurance that dentin is not overtly weakened by the bleaching protocol used in this study. However, the lack of a statistically significant difference cannot be used to state that there is no effect of bleach on dentin fracture toughness (this would risk committing a Type 2 error). An inadequate sample size or study design may have contributed to the lack of significant findings. The *in situ* specimens were subject to greater variability than the *in vitro* specimens in the form of different tray-wear patterns by the different participants, fluctuating intraoral temperatures, and varying intraoral conditions. An *in vivo* situation would add even more variability. Vital teeth would have an outward movement of fluid through dentinal tubules, which would tend to expel and buffer the applied bleach. Further studies with a greater sample size or different study design are needed to find more evidence to accept or reject the null hypothesis.

Although there was no statistical difference, the results of this study did show a slight trend for reduced dentin fracture toughness with the higher bleach concentration, which is in accordance with previously reported *in vitro* results.[18] Tooth bleaching materials are available over-the-counter and patients may overuse these products in an attempt to further whiten their teeth. Clinically, it is quite common for a patient to repeat the bleaching procedure several times for several weeks or to use higher bleach concentrations in order to achieve a satisfactory lightening of tooth color. It is not known whether repeated bleaching or use of a higher bleach concentration would cause a significant weakening of the dentin. The results of this study cannot be extrapolated to bleach concentrations or application times higher or longer than those used in this study. Until the specific effects of tooth bleach on dentin are clarified, it remains prudent to keep bleaching concentrations and times to a minimum and to avoid direct application of bleach to areas of exposed dentin, such as in gingival recession or occlusal attrition cases, whenever possible.

CONCLUSION

1. Mean fracture toughness ($\pm$ standard deviation) for the placebo, 10% CP, and 15% CP groups were 2.3 ± 0.9, 2.2 ± 0.7, and 2.0 ± 0.5 MPa*$m^{1/2}$, respectively. There were no significant differences in *in situ* dentin fracture toughness results among the groups ($p=0.241$). The results of this study therefore could provide some reassurance that dentin is not overtly weakened by the bleaching protocol used in this study. However, the lack of a statistically significant difference cannot be used to state that there is no effect of bleach on dentin fracture toughness.
2. Compared with the placebo group, the bleach groups reported a significantly greater increase in the tooth sensitivity score for the cold test after the study compared with before the study ($p=0.049$), a higher degree of maximum tooth sensitivity during the study period ($p=0.003$), and a fewer number of sensitivity-free days ($p=0.000$).
3. There was a significant difference in color-change results among the groups when measured by visual shade-matching ($p=0.008$) and by the Easy Shade spectrophotometer ($p=0.000$).

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Conflict of Interest Declaration

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

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Comparison of Acid Versus Laser Etching on the Clinical Performance of a Fissure Sealant: 24-Month Results

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Clinical Relevance

Etching of enamel with either acid or laser provides similar clinical performance of fissure sealants.

SUMMARY

Objective: **To compare the clinical performance of a pit and fissure sealant placed with the use of different enamel preparation methods, i.e. acid or Er,Cr:YSGG laser etching, over 24 months.**

Methods: **Sixteen subjects (15 female, 1 male) with no restorations or sealant present on their fissures and no detectable caries participated. Using a table of random numbers, a total of 112 sealants (56 with acid-etching, 56 with laser etching) were placed on the permanent premolar and molar teeth. All restorative procedures except for application of the laser were performed by the same dentist. After completion of the fissure preparation either with acid or laser, the adhesive was applied; then a pit and fissure sealant, Clinpro Sealant, was placed and polymerized. Clinical evaluations were done at baseline and at 6-, 12-, 18-, and 24-month follow-up visits by two calibrated examiners, who were unaware of which etching method had been used. The retention of sealants and caries were evaluated with the aid of a dental explorer and an intra-oral mirror. Each sealant was evaluated using the following criteria: 1=completely retained; 2= partial loss; 3= total loss. The Pearson chi-square test was used to evaluate differences in**

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the retention rates among the sealants used with different etching methods.

Results: **All patients attended the 24-month follow-up visit and all sealants were evaluated (total recall rate 100%). At the end of 24 months, 83.9% of the sealants from laser group and 85.7% of those from acid-etch group were recorded as "completely retained". There were no statistically significant differences in retention rates among the preparation methods after all evaluation periods ($p>0.05$). No statistically significant differences were found between the retention rates of premolar and molars at each evaluation period. No secondary caries was detected in association with any sealants. Conclusion: The clinical performance of fissure sealants placed after acid or Er,Cr:YSGG laser etching was similar.**

INTRODUCTION

Pit and fissure sealants are one of the most highly recommended and widely accepted preventive procedures; they were first reported by Cueto and Buonocore[1] in 1967.[2] It has been shown that fissure sealants are highly effective in preventing dental caries, reducing caries in the pits and fissures.[3] By late adolescence, almost 70% of youths have experienced dental caries. Most of these carious lesions are found in the pits and fissures of permanent posterior teeth, with molars being the most susceptible to caries.[4]

The effectiveness of a sealant is related to its retention, and a retained sealant has been shown to be 100% effective.[5] Retention rates differ according to the proper isolation of the working field, viscosity of the sealant material, preparation of enamel surfaces, and use of adhesive system.[6] Etch-and-rinse adhesives are the commonly used adhesives prior to the application of fissure sealants. Many studies have confirmed the benefits of adhesive systems used under sealants. Hitt and Feigal[7] first reported the benefit of adding an adhesive between the etched enamel and the sealant as a way of optimizing bond strength. Other studies also have shown that application of an adhesive under sealants increases bond strength,[8,9] reduces microleakage,[10] enhances flow of resin into fissures,[11] and improves short-term clinical success.[12]

The acid-etch technique is a well-accepted and standard method for roughening enamel surfaces. However, remaining debris and pellicle might not be removed from the base of fissures by the conventional prophylaxis and the etching procedures.[13,14] Therefore, alternative methods have been proposed for preparing fissures other than acid etching for sealant retention.[15–17] The use of a laser has been suggested as a pretreatment method to roughen enamel. Laser application in dentistry has been a research interest for the past 30 years and recently has risen in popularity. The main advantages that have been described with the use of laser technology are the lack of vibration and pain during the preparation of the tooth and no need for local anesthesia.[18,19] Previous lasers have been reported to produce major thermal effects on dentin, including cracks in the surrounding tissues, as well as increases in pulpal temperature.

With the development of the erbium, chromium: yttrium-scandium-gallium-garnet (Er,Cr:YSGG) laser, this problem has been solved. An Er,Cr:YSGG laser, with a 2.78 μm wavelength emission, can ablate dental hard tissue effectively because of its high absorbability in both water and hydroxyapatite. Moreover, no thermal effects on pulp tissue have been reported due to its water-cooled system, and this laser can be used in wet conditions.[20] The main advantage of the laser-etched surface is acid resistance. As the calcium/phosphorus ratio changes with the laser application, the enamel becomes more resistant to caries attack.[21,22] Considering these facts, the use of the Er,Cr:YSGG laser in fissure sealing is of increasing interest.[23] There are conflicting results about the effectiveness of laser etching during fissure preparation. While some authors[24–26] have reported that acid and laser etching cause similar results in terms of marginal adaptation and microleakage, some of them recommended the use of acid after laser application and also stated that the laser etching did not eliminate the need for acid etching.[27–29] Therefore, the search continues for the most effective enamel surface preparation to enhance sealant integrity and retention.

To the best of the authors' knowledge, only one clinical study has compared the retention rates of sealants placed after acid or Er,Cr:YSGG laser etching, and this clinical study was only over 14.5 months.[30] The aim of our 24-month clinical study was to evaluate the clinical performance of a fissure sealant, Clinpro, using two different enamel preparation methods, acid and laser etching. The null hypothesis tested was that no differences would be found between the two preparation methods.

MATERIALS AND METHODS

Subjects were recruited from among patients seeking routine dental care at the conservative dentistry

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clinics at Hacettepe University, Faculty of Dentistry. A total of 112 sealants, distributed in 63 molars and 49 premolars, were placed in 16 patients comprising 15 women and 1 man with a mean age of 21 years (range 20 to 23 years). The patients had good general and oral health and hygiene and no caries, bruxism, malocclusion, previously placed restorations, or allergy to resins. The protocol and consent form for this study were reviewed and approved by the University Human Ethics Committee, and written informed consent was obtained from all patients.

After taking bitewing radiographs of molar and premolars, the fissures of the teeth were cleaned with a slurry of pumice applied with a bristle brush in a slow-speed handpiece to remove salivary pellicle and any remaining plaque. A table of random numbers was used to assign the teeth for etching with either acid or laser. The teeth were isolated with cotton rolls and suction. For the acid-etch group, the fissures were etched with 35% phosphoric acid (Scotchbond, 3M ESPE, Seefeld, Germany) for 30 seconds, rinsed for 15 seconds, and dried for a few seconds until the surface was chalky white. For the laser group, fissures were prepared with an Er,Cr:YSGG laser system (Waterlase, BIOLASE Technology, San Clemente, CA, USA) emitting photons at a wavelength of 2.78 μm. Laser irradiation at 1.25 W (65% air and 75% water), in a noncontact mode, with a repetition rate of 10 Hz, was used. The treatment was performed with a 600-μm diameter tip aligned perpendicularly to the target area at a distance of 1–2 mm from the surface. The duration of exposure depended on the time needed to guide the laser beam evenly across the pits and fissures to be irradiated. After preparation of the fissures, an etch-and-rinse adhesive, Adper Single Bond 2 (3M ESPE), was used according to the manufacturer's instructions. The Clinpro Sealant (3M ESPE) was then applied to the fissures of teeth. To prevent voids and air entrapment, the sealant was gently teased through the fissure with the tip of a periodontal probe and was then polymerized using a quartz-tungsten-halogen light (Hilux, Benlioglu, Ankara, Turkey) for 40 seconds. Light output of the curing unit was found to exceed 550 mW/cm^2 prior to and after the study, as verified with a radiometer (Curing Radiometer Model 100; Demetron Research Corp, Danbury, CT,USA). The occlusion was checked with articulation paper. Finishing and polishing were performed using fine-grit diamond burs (Diatech, Swiss Dental, Heerbrugg, Switzerland) and rubber cups (Edenta AG, Au, Switzerland). The same dentist performed all operative procedures, except for the laser application.

All patients were available for all evaluations. Two calibrated examiners, who were unaware of which preparation method had been used and who were not involved in the treatment procedures, evaluated the restorations at baseline, and at 6-, 12-, 18-, and 24-month follow-up visits. The retention of sealants and caries occurrence was evaluated with the aid of a dental explorer and an intraoral mirror and visual inspection. The sealants were evaluated in terms of caries formation as present or absent and retention as:

1 = completely retained (CR)

2 = partial loss (PL)

3 = total loss (TL)

The Pearson chi-square test was used to evaluate the differences in retention rates between the two different etching methods at a 5% level of significance. Future follow-up visits at 36 months and 48 months are also planned.

RESULTS

Sixteen patients participated in this clinical study and all of them were available for all evaluations (total recall rate was 100%). Table 1 shows the distribution of preparation methods with regard to premolar and molar teeth.

The distribution of sealant retention rates is displayed in Table 2. A total of 112 restorations (56 for acid, 56 for laser) for 16 patients were evaluated at the 6-monthfollow-up visit. Only one fissure sealant placed after acid etching was totally lost, and two fissure sealants placed after laser application were partially lost after 6 months.

After 12 months, 111 fissure sealants of 16 patients were available for evaluation. No statistically significant differences were found between the retention rates of the acid group (91.1%) and the laser group (91.1%).

At the 18-monthfollow-up visit, 101 teeth were fully sealed with fissure sealant. The retention rate in the acid-etch group was 89.2%, while it was 91.1% in the laser group. Four sealants from each group were evaluated as partially lost. Two sealants from the acid-etch group were completely lost, while no sealants from the laser group were lost.

At the end of 24 months, 47 sealants from the acid group and 48 sealants from the laser group were

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Table 1: *Distribution of Preparation Methods With Regard to Premolar and Molar Teeth*

	Acid	Laser	Total
Premolar	24	25	49
Molar	32	31	63
Total	56	56	112

evaluated as completely retained; the retention rates were 83.9% and 87.5%, respectively. Only two sealants from the acid group were totally lost during the study, while none of them were "totally lost" in the laser group. Seven sealants from the acid-etch group and eight sealants from the laser group were partially lost. There were no significant differences in retention rates between these two preparation methods after 6, 12, 18, or 24 months.

The distributions of retention rates of premolars and molars are shown in Tables 3 and 4. No statistically significant differences were found between the retention rates of premolars and molars at each evaluation period. There was no caries development during the evaluation periods.

DISCUSSION

This clinical study examined the retention of a fissure sealant used with an adhesive system after acid or Er,Cr:YSGG laser etching in young adults. It is known that early placement of sealants protects teeth from caries development. Since recently erupted teeth are immature and less mineralized, the most appropriate time for application of fissure sealants is soon after the eruption of the permanent teeth.[31] Because clinical studies with children have some difficulties like isolation of the teeth, cooperation of the children, and bringing the children to scheduled appointments, this study evaluated sealant loss in patients with a mean age of 21 years. In 2002, Feigal[32] reported that caries risk on surfaces with pits and fissures might continue into adulthood; therefore, posteruptive age alone no longer should be used as a major criterion for making a decision about whether to place sealants. He also pointed out that any tooth at any age could benefit from sealants.

In the current study, there was no significant difference between the two enamel preparation methods. Therefore, the null hypothesis should be accepted. It has been reported that 5% to 10% of all sealants can be expected to fail annually.[33] In the present study, the percentage of the total loss of sealants was 3.5% for the acid-etch group and 0% for the laser group at the end of 24 months.

The laser used in this study was a hydrokinetic system. The main disadvantage of the previous lasers was the immediate increase in temperature, resulting in an inflammatory pulpal response. With the Er,Cr:YSGG laser system, not only could the temperature be suppressed but also cutting efficiency could be increased. Using a pulsed-beam system and fiber delivery, it has proved to be a valuable tool for ablating enamel and dentin. Since the handpiece of the Er,Cr:YSGG laser is light, its manipulation is easy. Unnecessary etching of the enamel is also prevented with the Er,Cr:YSGG laser.[34] There are some contradictory findings concerning the use of

Table 2: *Sealant Retention Rates: Number and percent of sealants at each time interval*

	6-Month		12-Month		18-Month		24-Month	
	Acid	Laser	Acid	Laser	Acid	Laser	Acid	Laser
CR	55 (98.2%)	54 (96.4%)	51 (91.1%)	51 (91.1%)	50 (89.2%)	51 (91%)	47 (83.9%)	48 (85.7%)
PL	0 (0%)	2 (3.5%)	3 (5.3%)	5 (8.9%)	4 (7.1%)	5 (8.9%)	7 (12.5%)	8 (14.2%)
TL	1 (1.7%)	0 (0%)	2 (3.5%)	0 (0%)	2 (3.5%)	0 (0%)	2 (3.5%)	0 (0%)
Total	56	56	56	56	56	56	56	56
p Value	>0.05		>0.05		>0.05		>0.05	

Abbreviations: CR, completely retained; PL, partial loss; TL, total loss.

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Table 3: *Distribution of Sealant Retention Rates (Number (percent)) of Acid-Etch Group for Premolars and Molars*

	ACID-ETCH							
	6-Month		12-Month		18-Month		24-Month	
	Premolar	Molar	Premolar	Molar	Premolar	Molar	Premolar	Molar
CR	23 (95.8%)	32 (100%)	22 (91.6%)	29 (90.6%)	22 (91.6%)	29 (90.6%)	21 (87.5%)	26 (81.2%)
PL	0 (0%)	0 (0%)	1 (4.1%)	2 (6.2%)	1 (4.1%)	2 (6.2%)	2 (8.3%)	5 (15.6%)
TL	1 (4.1%)	0 (0%)	1 (4.1%)	1 (3.1%)	1 (4.1%)	1 (3.1%)	1 (4.1%)	1 (3.1%)
Total	24	32	24	32	24	32	24	32
p Value	>0.05		>0.05		>0.05		>0.05	

Abbreviations: CR, completely retained; PL, partial loss; TL, total loss.

lasers for enamel etching. The majority of previous studies demonstrated that the roughened surface produced by the laser alone lacks the seal obtained with acid etching.[35,36] In contrast, some authors[37–39] reported that laser irradiation may be used to etch enamel. Borsatto and others[40] found that microleakage of fissure sealant with the Er:YAG laser application was higher than that following acid etching or with the laser together with acid etching. In another microleakage study, it was reported that the laser irradiation alone or in combination with acid etching resulted in higher microleakage.[41] In concurrence with these results, it has been suggested that the laser alone was not adequate for etching enamel prior to sealant application. Cehreli and others[26] reported that Er,Cr:YSGG laser pretreatment did not influence the resistance to microleakage of fissure sealants in primary teeth. In all these studies, it was concluded that conventional acid etching remains the most effective and simplest technique in sealants' success. Furthermore, Manhart and others[28] and Lepri and others[27] reported that if Er:YAG laser conditioning was followed by acid etching, the retention of the sealants was equal to that achieved with acid etching alone. Similarly, Sungurtekin and Oztas[29] reported that the micro-

Table 4: *Distribution of Sealant Retention Rates (Number (percent)) of Laser Group for Premolars and Molars*

	LASER							
	6-Month		12-Month		18-Month		24-Month	
	Premolar	Molar	Premolar	Molar	Premolar	Molar	Premolar	Molar
CR	25 (100%)	29 (93.5%)	25 (100%)	25 (80.6%)	25 (100%)	25 (80.6%)	25 (100%)	23 (74.1%)
PL	0 (0%)	2 (6.4%)	0 (0%)	6 (19.3%)	0 (0%)	6 (19.3%)	0 (0%)	8 (25.8%)
TL	0 (0%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)
Total	25	31	25	31	25	31	25	31
p Value	>0.05		>0.05		>0.05		>0.05	

Abbreviations: CR, completely retained; PL, partial loss; TL, total loss.

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leakage values in their laser plus acid etching group were similar to those of their acid-etching group. In a recent study assessing the effect of laser surface treatment on the bond strength of a sealant, it was found that the laser prepared the enamel surfaces for sealing but did not eliminate the need for acid etching.[42] As a result, it was concluded that acid etching following laser application enhances marginal seal and decreases microleakage of sealants. These contradictory findings may be due to the different outputs and experimental designs of the studies. As the studies mentioned above were *in vitro*, we cannot directly compare our results with theirs. *In vitro* studies could predict clinical success, but the real performance should be evaluated with clinical studies. In a recent clinical study, the two-year clinical performance of two minimally invasive cavity preparation techniques, bur and laser, in Class I occlusal resin composite restorations was compared. In that study, no significant differences were observed between the two techniques and both cavity preparation techniques performed equally, with excellent outcomes after 24 months. However, in that study, laser was used for cavity preparation and flowable resin composite was used.[43]

A split-mouth clinical trial was undertaken to compare the retention of fissure sealants placed using CO_2 laser or acid etching. After a mean follow-up period of 14.5 months, the retention rates were found to be statistically similar.[30] As there is a lack of studies evaluating the laser etching effect on sealant's retention, it is difficult to discuss our findings. In a recent study, stereoscopic observation revealed that the laser was capable of cleaning debris in pits and fissures completely. Lased cavities also showed unevenness or irregularity of the enamel surfaces similar to acid etching. They also showed the advantage of the laser in reaching the narrow and deepest parts of the fissures.[44] The removal of debris that accumulates in fissures could increase sealant retention.[14] Laser irradiation also reduces the carbonate to phosphate ratio, leading to the formation of more stable and less acid soluble compounds.[21,22,45,46] Even if there were partial or total loss of a sealant, the laser-prepared enamel surface would be less susceptible to acid attack and caries development. Taking into consideration these features of the laser, it can be speculated that the laser could be a good choice for preparation of enamel prior to the application of a fissure sealant. Long-term follow-up visits are also planned to determine if a difference in retention rates or caries development among the two preparation methods will occur at later sealant ages.

CONCLUSION

As preparation of enamel either with acid or laser did not affect the clinical performance of fissure sealants it might be advantageous to prefer laser with its benefits in caries prevention.

Conflict of Interest

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

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Laboratory Research

Resin-based Composite Light-cured Properties Assessed by Laboratory Standards and Simulated Clinical Conditions

N Ilie • H Bauer • M Draenert
R Hickel

Clinical Relevance

The irradiation technique, used to measure mechanical properties of resin-based composites according to international standards, consistently differs from clinically simulated conditions, calling into question whether laboratory findings can be unrestrictedly applied clinically, especially at short polymerization times. The study analyzes whether degree of conversion measurements at short post-polymerization time (five minutes) are able to predict the long-term material behavior.

SUMMARY

The following parameters were varied: 1) irradiation technique: top and bottom polymerization according to the ISO standard, and polymerization from only the top, simulating clinical situations; 2) polymerization time: 5, 10, 20, and 40 seconds; 3) storage conditions: 24 hours in distilled water, thermocycling followed by storage for four weeks in artificial saliva or alcohol. Flexural strength (FS), flexural modulus ($E_{flexural}$), indentation modulus (E), Vickers hardness (HV), and degree of conversion (DC) were measured.

The laboratory results were similar to those measured by mimicking clinical conditions only at high polymerization times and mild storage conditions (20 seconds and 40 seconds and storage for 24 hours in water, and 40 seconds with aging and storing in saliva). Significantly higher DC values were measured on the top than on the bottom of a 2-mm layer for all polymerization times. Overall, 5-second

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and 10-second irradiation times induced significantly lower DC values compared to the currently recommended polymerization times of 20 and 40 seconds at both the top and bottom of the samples.

The initial DC differences as a function of irradiation time are leveled at 24 hours of storage but seem to do well in predicting long-term material behavior. A minimum irradiation time of 20 seconds is necessary clinically to achieve the best mechanical properties with modern high-intensity light emitting diode (LED) units.

INTRODUCTION

Dentists' requests for a short chair time to prepare a restoration compel a continual reduction in polymerization time for curing resin-based composites (RBCs). When the first high-intensity visible light plasma arc curing (PAC) units were introduced in 1998, the manufacturer claimed to be able to fulfill these wishes, declaring a 3-second polymerization time as sufficient for an adequate polymerization. The manufacturer also claimed that curing for 3 seconds with a high-intensity PAC unit is equivalent to a 40- or 60-second exposure to a quartz-tungsten-halogen (QTH) light.[1] These statements were based on the radiant energy (dose) concept, calculated as a simple reciprocal relationship between irradiation and irradiation time, suggesting that the irradiation time can be shortened when a curing unit's irradiance is proportionally increased. Though generally valid, the radiant energy concept was put into perspective because, within a given dose, shorter exposure time at higher intensity proved to induce decreased properties (lower degree of cure, polymer cross-linking, and physical properties).[2,3] It was also shown that a polymerization of 3 seconds with the high-intensity PAC unit is too short; thus, multiple 3-second exposures are recommended for a clinically adequate performance.[4]

Due to consistent improvements in light emitting diode (LED) technology, a great increase in output power has been achieved in the second and third generation LED units,[1] again suggesting a shortening of irradiation time. While 20-second or 40-second irradiation times with modern LED units are still indicated by many manufacturers, some have recently suggested short polymerization times of up to 5 seconds.

Although time-saving, a fast cure with a high level of energy has a number of drawbacks, including increased polymerization shrinkage stress,[5,6] which is generally related to several negative clinical effects, such as less integrity of the restoration-cavity interface,[7] marginal staining, cusp fractures,[8], microleakage,[9] secondary caries, postoperative sensitivity, or pain. Also, a rise in temperature and risk of pulp damage[10] are associated with the use of curing units with very high intensity.

The quality of polymerization in RBCs is preliminarily assessed in laboratory tests. To measure macromechanical properties such as flexural strength, international standards (ISO 4049: 2009[11]) require curing 2-mm thick slabs from both sides, top and bottom. This double polymerization might generate good mechanical properties with low polymerization times, which could considerably differ from a clinical situation where curing occurs mainly from the top of a restoration. Additionally, the storage time for standard measurements is generally set at 24 hours, raising the question of the role of aging, especially in connection with low polymerization times.

Our study aimed to analyze the effect of aging on the macromechanical and micromechanical properties of a nano-hybrid RBC by varying the polymerization time, the irradiation technique, and the storage conditions. Furthermore, the variation in degree of cure as a function of polymerization time and position—top or bottom of the samples—is analyzed.

The null hypotheses tested in our study were as follows: 1) the irradiation technique—polymerization from both sides, top and bottom, according to ISO standards, and polymerization only from the top, simulating a clinical situation—would not affect the macromechanical (flexural strength and modulus) and micromechanical properties (Vickers hardness and modulus of elasticity); 2) the above mentioned mechanical properties and the degree of cure would not be influenced by the irradiation time and measuring location (top or bottom); 3) aging (24 hours in distilled water, thermocycling followed by storage for four weeks in artificial saliva or alcohol) would have no influence on the mechanical properties; and 4) the aging agent—saliva or alcohol solution—would not influence the mechanical properties.

MATERIALS AND METHODS

A nano-hybrid resin-based composite (IPS Empress Direct Dentin, Ivoclar Vivadent, Batch No. M68447, dimethacrylate resin matrix, Ba-Al-Si-glass, YbF_3,

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and SiO_2/ZrO_2-mixed oxide fillers, 75 wt%, 53 vol%) was analyzed by varying the direction of irradiation (top and bottom or only top), the irradiation time (5, 10, 20, and 40 seconds), the storing conditions (24 hours in distilled water or thermocycling followed by storage for four weeks in an aging medium) and the storage medium for aging (artificial saliva or alcohol). A total of 24 combinations of the above mentioned parameters were studied.

The flexural strength (FS) and flexural modulus ($E_{flexural}$) were determined in a three-point-bending test (n=15). Therefore, 360 samples were made by compressing the composite material between two glass plates with intermediate polyacetate sheets, separated by a steel mold having an internal dimension of $2 \times 2 \times 16$ mm. Irradiation occurred in two different ways: once on the top and bottom of the specimens, as specified in ISO 4049:2009 standards,[11] and once only on top, simulating a clinical situation. When testing, the load was applied to the top surface of these specimens. The times of the light exposures were 5, 10, 20, and 40 seconds, with three light exposures, overlapping one irradiated section no more than 1 mm of the diameter of the light guide (Elipar Freelight 2, 3M ESPE, Seefeld, Germany) to prevent multiple polymerizations. The irradiance of the curing unit (1241 mW/cm^2) was measured by means of a calibrated fiber optic spectrally resolving radiometer equipped with an integrating sphere (S2000, Ocean Optics, Dunedin, FL, USA). To assess possible variations in irradiation, a calibrated spectrometer (MARC, BlueLight analytics inc, Halifax, Canada) was used at the beginning, middle, and end of a sample preparation session.

After removal from the mold, the specimens were ground with silicon carbide sand paper (grit size P 1200/4000 [Leco]) to remove disturbing edges or bulges and stored for 24 hours in distilled water at 37°C. One third of the specimens were subsequently measured and considered as references, the rest were additionally aged (thermocycling for 5000 cycles at 5°C to 55°C) before storage for four weeks at 37°C in artificial saliva or a 1:1 ethanol-water mixture. The samples were loaded until failure in a universal testing machine (Z 2.5, Zwick/Roell, Ulm, Germany) in a three-point bending test device, which was constructed according to the guidelines of NIST No. 4877 with a 12-mm distance between the supports.[12] During testing, the specimens were immersed in distilled water at room temperature. The crosshead speed was 0.5 mm/min. The universal testing machine measured the force during bending as a function of deflection of the beam. The bending modulus was calculated from the slope of the linear part of the force-deflection diagram.

Micromechanical Properties

Fragments (n=12) of the three-point bending test specimens of each group were used to determine the micromechanical properties: Vickers hardness (HV) and indentation modulus (E) according to DIN 50359-1:1997[13] by means of a universal-hardness device (Fischerscope H100C, Fischer, Sindelfingen, Germany). Prior to testing, the samples were polished with a grinding system (EXAKT 400 CS, EXAKT, Norderstedt, Germany) using silicon carbide paper (P 2500 followed by P 4000). Measurements were done on the top (n=6) and the bottom (n=6) of the slabs (10 measurements per sample per side). The test procedure was carried out with controlled force; the test load increased and decreased with a constant speed between 0.4 mN and 500 mN. The load and the penetration depth of the indenter were continuously measured during the load-unload-hysteresis. The universal hardness is defined as the test force divided by the apparent area of the indentation under the applied test force. From a multiplicity of measurements, a conversion factor between universal hardness and Vickers hardness was calculated and implemented in the software, such that the measurement results were indicated in the more familiar HV units. The indentation modulus E was calculated from the slope of the tangent of indentation depth-curve at maximum force.

Degree of Cure

The degree of cure (DC) was analyzed by considering two different sample geometries: one 2-mm high increment measured in a white Teflon mold measuring 2 mm in height and 3 mm in diameter and a thin 100-μm composite film. The samples were cured for 5, 10, 20, and 40 seconds by applying the curing unit directly on the sample's surface (n=5). Measurements were made in real time (five minutes, 2 spectra/s, 4 cm^{-1} resolution) with a Fourier transform infrared (FTIR) spectrometer with an attenuated total reflectance (ATR) accessory (Nexus, Thermo Nicolet, Madison, WI, USA) by applying the nonpolymerized composite paste directly on the diamond ATR crystal. The spectra were recorded in this way at the bottom of the samples.

DC was calculated as the variation in peak height ratio of the absorbance intensities of methacrylate carbon double bond peak at 1634 cm^{-1} and that of internal standard peak at 1608 cm^{-1} (aromatic

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carbon double bond) during polymerization, in relation to the uncured material.

$$DC_{Peak}\% = \left[1 - \frac{1634\text{cm}^{-1}/1608\text{cm}^{-1})_{\text{Peak height after curing}}}{1634\text{cm}^{-1}/1608\text{cm}^{-1})_{\text{Peak height after curing}}}\right] \times 100$$

Statistical Analysis

The Kolmogorov-Smirnoff test was applied to verify that the data were normally distributed. The results were compared using one- and multiple-way analysis of variance (ANOVA) and Tukey honestly significant difference (HSD) post hoc-test (α=0.05). A multivariate analysis (general linear model with partial eta-squared statistics) assessed the effect of storage, irradiation time, and irradiation mode (ISO/clinical) on the considered properties. An independent *t*-test additionally analyzed the differences in mechanical properties as a function of irradiation technique (SPSS, version 19.0, SPSS Inc, Chicago, IL, USA). An additional Weibull analysis was performed for the flexural strength data.

A common empirical expression for the cumulative probability of failure P at applied stress is the Weibull model:

$$P_f(\sigma_c) = 1 - \exp\left[-\left(\frac{\sigma_c}{\sigma_0}\right)^m\right].$$

where σ_c is the measured strength, m is the Weibull modulus and σ_0 is the characteristic strength, which is defined as the uniform stress at which the probability of failure is 0.63. The double logarithm of this expression gives:

$$\ln\ln\frac{1}{1-P} = m\ln\sigma_c - m\ln\sigma_0$$

By plotting ln ln(1/(1–P)) vs ln sigma, a straight line results with the upward gradient m.

RESULTS

Post-hoc multiple pairwise comparisons with Tukey HSD test ($p<0.05$) showed a decrease in the parameters measured in the flexural strength test (FS, $E_{flexural}$, Figure 1; Table 1) after aging, with a considerable decrease in the properties after storing the samples in alcohol. Preparing the samples by mimicking clinical conditions weakened the properties for almost all polymerization times and storage conditions compared to the groups prepared according to the international standard ($p< 0.05$). Only a high polymerization time and a mild storage condition (20 seconds and 40 seconds with 24-hour water storage, and 40 seconds with aging and saliva storage) were able to equalize these differences.

Irrespective of irradiation time and storage, the difference between the micromechanical properties measured on the top and bottom of the samples was not statistically significant in the samples polymerized from both sides but was significantly lower at the bottom of the samples polymerized only from the top (Table 2).

As for the degree of cure, significantly higher values were measured for all polymerization times on the top of the samples, simulated by the use of a thin composite film, as in a depth of 2 mm ($p< 0.05$) (Figure 2; Table 3). For both conditions, 5-second and 10-second irradiation times induced significantly lower DC values compared to the currently recommended polymerization times of 20 seconds or 40 seconds.

The storage proved to have the greatest influence on all measured mechanical properties (Table 4), exercising the strongest effect on the material's reliability, expressed by the Weibull modulus, m. The effect of irradiation time was greater than the effect of irradiation technique with regard to the parameters measured in the flexural test; both effects were comparable with regard to the micromechanical properties.

The modulus of elasticity measured in both methods—the flexural test and the universal hardness test—correlated well (Pearson correlation coefficient = 0.8). There was also a good correlation between the micromechanical properties (E-HV = 0.97) and macromechanical properties (FS-$E_{flexural}$ = 0.81).

DISCUSSION

Laboratory experiments try to simulate clinical conditions as accurately as possible to obtain information that is directly applicable in daily practice. Therefore, our study compared two irradiation techniques—top and bottom, according to the ISO standard, and only top, to simulate clinical situations. The mechanical properties were assessed on a macroscopic scale, by determining the strength and the resistance the materials opposed to deformation (modulus of elasticity), and on a microscopic scale (hardness and modulus of elasticity) by means of a dynamic measuring principle, simultaneously recording the load and the corresponding penetration depth of the indenter.[13] With maximum indentation

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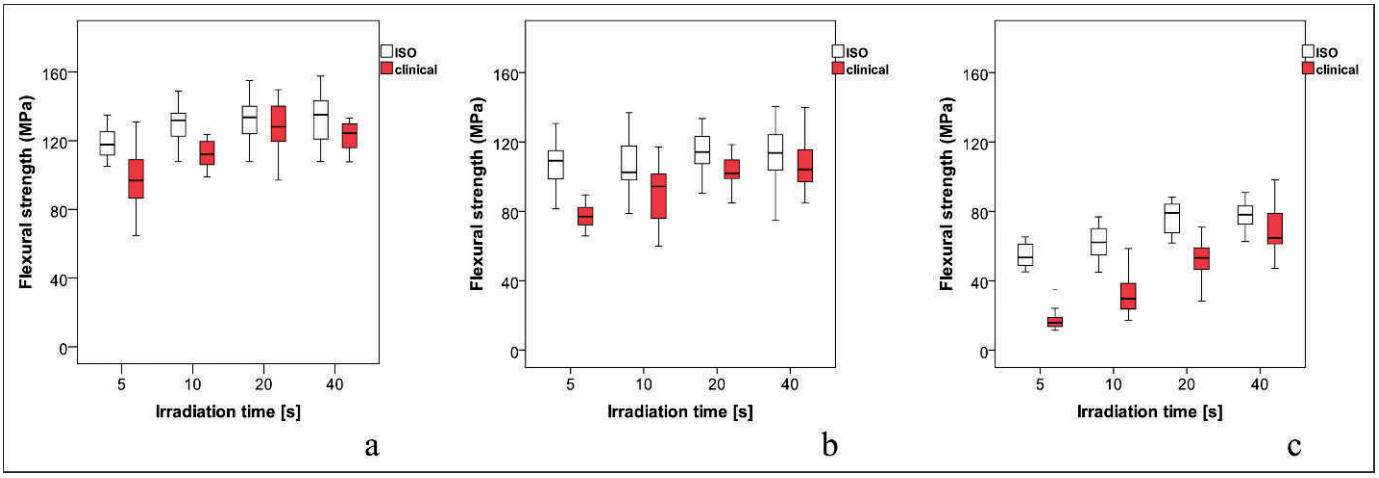

Figure 1. *Variation of flexural strength with irradiation time and irradiation technique after: 1) water storage for 24 hours; 2) thermocycling followed by storage in saliva for four weeks; and 3) thermocycling followed by storage in alcohol for four weeks.*

Table 1: *Flexural Strength (FS) and Modulus of Elasticity in Flexural Test ($E_{flexural}$) are Detailed in Mean Values and Standard Deviations (in Parentheses)*[a]

Storage	Time, s	FS, MPa		*p*	Weibull, *m*		$E_{flexural}$, GPa		*p*
		ISO	Clinical		ISO	Clinical	ISO	Clinical	
24h water	5	118.7^{de} (9.3)	98^{FG} (9.8)	< 0.001	4.09	2.81	4.7^{c} (0.7)	3.4^{C} (0.6)	< 0.001
	10	129.7^{e} (11.9)	110.6^{GH} (12.3)	< 0.001	4.28	3.23	5.6^{d} (0.6)	4.1^{D} (0.7)	< 0.001
	20	132.1^{e} (13.7)	128.5^{I} (14.5)	0.491	4.00	3.14	5.3^{cd} (0.9)	5.3^{EF} (0.7)	0.843
	40	133.1^{e} (15.3)	122.8^{HI} (8.4)	0.051	4.12	3.37	5.2^{cd} (0.8)	5.4^{F} (0.6)	0.349
Thermocycling + 4w saliva	5	105.9^{d} (15.8)	78.4^{DE} (8.9)	< 0.001	2.48	2.79	5.2^{cd} (0.6)	4.1^{D} (0.3)	< 0.001
	10	106.8^{d} (16.0)	90.4^{EF} (17.8)	0.012	2.22	2.08	5.2^{cd} (0.6)	4.7^{E} (0.4)	0.048
	20	113.4^{d} (13.0)	103.0^{FG} (10.8)	0.017	3.38	3.20	5.5^{d} (0.4)	5.3^{EF} (0.3)	0.036
	40	112.8^{d} (16.6)	106.7^{G} (14.5)	0.277	2.31	2.91	5.6^{d} (0.5)	5.7^{F} (0.4)	0.828
Thermocycling + 4w alcohol	5	53.1^{a} (8.9)	17.3^{A} (5.8)	< 0.001	1.95	0.94	2.5^{a} (0.2)	1.6^{A} (0.3)	< 0.001
	10	62.0^{ab} (9.2)	33.0^{B} (12.8)	< 0.001	2.32	0.80	2.7^{ab} (0.4)	2.3^{B} (0.3)	0.006
	20	76.3^{bc} (9.3)	51.4^{C} (10.6)	< 0.001	2.65	1.59	3.2^{ab} (0.4)	2.4^{B} (0.3)	< 0.001
	40	77.1^{c} (7.8)	69.0^{D} (13.2)	0.040	3.30	1.77	3.2^{b} (0.2)	3.2^{C} (0.2)	0.829

[a] *Superscript letters indicate statistically homogeneous subgroups within a column (Tukey HSD test, $\alpha = 0.05$). A* t*-test analyzed differences between the way of curing the samples—ISO or clinical—for each irradiation time. The Weibull parameter* m *is indicated.*

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Table 2: *Micromechanical Properties—Modulus of Elasticity (E) and Vickers Hardness (HV)—Are Detailed in Mean Values and Standard Deviations (in Parentheses)*[a]

Storage	Time, s	E, GPa			HV, N/mm^2		
		ISO	Clinical Top	Clinical Bottom	ISO	Clinical Top	Clinical Bottom
24h Water	5	12.4^{e*} (0.5)	12.4^{E*} (0.4)	6.8^{b} (2.1)	66.6^{e} * (4.5)	67.5^{E*} (4.9)	28.3^{a} (13.1)
	10	13.1^{g} (0.3)	12.4 ^{E} (1.6)	11.8^{ef} (0.8)	73.5^{g} * (1.7)	71.7^{F*} (6.5)	63.3^{g} (6.8)
	20	13.1^{g*} (0.3)	13.0^{FG*} (1.2)	12.4^{fg} (0.3)	74.8^{g} * (2.6)	76.0^{G*} (6.1)	67.2^{g} (2.8)
	40	13.6^{d} (0.3)	13.3^{G} (0.5)	12.8^{g} (0.3)	76.6^{h} (2.3)	75.0^{G} (3.4)	72.0^{h} (2.4)
Thermocycling + 4w saliva	5	12.6^{ef} (0.4)	11.4^{D*} (0.6)	8.1^{c**} (0.9)	68.5^{f} (2.2)	58.4^{D*} (4.1)	33.8^{bc**} (6.8)
	10	13.0^{g} (0.4)	12.4 ^{E*} (0.4)	9.8^{d**} (1.0)	70.2^{f} (4.8)	65.5^{E*} (4.1)	44.3^{e**} (8.9)
	20	12.9^{g} (0.4)	13.0^{FG*} (0.3)	11.2^{e**} (0.4)	69.2^{f} (3.5)	72.4^{F*} (1.8)	58.0^{f**} (3.8)
	40	13.1^{g} (0.4)	12.6^{EF*} (0.5)	11.3^{e**} (0.6)	73.3^{g} (2.1)	67.4^{E*} (4.5)	57.0^{f**} (6.1)
Thermocycling + 4w alcohol	5	7.1^{a*} (0.4)	7.2^{A*} (0.4)	5.9^{a} (0.3)	38.5^{a*} (2.3)	37.9^{A*} (4.1)	31.6^{ab} (2.9)
	10	7.6^{b*} (0.3)	8.0^{B*} (0.5)	6.9^{b} (0.3)	41.5^{b*} (3.0)	43.9^{B*} (2.4)	37.0^{cd} (2.3)
	20	8.4^{c*} (0.3)	8.5^{C*} (0.4)	7.1^{b} (0.4)	48.2^{c*} (1.6)	48.7^{C*} (2.5)	40.5^{de} (2.4)
	40	9.3^{d*} (0.5)	8.3^{BC*} (0.5)	7.3^{b} (0.3)	52.7^{d*} (2.7)	45.8^{B*} (3.7)	40.4^{de} (2.9)

[a] *Superscript letters indicate statistically homogeneous subgroups within a column and asterisks(*) indicate statistically homogeneous subgroups within a line. (Tukey HSD test, $\alpha = 0.05$). For the ISO way of cure, no statistical differences between top and bottom were measured.*

depth in the range of 7-15 μm, the measurements performed at microscopic scale reflect the material properties and not the properties of the individual material components (filler and filler agglomerates or matrix and matrix-reach areas). As for the degree of cure, the measurements were done at the same depth as the measurement of micromechanical properties, by simulating the top and bottom of clinically relevant 2-mm-thick increments, allowing thus a direct comparison among the measured parameters.

The results demonstrated that when samples were stored for 24 hours, the irradiation technique significantly affected the mechanical properties at short irradiation times (5 seconds and 10 seconds), lowering FS and $E_{flexural}$ in simulated clinical conditions when compared to the ISO standard, but showed similar results for both polymerization techniques at high irradiation times (20 seconds and 40 seconds). These results support the recommended irradiation time of at least 20 seconds, as indicated by the manufacturer and also often used in ISO 4049:2009,[11] for both *in vitro* and *in vivo* use. Within the above mentioned curing and storage conditions—irradiation of at least 20 seconds and storage for 24 hours in distilled water—the results of the ISO-irradiation can be directly applied clinically, since both tested irradiation techniques produced not only similar macromechanical properties (FS, $E_{flexural}$) but also similar micromechanical properties (HV, E). After aging and storing in artificial saliva, a polymerization time of 40 seconds is necessary to ensure statistically similar properties between the ISO and the clinically simulated irradiation, whereas storing in alcohol never produced comparable results (40 seconds). These results put in perspective whether the mechanical properties measured according to the current standards are able to reflect

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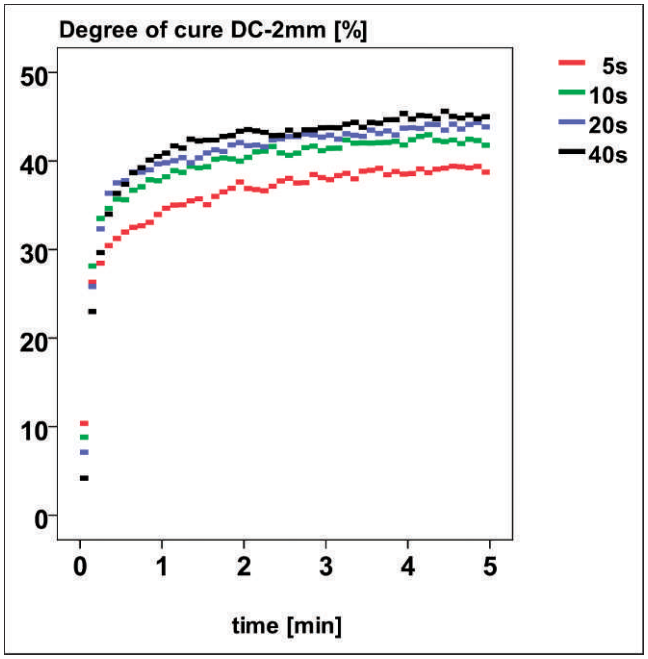

Figure 2. *Degree of cure measured at 2-mm depth in real-time for five minutes, as a function of polymerization time (mean values, n=5).*

the long-term behavior of an RBC filling and confirm the lack of correlation between the clinical behavior of restorative materials and laboratory results.[14] This statement should not, however, diminish the meaning of preliminary laboratory tests and standardized methods, since an initial selection of materials with adequate properties is of particular importance.[15]

Table 3: *Degree of Cure (DC) at 0.1 mm and 2 mm Are Detailed in Mean Values and Standard Deviations (in Parentheses)*[a].

Time, s	$DC_{0.1mm}$, %	DC_{2mm}, %	*p*
5	45.3[a] (3.5)	38.6[A] (2.8)	< 0.001
10	46.5[a] (3.0)	41.8[B] (2.3)	< 0.001
20	48.7[b] (2.4)	43.9[C] (1.5)	< 0.001
40	49.6[b] (4.2)	45.0[C] (2.7)	0.005

[a] *Superscript letters indicate statistically homogeneous subgroups within a column (Tukey HSD test, α=0.05). A t-test analyzed differences between the DC at 0.1 and 2 mm for each irradiation time.*

Table 4: *Influence of Storage, Irradiation Time and Way of Curing on Flexural Strength (FS), Modulus of Elasticity in Flexural Test ($E_{flexural}$), and Weibull parameter* m*, Vickers hardness (HV), and Modulus of Elasticity (E), as Well as Degree of Cure (DC)*[a]

Parameters	FS	$E_{flexural}$	*m*	HV	E	DC
Storage	.842	.829	.973	.825	.889	
Irradiation time	.402	.409	.776	.531	.483	.292
ISO/clinical	.342	.221	.901	.534	.490	.313

[a] *The higher the partial eta-squared values the higher is the influence of the selected variables on the measured properties.*

The study also showed that the most sensitive parameter to all of the above mentioned influences (Table 4, highest eta-squared values) was the Weibull modulus *m*, that means the reliability of the tested material, being lower in the samples cured by simulating a clinical situation compared to the groups polymerized according to the ISO standards, for all storage conditions. Furthermore, the material reliability was lowered with aging and with increased aggressiveness of the storage agent. Furthermore, not only curing time and irradiation condition but also the storage conditions and, thus, the softening effect due to aging and storage in saliva or alcohol were more strongly reflected in the reliability determined at a macroscopic scale (Weibull parameter *m*) than in the micromechanical properties E and HV. These observations highlight the importance of performing macromechanical tests with a higher number of samples to allow performance of a Weibull statistical analysis for acquiring sensitive and reliable observation on a material's behavior.

The surface of a restoration was simulated in our study by a 100-μm RBC layer used to assess the evolution of degree of cure as a function of irradiation time in real time. The measurements were done at the bottom of the film to avoid the influence of the oxygen-inhibition layer, which was shown to be less than 20 μm thick.[16] The micromechanical properties were also measured after the oxygen-inhibition layer was eliminated by grinding and polishing; thus, both tests were recorded at similar depth. Differences in DC between the 0.1-mm and 2-mm depths were statistically significant for all irradiation times. The

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postpolymerization, however, seems to have leveled these differences for longer irradiation times (20 seconds, 40 seconds), as reflected in the mechanical properties (top-bottom) measured 24 hours after storage in water. However, aging again emphasizes the importance of assessing the initial degree of cure since, especially after aging and storage in alcohol solution, the difference in micromechanical properties between the top and bottom of the samples cured from only one side became evident at all polymerization times. Similar trends were found also for the flexural strength, when both irradiation techniques were compared. Since softening tests in solvents such as ethanol and water are well-established methods of assessing the cross-link density of a polymer network,[17,18] the aging and storage in ethanol performed in this study can be taken as an indicator of the effect of reduced polymerization time as well as attenuated light at the bottom of the specimens. The softening effect of solvents was shown to be generally stronger in a more linear polymer structure than in a highly cross-linked polymer,[19] emphasizing the negative effect of short curing time on the measured mechanical properties. This statement is also consolidated by the measured degree of cure.

The results of the present study also validate the literature indicating that the minimum radiant energies necessary to properly cure RBCs are 16.8 J/cm^2 for a 1-mm increment[20] and 24 J/cm^2 for 2-mm increments.[21] Under the study conditions (light intensity 1241 mW/cm^2, polymerization time 5, 10, 20, and 40 seconds; 2-mm increments), this minimum irradiation was reached only for polymerization times of 20 seconds and 40 seconds.

The statements of the study are limited by having analyzed only one RBC. Though we randomly chose a modern nano-hybrid RBC with moderate mechanical properties,[22] the large diversity of RBCs, which contain complex fillers, organic matrices, and initiator systems, make it difficult to generalize the results but gives at least a reference note for the complex impact of polymerization on material behavior.

CONCLUSIONS

All tested null hypotheses are rejected. The properties measured according to ISO standards were similar to those measured by mimicking clinical conditions only at high polymerization times and mild storage conditions (20 seconds and 40 seconds with 24-hour water storage, and 40 seconds with aging and storing in saliva). The initial (5-minute) differences in DC measurements as a function of irradiation time are leveled at 24 hours of storage but seem to be a good indicator of the long-term material behavior.

A minimum irradiation time of 20 seconds is clinically necessary to achieve the best mechanical properties, also when modern high-intensity LED units are used.

Conflict of Interest

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

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Effect of Artificial Aging and Surface Treatment on Bond Strengths to Dental Zirconia

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FA Oliveira

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Clinical Relevance

In case the veneering ceramic of a zirconia-based restoration fractures in the mouth to expose the zirconia coping, a phosphate/carboxylate monomer–based primer may be a good adhesion enhancer to repair the fracture.

SUMMARY

The objective of this project was to study the influence of artificial aging and surface treatment on the microtensile bond strengths (μTBS) between zirconia and a phosphate monomer–based self-adhesive cement. Thirty zirconia disks (IPS e.max ZirCAD, Ivoclar Vivadent) were randomly assigned to two aging regimens: AR, used as received, which served as a control, and AG, artificial aging to simulate low-temperature degradation. Subsequently, the disks of each aging regimen were assigned to three surface treatments: NT, no surface treatment; CO, surface silicatization with CoJet sand (3M ESPE); and ZP, zirconia surface treated with Z-Prime Plus (Bisco Inc). Thirty discs were made of Filtek Z250 (3M ESPE) composite resin and luted to the zirconia discs using RelyX Unicem (3M ESPE). The specimens were sectioned with a diamond blade in X and Y directions to obtain bonded beams with a cross-section of 1.0 ± 0.2 mm. The beams were tested in tensile mode in a universal testing machine at a speed of 0.5 mm/min to measure μTBS. Selected beams were selected for fractographic analysis under the SEM. Statistical analysis was carried out with two-way analysis of variance and Dunnett T3 post hoc test at a significance level of 95%. The mean μTBS for the three AR subgroups (AR-NT, AR-CO, and AR-ZP) were significantly higher than those of the corresponding AG groups ($p<0.0001$). Both AR-CO and AR-ZP

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resulted in statistically significant higher mean bond strengths than the group AR-NT ($p<0.006$ and $p<0.0001$, respectively). Both AG-CO and AG-ZP resulted in statistically significant higher mean bond strengths than the group AG-NT (both at $p<0.0001$). Overall, AG decreased mean μTBS. Under the SEM, mixed failures showed residual cement attached to the zirconia side of the beams. CO resulted in a characteristic roughness of the zirconia surface. AR-ZP was the only group for which the amount of residual cement occupied at least 50% of the interface in mixed failures.

INTRODUCTION

Zirconia is a high-strength and flaw-tolerant material[1,2] that has shown potential as a biomaterial in orthopedics and dentistry.[3] The high strength of zirconia is derived from a stress-induced transformation from the metastable tetragonal form to the stable monoclinic form ($t \rightarrow m$).[2,4] This transformation enhances the mechanical properties of zirconia through an increase in crack resistance during the course of crack propagation, as a result of compressive stresses that form in the vicinity of the crack.[4,5]

Tetragonal zirconia stabilized with 3mol% yttria (or 3Y-TZP) is the material currently used in dentistry. Several commercial 3Y-TZP dental materials are available, including Cercon (Dentsply Prosthetics, York, PA, USA), IPS e.max ZirCAD (Ivoclar Vivadent, Principality of Liechtenstein), and Lava (3M ESPE, St Paul, MN, USA). The advent of CAD/CAM technology has given clinicians the opportunity to prescribe complex but precise 3Y-TZP–based restorations.[6,7]

Acid-resistant ceramics (such as alumina- and zirconia-based) are not etchable with hydrofluoric acid because they lack a vitreous phase. In lieu of etching, clinicians have used airborne particle abrasion to create micro-retentive features for the luting agent.[8,9] Chairside air abrasion with 30-μm silica-coated aluminum particles (tribochemical silica coating, also known as *silicatization*) has been recommended to create surface irregularities on acid-resistant ceramics.[9–11] The rationale behind silica coating is to embed the ceramic surface with silica particles,[12] which are chemically more reactive to the resin via priming agents.[13]

Several recent resin cements, known as self-adhesive cements, contain phosphorylated methacrylate monomers. These materials may interact chemically with the zirconia surface. With two phosphate groups and at least two double-bonded carbon atoms, self-adhesive cements are expected to bond to zirconia similarly to other phosphate-based adhesive materials.[14] In addition, new phosphate monomer–based primers are now available to promote chemical adhesion between resin materials and hydroxyl groups present on the zirconia surface.[15–17] One of these phosphate-based materials, Z-Prime Plus (Bisco Inc, Schaumburg, IL, USA), is a primer that includes a mixture of phosphate and carboxylate monomers to enhance the bonding to zirconia and metals, according to the respective manufacturer. This primer has been shown to enhance the adhesion of conventional and self-adhesive resin cements to air-abraded zirconia.[18]

Aging occurs experimentally in zirconia, mostly in humid atmosphere or in water.[19,20] The lack of long-term stability has been a major issue for medical use and has led to the replacement of several zirconia femoral heads in orthopedic patients.[21] Low-temperature degradation (LTD) has been associated with several 3Y-TZP–based biomaterials[22] but is difficult to simulate in the laboratory. Consequently, a standard *in vitro* hydrothermal accelerated aging test using steam and pressure has been developed.[23] The ISO Standard 13356 recommends that "the Y-TZP specimens are placed in a suitable autoclave and exposed to steam at (134 ± 2)°C under a pressure of 0.2 MPa for a period of 5 h."[23]

In addition to studying adhesion to zirconia as received from the dental laboratory, it is therefore clinically relevant to study adhesion to aged zirconia, in case there is a need to repair the fractured restoration when the zirconia coping becomes exposed to the oral environment. In fact, clinical studies have reported an incidence of chipping of the zirconia veneering porcelain as high as 10%-15% at 3 years.[24,25] Veneering zirconia with glass-based ceramic reduces the fracture resistance of zirconia copings compared with those copings that are subjected only to cyclic loading.[26] The reduction in fracture resistance associated with the hydrothermal degradation of 3Y-TZP[22] may be responsible, at least partially, for the clinical failures associated with chipping of the veneering ceramic.

The null hypotheses to test in this study were 1) artificial aging of zirconia does not result in a reduction of microtensile bond strengths (μTBS) of a phosphate-based self-adhesive resin cement, and 2) zirconia surface treatment with silicatization or with a specific primer does not increase μTBS of a phosphate-based self-adhesive resin cement.

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METHODS AND MATERIALS

Thirty IPS e.max ZirCAD (IPS, Ivoclar Vivadent) discs (d=13.0 mm; h=3.0) were prepared from CAD-CAM blocks and sintered following the manufacturer's instructions (Table 1). All disks were cleaned ultrasonically in distilled water for five minutes prior to use in this project.[14] The disks were randomly and equally assigned to two aging regimens: 1) AR, as received (control), and 2) AG, artificial aging to simulate LTD: disks were immersed in distilled water in an autoclave (SANOclav Las-3-13-MCS-J, Adolf Wolf SANOclav, Hausen, Germany) for five hours at a temperature of 134°C under a pressure of 0.2 bar, following ISO recommendations.[23]

Disks were further randomly assigned to one of three surface treatments: 1) NT, no treatment; 2) CO, silicatization with CoJet sand (3M ESPE) for 20 seconds, under a pressure of 2.5 bar, from a distance of 7 mm,[27] with a tip inclination of 45°; and 3) ZP, two coats of Z-Prime Plus (Bisco Inc) were applied with a microbrush (Microbrush International, Grafton, WI, USA) to uniformly wet the zirconia surface, followed by drying with air for three to five seconds.[28]

Thirty composite resin disks (d=13.0 mm; h=3.0) were made of Filtek Z250 shade A2 (3M ESPE) in a silicone mold. The composite was polymerized with an Elipar S10 curing light (3M ESPE) in four areas for 60 seconds each to include the entire disk surface. RelyX Unicem Aplicaps (3M ESPE) shade A2 was activated, mixed for 15 seconds, and applied on the irradiated surface of the composite disc, which was seated on top of the zirconia disc. This bonded setup was loaded with 50N for 60 seconds,[14] excess cement was wiped off, and the specimen was light polymerized from the composite side with the same curing unit at four different locations for 60 seconds each.

Bonded specimens were sectioned in X and Y directions with a 0.5-mm-thick diamond disk (Cafro SRL, Fino Mornasco, Como, Italy) in a Secotom-10 apparatus (Struers A/S, Ballerup, Denmark) to obtain approximately 20 beams per bonded disk, with a cross section of 1.0 ± 0.2 mm^2.

After 24 hours in distilled water at 37°C,[14] each beam was individually attached to a stainless-steel jig using cyanoacrylate glue (Zapit, Dental Ventures of America, Corona, CA, USA). The zirconia-resin μTBS were measured in MPa by applying a tensile load to the bonded interface using a universal testing machine (Shimadzu Autograph AG-IS, Tokyo, Japan) at a crosshead speed of 0.5 mm/min. A digital caliper (Mitutoyo Corp, Kanagawa, Japan) with an accuracy of 0.001 mm was used to measure the sides of the bonding interface and calculate the bonding area in millimeters squared. The load at fracture and the bonding surface area of the specimen were registered and μTBS calculated in MPa. Pretesting failures (PTFs), which corresponded to spontaneous debonding that occurred prior to testing, were computed as zero MPa.[29–31] The fractures were analyzed under a stereo microscope (Leica MZ6,

Table 1: *Composition of Materials Used in This Study (Batch Numbers in Parentheses)*

Material	Manufacturer	Batch Number	Composition
IPS e.max ZirCAD[a]	Ivoclar Vivadent	(M40290)	ZrO_2 (87.0–95.0wt%), Y_2O_3 (4.0–6.0wt%), HfO_2 (1.0–5.0wt%), Al_2O_3 (0.1-1.0wt%), Other oxides (<0.2wt%)
CoJet Sand	3M ESPE	(433711)	Aluminum oxide, amorphous silica
Z-Prime Plus	Bisco Inc	(1000003112)	Organophosphate monomer (MDP), carboxylic acid monomer (BPDM); HEMA, ethanol
RelyX Unicem Aplicap	3M ESPE	(435042), (431825)	Powder: glass powder, silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator; liquid: methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiator
Filtek Z250	3M ESPE	(N234284)	BisEMA, BisGMA, TEGDMA, UDMA, zirconium, silica, pigments, camphorquinone

Abbreviations: bis-GMA, bisphenol A diglycidyl methacrylate; BisEMA-(6), bisphenol A polyetheylene glycol diether dimethacrylate; BPDM, biphenyl dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethyleneglycol-dimethacrylate; UDMA, urethane dimethacrylate.
[a] Sintering temperature is 1500°C in a program of eight hours divided in three cycles: (1) 90 minutes, (2) two hours 45 minutes, (3) three hours 30 min.

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Leica Microsystems AG, Heerbrugg, Switzerland) at 20×. The mode of failure was classified as adhesive, mixed, and cohesive. Failures were considered adhesive if they occurred at the zirconia-cement interface, which included PTFs; of mixed nature if there was residual cement at the zirconia side of the interface; and of cohesive nature if the fracture occurred solely in composite resin.

Data were submitted to two-way analysis of variance (ANOVA). As Levene's statistics showed that there was an inhomogeneity of variances ($p>0.05$), the Dunnett T3 pairwise comparison test was used for the μTBS data ($p<0.05$). Failure modes were analyzed with chi-square followed by Cramer's V. The software PASW Statistics 18.0 (SPSS Inc, Chicago, IL, USA) was used for the analyses.

SEM Analysis

Randomly selected fractured beams (six for each experimental condition) were mounted on Al stubs (Ted Pella Inc, Redding, CA, USA) with carbon adhesive tape and colloidal silver paint (Ted Pella Inc). Then, specimens were sputter coated with gold-palladium by means of an E-5100 sputter coater (Polaron Ltd, Watford, UK) at 20 mA for 45 seconds and observed under an S-4700 FESEM (Hitachi High Technologies America Inc, Pleasanton, CA, USA) at an accelerating voltage of 5.0 kV and working distance of 11.8-12.2 mm.

RESULTS

Means, standard deviations, number of beams per group, and number of PTFs are displayed in Table 2. Two-way ANOVA found significant differences for both factors "aging" and "surface treatment" (both at $p<0.0001$). There were no significant interactions between these variables ($p>0.097$).

The highest mean μTBS was obtained with group AR-ZP (17.4 MPa), which was statistically higher than any other mean μTBS. The lowest mean μTBS was obtained with group AG-NT (0.7 MPa), which also resulted in the highest number of PTFs (91.9% of the beams). AG-CO was the only other group that had PTFs (36.8% of the beams).

The three groups for which zirconia was used as received (AR) resulted in statistically higher mean μTBS than the corresponding AG groups (AR-NT > AG-NT; AR-CO > AG-CO; AR-ZP > AG-ZP).

Table 3 displays the statistical significance for each pair of means. For the AR groups, AR-NT resulted in statistically lower mean μTBS than either AR-CO or AR-ZP ($p<0.006$ and $p<0.0001$, respectively). The means for AR-CO were statistically lower than those of AR-ZP at $p<0.022$. For the AG groups, mean μTBS for AG-NT were statistically lower than those of either AG-CO or AG-ZP ($p<0.0001$). The mean μTBS for AG-CO were statistically lower than those for AG-ZP at $p<0.009$.

Most failures were of adhesive nature (Table 4). There was a statistical difference for failure modes among groups ($p<0.0001$, Cramer's V). Overall, AR specimens had a statistically lower number of adhesive failures (and greater number of mixed failures) than AG specimens.

SEM Analysis

Adhesive failures displayed residual scratches from the zirconia manufacturing finishing procedures

Table 2: *Means ± SD, Number of Bonded Beams, and PTFs*

	Surface Treatment	Mean ± SD (MPa)	Number of Beams	PTFs (%)
As received (AR), n=15	No treatment	9.2 ± 4.7	99	0
	CoJet	13.1 ± 7.2	96	0
	Z-Prime Plus	17.4 ± 8.8	112	0
Aged (AG), n=15	No treatment	0.7 ± 2.7	99	91.9
	CoJet	7.7 ± 7.8	114	36.8
	Z-Prime Plus	11.0 ± 5.8	101	0

Abbreviation: PTFs, pretesting failures.

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Table 3: *Statistical Significance Between Pairs of Microtensile Bond Strengths Means*

	AR-NT	AR-CO	AR-ZP	AG-NT	AG-CO	AG-ZP
AR-NT		0.006	**0.0001**	**0.001**	0.836	0.325
AR-CO	**0.006**		**0.022**	**0.0001**	**0.0001**	0.601
AR-ZP	**0.0001**	**0.022**		**0.0001**	**0.0001**	**0.0001**
AG-NT	**0.0001**	**0.0001**	**0.0001**		**0.0001**	**0.0001**
AG-CO	0.836	**0.0001**	**0.0001**	**0.0001**		**0.009**
AG-ZP	0.325	0.601	**0.0001**	**0.0001**	**0.009**	

The p-values in bold denote a statistical significant difference between the respective pair of means.

(Figure 1). Mixed failures showed residual cement attached to the zirconia side of the beams (Figure 2a). CO resulted in a characteristic roughness of the zirconia surface (Figure 2b). AR-ZP was the only group for which the amount of residual cement occupied at least 50% of the interface in mixed failures (Figure 3a). For the same group AR-ZP, areas of the zirconia side of the interface displayed 3- to 5-μm-long neddle-like crystals (Figure 3b). For AG-NT specimens, there were no specific morphologic features. AG-CO specimens displayed a similar surface roughness compared with their AR counterparts with areas showing the zirconia crystals intermingled with others without signs of surface abrasion (Figure 4). Mixed failures in the AG-ZP group displayed a minimal amount of residual cement at the interface compared with AR-ZP (Figure 5).

DISCUSSION

The use of toughened ceramics such as 3Y-TZP has enabled the use of all-ceramic restorations in posterior regions where high-strength structures are required. Ceramic materials applied in dentistry may exhibit significant subcritical crack growth due to thermal fatigue and the aqueous environment during mastication.[32]

Veneering of the zirconia coping is usually carried out at a relatively high temperature (750–900°C). During this process, the zirconia frameworks are exposed to moisture from the veneering porcelain. The firing process is repeated two to five times.[3] Residual stresses accumulate each time the zirconia is fired at high temperatures followed by cooling to ambient temperature during the laboratory process of making crowns and bridges.[33] A study evaluated the fracture resistance of 3Y-TZP bridge frameworks after mechanical fatigue, heat treatment similar to that used during veneering of the zirconia or after veneering.[26] Multiple firings may affect the resistance of the framework to fracture and the adhesion of the veneering porcelain to the zirconia coping.[26]

In addition to this reduction in physical properties from firing, zirconia undergoes a slow transformation process under a humid environment (known as LTD) from the tetragonal phase to the monoclinic phase.[34,35] The fracture of the veneering ceramic

Table 4: *Type of Fractures per Group (%)*

	AR-NT	AR-CO	AR-ZP	AG-NT	AG-CO	AG-ZP
Adhesive	69.7	68.8	70.4	99.0	80.7	89.1
Cohesive	0	0	0	0	0	0
Mixed	30.3	31.2	29.6	1.0	19.3	10.9

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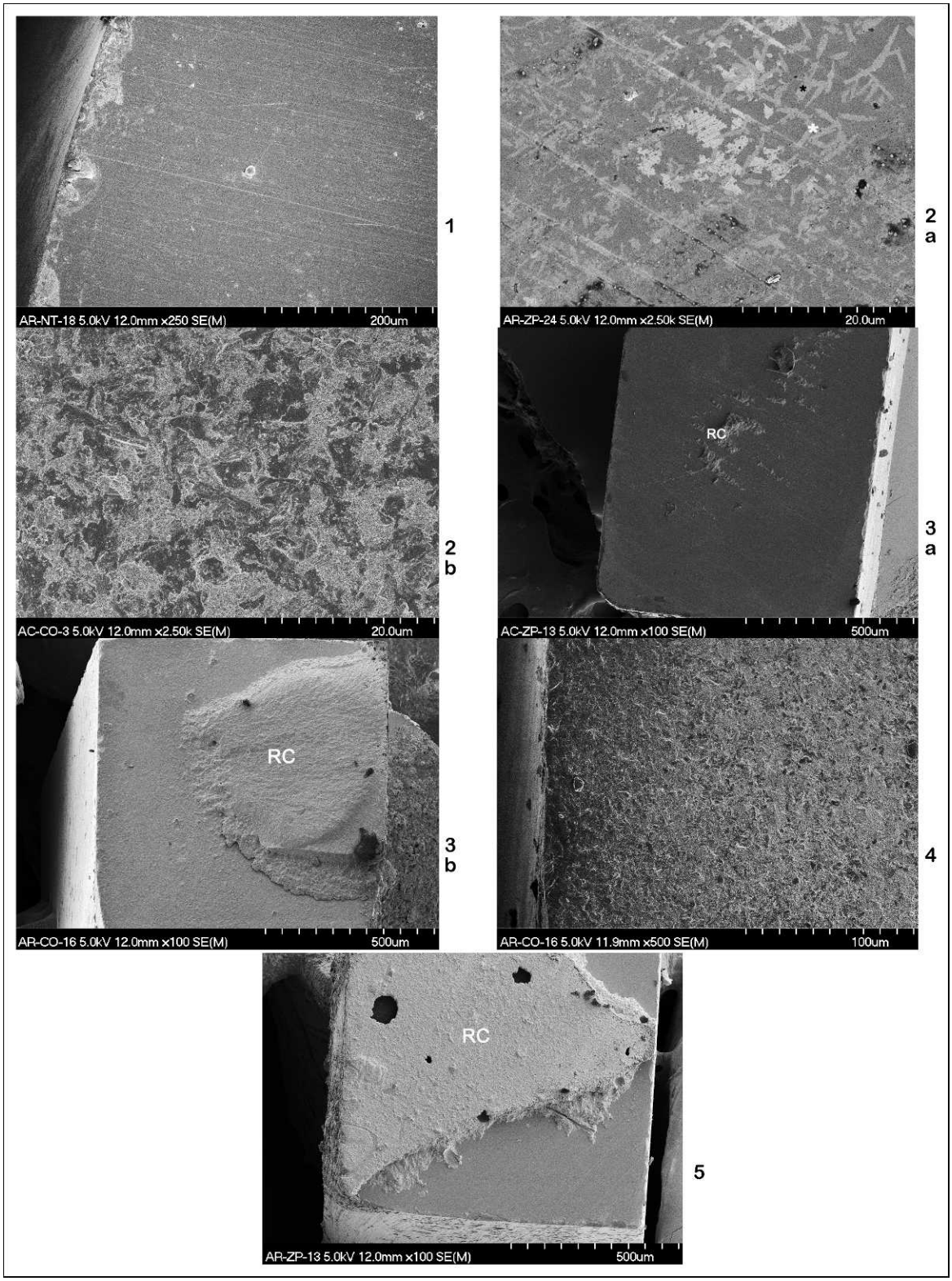

Figure 1. *Adhesive failure (zirconia side) of AR-NT specimen. Original magnification = 250×.* Figure 2. *(a): Mixed failure (zirconia side) of AR-CO specimen. RC, resin cement. Original magnification = 100×. (b): Higher magnification of the same specimen showing the zirconia surface roughness as a result of CO abrasion. Original magnification = 500×.* Figure 3. *(a): Mixed failure (zirconia side) of AR-ZP specimen. RC, resin cement. Original magnification = 100×. (b): Higher magnification of the same specimen showing needle-like deposits (asterisks) on the zirconia surface. Original magnification = 2500×.* Figure 4. *Morphological characteristics of the effect of CO on AG zirconia. Original magnification = 2500×.* Figure 5. *Mixed failure (zirconia side) of AG-ZP specimen showing small islands of resin cement (RC). Original magnification = 100×.*

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may expose the core framework to the aqueous environment of the mouth. The long-term lifetime of zirconia-based bridges is then governed by the water-assisted subcritical crack growth behavior of the framework material.[31]

The phosphate groups make self-adhesive cements acidic immediately upon mixing. RelyX Unicem (3M ESPE) reaches a pH close to neutrality after mixing.[36] Some authors have reported that RelyX Unicem has the ability to bond to zirconia, regardless of the zirconia surface treatment (no treatment, sandblasting with Al_2O_3 particles, or silicatization).[37] Other studies have shown that phosphate monomer–based resins bond better to zirconia than resins without these monomers.[38] We used only one resin cement in our study; therefore, it is not possible to generalize to other materials. In agreement with previous studies,[14,37] the resin cement used in our study has the ability to interact with zirconia without the need for additional surface treatment, as observed in the AR-NT subgroup (9.2 ± 4.7 MPa). However, this likely chemical interaction was lost when RelyX Unicem was applied on aged (AG) zirconia, for which the majority of the bonds failed spontaneously (0.7 ± 2.7 MPa, 91.9% PTFs). The use of CO improved the bonding ability of RelyX Unicem to AG zirconia (7.7 ± 7.8 MPa), but the number of spontaneous PTFs was still relatively high (36.8%). Although ZP was able to restore the bonding ability to the AG zirconia substrate, the bond strengths obtained for AG-ZP (11.0 ± 5.8 MPa) were only 62.5% of those measured when ZP was applied to AR zirconia (17.4 ± 8.8 MPa). In addition, mixed failures for AR-ZP specimens under the SEM displayed wider areas of residual fractured cement compared to AG-ZP specimens. This decrease in bond strengths and the different fracture pattern highlight the negative effects of artificial aging (LTD) on the bond strength of the self-adhesive cement to zirconia. The lower bond strengths to AG zirconia may have been a result of the structural changes induced by artificial aging or simulated LTD on the zirconia surface. When 3Y-TZP disks were implanted in a denture and worn for 24 hours/day for one year, the material underwent a degradation process in which the increase in the percentage of monoclinic phase was similar to that of zirconia aged in an autoclave at 134°C for six hours.[39] Aging through LTD has been reported to activate the transformation from the tetragonal to the monoclinic phase ($t \rightarrow m$), which is always accompanied by a slight volume change inducing stresses leading to microfissures on the zirconia surface.[35] This phenomenon may have clinical relevancy in case a zirconia-based restoration fractures in the mouth and needs to be repaired.

Other studies have investigated the influence of hydrothermal aging, thermocycling, and mechanical fatigue on bond strengths to zirconia.[40,41] In general, thermal and mechanical fatigue result in a reduction in bond strengths, which is in agreement with our study. Chevalier and others[35] observed nucleation sites in zirconia crystals with formation of nanofissures after the material had been submitted to an aging regimen similar to that used in our study.

Surface abrasion with a tribochemical silica-coated alumina (silicatization) has been shown to be an effective treatment method for zirconia by physically roughening the surface while also leaving behind physically bound silica. Silica coating, therefore, combines micromechanical retention, produced by airborne-particle abrasion, with chemical bonding, and the process is well documented.[40,42,43] However, the silica content on the Y-TZP surface after silica coating is too low for effective silanization.[17] In addition, one of the problems associated with the use of an air brasion method on zirconia has to do with the possibility of air brasion inducing the $t \rightarrow m$ transformation.[44] The presence of the monoclinic phase on the zirconia surface may result in surface alterations that compromise the establishment and durability of reliable micromechanical adhesion.

Piascik and others[45] obtained a mean μTBS of 18.6 MPa to silica-coated zirconia. In our study, the corresponding condition would be the subgoup AR-CO, for which we obtained a mean of 13.1 MPa. The difference may reside in the size of the microtensile specimens. While the cross section of our specimens was approximately 1.0 mm^2, those of Piascik and others were 1.5 mm × 1.5 mm.Z-Prime Plus includes both phosphate monomers and carboxylate monomers. Organophosphate monomers resemble silanes because their methacrylate groups can copolymerize with monomers in the resin cement.[17] Phosphate radicals may bond to metal oxides such as zirconia.[18] In the case of ZP, the carboxylate groups may also enhance the bonding ability of this primer to zirconia.[18] In our study, ZP improved mean μTBS significantly compared with those to NT and CO zirconia surfaces for each of the two aging regimens. Although studies have reported that primers containing phosphorylated monomers improve bonding to zirconia,[15–17] ZP, in the present study, may have also acted as a wetting agent for the resin cement, which would have promoted adhesion as reported for other phosphate monomers applied to alumina.[46] The resistance of this possible chemical bonding to

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hydrolysis and thermal fatigue in the mouth has not been established.[47]

The present study has several limitations, including the absence of mechanical fatigue to simulate clinically relevant loads. Furthermore, we used only one resin cement for two reasons. First, this specific self-adhesive resin cement has been shown to bond better to zirconia than a conventional dual-cured resin cement.[48,49] Second, our experimental hypotheses were not related to the type of resin cement used but to the effect of artificial aging and surface treatment of zirconia.

The results of the present study suggest that new developments in bonding to zirconia may depend on research on new surface treatments that establish chemical interaction with both luting cements and zirconia surfaces. Further studies must test luting agents and primers that include phosphate monomers and silanes in the same solution.

CONCLUSION

Within the limitations of this study, in which zirconia was not veneered with glass-based ceramic as in clinical situations, we have to reject both null hypotheses, as 1) artificial aging of zirconia resulted in a reduction of microtensile bond strengths and 2) zirconia surface treatment with silicatization and with Z-Prime Plus increased μTBS of a phosphate-based self-adhesive resin cement to zirconia.

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 Relationship of Hydrogen Peroxide Exposure Protocol to Bleaching Efficacy

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Clinical Relevance

The use of a linear low-density polyethylene wrap as advocated in the sealed bleaching technique can minimize hydrogen peroxide penetration into the pulp cavity without compromising bleaching efficacy *in vitro*.

SUMMARY

The purpose of this study was to compare two in-office bleaching methods with respect to tooth color change and level of hydrogen peroxide penetration into the pulp cavity and to evaluate relationships between penetration level and color change. Eighty extracted canines were exposed to two different bleaching regimens (conventional vs sealed bleaching technique). After exposure to 38% hydrogen peroxide gel for one hour, hydrogen peroxide amount was estimated spectrophotometrically. Color change was measured per Commission Internationale de l'Eclairage methodology. Linear regression was used to evaluate factors affecting color change, including bleaching technique. The conventional and sealed bleaching groups showed no difference for any color change parameters (ΔL, Δa, Δb, ΔE); however, there was significantly greater hydrogen peroxide penetration in the conventional bleaching group ($p<0.05$). Linear modeling of the change in lightness (ΔL) showed that the increase in lightness tended to be greater for teeth with lower initial L* values ($r=-0.32$, $p<0.05$). After adjustment for initial L*, there was no evidence that ΔL differed with hydrogen peroxide penetration

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levels ($p>0.05$) or bleaching technique (mean group difference in $\Delta L=0.36$; $p>0.05$).

INTRODUCTION

Tooth bleaching is a conservative and highly effective method to whiten discolored teeth. It is a treatment option to enhance the esthetics of the teeth that has been practiced in dentistry for more than 100 years. Thus, the safety and efficacy of this procedure have been well established.[1]

In-office bleaching is generally preferred by both dentists and patients in that the responsibility for the procedure of bleaching teeth is transferred to the dental office.[2] In-office bleaching produces immediate bleaching results and can also be used as a kick start so that patients better comply with home bleaching procedures.

During in-office bleaching, the highly concentrated hydrogen peroxide bleaching gel is usually left on the tooth surface for 5 to 20 minutes and replenished according to the manufacturer's directions. However, irritation to the nasal mucosa caused by evaporation of volatile components in the bleaching gel, inadvertent exposure to the highly concentrated bleaching gel, as well as inconvenience and increased costs associated with multiple replenishment of bleaching gel during one bleaching session have been pointed out as disadvantages of conventional in-office bleaching procedures.[3] To prevent evaporation and desiccation of active agents, placement of a linear low-density polyethylene (LLDPE) wrap onto the bleaching gel had been described as the sealed bleaching technique.[3]

Bowles and Ugwuneri[4] were the first to show that in extracted teeth exposed to hydrogen peroxide, significant levels of hydrogen peroxide could be detected in the pulp cavity. Many studies followed, adopting the newly introduced *in vitro* model to investigate various factors that might influence the amount of hydrogen peroxide penetration into the pulp cavity. Studies have shown that higher concentrations of hydrogen peroxide,[4] heat and prolonged bleaching time,[5] light activation,[6] altered surface due to restorations,[7] and characteristics such as large open dentinal tubules of young teeth[8] facilitate the diffusion and penetration of hydrogen peroxide molecules from the outer tooth surface into the pulp cavity.

However, the effect of different in-office bleaching protocols on the amount of hydrogen peroxide penetration into the pulp cavity has not been investigated. It is also not known whether there is a relationship between hydrogen peroxide penetration levels and the color change of the tooth.

Thus, the purpose of this *in vitro* study was to compare the relationship of the amount of hydrogen peroxide penetration into the pulp cavity between the conventional and sealed bleaching technique and correlate penetration levels with the color change of the tooth. The null hypotheses to be tested were that color change and hydrogen peroxide penetration levels would not differ between the two in-office bleaching methods and that there would be no correlation between hydrogen peroxide penetration levels and tooth color change.

METHODS AND MATERIALS

Sample Selection and Preparation

Eighty extracted human canines were collected three months prior to the study and stored in 0.2% Thymol (Sigma-Aldrich, St Louis, MO, USA) and distilled water at 4°C. All teeth were cleaned and observed for the absence of developmental anomalies, caries, existing restorations, deep crack lines, or severe attrition. The roots were trimmed 3 mm apical to the cementoenamel junction (Figure 1a), and the pulpal tissue was removed with #25 to #40 H-files (Maillefer files, Dentsply Maillefer, Ballaigues, NA, Switzerland). The pulp chamber was slightly enlarged with a round carbide bur (Midwest, Dentsply Professional, Des Plaines, IL, USA) toward the lingual to maintain intact labial tooth structure and still be able to encompass 30 μL of acetate buffer (Figure 1b).

Bleaching Protocol

Fifty maxillary and 30 mandibular canines were randomly assigned to the conventional bleaching group and the sealed bleaching group. Tooth thickness was measured from the outer labial surface to the outer boundary of the pulp cavity at the cross-sectioned root 3 mm below the cementoenamel junction using an electronic digital caliper (Harbor Freight Tools, Pittsburgh, PA, USA; Figure 1c). A jig was fabricated for each tooth by gently placing the lingual surface of the tooth into a polyvinylsiloxane putty impression material (Exaflex, GC America Inc, Alsip, IL, USA) at a 30° angle from the bottom. The baseline color was measured with a spectrophotometer (Spectroshade Micro, MHT, Niederhasli, Switzerland) to provide a topographical color map of the entire tooth in one image (Figure 1d). A resin barrier (OpalDam, Ultradent Products Inc, South Jordan, UT, USA) was placed to cover 0.5 mm of tooth

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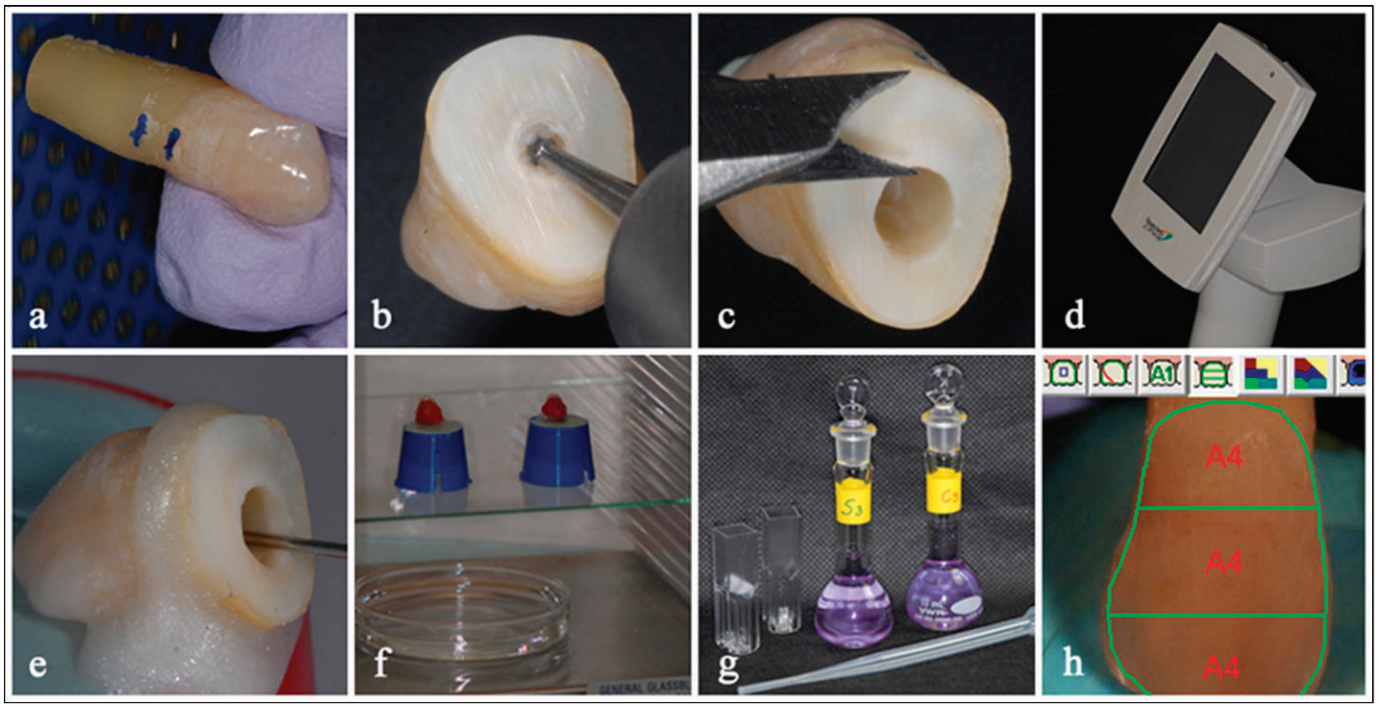

Figure 1. *Step-by-step procedures. (a): The roots of canine teeth were trimmed 3 mm apical to the cementoenamel junction. (b): The pulp chamber was enlarged to encompass 30 μL of acetate buffer. (c): Tooth thickness was measured from the outer labial surface to the outer boundary of the pulp cavity at the cross-sectioned root. (d): Tooth color was measured with a spectrophotometer. (e): Acetate buffer is placed into the pulp cavity. (f): Teeth were placed in a closed humid chamber during the bleaching process. (g): The mixture in the volumetric flask is transferred to cuvettes to be measured in a UV/Visible Spectrophotometer. (h): The color difference is measured with a software analysis program.*

coronal as well as 2 mm of root apical to the cementoenamel junction and light cured for 20 seconds (Elipar S10 LED curing light, 3M ESPE, St Paul, MN, USA).

The pulp cavities were rinsed twice with 30 μL of distilled water and dried with coarse paper points prior to the placement of freshly mixed 30 μL, 2M acetate buffer (pH 4.5; Figure 1e). The acetate buffer acted as a stabilizing agent of hydrogen peroxide that might have diffused into the pulp cavity. Thirty-eight percent hydrogen peroxide gel (Opalescence Boost, Ultradent Products Inc, South Jordan, UT, USA) was then placed onto the labial surface of the canines and subjected to the following groups.

Conventional Bleaching Group—The bleaching gel (80 μL) was applied onto the tooth surface and replenished every 20 minutes for three times according to the manufacturer's directions. A microbrush was used for the removal of the bleaching gel, but no irrigation was performed to avoid any contamination with the acetate buffer in the cavity.

Sealed Bleaching Group—The bleaching gel (80 μL) was applied onto the tooth surface and covered with a linear low-density polyethylene wrap (Professional Plastic Food Wrap Film, Bakers & Chefs Food Equipment Pte Ltd, Singapore) without replenishment of the gel during the bleaching procedure (60 minutes).

All teeth were kept in a closed humid chamber (General Glassblowing Co. Lab Apparatus, Richmond, CA, USA) at room temperature (25°C) with 100% relative humidity during the bleaching procedure (Figure 1f). At the end of the bleaching procedure, the acetate buffer was retrieved and placed in 10 mL volumetric flasks. The pulp cavities were thoroughly rinsed twice with 30 μL of distilled water, and the washes were added to the flasks. After removal of the acetate buffer, the bleaching gel was removed with microbrushes, and the teeth were rinsed with distilled water and stored in individual glass vials for two hours prior to taking postoperative shades with the spectrophotometer.

Measurement of Hydrogen Peroxide Penetration Levels

Hydrogen peroxide penetration levels were estimated according to the method of Mottola and others.[9]

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Table 1: *Baseline Data (Mean/Median [SD]) for Conventional and Sealed Bleaching Group*[a]

Baseline Parameter	CBG	SBG	*p* Value*
L_1*	70.2/70.4 (3.43)	69.8/69.6 (3.78)	0.56
a_1*	2.9/2.7 (1.58)	3.2/3.0 (1.65)	0.54
b_1*	23.7/23.7 (2.62)	24.0/24.2 (2.40)	0.51
Tooth thickness (mm)	2.6/2.2 (0.20)	2.64/2.4 (0.19)	0.95

Abbreviations: CBG, conventional bleaching group; SBG, sealed bleaching group.
[a] *n = 40 in each group.*
* *Significance probability associated with Wilcoxon rank sum test.*

One milliliter of leucocrystal violet solution (0.5 mg/mL), 0.5 mL of horseradish peroxidase solution (1 mg/mL) was added to the volumetric flasks containing the acetate buffer retrieved from the cavity. After addition of 4 mL acetate buffer, the total volume was adjusted to 10 mL with distilled water, and the intensity of the color was measured in a UV/Visible Spectrophotometer (Model Lambda 20, Perkin Elmer, Branford, CT, USA) at a wavelength of 596 nm (Figure 1g). The evaluator taking the spectrophotometer reading was blinded regarding the treatment group. A standard calibration curve with known amounts of hydrogen peroxide was used to determine the amount of hydrogen peroxide in microgram equivalents in the samples.

Determination of Color Change

The color difference of the tooth before and after bleaching was measured as ΔE from the Commission Internationale de l'Eclairage. It was calculated from the following equation: $\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$ with the use of a software analysis program (MHT Software Analysis version 2.43; Figure 1h).

Statistical Methods

Measurements of color change included overall color change (ΔE) as well as changes in lightness (ΔL), the red-green dimension (Δa), and the blue-yellow dimension (Δb). Other measures of interest included hydrogen peroxide penetration and tooth thickness. The nonparametric Wilcoxon rank sum (Mann-Whitney) procedure was used to assess whether the two treatment groups differed at baseline with respect to L*, a*, b*, and tooth thickness. This procedure was also used to evaluate group differences in color change and H_2O_2 penetration following treatment. Multiple linear regression was used to evaluate factors affecting color change, which was measured as ΔL. Candidate covariates entertained in the modeling of a given color change outcome included bleaching technique, tooth thickness, H_2O_2 penetration, and the relevant baseline values of the particular color dimension. Standard residual analyses were carried out to assess validity of assumptions associated with the regression modeling, including residual plots and Shapiro-Wilk tests of normality. Throughout, the level of significance was set at $\alpha = 0.05$.

RESULTS

The conventional and sealed bleaching groups were similar at baseline with respect to the L_1*, a_1*, and b_1* color dimensions, as well as tooth thickness (Table 1; $p > 0.05$ in all instances, Wilcoxon rank sum test).

Based on the Wilcoxon rank sum test, there was no evidence that the two groups differed for any color change measurement (Table 2; Figure 2). In con-

Table 2: *Color Change Data and Hydrogen Peroxide Penetration (Mean/Median [SD]) by Bleaching Group*

	ΔL	Δa	Δb	ΔE	HPP (μg)
CBG	2.35/2.00 (1.58)	−0.87/−0.77 (0.60)	−2.06/−2.13 (1.23)	3.60/3.36 (1.37)	0.54/0.50 (0.20)
SBG	2.05/2.11 (1.49)	−0.73/−0.70 (0.44)	−1.83/−1.87 (1.19)	3.11/2.92 (1.48)	0.33/0.31 (0.16)
p value*	0.62	0.66	0.38	0.15	<0.0001

Abbreviations: CBG, conventional bleaching group; HPP, hydrogen peroxide penetration level; SBG, sealed bleaching group.
* *Significance probability associated with Wilcoxon rank sum test.*

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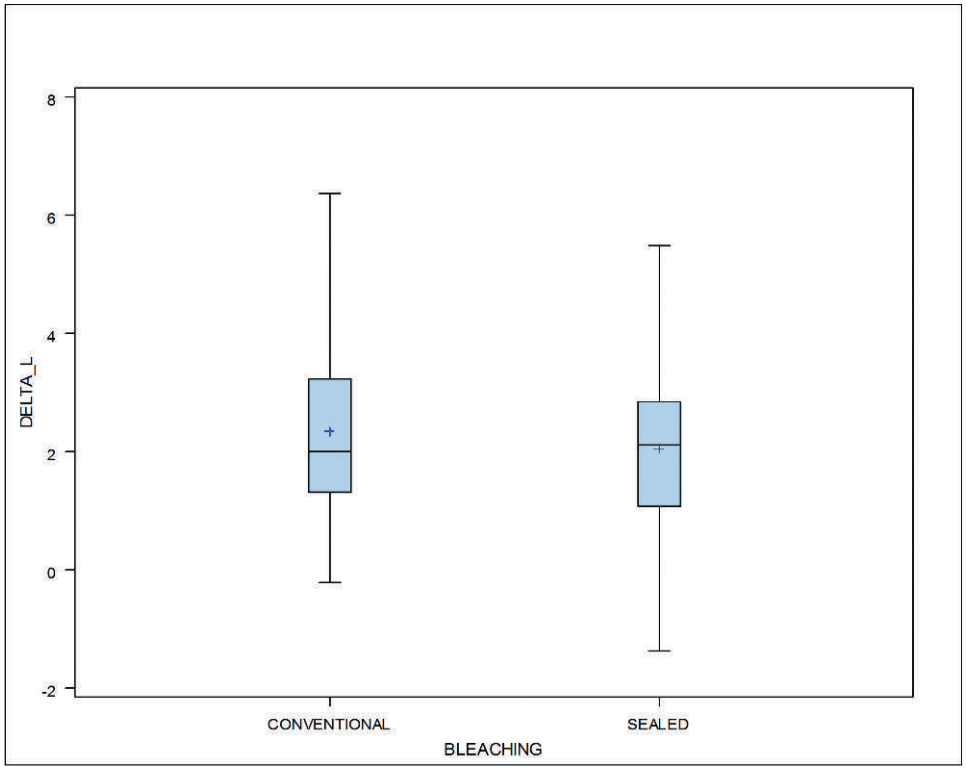

Figure 2. *Box plots for change in lightness (ΔL) relative to baseline by bleaching group.*

trast, the two groups were found to differ significantly in the level of hydrogen peroxide penetration following bleaching ($p<0.05$). In the conventional bleaching group, the mean level of H_2O_2 penetration was significantly higher than in the sealed bleaching group (Table 2). The distribution of hydrogen peroxide penetration levels is illustrated in Figure 3.

There was interest in whether color change was also related to other measures, such as tooth thickness and H_2O_2 penetration. Pearson correlation coefficients were used as measures of bivariate association (Table 3). None of the four color change measures appeared to be correlated with hydrogen peroxide penetration. Noteworthy are the highly significant correlations of ΔL with baseline lightness ($r=-0.32$, $p<0.05$) and the correlation of Δa with baseline values of a* ($r=-0.42$, $p<0.05$). In addition, changes in the red-green color dimension (Δa) were strongly correlated with tooth thickness ($r=0.37$, $p<0.05$).

These bivariate correlations indicate that those teeth that were initially darker tended to show greater increases in lightness after bleaching treatment. In the case of the a* dimension, the changes were primarily negative, that is, toward the green end of the red-green dimensional scale. Those teeth that showed the greatest change tended to be those that had the highest baseline a* levels and the smallest tooth thicknesses.

Change in the blue-yellow color dimension (Δb) was also correlated with tooth thickness ($r=0.25$, $p<0.05$). These changes were also overwhelmingly negative, that is, shifted toward the blue end of the blue-yellow dimensional scale. Those teeth that showed the greatest change tended to be those that had the smallest tooth thicknesses.

The issue of group comparisons was therefore revisited in the context of multiple linear regression, which made it possible to reassess group differences after adjustment for covariates.

Linear modeling of the change in lightness (ΔL) showed that the increase in lightness tended to be greater for teeth with lower initial L* values ($r=-0.32$, $p<0.05$). After adjustment for initial L*, there was no evidence that ΔL differed with bleaching technique ($p>0.05$). The mean difference in ΔL between the two treatment groups was 0.36 lightness units. The adjusted (for baseline L) means for ΔL were 2.38 for the conventional bleaching group and 2.02 for the sealed bleaching group. No

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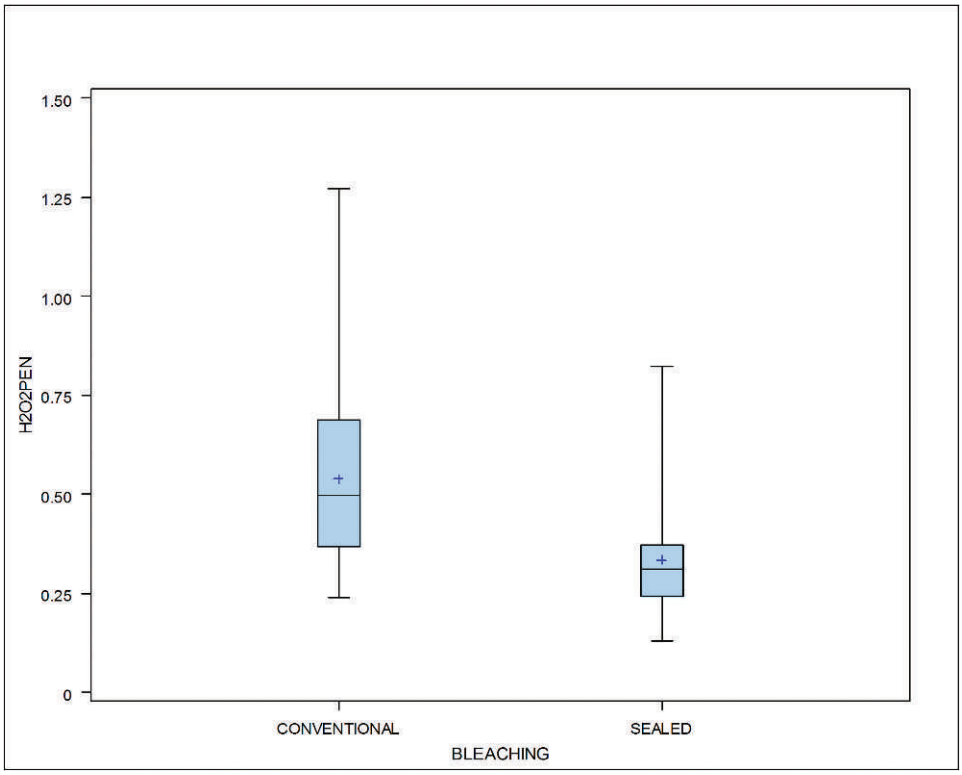

Figure 3. *Box plots for hydrogen peroxide penetration level by bleaching group.*

other covariate was significantly associated with change in lightness (Figure 4).

DISCUSSION

This study compared two in-office bleaching techniques, the conventional and sealed bleaching group, with respect to four color change parameters and hydrogen peroxide penetration levels. Following bleaching treatment, the two groups were similar in terms of color change relative to baseline. The mean overall color changes for the conventional and sealed bleaching groups were 3.60 and 3.11, respectively, which is considered to be discernible to the naked eye[10] and reflects the clinical relevance of this study.

The results indicated that the application of a 38% hydrogen peroxide gel for one hour without replenishment was as effective as three 20-minute applications. Similar results were obtained in an *in vitro* pilot study by Marson and others.[11] They reported no difference in lightness change after bleaching between a single 45-minute and three 15-minute applications. In their chemical analysis to quantify

Table 3: *Correlations of Color Change Parameters With Tooth Thickness, HPP, and baseline Color Measurements*[a]

	Tooth Thickness	HPP	L_1^*	a_1^*	b_1^*
ΔL	−0.03 (0.8169)	0.16 (0.1544)	−0.32 (0.0038)	0.41 (0.0001)	0.07 (0.5551)
Δa	0.37 (0.0009)	−0.02 (0.8935)	0.26 (0.0187)	−0.42 (<0.0001)	−0.22 (0.0469)
Δb	0.25 (0.0249)	0.09 (0.4135)	−0.04 (0.7388)	−0.02 (0.8477)	−0.17 (0.1325)
ΔE	−0.19 (0.0840)	0.12 (0.2757)	−0.29 (0.0104)	−0.39 (0.0004)	0.15 (0.1824)

Abbreviation: HPP, hydrogen peroxide penetration.
[a] *Pearson correlation coefficients, n=80, Prob>|r| under H_O:Rho=0,* p *value in parentheses.*

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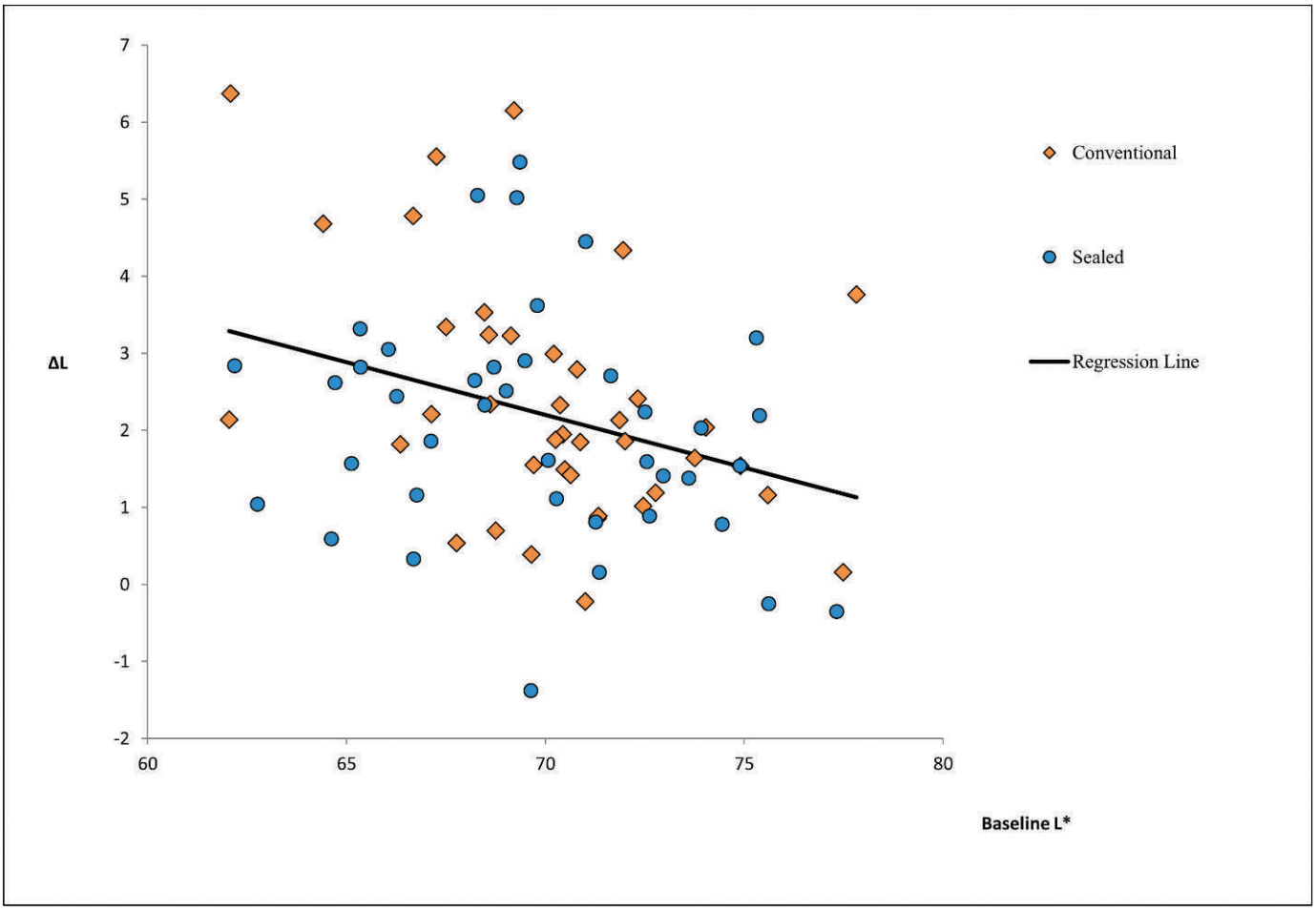

Figure 4. *Linear regression of change in lightness (ΔL) on baseline L*.*

the concentration of hydrogen peroxide as a function of time, they showed only a minor change from 34% at baseline to 29% after 40 minutes. This may suggest the rationale of not having to replenish the gel during a single in-office bleaching session.

However, Reis and others[12] reported contradictory results in their recent clinical trial. In-office bleaching was performed in 30 participants with 35% hydrogen peroxide. They reported superior bleaching results and less sensitivity with replenishment of the bleaching gel compared with a single prolonged application. The difference of these results may be explained by the use of the LLDPE used in our study as advocated in the sealed bleaching technique. The LLDPE wrap may prevent dehydration of the gel and rapid degradation of active agents in the gel, thus making replenishment less critical.

The two in-office bleaching methods were found to differ significantly in the level of hydrogen peroxide penetration following bleaching. In the conventional bleaching group, the mean level of H_2O_2 penetration was higher (ie, 0.54 μg). In the sealed bleaching group, the mean level of penetration was significantly lower, 0.33 μg. The mean hydrogen peroxide penetration levels of both groups are much lower than in a previous study by Bowles and Ugwuneri,[4] who applied 30% hydrogen peroxide for 15 minutes at 37°C and detected hydrogen peroxide levels of 25.4 ± 8.5 μg. This difference can be explained by the difference of cavity preparation, tooth selection, hydrogen peroxide delivery method, and temperature settings. First, the cavity preparation was more conservative in our study by encompassing a smaller amount of acetate buffer and confining the enlargement of the pulp cavity to the lingual side. Second, central and lateral incisors were used in the study by Bowles and Ugwuneri,[4] whereas bulkier canines were selected in this study. Third, the facial surfaces of teeth were immersed in liquid hydrogen peroxide at 37°C, which might have created hydrogen peroxide penetration by capillary action directly into the

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pulp cavity rather than from diffusion from the outer surface in Bowles and Ugwuneri's study.

There are many factors affecting the hydrogen penetration level, and the levels seem to differ according to the experimental protocol employed. It is important to understand the clinical significance of hydrogen peroxide penetration into the pulp cavity and the possible risk associated with significant levels of hydrogen peroxide penetration. The threshold for pulpal enzyme inhibition, which was calculated to be in the range of 50 mg, explains why pulpal damage resulting from the clinical use of in-office bleaching procedures has been remarkably low.[4] However, considering the lack of knowledge of the effect of hydrogen peroxide penetration at the molecular level within the cell and connective tissue of the pulp,[13] it is challenging to assess the actual comparative clinical difference between the small values of recovered hydrogen peroxide in our study.

Bleaching involves a series of complex changes that alter a set of separate color parameters, of which L* is generally regarded as the primary one and also the most used to assess the effectiveness of a bleaching procedure.[14] Modeling of ΔL showed that it was not affected by other covariates except for initial lightness values, which seem to make it a consistent measure for evaluating bleaching efficacy. It is also noteworthy to point out the importance of taking initial baseline values into consideration when comparing different treatment groups in bleaching studies since they affect the amount of change in lightness (ΔL).

This *in vitro* model is representative of the *in vivo* process, although it is not known how closely it would compare to the *in vivo* absorption of hydrogen peroxide in teeth with vital pulps exhibiting positive pulpal pressure during the bleaching process.[4]

Another limitation of this study was that it did not consider the cumulative effect of color change with repeated conventional vs sealed bleaching technique. Although an *in vitro* study by Rosenstiel and others[15] has shown that color changes beyond the first in-office bleaching treatment were small, repeated bleaching with a different bleaching regimen might result in other findings.

This study explored the tooth color change and the amount of hydrogen peroxide penetration levels into the pulp cavity by comparing two different in-office bleaching treatments, and the findings supported the null hypothesis that the color change would not differ between the two techniques. However, there was a significant difference between hydrogen peroxide penetration levels, so the second null hypothesis had to be rejected. There was no correlation between penetration levels and tooth color change, which led to the acceptance of the third null hypothesis.

Based on these findings, further studies should be employed to evaluate the significance of hydrogen peroxide penetration levels at the molecular level of pulpal cells and the clinical significance of these penetration levels. Different bleaching agent concentrations and various delivery methods should be assessed regarding hydrogen peroxide penetration levels as well as color change and ultimately suggest a bleaching regimen with minimal hydrogen peroxide penetration and maximum bleaching efficacy.

CONCLUSION

Within the limitation of this study, the sealed bleaching technique compared with the conventional in-office technique exhibited lower hydrogen peroxide penetration levels without compromising bleaching efficacy in terms of all parameters in color change. Change in lightness was not affected by hydrogen peroxide penetration levels or bleaching techniques after adjustment for initial L*.

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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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In Vitro Shear Bond Strength of Three Self-adhesive Resin Cements and a Resin-Modified Glass Ionomer Cement to Various Prosthodontic Substrates

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Clinical Relevance

Self-adhesive resin cements demonstrated superior bond strength to a variety of prosthodontic substrates relative to resin-modified glass ionomer cement, indicating that they are able to provide a wider array of clinical applications. However, selection of the cement should be determined largely by the type of substrate and setting reaction.

SUMMARY

Objective: To evaluate the shear bond strength (SBS) of three self-adhesive resin cements and a resin-modified glass ionomer cement (RMGIC) to different prosthodontic substrates.

Materials and Methods: The substrates base metal, noble metal, zirconia, ceramic, and resin composite were used for bonding with different cements (n=12). Specimens were placed in a bonding jig, which was filled with one of four cements (RelyX Unicem, Multilink Automix, Maxcem Elite, and FujiCEM Automix). Both light-polymerizing (LP) and self-polymerizing (SP) setting reactions were tested. Shear bond strength was measured at 15 minutes and 24 hours in a testing device at a test speed of 1 mm/min and expressed in MPa. A Student *t*-test and a one-way analysis of variance (ANOVA) were used to evaluate differences between setting reactions, between testing times, and among cements irrespective of other factors. Generalized linear regression model and Tukey tests were used for multifactorial analysis.

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Results: Significantly higher mean SBS were demonstrated for LP mode relative to SP mode ($p<0.001$) and for 24 hours relative to 15 minutes ($p<0.001$). Multifactorial analysis revealed that all factors (cement, substrate, and setting reaction) and all their interactions had a significant effect on the bond strength ($p<0.001$). Resin showed significantly higher SBS than other substrates when bonded to RelyX Unicem and Multilink Automix in LP mode ($p<0.05$). Overall, FujiCEM demonstrated significantly lower SBS than the three self-adhesive resin cements ($p<0.05$).

Conclusions: Overall, higher bond strengths were demonstrated for LP relative to SP mode, 24 hours relative to 15 minutes and self-adhesive resin cements compared to the RMGICs. Bond strengths also varied depending on the substrate, indicating that selection of luting cement should be partially dictated by the substrate and the setting reaction.

INTRODUCTION

The long-term success of indirect restorations depends on several factors, including an adequate design, preparation, and selection of the restorative material. An aspect equally important to the longevity of indirect restorations is the integrity of the bonded interface between the tooth and the restoration. Currently, most resin cements use an etch-and-rinse or a self-etch adhesive in combination with a low-viscosity dual polymerizing resin cement.[1] However, this multi-step bonding procedure is complex, technique sensitive, and it involves significant chair time. A new generation of self-adhesive resin cements has been developed recently that eliminates the need for etching, priming, and bonding as separate steps. These self-adhesive resin cements are based on new monomer, filler, and initiator formulations. The acidic monomer replaces the previous three steps by combining the use of adhesive and cement into a single application. These multi-functional phosphate-based acidic methacrylates can react with the basic fillers in the luting cement and the hydroxyapatite of the hard tooth tissue.[2] Self-adhesive resin cements combine the high-strength and low-solubility advantages of resin cements with the characteristic ease of use of self-adhesive systems, making them highly attractive to the clinician.

Evidence is limited as to how the bond strength of newer self-adhesive resin cements compares to that of conventional self-adhesive resin-modified glass ionomer cements (RMGICs) when bonded to a variety of prosthodontic materials under multiple testing conditions. While RMGICs are self-adhesive and provide simultaneous fluoride release, aspects such as water absorption with the associated setting expansion, potential for crack development, and less associated esthetics make these cements less than ideal for situations such as the cementation of all-ceramic crowns.[3] Recent studies have shown higher bond strengths for self-adhesive resin cements compared to RMGICs when bonded to a variety of materials such as noble and non-noble alloys, zirconia, aluminum oxide ceramic, and pressable ceramic.[4,5]

Different studies have reported on the bond strength of self-adhesive resin cements to enamel and dentin,[1,2,6-9] as well as different substrates such as alloys,[10-13] ceramics,[4,14] and polymers.[15] However, most of these studies concentrate on a single substrate, type of setting reaction, or testing time. As self-adhesive cements continue to gain popularity for the cementation of indirect restorations, large comparative studies are needed to gain a better understanding of the overall behavior of these cements under multiple testing conditions and when bonded to a variety of prosthodontic substrates.

Therefore, the objective of this study was to evaluate the shear bond strength (SBS) of three dual polymerized self-adhesive resin cements and a RMGIC to a variety of prosthodontic substrates (base metal, noble metal, zirconia, ceramic, and resin composite). Furthermore, this study aimed to evaluate differences in SBS values between 15 minutes and 24 hours and between self-polymerizing (SP) and light-polymerizing (LP) setting reactions for the different cement-substrate combinations. The null hypothesis was that there would be no significant difference in mean SBS among the tested cements, between SP and LP modes, and between 15 minutes and 24 hours.

MATERIALS AND METHODS

Bonding Substrates

The bonding substrates, including commercial names and composition are summarized in Table 1. One hundred ninety-two specimens were prepared for each of the following substrates: base metal, noble alloy, densely sintered yttrium-stabilized zirconia, lithium disilicate glass ceramic, and resin composite. For the metallic substrates, noble metal rectangular pieces (15 mm long $\times$ 5 mm wide $\times$ 1 mm high) and base metal cylindrical blocks (10 mm diameter and 5 mm thick) were used. The original

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Table 1: *Tested Materials, Type, Composition and Batch Numbers as Per Manufacturer's Descriptions*

Group	Type	Composition	Lot No.	Manufacturer
Self-adhesive Cements				
RelyX Unicem	Dual polymerized self-adhesive resin cement	55%-65% Glass powder 15%-25% Methacrylated phosphoric acid esters 10%-20% TEGDMA 1%-5% Silane-treated silica 1%-5% Sodium persulfate	337810	3M ESPE (St Paul, MN, USA)
Multilink Automix	Dual polymerized adhesive resin cement with self-etching primer	22%-26% Dimethacrylates 6%-7% HEMA <1% Benzoyl peroxide 40% Barium glass, YF_3, spheroid mixed oxide	L27890	Ivoclar Vivadent (Schaan, Liechtenstein)
Maxcem Elite	Dual polymerized self-adhesive resin cement	19%-40% Methacrylate esther monomers Other—inert mineral fillers, activators stabilizers, colorants, YF_3	3100070	Kerr Corporation (Orange, CA, USA)
FujiCEM Automix	Resin-modified glass ionomer cement	30%-40% Polyacrylic acid 30%-40% Distilled water 2% Silica powder 20% Silicone dioxide 2%-3% Benzensulfonic acid sodium salt	0404091	GC America (Alsip, IL, USA)
Bonding Substrates				
Base metal	Identalloy	Co 60% Cr 30% Other 10%		Ivoclar Vivadent (Schaan, Liechstenstein)
Noble metal	Harmony Medium	Au 77% Ag 13% Cu 8% Other 2%		Ivoclar Vivadent (Schaan, Liechstenstein)
Zirconia	IPS e.max ZirCAD	87% ZrO_2, Y_2O_3, HfO_2, Al_2O_3		Ivoclar Vivadent (Schaan, Liechstenstein)
Ceramic	IPS e.max CAD	> 57% SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, $Al2O_3$, MgO and pigments		Ivoclar Vivadent (Schaan, Liechstenstein)
Composite resin	Z100	80%-90% Silane-treated ceramic 1–10% BisGMA 1–10% TEGDMA <1% 2-Benzotriazolyl-4-methylphenol		3M ESPE (St Paul, MN, USA)

Abbreviations: Bis-GMA, bisphenol A glycidyl dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; YF_3, ytterbium trifluoride.

zirconia and ceramic blocks were cut using a low-speed saw (Isomet, Buehler, Lake Bluff, IL, USA) to obtain square blocks (10 mm × 10 mm × 2 mm). The zirconia specimens were sintered following manufacturer's recommendations. The composite specimens were fabricated using a ring-shaped mold (10 mm diameter and 2 mm height) and light polymerized.

All substrates were embedded in a chemically polymerized methacrylate (Fastray, HJ Bosworth, Skokie, IL, USA). The exposed surfaces were sequentially polished with 320-, 400-, and 600-grit silicon carbide abrasive paper (SiC sandpaper, Buehler) under water and air abraded with 50-μm aluminum oxide particles at 1 bar and a distance of 10 mm for 10 seconds. The specimens were stored in dry conditions at room temperature until ready to be bonded.

Bonding and Testing

The cements tested are listed in Table 1. A sample size of 12 specimens per study group (n=12) were

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prepared. All cement systems were mixed and applied according to the manufacturer's recommendations for each substrate. For those cements requiring the use of a primer, as for Multilink Automix, the corresponding primer (Monobond Plus, Ivoclar Vivadent, Amherst, NY, USA) was used before application of the cement. All bonding procedures were carried out in a temperature-, humidity-, and light-controlled room with overhead lighting that used orange filters to avoid polymerization of the materials due to ambient light photoactivation. To avoid bias during the bonding procedures, study groups were randomized.

The SBS for each cement-substrate combination was tested at 15 minutes and 24 hours in both SP and LP setting reactions. The specimens were secured using a specially fabricated jig (Bonding jig, Ultradent, South Jordan, UT, USA) with a cylindrical mold of 2.38 mm in diameter. The corresponding cement was injected into the cylindrical mold, which was not filled to the top. For the LP groups, specimens were polymerized following manufacturer's recommendations with a light curing unit (Bluephase C8, Ivoclar Vivadent). A minimum power density of 800 mW/cm^2 was ensured by periodically monitoring the unit's output with a radiometer (Demetron, Kerr, Orange, CA, USA). All specimens were stored at 37°C and 100% humidity until ready to be tested.

Shear bond strength was measured using a testing machine (Ultratester, Ultradent) at a test speed of 1 mm/min. A notched crosshead designed to match the diameter of the bonded specimen was used to apply the testing load (Figure 1). Specimens were stabilized in a testing jig, which was free to move to facilitate positioning under the load. The test base was then positioned so that the notched crosshead was placed against the specimen surface, and the notch was fitted on the diameter of the bonded specimen. The load required to debond the specimen was recorded and expressed in MPa by dividing the load by the surface area of the bonded specimen, and the mean SBS for each study group was calculated.

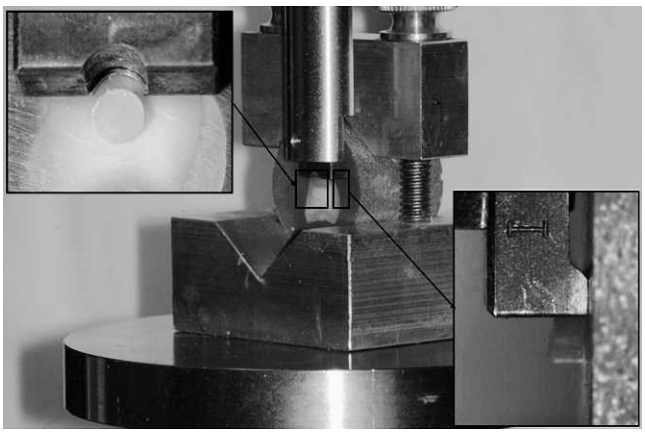

Figure 1. *Shear bond strength universal testing machine with a notched cross-head matching the diameter of the bonded specimen.*

Statistical Analyses

A Student *t*-test was used to determine whether significant differences existed between setting reactions (SP vs LP) and between testing times (15 minutes vs 24 hours) regardless of other variables. A one-way analysis of variance (ANOVA) was performed to evaluate whether significant differences existed among cements stratified by setting reaction and testing time irrespective of the substrate. A multifactorial analysis with generalized linear model was used to evaluate the effect of multiple covariates (substrate, cement, and setting reaction) and all their interactions on SBS at each testing time. *Post hoc* analysis with Tukey test was conducted to explore the presence of significant differences between specific substrate-cement combinations for each testing condition (setting reaction and testing time). For each substrate-cement combination, we also reported pretesting failures or samples spontaneously debonded prior to testing. A significance level of 0.05 was used for all tests. All statistical analysis was performed with Statistical Package for Social Sciences (SPSS) version 16.0 (SPSS Inc, Chicago, IL, USA).

RESULTS

Effect of the Setting Reaction

Student *t*-test revealed significantly higher mean SBS values for LP relative to SP groups at both testing times irrespective of substrate-cement interactions ($p<0.001$). At 15 minutes, mean SBS values were 11.4 ± 0.5 and 3.3 ± 0.1 for LP and SP, respectively. At 24 hours, mean SBS values were 15.8 ± 0.8 and 11.6 ± 0.4 MPa for LP and SP, respectively. However, when specific interactions were considered by a one-way ANOVA, a few exceptions were observed. FujiCEM did not show significant differences between SP and LP modes when evaluated at either 15 minutes ($p=0.40$) or at 24 hours ($p=0.54$). All self-adhesive resin cements showed significant differences between SP and LP modes irrespective of the substrate at both testing times. The only exception was RelyX Unicem, which

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Table 2: *Mean Shear Bond Strength in MPa for the Different Cements by Setting Reaction and Testing Time**

Cements	SP/15 min Mean (SE)	SP/24 h Mean (SE)	LP/15 min Mean (SE)	LP/24 h Mean (SE)
RelyX Unicem	1.5 (0.1)[a]	18.5 (0.8)[a]	11.4 (0.6)[a]	18.7 (1.3)[a,b]
Maxcem Elite	5.1 (0.2)[b]	11.7 (0.6)[b]	10.6 (0.4)[a]	16.8 (0.5)[b]
Multilink Automix	2.6 (0.3)[c]	8.9 (0.3)[c]	19.0 (1.1)[b]	21.8 (2.1)[a]
FujiCEM Automix	4.0 (0.1)[d]	5.3 (0.3)[d]	4.0 (0.2)[c]	5.6 (0.3)[c]

Abbreviations: LP, light-polymerizing mode; SE, standard error; SP, self-polymerizing mode.
** Different superscript letters represent significant differences between cements for each testing condition irrespective of substrate (Tukey test).*

demonstrated no differences between SP and LP modes when evaluated at 24 hours (p=0.89).

Effect of the Testing Time

Student t-test also demonstrated significantly higher mean SBS values at 24 hours relative to 15 minutes for both setting reactions irrespective of substrate-cement combination ($p<0.001$). However, when specific substrate-cement interactions were considered in a *post hoc* analysis, a few exceptions showed no significant differences between testing times. In LP mode, no significant differences were detected between 15 minutes and 24 hours for Multilink Automix bonded to base metal (p=0.87) and noble metal (p=0.12), RelyX Unicem bonded to ceramic (p=0.97), and FujiCEM bonded to base metal (p=0.47) and zirconia (p=0.99). In SP mode, no significant differences between 15 minutes and 24 hours were found for FujiCEM bonded to zirconia (p=0.13) and ceramic (p=0.96).

Effect of the Cement

Table 2 summarizes mean SBS values for the different cements under the different testing conditions. One-way ANOVA revealed significant differences in mean SBS values among cements

Table 3: *Generalized Linear Model for Multiple Comparisons of Substrates, Cements, and Setting Reactions, as Well as Their Interactions (n=941)*

Variable	Type III Sum of Squares	*df*	Mean Square	F	Sig
Intercept	100378.499	1	100378.499	3523.329	0.000
Cement	10126.694	3	3375.565	118.484	0.000
Substrate	7018.774	4	1754.693	61.590	0.000
Setting reaction	9268.924	1	9268.924	325.343	0.000
Cement * substrate	7024.988	12	585.416	20.548	0.000
Cement * setting reaction	6245.785	3	2081.928	73.077	0.000
Substrate * setting reaction	3994.508	4	998.627	35.052	0.000
Cement * substrate * setting reaction	5197.472	12	433.123	15.203	0.000

Abbreviations: Adjusted R^2,0.643; df*, degree of freedom; F, F statistic; Sig, significance level.*

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irrespective of substrate for each setting reaction and testing time ($p<0.001$). *Post hoc* analysis with Tukey test revealed that all cements were significantly different from each other ($p<0.001$) except for RelyX Unicem and Maxcem Elite in LP mode at 15 minutes ($p=0.85$). In LP mode at 24 hours, no significant differences were shown between RelyX Unicem and Multilink Automix ($p=0.34$) and RelyX Unicem and Maxcem Elite ($p=0.71$). The highest mean SBS was shown for RelyX Unicem and Multilink Automix in LP mode at 24 hours with values of 18.7 and 21.8 MPa, respectively.

Effect of Multiple Factors

Table 3 summarizes the results from the multifactorial analysis. The generalized linear model revealed that all factors (cement, substrate, and setting reaction) as well as all of their interactions were found to have a significant effect in the SBS ($p<0.001$).

Mean SBS values for the different cement-substrate combinations under different testing conditions are summarized in Figure 2 and Table 4. Significant differences were evidenced between cements for each substrate under the same testing conditions (letters in Table 4), and between substrates for each cement under the same testing conditions (letters in Figure 1). As shown in Figure 1, resin specimens bonded to RelyX Unicem and Multilink Automix at 24 hours in LP mode showed significantly higher SBS than the other substrates ($p<0.05$). Resin specimens bonded to RelyX Unicem also demonstrated significantly higher SBS than the other substrates at 24 hours in SP mode ($p<0.05$). Base metal bonded to Maxcem Elite showed significantly higher SBS than the other substrates at 24 hours ($p<0.05$). Post hoc analysis with Tukey test shown in Table 4 revealed that FujiCEM Automix consistently showed significantly lower mean SBS than all other self-adhesive resin cements ($p<0.05$). The only exception was when the different substrate-cement combinations were evaluated in SP mode at 15 minutes.

Pretesting failures or specimens spontaneously debonded prior to testing were observed in some groups. As shown in Table 4, Rely X Unicem and Multilink Automix showed debonding of only one specimen from noble and base metal, respectively,

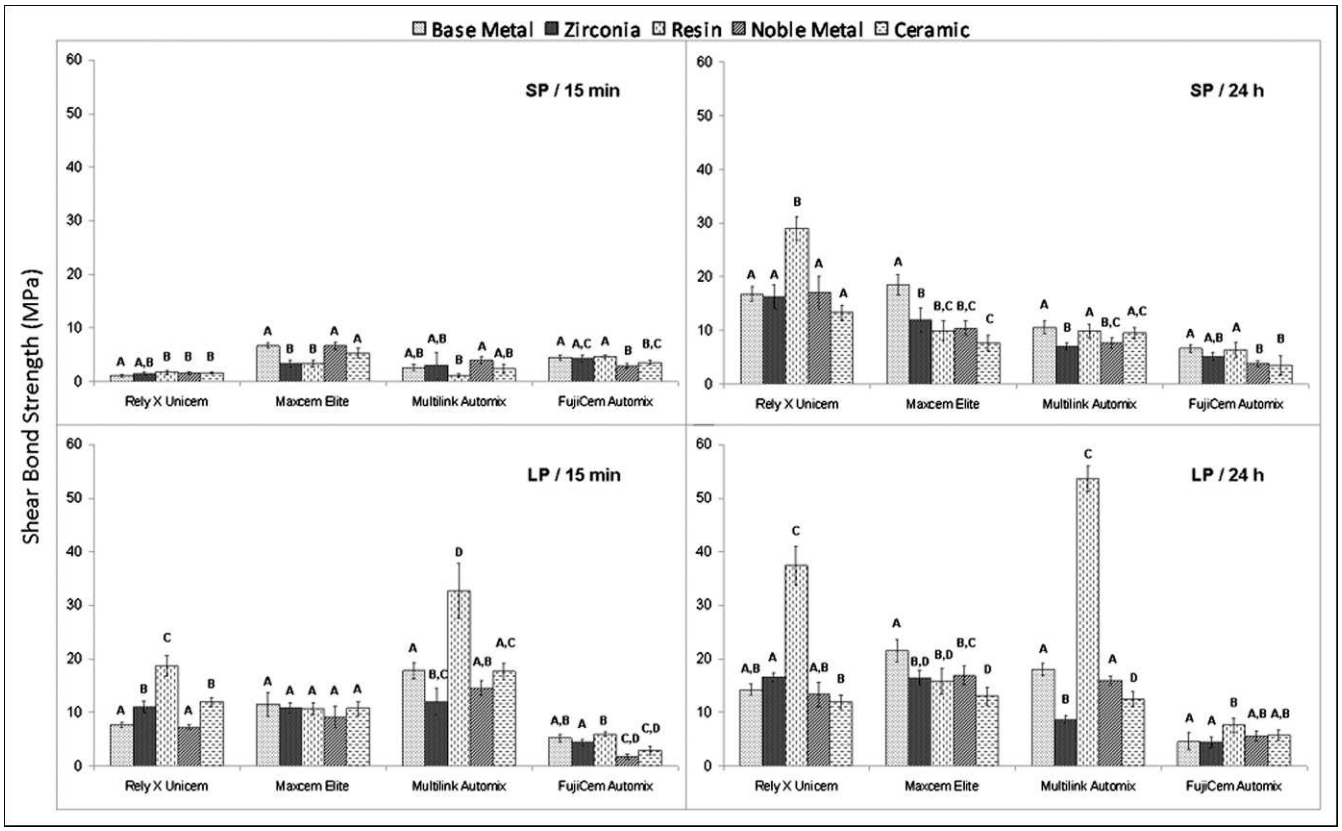

Figure 2. *Mean shear bond strengths in MPa for the different study groups. For each time, polymerization method, and cement, same letter indicates substrates that are not statistically different under the same testing conditions (setting reaction and testing time).*

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Table 4: *Mean Shear Bond Strengths in MPa for Each of the Tested Groups With Number of Pretest Failures or Samples Spontaneously Debonded Prior to Testing**

Substrate	Time	Mode	RelyX Unicem		Maxcem Elite		Multilink Automix		FujiCEM Automix	
			Mean (SE)	No. PTF	Mean (SE)	No. PTF	Mean (SE)	No. PTF	Mean (SE)	No. PTF
Base metal	15 min	SP	1.0 (0.1)^{a}	0	6.7 (0.2)^{b}	0	2.6 (0.3)^{c}	0	4.5 (0.2)^{d}	0
		LP	7.6 (0.2)^{a}	0	11.5 (1.1)^{b}	0	17.8 (0.8)^{c}	0	5.2 (0.3)^{d}	0
	24 h	SP	16.8 (0.7)^{a}	0	18.5 (1.0)^{a}	0	10.6 (0.6)^{b}	0	6.6 (0.4)^{c}	0
		LP	14.3 (0.5)^{a}	0	21.6 (1.1)^{b}	0	18.0 (0.5)^{c}	1	4.6 (0.8)^{d}	0
Noble metal	15 min	SP	1.6 (0.1)^{a}	0	6.6 (0.4)^{b}	0	4.0 (0.3)^{c}	0	2.9 (0.2)^{d}	0
		LP	7.2 (0.2)^{a}	1	9.2 (1.0)^{a}	0	14.6 (0.7)^{b}	0	1.8 (0.2)^{c}	2
	24 h	SP	17.1 (1.6)^{a}	0	10.4 (0.7)^{b}	0	7.7 (0.5)^{b}	0	3.8 (0.4)^{c}	0
		LP	13.4 (1.2)^{a}	0	16.9 (0.8)^{b}	0	15.9 (0.4)^{b}	0	5.6 (0.6)^{c}	2
Zirconia	15 min	SP	1.4 (0.1)^{a}	0	3.3 (0.3)^{a,b}	0	3.0 (1.3)^{a,b}	0	4.3 (0.3)^{b}	1
		LP	11.0 (0.6)^{a}	0	10.9 (0.5)^{a}	0	12.1 (1.3)^{a}	0	4.4 (0.3)^{b}	0
	24 h	SP	16.2 (1.2)^{a}	0	11.9 (1.1)^{b}	0	7.1 (0.3)^{c}	0	5.1 (0.4)^{c}	0
		LP	16.6 (0.4)^{a}	0	16.5 (0.7)^{a}	0	8.7 (0.4)^{b}	0	4.4 (0.5)^{c}	0
Ceramic	15 min	SP	1.7 (0.1)^{a}	0	5.3 (0.4)^{b}	0	2.4 (0.5)^{a}	0	3.6 (0.1)^{c}	0
		LP	12.0 (0.4)^{a}	0	10.8 (0.7)^{a}	0	17.6 (0.8)^{b}	0	2.9 (0.1)^{c}	2
	24 h	SP	13.4 (0.7)^{a}	0	7.7 (0.7)^{b}	0	9.5 (0.5)^{b}	0	3.6 (0.9)^{c}	7
		LP	12.0 (0.7)^{a}	0	13.0 (0.9)^{a}	0	12.5 (0.7)^{a}	0	5.8 (0.4)^{b}	0
Resin	15 min	SP	1.7 (0.2)^{a}	0	3.4 (0.3)^{b}	2	1.1 (0.2)^{a}	0	4.6 (0.1)^{c}	0
		LP	18.6 (1.0)^{a}	0	10.7 (0.6)^{b}	0	32.7 (2.6)^{c}	0	6.0 (0.2)^{d}	0
	24 h	SP	29.0 (1.2)^{a}	0	10.0 (1.1)^{b}	0	9.9 (0.7)^{b}	0	6.5 (0.7)^{c}	0
		LP	37.4 (1.8)^{a}	0	15.8 (1.3)^{b}	0	53.6 (1.2)^{c}	0	7.6 (0.7)^{d}	0

Abbreviations: LP, light-polymerizing mode; PTF, Pretesting failures; SE, standard error; SP, self-polymerizing .
** Groups with the same superscript letter indicate cements that are not significantly different for each substrate under the same conditions (Tukey test).*

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and Maxcem Elite showed debonding of two resin specimens. FujiCEM showed a much higher rate of pretesting failures predominantly to noble metal and ceramic, with four and nine samples debonded, respectively.

DISCUSSION

This study evaluated the shear bond strength of a number of cements bonded to a variety of prosthodontic substrates tested at different times (15 minutes and 24 hours) and undergoing different setting reactions (SP and LP). The null hypothesis was rejected as significant differences in SBS were detected among cements, between SP and LP modes, and between 15 minutes and 24 hours. Furthermore, results from the multiple comparisons revealed that all interactions between the tested factors were also found to be significant with certain combinations of cement, substrate, and setting reaction showing improved bond strengths.

Effect of the Setting Reaction

Overall, significantly higher SBS were demonstrated when specimens were light-activated compared to values generated when the cements were allowed to self-polymerize. Similar findings have been reported in the literature.[4,16-18] Light polymerization yielded improved SBS of the three self-adhesive resin cements, which was also shown to be dependent on the cement. Different monomer composition and polymerization conditions have been shown to alter the degree of conversion, resulting in variations in bond strength results.[19] FujiCEM was the only cement that did not show differences between setting reactions at either 15 minutes or 24 hours. A recent report demonstrated that RMGIC acid-base and visible light polymerization reactions inhibit one another during the early phases of setting,[20] which may help explain why no differences were observed between SP and LP mode for FujiCEM. With the exception of FujiCEM, all substrate-cement combinations demonstrated higher SBS when light-activated. The only exception was RelyX Unicem, which demonstrated no differences between SP and LP modes when evaluated at 24 hours. Both RMGIC and self-adhesive resin cements set by an acid-base reaction as well as a free radical polymerization reaction. While dual polymerizing systems are known to compensate for light attenuation through the thickness of the indirect restoration,[21] it has been shown that, compared to RMGIC, resin cements typically exhibit higher bond strengths when light-activated.[4] A number of studies have reported higher degree of conversion under light-polymerization conditions for resin-based materials.[17,18,22]

Effect of the Testing Time

The SBS of all cements were also shown to be higher after 24 hours relative to 15 minutes irrespective of the substrate and type of setting reaction. This might have been the result of the continued post-irradiation polymerization reaction known to take place after the reaction is initiated and that lasts for up to 24 hours.[23,24] With the exception of a few groups, most substrate-cement combinations demonstrated an increase in SBS after 24 hours when evaluated in both setting reactions. In general, these differences remained significant and were more apparent for the three self-adhesive resin cements than the RMGIC. Similar findings of increased bond strength after 24 hours have been reported previously for different cements.[25] FujiCEM showed only a slight increase in mean SBS from 15 minutes to 24 hours. Some FujiCEM groups showed no change (FujiCEM bonded to ceramic in SP mode and FujiCEM bonded to zirconia in LP mode) or even a decrease in SBS values (FujiCEM bonded to base metal in LP mode) after 24 hours. Although our study did not formally measure the extent of the polymerization reaction, the slight-to-no increase in SBS values for FujiCEM after 24 hours suggests an apparent contradiction with previous studies, which have demonstrated that the RMGIC acid-base reaction continues overtime if undisturbed.[26,27]

Effect of the Cement and Multiple Interactions

Significantly higher SBS were evidenced for the three self-adhesive resin cements compared to FujiCEM for all testing conditions except in SP mode at 15 minutes. This is in agreement with previous studies, which have shown higher bond strengths for resin-based cements relative to RMGIC.[4,28] The similar SBS values for all cements when evaluated in SP mode at 15 minutes may have been the result of a slow initial cross-linking of the resin-based materials when they were allowed to self-polymerize. Multilink Automix and RelyX Unicem yielded the highest SBS irrespective of the substrate. A recent study demonstrated similar findings, with RelyX Unicem showing higher bond strengths compared to FujiCEM when bonded to base and noble metals, ceramic, and zirconia substrates.[5] Another study by Zhang and Degrange[29] showed higher bond strengths for Multilink Automix

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compared to other self-adhesive resin cements regardless of the restorative substrate. The same study also found that the bond strengths for many of the tested cements were dependent on the nature of the restorative substrate.[29] This is coincident with the results from our study which demonstrated that the interactions between cement, substrate, and setting reaction were also found to have a significant effect in the bond strength. The synergistic behavior whereby certain combinations of cement, substrate and setting reaction are more favorable than others indicates that the selection of the luting cement should be partially dictated by the substrate and the setting reaction. Resin bonded to Multilink Automix and RelyX Unicem in LP mode, and resin bonded to RelyX Unicem in SP mode showed higher SBS values than any of the other combinations in all testing conditions. Similar findings have been reported for RelyX Unicem.[4] Compatibility between the resinous components in the matrix of cements Multilink Automix and RelyX Unicem and those of composite Z100 may have been partially responsible for the observed results. Similarly, Maxcem Elite showed higher SBS values to base metal relative to all other substrates in all testing conditions. This could have been the product of the surface oxides known to make the base metal more reactive by providing potential for chemical bonding.[5] Presumably, a greater chemical affinity between the components of self-adhesive resin cements and those of specific prosthodontic substrates may have been responsible for the observed results. However, only general estimations can be made based on the information provided by the manufacturer because specific details regarding the material's chemical composition are proprietary. As recommended in the cements' directions for use, air abrasion with 50-μm aluminum oxide particles was used for surface roughening of all the substrate materials prior to bonding. Since the manufacturers do not specify additional surface treatment before application of the cement, no further surface treatments such as acid-etching or silanization were used as this might have led to different results. Only when bonding with Multilink Automix, was Monobond Plus primer used after air abrasion as per manufacturer's instructions.

As self-adhesive cements continue to gain acceptance in the market, large comparative studies are needed to evaluate their behavior when bonded to a variety of prosthodontic substrates and tested under different testing conditions. Bond strength studies represent valuable initial screening tests to assess the overall behavior and predict future clinical performance of the materials and techniques under investigation. However, care should be exercised when extrapolating the results obtained from laboratory studies to the expected clinical outcomes as *in vitro* tests are subject to a number of limitations. In the present study, a 24-hour immersion in a 37°C water bath was used prior to bond strength testing since this represents the standard short-term storage protocol recommended by the International Organization for Standardization (ISO/TR 11405).[30] Although the effects of thermal cycling and long-term storage on the bond strength were not evaluated as a part of this investigation, they are important in the simulation of clinical conditions and should be investigated in future laboratory studies incorporating multiple variables such as those included in the present study. Furthermore, a direct comparison among studies seems unfair since a number of aspects relative to the design and methodology are known to vary between studies. Since the aim of our study was to isolate specific interactions between the tested cements and different substrates, a simplified interfacial design was used, whereby the luting cement was directly bonded onto the substrate. A different methodology used in some studies involves two substrates (adherends) which are joined together by a luting cement (adhesive).[31] While this design resembles more closely the clinical situation, whereby a prepared tooth receives a laboratory-processed restoration, it represents a more complex interface since three different materials are joined together making it difficult to isolate the specific interactions taking place between the different components of the interface and perhaps compromising the validity of the results.

Further research is needed to validate the long-term behavior of the different substrate-cement combinations when tested in a variety of testing conditions. No conclusions can be drawn based solely on the results from bond strength studies. Combining the results from bond strength studies with those from microleakage and marginal adaptation studies may provide a more comprehensive assessment of the performance of the systems under investigation. Furthermore, inclusion of failure mode analysis routinely in bond strength studies may significantly contribute to a more accurate interpretation of the obtained results, as well as facilitate a better understanding of the mechanical behavior and stress distribution of adhesive interfaces during failure.

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CONCLUSIONS

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

1. The performance of the cements was greatly dependent on the type of setting reaction, with light-polymerized mode displaying significantly higher bond strengths than self-polymerized mode. The performance of the cements was also dependent on testing time. After 24 hours, all cements matured showing higher bond strengths than initial values obtained at 15 minutes.
2. Overall, self-adhesive resin cements demonstrated higher bond strengths than RMGIC FujiCEM Automix irrespective of the substrate for all testing conditions. The best performance was achieved for RelyX Unicem at 24 hours (SP and LP modes) and Multilink Automix in LP mode (15 minutes and 24 hours).
3. The bond strength of the cements also varied depending on the prosthodontic substrate, indicating that selection of the cement should be dictated partially by the substrate. Overall, Multilink Automix and RelyX Unicem demonstrated higher SBS when bonded to resin, and Maxcem Elite demonstrated higher SBS when bonded to base metal.

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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Visibility of Artificial Buccal Recurrent Caries Under Restorations Using Different Radiographic Techniques

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Clinical Relevance

Considering the difficulties in detecting buccal recurrent caries under restorations due to the compression of structures in intraoral radiography and occurrence of metal artifacts in cone beam computed tomography (CBCT), it is clinically useful to assess the performance of intraoral film and digital radiography and two different CBCT systems in terms of the visibility of artificial buccal secondary caries lesions under various restorative materials.

SUMMARY

The aim of the present study was to assess intraoral images and two cone beam computed tomography (CBCT) systems for detection of artificial buccal recurrent caries under restorations. Class V cavities were made for composite (30 teeth) and amalgam (30 teeth). Full restorations with thermoplastic polymer (30 teeth) and nickel-chromium metal crown (30 teeth) were constructed. In 60 teeth, artificial buccal recurrent caries were simulated; 60 other teeth served as controls. Intraoral film, intraoral digital, Veraviewepocs 3D, and Kodak 9000 images were scored twice. κ Coefficients were calculated and Az values were compared using *Z*-tests, with a significance level of α=0.05. Higher interobserver agreement was obtained from the CBCT images compared with the intraoral images. The Az values of both readings of all three observers were highest for the Veraviewepocs 3D followed by Kodak 9000 except for the second reading of the third observer. CBCT outperformed intraoral radiography in detection of artificial buccal recurrent caries under restorations.

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INTRODUCTION

Development of recurrent caries under different types of restorations is considered a major cause of restorative failure and replacement. It is important to diagnose early lesions in order to prevent severe destruction of hard tissue and to enhance the prognosis for a successful treatment outcome.[1–3] Radiographic detection is the most useful method to diagnose recurrent caries adjacent to restorations in conjunction with clinical examination. Intraoral film and digital radiography are commonly available methods in routine clinical dentistry. Conventional intraoral film consists of silver halide crystals in order to produce analog images. On the other hand, digital intraoral systems include a solid state silicon chip or a photostimulable phosphor plate (PSP). Solid state detectors use a scintillator layer to convert x-rays to light and include a charge-coupled device or a complementary metal oxide semiconductor.[4–7] PSPs absorb and store energy from x-rays. This energy is then released as phosphorescent when stimulated by another light of an appropriate wavelength.[8]

Regardless of the intraoral system used, the two-dimensional (2D) nature of the images limits the information that can be obtained, and their diagnostic value is dependent upon beam angulation, superimposition of anatomical structures, and patient-related factors.[9] Due to their 2D nature, intraoral techniques may fail to provide enough information in certain cases.[4–9] For example, buccal recurrent caries lesions under restorations are difficult to detect in radiographic examination. Radiopacity, which is greater in amalgam restorations than in enamel, can interfere with the detection of lesions in the lingual and buccal areas.[10]

Introduction of cone beam computed tomography (CBCT) enabled dentists to visualize teeth in axial, coronal, and sagittal views with a reduced radiation dose compared with medical CT. CBCT uses a cone-shaped x-ray beam centered on a 2D sensor to scan a 180°-360° rotation around the patient's head to acquire a full three-dimensional (3D) volume of data.[11] CBCT systems offer different sensor types, fields of view (FOV), and exposure settings. However, beam hardening and metal artifacts that occur in CBCT images are thought to be a limiting factor in detection of recurrent caries under restorations.[12,13] Although CBCT eliminates many disadvantages of intraoral radiography, it must be taken into consideration that patients receive higher radiation doses compared with intraoral and panoramic radiography.[14,15] Therefore, available CBCT images obtained for different purposes should be used only if conventional methods are not useful for diagnostic accuracy.

Considering the difficulties in detection of buccal recurrent caries under restorations due to the compression of structures in intraoral radiography and occurrence of metal artifacts in CBCT, the goal of this *ex vivo* study was to assess the performance of intraoral film and digital radiography and two different CBCT systems for the visibility of artificial buccal recurrent caries lesions under various restorative materials. Our null hypothesis was that there was no difference between CBCT and intraoral radiography systems for detection of artificial buccal recurrent caries lesions under various restorative materials.

MATERIALS AND METHODS

A total of 120 caries-free teeth (mandibular premolars and molars) extracted for periodontal and orthodontic reasons were used. Teeth of people who gave informed consent to donate their teeth for research and teaching were obtained from our hospital collection.

Preparation of Specimens

In 60 teeth, Black Class V cavities were made in the middle third of buccal surfaces for composite (Valux Plus, 3M ESPE, St Paul, MN, USA; 30 teeth) and amalgam restorations (Cavex, Haarlem, Netherlands; 30 teeth). In the remaining 60 teeth, chamfer margin preparations were made and full restoration with a thermoplastic polymer (Meliodent, Heraeus Kulzer, Hanau, Germany; 30 teeth) and full nickel-chromium (Ni-Cr) metal crown (30 teeth) was completed. Artificial buccal recurrent caries were simulated with the aid of a 1-mm-diameter carbide bur, sealed with 1-mm-diameter red wax under the restorations on the buccal shoulder of 30 teeth with chamfer margin preparations and on the buccal shoulder of 30 teeth with buccal Black Class V cavities. Another 60 teeth with restorations were left without simulated caries lesions. Distribution of an equal number of teeth with and without buccal caries for all types of restorations was ensured (15 teeth with caries and 15 teeth without caries for each type of restoration).

Image Acquisition

All teeth were randomly placed in the alveolar sockets of a dry human mandible in groups of eight (two premolars and two molars on left and right

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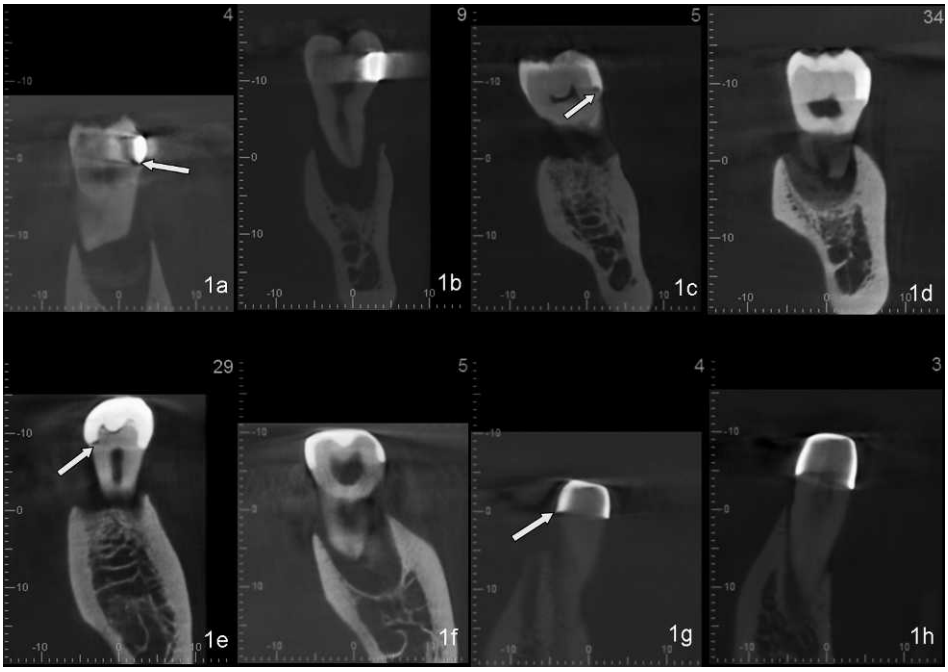

Figure 1. *Cross-sectional CBCT images obtained by Veraviewepocs 3D (Morita), 40 × 40 mm FOV (0.125-mm^3 voxel size). (a): Amalgam restoration with buccal recurrent caries. (b): Amalgam restoration without buccal recurrent caries. (c): Composite restoration with buccal recurrent caries. (d): Composite restoration without buccal recurrent caries. (e): Acrylic restoration with buccal recurrent caries. (f): Acrylic restoration without buccal recurrent caries. (g): Metal restoration with buccal recurrent caries. (h): Metal restoration without buccal recurrent caries.*

hemimandibles), and a 2-cm-thick plastic glove filled with distilled water was placed around the dry mandible in order to simulate soft tissue. Thereafter, teeth were imaged with intraoral conventional radiography, intraoral digital image receptors and two different CBCT units. Intraoral conventional radiographies and intraoral digital images were exposed ortho-radially with a Trophy Trex x-ray unit (Croissy, Beaubourg, France) operated at 65 kVp and 8 mA with a standardized paralleling technique and a focus-receptor distance of 20 cm. Repeated exposures after individual adjustment of the jaw/beam for each tooth were performed under reproducible conditions. Intraoral conventional radiographs were taken with Kodak Insight Film (size 2, E/F sensitivity, Eastman Kodak Co, Rochester, NY, USA) and an exposure time of 0.40 seconds. Films were automatically processed on the same day with fresh chemicals (Hacettepe, Ankara, Turkey) using an Extra-x Velopex (Medivance Instruments Ltd, London, England) in accordance with the manufacturer's instructions. Digital images were recorded using a Digora Optime (Soredex, Tuusula, Finland) PSP digital intraoral system, which includes a feature that automatically erases residual image signals. Image recording was set at a 40-μm pixel size, 14-bit grayscale, 12.5 line pairs per millimeter (lp/mm) spatial resolution and an image-exposure time of 0.20 second. A size 2 imaging plate was used, and the exposed phosphor plates were scanned immediately after exposure. Images of the teeth were obtained from two different CBCT units: 1) Veraviewepocs 3D model X550 (J Morita Mfg Corp, Kyoto, Japan) with a flat-panel detector offering digital 3D, panoramic, and cephalometric imaging options. With the Veraviewepocs 3D system, images were obtained at 60–90 kVp, 3 mA, and an exposure time of 9.4 seconds with a 40 × 40-mm FOV (0.125-mm^3 voxel size). 2) With the Kodak 9000 Extra-oral imaging system (Eastman Kodak), images were obtained at 60 kVp, 3 mA, and an exposure time of 13.2 seconds with an 50 × 37-mm FOV (76×76×76-μm voxel size isotropic voxel). Axial scans and multi-planar reconstructions were obtained, and volumetric data were reconstructed using the systems' software programs to provide serial cross-sectional views. A total of four image sets were obtained: 1) Veraviewepocs 3D (Morita), 40 × 40 mm FOV (0.125-mm^3 voxel size); 2) Kodak 9000, 50 × 37 mm FOV (76×76×76-μm voxel size); 3) intraoral digital images (Digora Optime PSP, Soredex); and 4) intraoral conventional film images (Kodak Insight Film). Figures 1 and 2 show examples of cross-sectional images obtained by Veraviewepocs 3D and Kodak 9000, respectively. Buccal recurrent caries–like lesions shown by arrows can be detected under each restoration. Figure 3 shows examples of intra-

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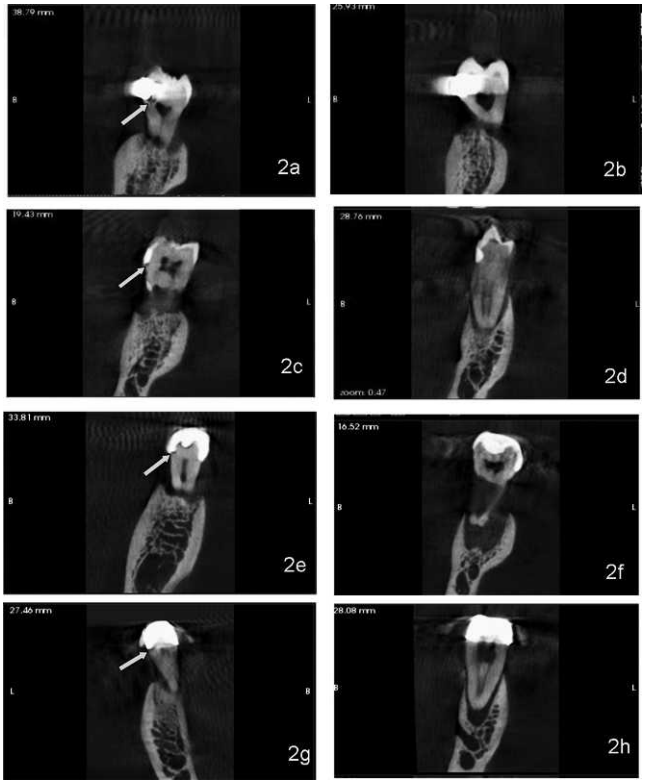

Figure 2. *Cross-sectional CBCT images obtained by Kodak 9000, 50 × 37 mm FOV (76×76×76-μm voxel size). (a): Amalgam restoration with buccal recurrent caries. (b): Amalgam restoration without buccal recurrent caries. (c): Composite restoration with buccal recurrent caries. (d): Composite restoration without buccal recurrent caries. (e): Acrylic restoration with buccal recurrent caries. (f): Acrylic restoration without buccal recurrent caries. (g): Metal restoration with buccal recurrent caries. (h): Metal restoration without buccal recurrent caries.*

oral images obtained by Digora Optime PSP (Soredex). Buccal recurrent caries–like lesions shown by arrows can barely be detected under each restoration. For all four methods, the exposure parameters used for image acquisition were based on pilot studies to ensure optimal image quality with good visibility of the pulpal root canal, enamel, and dentin.

Image Interpretation

A specific calibration session using 10 images was conducted prior to the study. Image sets were viewed separately by three calibrated and experienced observers (two dentomaxillofacial radiologists and a prosthodontist) in a dimly lit room. No time restriction was placed on the observers. Image sets were viewed at one-week intervals, and evaluations of each image set were repeated one week after the initial viewings. All radiographs were randomized within each imaging modality. All conventional intraoral images were evaluated using a light box and magnifier (2×). Digital intraoral and CBCT images were evaluated on a 22-inch LG Flatron monitor (LG, Seoul, Korea) set at a screen resolution of 1440 × 900 pixels and 32-bit color depth by using the systems' own software: DfW2.5 (Digora Optime, Soredex), i-Dixel (Veraviewepocs 3D, Morita), and Kodak Dental Imaging Software (Kodak 9000, Kodak). Built-in enhancement tools of the software were used if deemed necessary. Observers constructed cross-sectional images themselves. Cross-sectional images were not exported because by using the software, calibrated observers were able to identify buccal artificial lesions by scrolling through different cross-sectional images. One of the researchers who knew the study design and created artificial caries lesions guided viewing sessions by showing the observers which tooth in the arch would be scored. Also, the same researcher recorded the scores given by the observers. The buccal aspects of each restored tooth were randomly evaluated for the presence/absence of buccal caries and were scored using a 5-point scale as follows: 1 = *caries definitely present*; 2 = *caries probably present*; 3 = *uncertain/unable to tell*; 4 = *caries probably not present*; and 5 = *caries definitely not present*. A total of 120 buccal surfaces of 120 teeth were assessed.

Statistical Analysis

Weighted κ coefficients were calculated to assess the intraobserver and interobserver agreement for each image set. κ Values were calculated to assess intraoberserver and interobserver agreement according to the following criteria: <0.10 = *no agreement*; 0.10-0.40 = *poor agreement*; 0.41-0.60 = *moderate agreement*; 0.61-0.80 = *strong agreement*; and 0.81-1.00 = *excellent agreement*. κ Values were calculated using the MedCalc statistical software (MedCalc Software, Mariakerke, Belgium). Scores obtained from intraoral film and two different CBCT images were compared with the gold standard using the receiver operating characteristic (ROC) analysis to evaluate the observers' ability to differentiate between teeth with and without buccal caries. The areas under the ROC curves (Az values) were calculated using SPSS 15.0 (SPSS Inc, Chicago, IL, USA), and the Az values for each image type, observer, and reading and restoration type were compared using *Z*-tests, with a significance level of α=0.05. Bonferroni adjustment was used to evaluate the statistically significance. Sensitivity (Se), specificity (Sp), positive predictive value (PPV), negative predictive value (NPV), and false positive ratio (FPR) for each observer and their two readings were also calculated for each restoration type.

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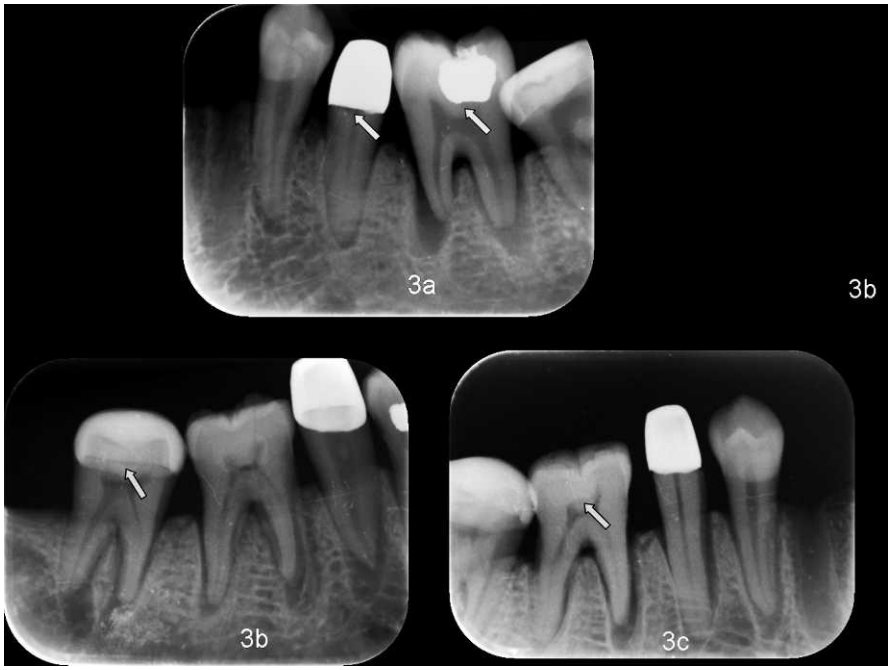

Figure 3. *Digital intraoral images obtained by Digora Optime PSP, (Soredex). (a): Digora Optime PSP image taken for the second premolar tooth. From left to right: first premolar without buccal recurrent caries under composite restoration, second premolar with buccal recurrent caries under metal restoration, and first molar with buccal recurrent caries under amalgam restoration. (b): Digora Optime PSP image taken for the first molar tooth. From left to right: second molar with buccal recurrent caries under acrylic restoration, first molar without buccal recurrent caries under composite restoration, and second premolar without buccal recurrent caries under metallic restoration. (c): Digora Optime PSP image taken for the first molar tooth. From left to right: first molar with buccal recurrent caries under composite restoration, second premolar without buccal recurrent caries under metallic restoration, and first premolar without buccal recurrent caries under composite restoration.*

RESULTS

Table 1 shows the intraobserver κ coefficients calculated for each observer by image type. Intraobserver κ coefficients ranged from 0.536-0.609 for the intraoral film (Kodak Insight), from 0.517-0.691 for the digital intraoral (Digora Optime), from 0.613-0.649 for the Veraviewepocs 3D, and from 0.582-0.628 for Kodak 9000 images, suggesting moderate and strong intraobserver agreement for observers 1 and 2 and strong intraobserver agreement for observer 3. Tables 2 and 3 show the interobserver κ coefficients for both the first and second readings by image type, respectively. Higher interobserver agreement was obtained from the CBCT images when compared with the intraoral images. Poor and moderate interobserver agreement was found for the first and second readings for the intraoral Kodak Insight film images (from 0.339-0.470) and for the digital intraoral Digora Optime images (from 0.337-0.522). In general, moderate interobserver agree-

Table 1: *Intraobserver Agreement Calculated for Each Observer by Image Type*

	Observer 1 Weighted κ–Se	**Observer 2 Weighted κ–Se**	**Observer 3 Weighted κ–Se**
Kodak Insight Film	0.583-0.058	0.536-0.074	0.609-0.062
Digora Optime	0.517-0.066	0.538-0.082	0.691-0.079
Veraviewepocs 3D (Morita)	0.647-0.079	0.613-0.092	0.649-0.086
Kodak 9000	0.590-0.078	0.582-0.082	0.628-0.085

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Table 2: *Interobserver κ Coefficients Among Observers for the First Readings*

	Obs 1-Obs 2 Weighted κ–Se	Obs 1-Obs 3 Weighted κ–Se	Obs 2-Obs 3 Weighted κ–Se
Kodak Insight Film	0.339-0.069	0.355-0.052	0.363-0.054
Digora Optime	0.395-0.051	0.356-0.066	0.337-0.070
Veraviewepocs 3D (Morita)	0.603-0.069	0.536-0.068	0.545-0.069
Kodak 9000	0.458-0.069	0.617-0.069	0.466-0.076

Abbreviation: Obs, observer.

ment was found for the first and second readings for the Veraviewepocs 3D (from 0.465-0.603) and Kodak 9000 (from 0.458-0.617).

The areas under the ROC curves (Az values) for the different observers, readings, and image types were calculated and are given in Table 4. The Az values of both readings of all three observers were highest for the Veraviewepocs 3D followed by the Kodak 9000, except for the second reading of the third observer. Az values of the CBCT images were higher than those of the intraoral images. Se, Sp, PPV, NPV, and FPR for each observer and their two readings are presented in Table 5. Also, higher sensitivity values for CBCT systems compared with intraoral images were obtained.

Comparisons between modalities are given in Table 6. No differences ($p>0.05$) were found between the Az values of the Kodak Insight film and those of the digital intraoral Digora Optime images for all observers. Also, there was no statistically significant difference ($p>0.05$) between the two CBCT systems (Kodak 9000 and Veraviewepocs 3D). Statistically significant differences between Az values for the intraoral Kodak Insight film images and Veraviewepocs 3D images were found for both readings of observer 1 (first reading: $p=0.007$, second reading: $p=0.011$) and observer 2 (first reading: $p=0.003$, second reading: $p=0.023$). Statistically significant differences were also found between the Az values for the digital intraoral Digora Optime and Veraviewepocs 3D images for both readings of observer 1 (first reading: $p=0.001$, second reading: $p=0.027$), observer 2 (first reading: $p<0.001$, second reading: $p=0.001$), and observer 3 (first reading: $p=0.002$). There was only a significant difference for the second reading of observer 2 between Kodak 9000 and Kodak Insight film ($p=0.027$) and between Kodak 9000 and the digital intraoral Digora Optime system ($p<0.001$).

When visibility of buccal recurrent caries under four different restorative materials for each imaging modality was taken into consideration for all observers, no statistically significant difference ($p>0.05$) was found among different restorative materials for each imaging modality except for the comparison of composite and amalgam restorations

Table 3: *Interobserver κ Coefficients Among Observers for the Second Readings*

	Obs 1-Obs 2 Weighted κ–Se	Obs 1-Obs 3 Weighted κ–Se	Obs 2-Obs 3 Weighted κ–Se
Kodak Insight Film	0.470-0.051	0.428-0.064	0.438-0.053
Digora Optime	0.446-0.066	0.440-0.083	0.522-0.082
Veraviewepocs 3D (Morita)	0.465-0.078	0.494-0.082	0.486-0.094
Kodak 9000	0.509-0.078	0.521-0.088	0.525-0.081

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Table 4: *Az Values, Their Standard Errors (SE), 95% Confidence Intervals (CI), and Significance Levels (p) for Each Observer*

	Observer 1		Observer 2		Observer 3	
	1st Reading	2nd Reading	1st Reading	2nd Reading	1st Reading	2nd Reading
Kodak Insight Film						
Az (SE)	0.597 (0.060)	0.592 (0.060)	0.575 (0.058)	0.524 (0.063)	0.647 (0.061)	0.558 (0.063)
95% CI	0.504- 0.685	0.499-0.680	0.484-0.757	0.431-0.616	0.555-0.732	0.465-0.648
p	0.063	0.663	0.003	0.041	0.113	0.427
Digora Optime						
Az (SE)	0.538 (0.064)	0.590 (0.062)	0.509 (0.064)	0.587 (0.062)	0.558 (0.063)	0.542 (0.060)
95% CI	0.426-0.647	0.478-0.697	0.397-0.619	0.474-0.693	0.445-0.666	0.530-0.744
p	0.266	0.246	0.849	0.383	0.485	0.034
Veraviewepocs 3D (Morita)						
Az (SE)	0.815 (0.048)	0.744 (0.056)	0.896 (0.036)	0.815 (0.048)	0.777 (0.052)	0.583 (0.065)
95% CI	0.711-0.895	0.631-0.836	0.805-0.954	0.710-0.894	0.667-0.864	0.465-0.694
p	<0.001	<0.001	<0.001	<0.001	<0.001	0.215
Kodak 9000						
Az (SE)	0.720 (0.058)	0.677 (0.061)	0.760 (0.062)	0.810 (0.049)	0.681 (0.060)	0.681 (0.060)
95% CI	0.606-0.816	0.561-0.780	0.643-0.764	0.704-0.890	0.564-0.782	0.565-0.783
p	0.001	0.008	0.017	<0.001	0.007	0.007

obtained from Veraviewepocs 3D images (observer 1; $p=0.011$ and observer 2; $p=0.003$).

DISCUSSION

To our knowledge, up until now, no previous study has compared CBCT and intraoral radiography in detecting buccal recurrent caries under different types of restorations. In the present study, composite restorations and thermoplastic polymer were used as nonradiopaque restorations, whereas amalgam and full crown were used as radiopaque restorations. Comparison between radiopaque and nonradiopaque restorations was considered useful in terms of assessing beam hardening and metal artifacts. Metal artifacts, which are seen as dark and light streaks on tomographic images, can seriously degrade the visual quality and interpretability of CBCT images. It is accepted that image degradation increases with the number of metal restorations in the jaws, whereas small voxel size, limited beam, and true alignment of x-ray beam decreases image degradation.[16] Although metal artifacts seen in CBCT images are claimed to be limiting factors in the diagnosis of caries under restorations, we found better Az values for CBCT images compared with intraoral images. This can be explained by the fact that with CBCT, it is possible to view teeth and related structures in axial, coronal, and cross-sectional views. Besides, in the present

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Table 5: *Sensitivity (Se), Specificity (Sp), Positive Predictive Value (PPV), Negative Predictive Value (NPV), and False Positive Ratio (FPR) for Each Observer and Their Two Readings*

	1st Reading Se	2nd Reading Se	1st Reading Sp	2nd Reading Sp	1st Reading PPV	2nd Reading PPV	1st Reading NPV	2nd Reading NPV	1st Reading FPR	2nd Reading FPR
Observer 1										
Kodak Insight Film	0.588	0.265	0.605	0.814	0.602	0.587	0.598	0.526	0.395	0.186
Digora Optime	0.676	0.765	0.209	0.372	0.458	0.546	0.388	0.606	0.791	0.628
Veraviewepocs 3D (Morita)	0.853	0.853	0.698	0.581	0.739	0.669	0.823	0.794	0.302	0.419
Kodak 9000	0.853	0.647	0.535	0.721	0.648	0.698	0.782	0.672	0.465	0.279
Observer 2										
Kodak Insight Film	0.588	0.421	0.767	0.814	0.719	0.688	0.652	0.582	0.233	0.186
Digora Optime	0.382	0.421	0.605	0.698	0.493	0.583	0.495	0.546	0.395	0.302
Veraviewepocs 3D (Morita)	0.912	0.882	0.698	0.647	0.752	0.715	0.886	0.844	0.302	0.353
Kodak 9000	0.735	0.912	0.581	0.674	0.637	0.733	0.690	0.881	0.419	0.326
Observer 3										
Kodak Insight Film	0.235	0.206	0.930	0.953	0.774	0.807	0.550	0.547	0.07	0.047
Digora Optime	0.441	0.529	0.698	0.837	0.594	0.768	0.555	0.641	0.302	0.163
Veraviewepocs 3D (Morita)	0.765	0.676	0.721	0.535	0.733	0.591	0.757	0.623	0.279	0.465
Kodak 9000	0.735	0.647	0.558	0.698	0.627	0.684	0.682	0.666	0.442	0.302

study, CBCT units with limited FOVs and small voxel sizes were chosen that could reduce metal artifacts. With the Veraviewepocs 3D, slightly higher values were found compared with the Kodak 9000. This difference, although without significance, may be due to sensor, software, or hardware specifications. In our notion, digital intraoral and CBCT systems must be evaluated by using their dedicated software as in the present study because software is probably the most important component of the digital systems. Software capability can not be detached from digital radiographic imaging systems. In addition, when the visibility of buccal recurrent caries under four different restorative materials for each imaging modality was taken into consideration for all observers, no statistically significant difference ($p>0.05$) was found.A study[17] found no difference between intraoral film (Ekstraspeed Plus, Eastman Kodak) and direct digital radiography (Sidexis, Siemens, Bensheim, Germany) in the detection of small artificial lesions induced by a demineralization buffer gel system at the crown margin, similar to our findings. Authors recommended the use of hydroxyethyl cellulose for creating artificial caries lesions. Also, artificial cementum and dentin lesions were slightly easier to diagnose than enamel lesions. Authors concluded that radiography was not considered a reproducible and safe method for characterization of the demineralization process localized at the crown margin—especially in enamel—due to the

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Table 6: *Modalities Compared by Using Z-Test, With a Significance Level of $\alpha = 0.05$ (Statistically Significant* p *Values Are Written in Bold*)

	Obs 1		Obs 2		Obs 3	
	1st Reading, *p*-Value	2nd Reading, *p*-Value	1st Reading, *p*-Value	2nd Reading, *p*-Value	1st Reading, *p*-Value	2nd Reading, *p*-Value
Kodak Insight Film-Digora Optime	0.273	0.869	0.225	0.072	0.210	0.479
Kodak Insight Film- Veraviewepocs 3D (Morita)	***0.007***	**0.011**	**0.003**	**0.023**	0.065	0.978
Kodak Insight Film- Kodak 9000	0.254	0.077	0.594	**0.027**	0.370	0.277
Digora Optime- Veraviewepocs 3D (Morita)	**0.001**	**0.027**	<**0.001**	**0.001**	**0.002**	0.426
Digora Optime- Kodak 9000	0.070	0.215	0.085	<**0.001**	0.056	0.818
Kodak 9000- Veraviewepocs 3D (Morita)	0.162	0.405	0.301	0.929	0.287	0.304

Abbreviation: Obs, observer.

low interexaminer agreement among three calibrated observers. Therefore, the importance of a thorough visual and tactile examination was emphasized.[17] In the present study, a standard 1-mm-diameter red wax was used under restorations in order to simulate small buccal recurrent caries that are difficult to diagnose in routine clinical and radiographic examination. A similar method in an attempt to create artificial caries lesions was used in a previous study.[18] Standardization of artificial buccal caries lesions was thereby provided to some extent, because our aim was only to compare different radiographic techniques in detecting artificial lesions, instead of histological validation of caries. In the present study, higher Az values for CBCT images compared with intraoral images may be attributed to the sharp round margins of the defects; however, sharp-margined defects were imaged by all systems assessed.

Another study[19] found that a thorough clinical examination was more reliable than intraoral radiography in detecting recurrent interproximal caries at crown margins of full restorations. This may be due to the masking effect of full restorations and limited information gathered from intraoral radiography. On the other hand, for nonrestored teeth, radiographs often rendered evidence of caries lesions that were not diagnosed during the clinical examination.[19] However, findings of the mentioned study are debatable because only one observer clinically and radiographically evaluated teeth. Therefore, it is not possible to assess the reproducibility of the results. The present study only compared different radiographic systems in detecting artificial buccal recurrent caries under different restorations. Visual and clinical examination findings were not in the scope of the present research.

It has been postulated that the marginal gap between the restoration and dentin is the main reason for recurrent caries' development, despite the fact that even with highly sophisticated technology, there is always a marginal gap.[1] However, existence of a clinical marginal defect alone is not a reason to replace a restoration, given that not all defective margins cause recurrent caries under restorations. Of the 822 teeth with defective restorations in the total sample, 86% (709 of 822) were free of radiographic recurrent caries.[1] On the other hand, an increased likelihood of defective over intact restorations to display radiographic recurrent caries was found. Approximately 14% of the defective restorations were associated with radiographic recurrent caries, compared with 5% for the intact restorations.[20] Similarly, colors next to restorations are not always predictive of secondary caries. Stained composite margins and ditched amalgam margins are not necessarily signs of decay, although they indicate a greater risk.[21,22] In light of these findings, radiographic diagnosis of caries under restorations is an important aid to clinical examination.

It must be taken into consideration that patients receive higher radiation doses with CBCT compared

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with intraoral and panoramic radiography. Radiation doses from CBCT scans vary substantially among devices, FOVs, and other technical factors.[14,15] In view of concerns regarding radiation exposure, a smaller FOV results in a less effective dose and should be used for dental images, whereas a larger FOV should be restricted to cases in which a wider view is required.[23] Although radiation exposure was not an issue for this *ex vivo* research, we used CBCT with a limited FOV and small voxel sizes in order to assess teeth that could possibly increase the observer's ability to detect artificial buccal recurrent caries lesions under restorations. Values produced in the present study may not apply to CBCT images taken for other indications and with different settings.

CONCLUSION

Higher Az and sensitivity values were obtained with Veraviewepocs 3D and Kodak 9000 images compared with both intraoral images, which performed similarly in the diagnosis of artificial buccal recurrent caries under restorations. Available CBCT units and images can be useful in the diagnosis of buccal recurrent caries under restorations.

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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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Effects of Surface Treatments, Thermocycling, and Cyclic Loading on the Bond Strength of a Resin Cement Bonded to a Lithium Disilicate Glass Ceramic

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AR Costa • GA Borges • MAC Sinhoreti
L Correr-Sobrinho

Clinical Relevance

The current results indicate that acid etching is a better option than air abrasion when bonding IPS e.max Press ceramic to dual-cure RelyX ARC resin cement. However, both fatigue and thermocycling decrease the bond strength.

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SUMMARY

Objectives: **The aim of this present study was to investigate the effect of two surface treatments, fatigue and thermocycling, on the microtensile bond strength of a newly introduced lithium disilicate glass ceramic (IPS e.max Press, Ivoclar Vivadent) and a dual-cured resin cement.**

Methods: **A total of 18 ceramic blocks (10 mm long × 7 mm wide × 3.0 mm thick) were fabricated and divided into six groups (n=3): groups 1, 2, and 3—air particle abraded for five seconds with 50-μm aluminum oxide particles; groups 4, 5, and 6—acid etched with 10%**

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hydrofluoric acid for 20 seconds. A silane coupling agent was applied onto all specimens and allowed to dry for five seconds, and the ceramic blocks were bonded to a block of composite Tetric N-Ceram (Ivoclar Vivadent) with RelyX ARC (3M ESPE) resin cement and placed under a 500-g static load for two minutes. The cement excess was removed with a disposable microbrush, and four periods of light activation for 40 seconds each were performed at right angles using an LED curing unit (UltraLume LED 5, Ultradent) with a final 40 second light exposure from the top surface. All of the specimens were stored in distilled water at 37°C for 24 hours. Groups 2 and 5 were submitted to 3,000 thermal cycles between 5°C and 55°C, and groups 3 and 6 were submitted to a fatigue test of 100,000 cycles at 2 Hz. Specimens were sectioned perpendicular to the bonding area to obtain beams with a cross-sectional area of 1 mm^2 (30 beams per group) and submitted to a microtensile bond strength test in a testing machine (EZ Test) at a crosshead speed of 0.5 mm/min. Data were submitted to analysis of variance and Tukey post hoc test ($p \leq 0.05$).

Results: **The microtensile bond strength values (MPa) were 26.9 ± 6.9, 22.2 ± 7.8, and 21.2 ± 9.1 for groups 1–3 and 35.0 ± 9.6, 24.3 ± 8.9, and 23.9 ± 6.3 for groups 4–6. For the control group, fatigue testing and thermocycling produced a predominance of adhesive failures. Fatigue and thermocycling significantly decreased the microtensile bond strength for both ceramic surface treatments when compared with the control groups. Etching with 10% hydrofluoric acid significantly increased the microtensile bond strength for the control group.**

INTRODUCTION

Ceramics are used for dental restorations and have excellent properties, such as chemical stability, biocompatibility, low thermal conductivity, high compressive strength, thermal diffusivity, translucence, fluorescence, and a coefficient of thermal expansion similar to that of tooth structure.[1–3] However, ceramic is brittle, a property that can be attributed to the presence of microcracks on the surface, making the material susceptible to fracture during luting and under occlusal forces.[2,4] To compensate for brittleness, ceramic has been associated with a metal substructure, and the system has served dentistry for approximately 50 years. Despite the high fracture resistance of traditional metal-ceramic crowns,[5] limitations are imposed on the systems because the metal core can reduce the translucency and affect the esthetics of restorations. Several technological advances have recently led to the development of new materials that possess high strength, such as glass-infiltrated, heat-pressed, and copy-milled ceramics.

The clinical success of ceramic restorations depends on a number of factors, such as the cementation procedure and composition of the ceramic material. Different ceramic surface treatments have been introduced to improve resin bonding to ceramic. A newly introduced lithium disilicate glass ceramic (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) may be adhesively cemented, but when the retentive area is small, retention may be inadequate. Bonding of the resin cement to the tooth is aided by acid etching of the enamel or dentin and by the use of a dentin adhesive.[6] Techniques for bonding to ceramic IPS e.max Press take advantage of the formation of chemical bonds and micromechanical interlocking at the resin-ceramic surface. Etching with hydrofluoric acid is used to create a rough surface on the bonding area of the ceramic material to enhance bonding between the ceramic and resin cement. Hydrofluoric acid removes the glass matrix and the second crystalline phase, thus creating irregularities within the lithium disilicate crystals of the IPS e.max Press for bonding.[3,7–8] Another treatment recommended for ceramic surfaces involves airborne particle abrasion with 50-μm aluminum oxide (Al_2O_3) particles to aid in mechanical retention.[7–11] After air abrasion, the ceramic surface must be coated with a suitable silane, which forms chemical bonds between the inorganic phase of the ceramic and the organic phase of the resin cement.[8,12–14]

Clinically, when ceramic restorations are cemented and exposed to the oral environment, factors that could result in fatigue may influence their physical and mechanical properties. Fatigue fracture is a form of failure that occurs in structures with microscopic cracks subjected to dynamic and fluctuating stresses.[15] Continued loading during mastication results in stress concentration, whereas thermal variations induce fatigue, and these cracks propagate and weaken the restoration. Catastrophic fracture results from a final loading cycle that exceeds the load-bearing capacity of the remaining sound portion of the material.[16–17] Thermal variations and the evaluation of fatigue resistance of dental ceramics could provide a more detailed understanding of clinical failures.[18]

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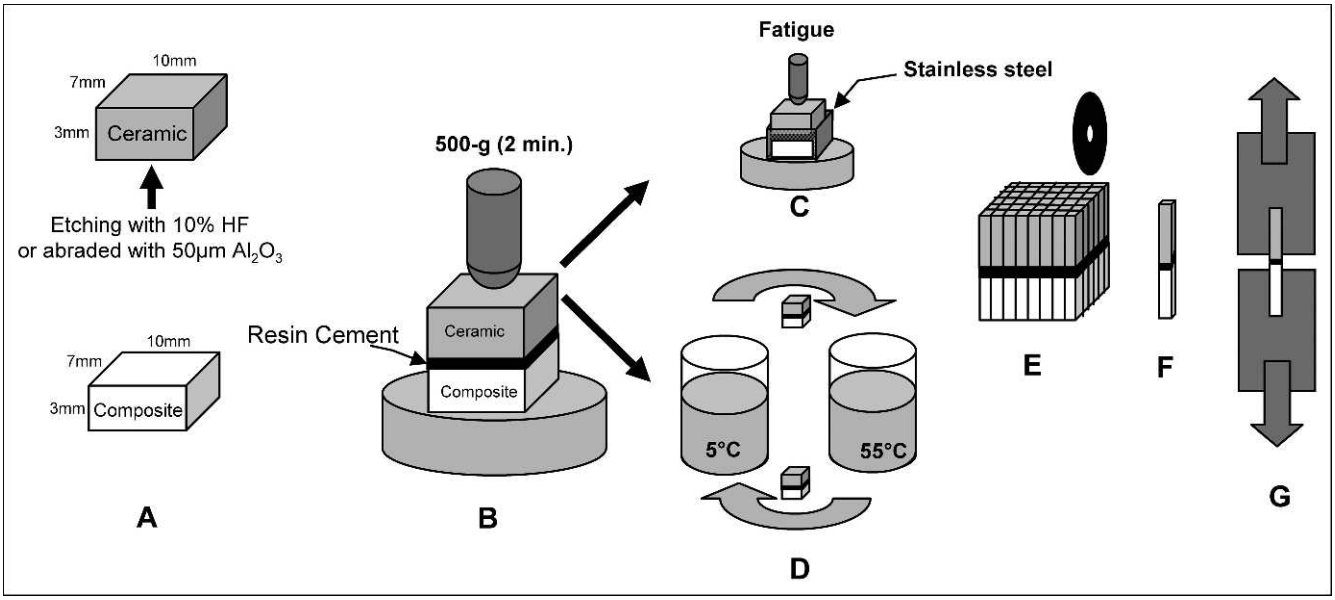

Figure 1. *Experimental setup of the study. (A): The surfaces of ceramic blocks were abraded with 50-µm Al_2O_3 particles (AOP) or etched with 10% hydrofluoric acid (HF) and one layer of silane was applied. (B): The ceramic blocks were bonded to a block of the composite under a 500-g static load for two minutes and light activated. (C): The specimens were put into a stainless steel box with a layer of elastomer and then submitted to a fatigue test of 100,000 cycles with an 8-mm-diameter stainless ball. (D): The specimens were submitted to 3,000 thermal cycles between 5°C and 55°C. (E): Beams obtained after perpendicular sectioning. (F): Beam with bonding area of 1 mm^2. (G): Beam was positioned on the testing machine and submitted to a microtensile bond test.*

Therefore, the aim of this present study was to investigate the effect of two surface treatments, fatigue and thermocycling, on the microtensile bond strength of the ceramic IPS e.max Press luted with a dual-cured resin cement. The hypotheses tested were 1) the surface treatments do not affect the microtensile bond strength of the ceramic; and 2) the fatigue and thermocycling do not affect the microtensile bond strength of the ceramic.

MATERIALS AND METHODS

Ceramic Blocks

A total of 18 rectangular blocks (10 mm long × 7 mm wide × 3.0 mm thick) of IPS e.max Press ceramic (Ivoclar Vivadent), shade LT D3, were fabricated (Figure 1A) in accordance with the manufacturer's instructions. Rectangular wax patterns were made, sprued, and attached to a muffle base with a surrounding paper cylinder. The wax patterns were invested with phosphate-based material (IPS PressVest Speed, Ivoclar Vivadent), and the wax was eliminated in an automatic furnace (Vulcan A-550, Degussa-Ney, Yucaipa, CA, USA) at 850°C for one hour. The IPS e.max Press ceramic ingots were pressed into the molds in an automatic press furnace (EP 600, Ivoclar Vivadent). After cooling, the specimens were divested and submitted to wet polishing with 600- and 1200-grit silicon carbide abrasive papers (Norton SA, São Paulo, Brazil) to obtain a flat surface.

Composite Blocks

A total of 18 rectangular blocks (10 mm long × 7 mm wide × 3.0 mm thick) of composite Tetric N-Ceram (Ivoclar Vivadent, shade A3) were fabricated (Figure 1A). The composite (Tetric N-Ceram) was bulk inserted into an elastomeric mold (Express STD, 3M ESPE, St Paul, MN, USA), and a Mylar strip was placed on the composite surface and manually pressed using a microscope slide to remove excess composite. The composite was light activated for 80 seconds from the top surface using an LED source (UltraLume 5, Ultradent, South Jordan, UT, USA) with an irradiance of 1100 mW/cm^2.

Surface Treatments of the Ceramic Blocks

The 18 ceramic blocks were randomly divided into six groups (n=3). In groups 1, 2, and 3, the test surfaces of the ceramic blocks were air particle abraded with 50-µm Al_2O_3 particles (AOP) (Bioart, São Carlos, Brazil) for five seconds under two bars of pressure using a sandblasting device (Microetch, Bioart) held at a distance of 10 mm and perpendicular to the ceramic surface (Figure 1A). Specimens were then rinsed, ultrasonically cleaned in distilled water for 20 minutes, and dried with compressed air. The test surfaces of groups 4, 5, and 6 were etched

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with 10% hydrofluoric acid (HF) (Dentsply, Petrópolis, Brazil) for 20 seconds, followed by rinsing with distilled water for one minute (Figure 1A). The specimens were then rinsed, ultrasonically cleaned in distilled water for 20 minutes, and dried with compressed air. One layer of a silane coupling agent (RelyX Ceramic Primer, 3M ESPE) was applied onto all ceramic specimens and allowed to air dry for five seconds.

Cementing the Composite to the Ceramic

One coat of Single Bond Adhesive (3M ESPE) was applied to all composite blocks, air dried for five seconds, and light activated for 10 seconds (UltraLume 5, Ultradent). A dual-cured resin luting agent (RelyX ARC, 3M ESPE), shade A3, was manipulated according to the manufacturer's instructions and applied to the ceramic surface. The ceramic blocks were bonded to a block of the composite Tetric N-Ceram and placed under a 500-g static load for two minutes (Figure 1B); the excess cement was removed with a disposable microbrush. Light-activation was performed for 40 seconds at right angles to each of the IPS e.max Press/Tetric N-Ceram margins (four activations) using an LED source (UltraLume LED 5, Ultradent), with a final 40-second light exposure from the top surface.

Conditioning the Specimens

All specimens were stored in distilled water at 37°C for 24 hours. The specimens of groups 2 and 5 were submitted to 3000 thermal cycles (MSCT 3, Marnucci ME, São Carlos, Brasil) between 5°C and 55°C (dwell time of 30 seconds) (Figure 1D). The specimens of groups 3 and 6 were put into a stainless steel box with a layer of polyether impression material (Impregun F, 3M ESPE, Seefeld, Germany) with 1 mm thickness. This layer was placed on the bottom as well as on the sides to stabilize the specimen. Then, the specimen was submitted to a fatigue test of 100,000 cycles (ER37000, ERIOS, São Paulo, Brazil) that consisted of cyclic loading with an 8-mm-diameter stainless steel ball applied on the central area of the ceramic side of the specimen with load of 80 N in a wet environment prior to microtensile bond testing. The cyclic loading had a force profile in the form of a sine wave at 2 Hz (Figure 1C).

Microtensile Bond Strength Testing

After the experimental procedures, the specimens were sectioned perpendicular to the bonding interface area (Figure 1E) to obtain beams with a bonding area of 1 mm^2 (Figure 1F) using a water-cooled diamond blade (EXTEC Corporation, Enfield, CT, USA) in a low-speed saw machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). The cross-sectional area of the bond interface of each beam was measured using a digital caliper (Mitutoyo Corporation, Tokyo, Japan). Each beam was fixed to the grips of a microtensile device using a cyanoacrylate adhesive (Zapit, Dental Ventures of America Inc, Corona, CA, USA), and the microtensile bond test was conducted in a testing machine (EZ Test, EZS, Shimadzu, Tokyo, Japan) at a crosshead speed of 0.5 mm/min until failure (Figure 1G).

Statistical Analysis

Bond strength values were calculated and the data supplied in megapascals. The experimental unit was the ceramic/composite block. Each group contained three blocks, and each block generated an average of 10 beams, for a total of 30 beams per group. Thus, the mean of the bond-strength values in each group represented the sum of the three experimental units. Microtensile bond-strength data were submitted to two-way analysis of variance, and multiple comparisons were performed using the Tukey *post hoc* test ($p<0.05$).

Failure Analysis

The fractured specimens were observed under optical microscopy (Olympus Corp, Tokyo, Japan) at 40× magnification. The mode of failure was classified as follows: adhesive (mode 1); cohesive within ceramic (mode 2); cohesive within composite (mode 3); and mixed, involving cement, ceramic, and composite (mode 4). The specimen surfaces were gold coated with a sputter coater (Balzers-SCD 050, Balzers Union, Aktiengeselischaft, Fürstentun, Liechtenstein) for 180 seconds at 40 mA. The specimens were then mounted on coded brass stubs and examined using scanning electron microscopy (SEM; LEO 435 VP, Cambridge, England), operated at 20 Kv, by the same operator.

RESULTS

Bond Strength Testing and Failure Analysis

The mean values of the microtensile bond strength testing are shown in Table 1. For the control group, the bond strength of the specimens etched with 10% HF was significantly higher than the specimens that received AOP ($p\leq0.05$). For the fatigue-tested group, no statistical difference was found between the specimens etched with HF and AOP ($p\geq0.05$).

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Table 1: *Microtensile Bond Strength Means ± Standard Deviations (MPa) for All Groups*[a]

Surface Treatments	Bond Strength (MPa)		
	Control Group	Fatigue Tested	Thermocycled
10% hydrofluoric acid	35.0 (9.6) A,a	23.9 (6.3) B,a	24.3 (8.9) B,a
50 μm Al_2O_3	26.9 (6.9) A,b	21.2 (9.1) B,a	22.2 (7.8) B,a

[a] *Means followed by different capital letters in the same row and/or small letters in the column are significantly different at* $p < 0.05$ *(Tukey test).*

Similar results were observed for the thermocycled groups.

The microtensile bond strength of the control group was significantly higher than that for the fatigue-tested and thermocycled groups ($p \leq 0.05$). No statistical difference was found between the fatigue-tested and thermocycled groups ($p \geq 0.05$).

The distribution of failure modes is shown in Table 2. The control, fatigue-tested, and thermocycled groups showed a predominance of failure mode 1 for the HF surface treatment and modes 1 and 4 for the AOP surface treatment.

The SEM micrographs showed that the control group (Figure 2) had fewer irregularities. On the other hand, the acid-etched group (Figure 3) presented more retentive irregularities when compared with the air-abraded group (Figure 4).

DISCUSSION

The clinical success of a ceramic restoration depends on the quality and durability of the bond between the ceramic and the resin cement. The quality of this bond is determined by the bonding mechanisms, which are controlled in part by the specific surface treatment used to promote micromechanical or chemical retention to the ceramic substrate.[19] The micromechanical retention of the ceramic surface plays an important role in bonding with a resin luting cement. Morphology modification on the ceramic surface may be performed to promote a better bond strength.[7] After surface treatment, the resin cement is applied on the ceramic surface, and the penetration of the cement and its polymerization is responsible for bonding.[20–21]

Table 2: *Failure Modes Analysis of the Debonded Specimens (%) Among Groups*[a]

Groups	Mode 1		Mode 2		Mode 3		Mode 4	
	HF	AOP	HF	AOP	HF	AOP	HF	AOP
Control	47	53	5	0	24	0	24	47
Fatigue tested	52	73	0	18	11	0	37	9
Thermocycled	80	91	0	9	5	0	15	19

Abbreviations: HF, hydrofluoric acid; AOP, aluminum oxide particles.
[a] *Mode 1: adhesive; Mode 2: cohesive within ceramic; Mode 3: cohesive within composite and Mode 4: mixed, involving cement, ceramic, and composite.*

In the present study, two ceramic surface treatments were evaluated. The results indicated that the first hypothesis was partly accepted. The etching procedure with HF resulted in the highest tensile bond strength with a statistically significant difference when compared with the AOP group or the control group. No statistical difference was found after fatigue and thermocycling.

The difference in tensile bond strength can be explained on the basis of morphology created on the AOP and HF specimens (Figures 2–4). Etching the ceramic surface with 10% HF promoted dissolution in the glassy matrix of the specimens to the depth of a few microns, enabling the lithium disilicate crystals to protrude from the glass matrix. Elongated crystals and shallow irregularities were clearly observed (Figure 3). The change in the surface morphology treated with 10% HF increased the surface area and facilitated the penetration and retention of resin cement into the microretentions of the treated surface.[7,8,22] It has been shown that this treatment has an efficient result for other kinds of dental ceramics.[3,7,8,12,19–20,23] The lowest mean tensile bond strength was obtained for the specimens that were air abraded with 50-μm Al_2O_3. The present results indicate that this treatment does not provide a mechanically retentive surface that is as efficient as etching with HF. This treatment promoted morphologic alterations of the ceramic surface, resulting in an increase in the number of potential

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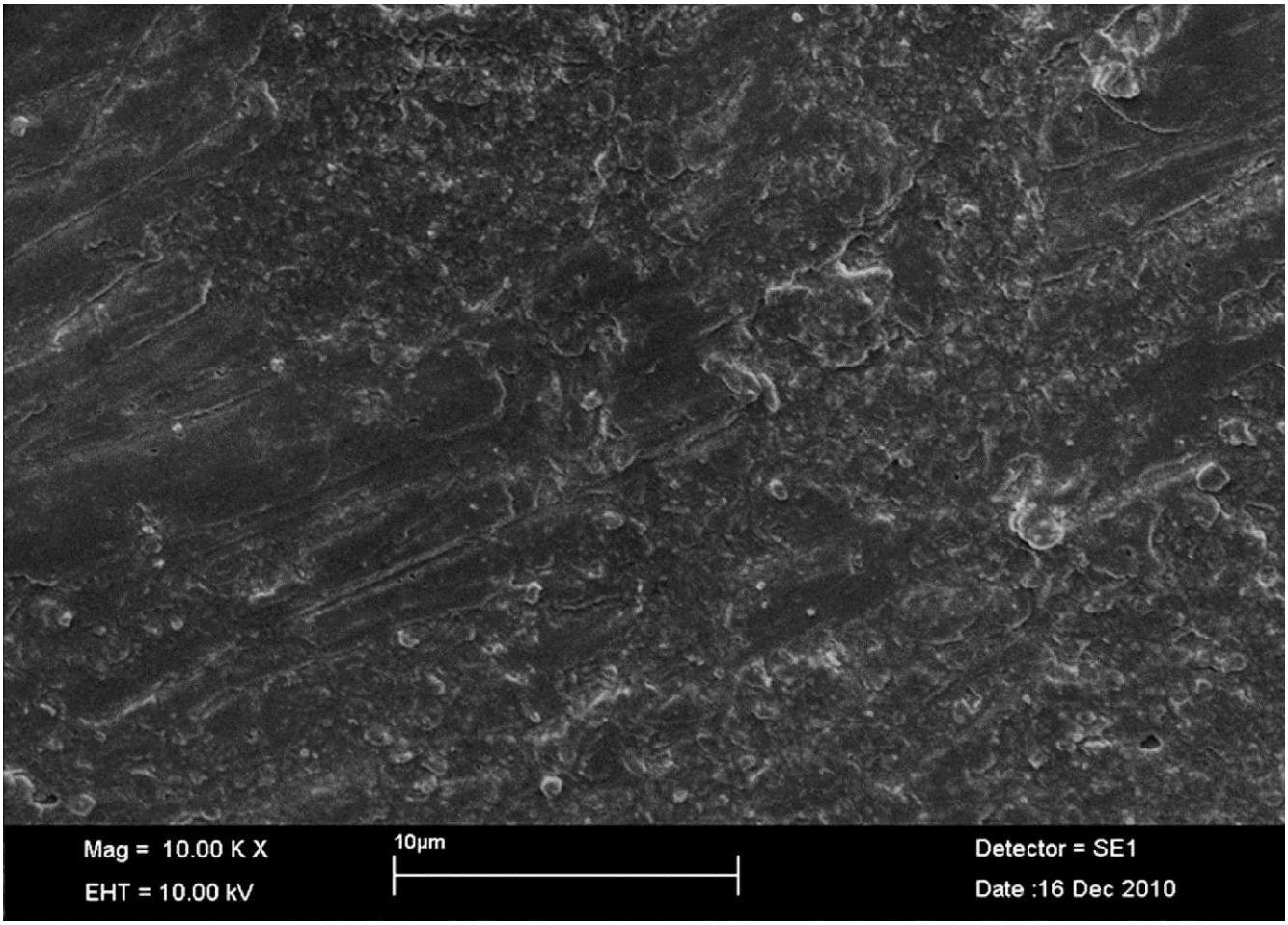

Figure 2. *SEM morphological aspect of a ceramic surface without additional treatment (10,000×).*

retention areas and surface area (Figure 4). Salvio and others,[7] Spohr and others,[8] Ayad and others,[20] and Attia[23] also demonstrated that there is a decrease in shear bond strength when the ceramic surface was air abraded with 50-µm Al_2O_3 when compared with a ceramic surface etched with 10% HF. However, Panah and others[24] showed no significant differences between a ceramic surface etched with 10% HF and one air abraded with 50-µm Al_2O_3.

A variable that can contribute to failure of a ceramic is the oral environment. It is known that the oral environment is able to induce physicochemical alterations in dental materials.[25] Mechanical fatigue and temperature alterations of materials provide conditions for their degradation in an aqueous environment.

In the present study, fatigue testing and thermal cycling were evaluated. The results indicated that the second hypothesis was not accepted. The groups etched with 10% HF or air abraded with 50-µm Al_2O_3 and submitted to fatigue testing or thermal cycling decreased in tensile bond strength, with statistically significant differences when compared with the control groups. It is known that fatigue results in material alterations in areas of stress concentration.[17] It is possible that when the specimens were submitted to a cyclical loading under wet conditions, the propagation of small cracks in the interface between the ceramic surface and the resin luting agent might have significantly reduced the bond strength.

In this present study, the effect of thermocycling on microtensile bond-strength testing was determined. Kamada and others[13] related that the durability of the bond strength between a silane-treated ceramic surface and the resin cement decreased with thermocycling and water storage. Oyafuso and others[25] showed a decrease in resistance to fracture after both ceramic-gold and ceramic-commercially pure titanium combinations were

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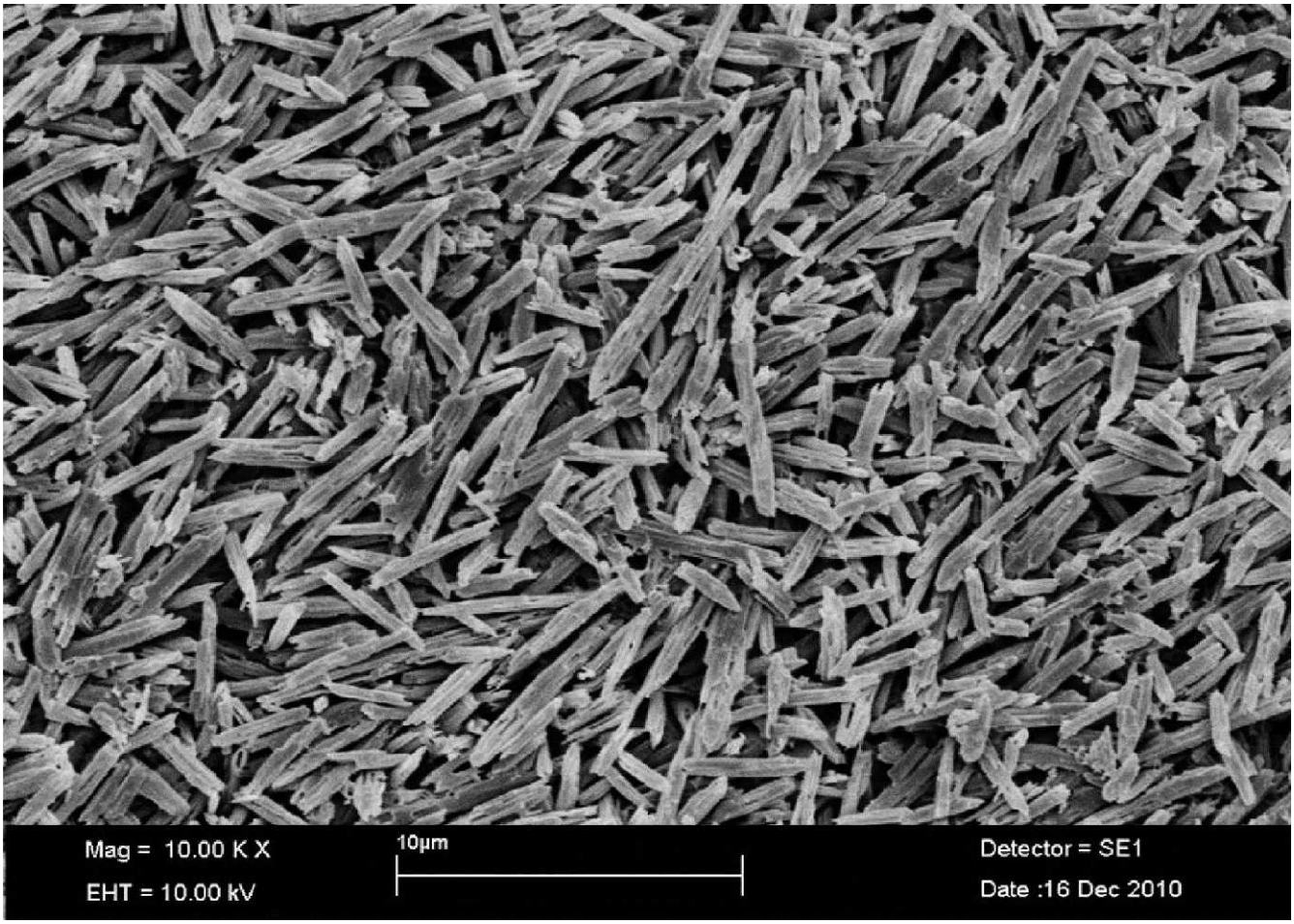

Figure 3. *SEM morphological aspect of a ceramic surface treated with 10% HF for 20 seconds (10,000×).*

subjected to thermocycling and mechanical cycling. Different studies have demonstrated that, depending on the silane used, thermocycling might have a significant effect on a reduction of bond strength between the resin and ceramic interface.[27–31] A degree of hydrolysis is responsible for the efficacy of the silane product; the higher the degree of hydrolysis, the better the bond provided by the silane coupling agent.[32] The permeability of the silane results from hydrolysis of the silicon-oxygen bonds at the ceramic-silane interface through water absorption.[33] However, this may also be responsible for the level of degradation of the bond strength between the ceramic-resin interface during thermocycling. Salvio and others[7] reported that the application of the Monobond S silane (Ivoclar Vivadent) followed by RelyX ARC resin cement was partially effective in water storage conditions, given that the mean bond strength decreased after 1 year. On the other hand, Spohr and others[8] showed that Scotchbond Ceramic Primer (3M ESPE), when used with the Single Bond adhesive system and Rely X resin cement, was effective after thermocycling procedures. However, in that study, the specimens were submitted to only 500 thermal cycles.

Another possible factor for the reduced bond strength seen in this current study is the reduction in the mechanical properties of the resin cement when submitted to fatigue testing and thermocycling. The reduction of mechanical properties of the resin cement is probably a result of a continuous action of water on the interface of the ceramic-resin cement. The mechanism of water transport and its effects on the mechanical properties of polymers depend on several factors.[34–35] Monomer ratio and composition vary according to the specific applications and the manufacturer's goals,[36] and variability will define the chemical stability of a resin in a specific environment.[37] The sensitivity of resin-based materials to water depends on the degree of monomer conversion,[38] degree of polymer cross-linking, volume fraction of intrinsic nanometer-sized

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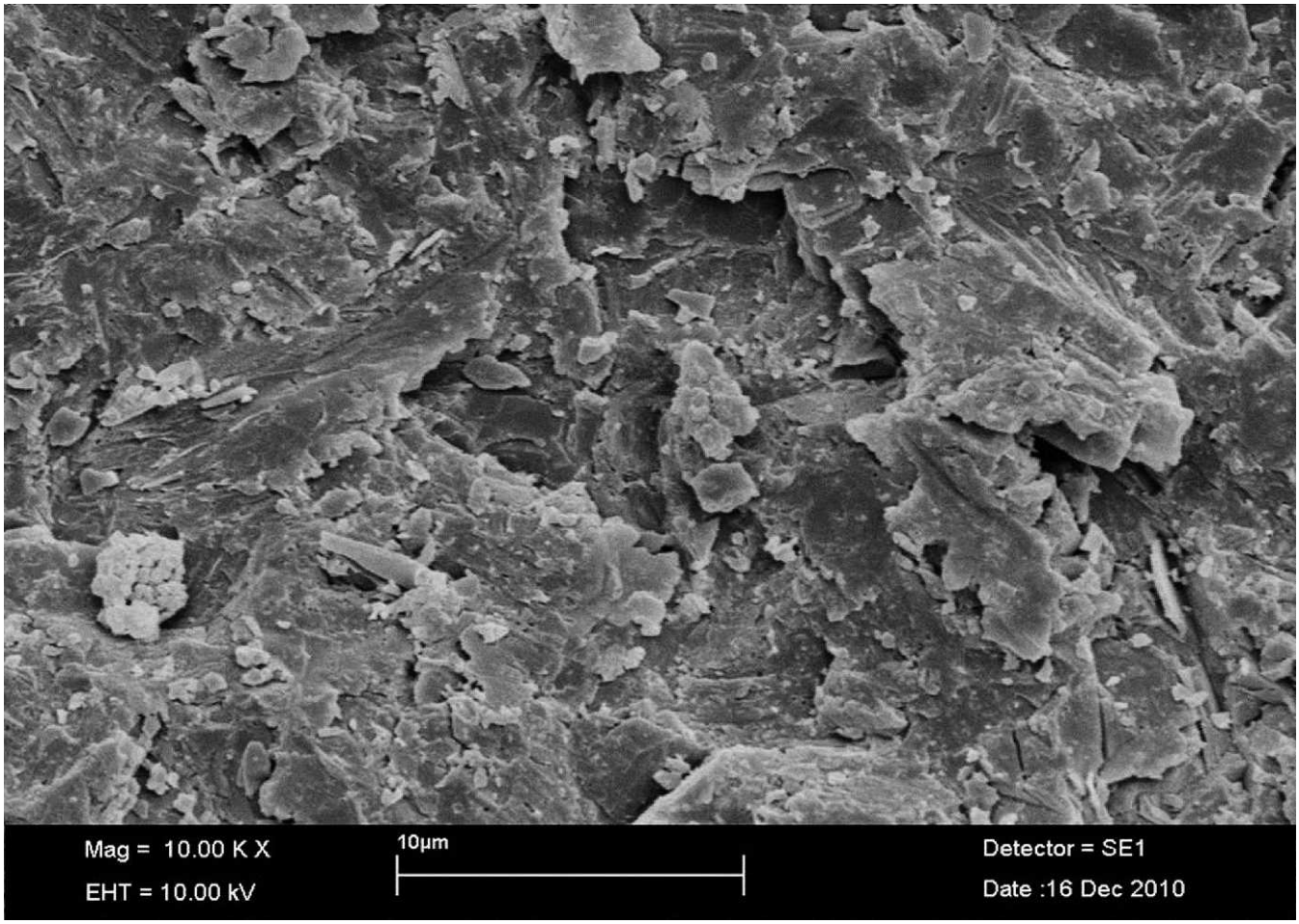

Figure 4. *SEM morphological aspect of a ceramic surface treated with air particle abrasion for five seconds with 50-µm aluminum oxide particles (10,000×).*

pores, and the quantity and presence of fillers.[34] One study found that by increasing the ratio of triethyleneglycol dimethacrylate (TEGDMA) and urethane dimethacrylate to bisphenol-A-glycidyl dimethacrylate, an increase in water sorption was observed.[39–40] It is possible that the presence of TEDGMA in the resin cement used in the present research contributed to the acceleration of water sorption and affected the mechanical properties of the resin cement after fatigue testing and thermocycling.

The mode of failure did not correlate directly with the bond-strength results, as can be seen in Table 2. Even though the control group showed bond strengths that were higher than the fatigued and thermocycled specimens for both treatments, the adhesive mode failure was predominant. This could be explained by the fact that the microtensile test evaluates a small area and the resin cement was much more strongly bonded with the resin composite than the ceramic, even though the bond with the ceramic material is a chemical union. Hence, more detailed studies would clarify the failures at microscopic levels. Thus, a closer evaluation using a SEM and an energy-dispersive spectrometer of the debonded surface could obtain more definitive information.

The present study evaluated one silane agent and one resin cement with a silicate-based ceramic. Further studies should be conducted to evaluate different materials. The current results showed the efficacy of surface treating a disilicate ceramic with 10% HF or with air abrasion using 50-µm Al_2O_3 particles. These results may be clinically useful when choosing a specific surface treatment of disilicate ceramics prior to luting with a resin cement.

CONCLUSION

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

1. Fatigue and thermal cycling significantly de-

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creased the microtensile bond strength for both ceramic surface treatments when compared with the control groups.
2. For the control group, the 10% HF surface treatment showed higher microtensile bond strength values when compared with the specimens treated by sandblasting with 50-μm Al_2O_3 particles.

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Conflict of Interest Declaration

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

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Effect of Delaying Toothbrushing During Bleaching on Enamel Surface Roughness: An *In Vitro* Study

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M Ghojazadeh • PA Oskoee

Clinical Relevance

Although daily toothbrushing immediately after bleaching increased enamel surface roughness, postponing the procedure for one or two hours after daily bleaching resulted in no changes in enamel surface roughness.

SUMMARY

This study aimed to evaluate the effect of toothbrushing on enamel surface roughness at three different intervals after daily bleaching treatment. Eighty enamel slabs were initially evaluated for surface roughness and then randomly divided into four groups. The bleaching procedure was carried out for 21 days, six hours daily. In the control group (group 1), the specimens were not brushed after bleaching, but in groups 2–4, they were brushed with toothpaste immediately, one hour, or two hours after bleaching, respectively. Then the specimens were stored in artificial saliva. Enamel surface roughness was reevaluated at the end of the period. Kruskal-Wallis and Mann-Whitney U tests showed statistically significant differences in the means of surface roughness values between the immediately brushed group and the three other groups ($p<0.001$). Daily toothbrushing immediately after bleaching increased enamel surface roughness; however, postponing the procedure for one or two hours after daily bleaching and exposing the specimens to artificial saliva during the study period resulted in enamel surface roughness comparable to that of the control group.

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INTRODUCTION

In recent years bleaching the teeth has gained utmost importance as a result of an increase in demand for esthetics and beautiful teeth in the community. Additional research is under way to achieve more appropriate results.

Carbamide peroxide, which is recommended for at-home bleaching procedures, is generally used in a tray for six to eight hours during the night.[1] One routine for oral hygiene procedures during the at-home bleaching period is to remove the tray after daily bleaching is finished and then brush the teeth with toothpaste. This way the bleaching agent is removed from tooth surfaces.[2] Considering the penetration of bleaching agents into tooth hard tissues, there is a possibility for changes in tooth structures. Some studies have demonstrated surface deterioration, formation of defects on the surface and porosity in electron microscope studies, and decreases in enamel hardness.[3–11] Therefore, the patient's oral hygiene procedures during the bleaching period can have a substantial role in creating subsequent complications, including tooth hypersensitivity. One study demonstrated that brushing teeth bleached with 10% carbamide peroxide using abrasive toothpastes increases enamel surface roughness.[2] On the other hand, using a fluoride varnish or mouthwash after exposing the teeth to carbamide peroxide can remineralize the enamel.[11–13]

Because use of saliva or artificial saliva in laboratory studies can also have a role in the remineralization of teeth,[2,7,14] the present study attempted to evaluate changes in enamel surface roughness subsequent to bleaching and brushing at three time intervals and storing the specimens in artificial saliva during the study period. The null hypothesis was that enamel surface roughness is not influenced by when toothbrushing is initiated after bleaching (immediately, at one hour, and at two hours after bleaching).

MATERIALS AND METHODS

In the present *in vitro* study, the specimens were prepared from impacted human third molars extracted surgically. The teeth were gathered after obtaining informed consent and approval from the deputy dean of research at Tabriz University of Medical Sciences. The teeth were then stored in 0.5% chloramine T solution (pH 8-11) until used. Impacted third molars were included in the study because there are no changes on enamel surface in such teeth. Teeth that had enamel surface abnormalities or cracks or fractures that occurred during surgical extraction were excluded from the study. The study design is illustrated in Figure 1.

Subsequent to cleaning the teeth the roots were cut away at the cementoenamel junction. Two 2-mm-thick enamel slabs measuring 6 × 4 mm were prepared from the middle third of the buccal and lingual aspects with the use of double-sided diamond disks (D&Z, Berlin, Germany). Eighty enamel slabs were prepared from 40 human third molars. The tooth sections were rechecked for any cracks or fractures under a stereomicroscope (Nikon, Tokyo-Japan) at ×20 and discarded if defective. Water spray was used during specimen preparation to avoid dehydrating the specimens. The specimens were stored in distilled water at 37°C after cutting and then placed, with the enamel surface on top, inside cold-cured acrylic resin in a cylindrical mold with a diameter of 1.5 cm. Subsequent to removal of the slabs from the mold, the side of a flat-end tapered diamond bur (Teezkavan, Tehran, Iran) in a high-speed handpiece under water spray was used to make the surface of the enamel slab horizontal so that it could be properly placed under the device that measures surface roughness. Then the enamel surface was smoothed using an enamel adjustment kit (Shofu Dental Corp, Kyoto, Japan) containing Dura White stones for adjusting and finishing and Ceramiste Points in standard, ultra, and ultra II grits for polishing. These stones and points were used in a low-speed handpiece under water spray. Finally, a disk composed of felt polishing cloth (Super-Snap Buff Disk, Shofu Dental Corp, Kyoto, Japan) was used in a low-speed handpiece along with first a 6- and then a 1-μm abrasive diamond paste (Microdent, São Paulo, Brazil) for the final polish of the enamel surfaces.

The specimens were placed in an ultrasonic device containing distilled water for 10 minutes to remove polish debris. Then the slabs were randomly divided into four groups of 20 specimens each, as follows:

Group A (control): No toothbrushing after bleaching
Group B: Toothbrushing immediately after bleaching
Group C: Toothbrushing one hour after bleaching
Group D: Toothbrushing two hours after bleaching

The initial surface roughness values of all the specimens were measured and recorded before the study using a stylus profilometer (MARSURF-PS1, Mahr, Göttingen, Germany) that uses the contact method. The measurements were randomly performed on the surface of enamel slabs. To this end,

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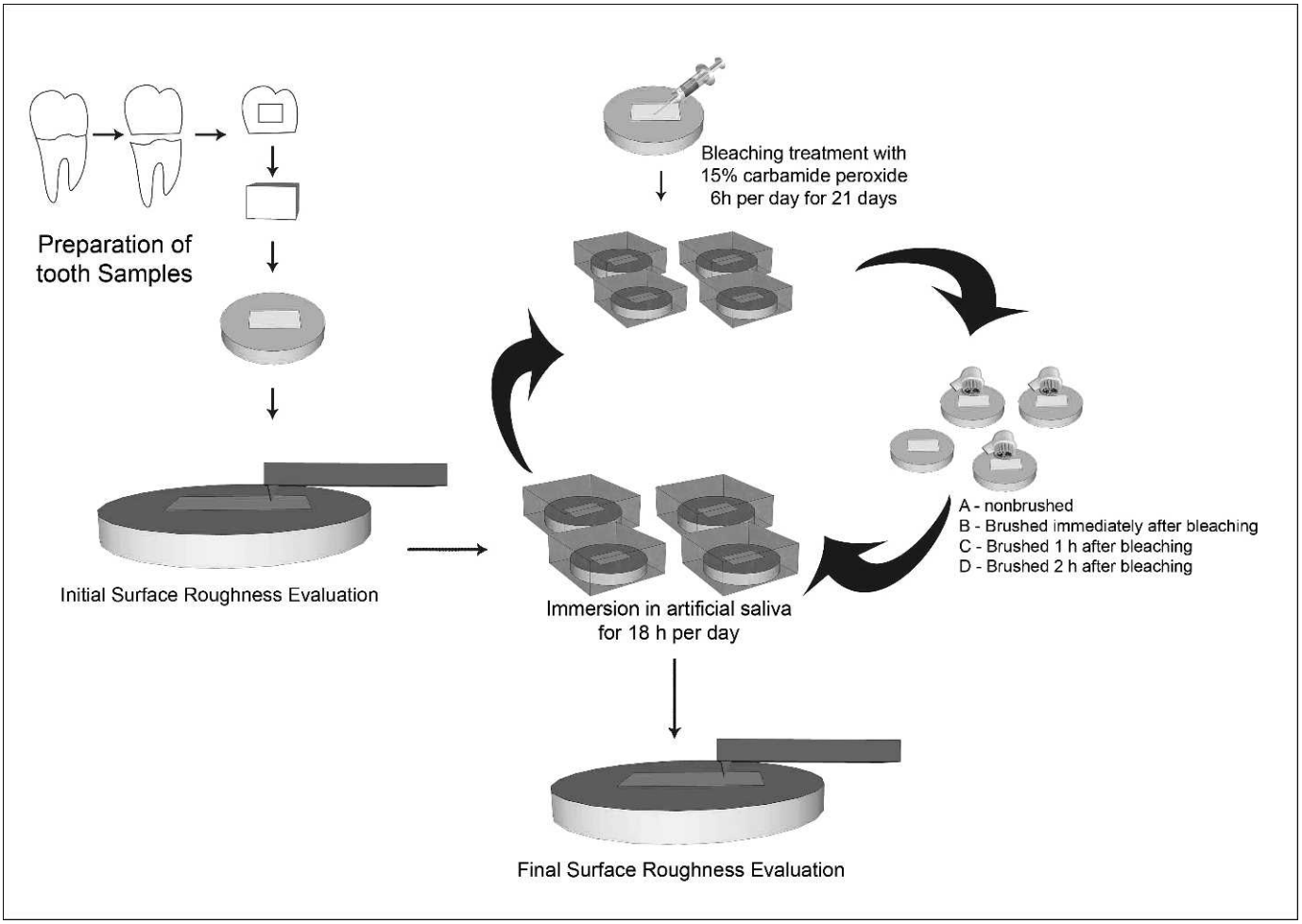

Figure 1. *Experimental design of the study.*

a 2-μm diamond bar scanned the surface at a rate of 0.1 mm/s and a force of 0.7 mN. Evaluation and cutoff lengths were 1.25 mm and 0.25 mm, respectively. The average roughness (Ra), which describes the overall surface roughness and can be defined as the arithmetic mean of all absolute distances of the roughness profile from the center line within the evaluation length,[15] was registered by profilometer in micrometers. Three tracings were made on each specimen at different locations. The mean roughness value, achieved after three measurements on each slab, was statistically analyzed. Because this system had an integrated calibration standard there was no need for external calibration.

After the initial surface roughness was measured, the bleaching process was instituted. To this end, a special tray was fabricated for each specimen in a vacuum apparatus; each tray was made of ethylvinyl acetate and was 1 mm thick. Then 0.02 mL of 15% carbamide peroxide gel (Opalescence PF, Ultradent Products, South Jordan, UT) was placed inside each tray, and the tray was placed on each specimen for six hours daily. During the process each specimen covered with the tray was placed in a separate vial containing artificial saliva, which was replaced daily. The composition of the artificial saliva was as follows: 1.0 mM $CaCl_2$, 3.0 mM KH_2PO_4, and 100 mM NaCl; the pH value was 6.30 and was adjusted with NaOH solution.[16]

After the daily bleaching procedure, the specimens were rinsed with deionized distilled water for five seconds. Then the subsequent steps for each group were carried out as follows.

Group A: The specimens in group A were placed in 1 mL of artificial saliva at 37°C in an incubator for 18 hours after bleaching and rinsing.
Group B: In group B the specimens were brushed immediately after they were bleached for six hours and rinsed for five seconds; the specimens were

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Table 1. *Mean Differences in Surface Roughness Values Before and After Intervention (ΔRa = final value – baseline value)*

Group	Baseline Value		Final Value		Change in Roughness (ΔRa)				
	Mean	SD	Mean	SD	Mean	SD	Median	Lowest	Highest
A	0.63	0.18	0.48	0.18	–0.15	0.22	–0.13	–0.55	0.20
B	0.51	0.16	0.64	0.29	0.13	0.25	0.14	–0.45	0.86
C	0.52	0.13	0.32	0.06	–0.20	0.12	–0.20	–0.45	–0.02
D	0.55	0.19	0.44	0.12	–0.11	0.19	–0.05	–0.49	0.10

brushed with a powered brush (Oral-B Vitality Precision model, Oral-B Corp, Belmont, CA) inside a reservoir of freshly prepared toothpaste slurry (Opalescence whitening toothpaste, Ultradent Products) with one part (50 g) of toothpaste to three parts (150 g) of deionized distilled water. The brush was fixed on a bar with a clamp, and brushing was carried out once daily for three minutes with a typical force of 200 g. The amount of force applied was measured with an orthodontic gauge. The brush head was made of nylon and was multitufted. A separate and specific brush was used for each specimen. The specimen was placed inside the toothpaste slurry, which was agitated before use. The toothpaste slurry was replaced every two days so that a neutral pH was maintained. After daily brushing, the specimens were rinsed with distilled water and stored in special containers containing artificial saliva at 37°C for the rest of the day.

Group C: The same brushing procedure described for group B was repeated for group C, except that after bleaching and rinsing, the specimens were kept in artificial saliva for one hour, after which the brushing procedure was carried out. Then the specimens were once again stored in artificial saliva until the next day.

Group D: The procedure for group D was the same as for group C, but after bleaching there was a time interval of two hours before toothbrushing occurred.

The bleaching, brushing, and rinsing procedures continued for 21 days in all the groups. At the end of this period the surface roughness values of the specimens were once again measured, recorded, and compared with the initial values. Data were analyzed with a nonparametric Kruskal-Wallis test. A nonparametric Mann-Whitney U test was used for two-by-two comparison of the groups. Statistical significance was defined at $p<0.05$.

RESULTS

Table 1 demonstrates the descriptive statistics of mean differences in surface roughness values before and after intervention in the groups under study.

Before the study was initiated, the means of surface roughness values in the four groups were compared. The nonparametric Kruskal-Wallis test showed that there were no significant differences in the means of surface roughness values before intervention among the four groups ($p=0.12$).

Because data were widely dispersed, logarithmic transformation of the data was considered, and then a nonparametric Kruskal-Wallis test was used to evaluate the differences. The results showed that the differences in the means of surface roughness values before and after intervention were statistically significant among the groups under study ($p<0.001$). Mann-Whitney U test revealed significant differences in the means of surface roughness values between the immediately brushed group and the three other groups ($p<0.001$). There were no significant differences between the other groups ($p\geq0.06$).

The linear and bar graphs of the mean differences in enamel surface roughness values before and after intervention in the groups are presented in Figure 2.

DISCUSSION

Surface roughness, a measure of the texture of a surface, is one of the test methods used to evaluate the effects of different bleaching materials and oral hygiene procedures on tooth hard tissues.[2,9,10,17,18] The oral cavity is inhabited by many diverse microbial species, and most of these microorganisms, especially those responsible for caries or periodontitis, can only survive in the oral cavity when they

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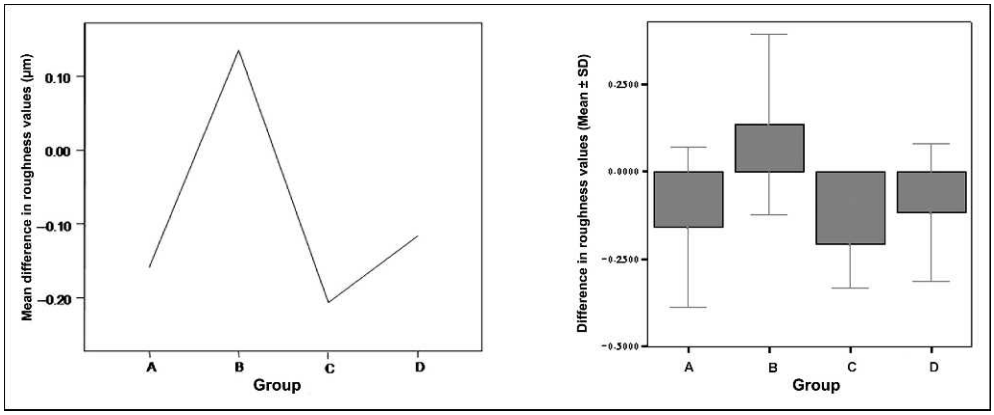

Figure 2. *The linear and bar graphs of differences in surface roughness values before and after intervention in the groups under study.*

adhere to rough surfaces. Therefore, the roughness of intraoral hard tissues can promote formation, maturation, and retention of plaque, resulting in increased risk of dental caries, periodontitis, or tooth discoloration.[19,20]

According to the results of the present study, the only group demonstrating significant increases in surface roughness after bleaching compared with the control group was group B (toothbrushing immediately after bleaching). In other words, postponement of toothbrushing for one or two hours after daily bleaching procedures and storing the specimens in artificial saliva during the wait period led to surface roughness values comparable to those in the control group; therefore, the null hypothesis was rejected.

Bleaching agents might exert negative influences on the integrity of organic structures of the tooth, including proteins and collagen. By-products of carbamide peroxide breakdown are urea and hydrogen peroxide. Urea can denature tooth structure proteins by penetrating the enamel structure and influencing prismatic and interprismatic structure and also by increasing permeability and ultrastructural changes. These processes result in pore formation and increases in the diameter of enamel and dentin surface pores. On the other hand, the free oxygen present in hydrogen peroxide increases surface porosity and reacts with the organic structure of dental tissues. Increases in the surface porosity facilitate the passage of oxygen radicals beyond enamel and dentin and the breakdown of stained macromolecules into smaller and light-colored molecules.[5,10]

In the present study, 15% carbamide peroxide gel (Opalescence PF, Ultradent Products) was used. This gel contains 3% potassium nitrate and 0.11 wt% (equal to 1100 ppm) fluoride ion. According to the manufacturer, the incorporation of fluoride ions and potassium nitrate into the structure of this gel has helped reduce the odds of caries and tooth hypersensitivity during bleaching and has improved enamel health and integrity. In addition, according to some studies, bleaching with these gels does not increase enamel susceptibility to caries or acidic erosion or increase the odds of demineralization.[21–24] However, some studies have reported significant increases in enamel surface roughness subsequent to the use of bleaching agents.[8,9] Some of the complications of applying bleaching agents in tooth hard tissues are changes in tooth chemical structure, demineralization and decreases in the mineral content including calcium and phosphate,[14,25,26] decreases in the fluoride content of enamel,[11] and changes in the micromorphology of enamel.[9,27,28] However, other studies have reported different results with no statistically significant differences in tooth hard tissue characteristics, including enamel and dentin surface roughness, after bleaching with 10% and 15% carbamide peroxide.[17,18,29–31]

The discrepancies in the results of various studies might be attributed to differences in the formulation and concentration of bleaching agents; the duration of daily applications or treatment protocols; differences in study designs, including the environments in which the specimens are stored (eg, artificial or human saliva); the use of remineralizing agents; continuous use of fluoridated toothpastes; and the technique of toothbrushing during the procedure and after bleaching.[2,14,30,32,33] In the present study, tooth structures underwent surface changes after bleaching, and toothbrushing immediately after bleaching had a substantial role in increasing enamel surface

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roughness. In other words, under such conditions, bleaching might have a synergistic effect with toothbrushing, contributing to increases in enamel surface roughness. However, in our previous study, which measured enamel microhardness in the same experimental conditions, it was shown that toothbrushing immediately after bleaching has no detrimental effect on enamel microhardness.[34]

Another study has demonstrated that toothbrushing with abrasive toothpastes with or without fluoride after bleaching with 10% carbamide peroxide increases enamel surface roughness.[2] Therefore, in the present study a low-abrasive fluoridated toothpaste recommended by the bleaching agent manufacturer was used for toothbrushing in all the groups. According to some studies, despite the probable destructive role of toothbrushing, the fluoride present in toothpastes can play a balancing role between remineralization and demineralization when used daily after the bleaching procedure.[12,13] When fluoride is used, formation of calcium fluoride prevents enamel surface demineralization.[12,35,36] Because the toothbrushing regimen was the same in all the groups in the present study, improvements in enamel surface roughness cannot be attributed to the role of fluoride in the toothpaste.

Saliva can have a notable role in remineralization.[2,18] In most studies on bleaching, specimens have been stored in remineralizing solutions containing PO_4^{-3} and Ca^{+2} with concentrations similar to that of human saliva. According to some studies, artificial saliva can mimic oral saliva *in vitro* and plays a role in remineralization.[16] Considering the results of the present study, if teeth have the opportunity to be in contact with artificial saliva after bleaching and before toothbrushing, surface roughness will be comparable to that of the control group. It is likely that precipitation of minerals present in the artificial saliva on tooth surfaces can play a role in decreasing the surface roughness of bleached enamel[18]; however, further studies are necessary to evaluate the effects of different storage environments, such as artificial saliva and plain water, on changes in surface roughness of bleached enamel. In the present study the specimens were kept in artificial saliva for one or two hours before toothbrushing, which did not result in significant differences ($p \geq 0.06$).

One of the factors influencing changes in tooth surfaces during the bleaching period is the pH of the bleaching agent. More acidic pH values increase the likelihood of surface changes and demineralization.[37] The carbamide peroxide gel used in the present study had a pH value of 6.5. According to the results of several studies, two factors can neutralize this pH. The first factor is the urea produced by the breakdown of carbamide peroxide after its use, which is mainly responsible for an increase in the oral pH value to more than 8 for a few hours. The second factor is saliva, which is believed to have a role in neutralizing the acidity of the bleaching gel.[37] An *in vivo* study has shown that the low pH of bleaching agents in the first five minutes results in a decrease in the pH value of the patient's saliva. In 15 minutes, the pH value will be higher than the baseline value; this is attributed to the chemical reaction between carbamide peroxide and salivary bicarbonate ion as a result of the buffering capacity of saliva, which neutralizes the acidity of the bleaching agent.[38] Another positive role of saliva becomes evident when, similar to the present study, fluoridated bleaching agents are used. Fluoridated bleaching gels reinforce the fluoride-containing elements of tooth enamel. Although this influence is less effective than that of pure fluoride, fluoride-containing gel strengthens and restores microstructural defects on tooth surfaces by surface absorption and precipitating fluorapatite from calcium and phosphate ions of saliva.[21,39] However, further studies, especially clinical experiments, should be carried out to explore this further.

In the present study, enamel samples were smoothed and polished to allow standardized profilometric measurements with flat reference surfaces. Considering that the specimen preparation process in surface roughness tests might have influenced the results, it might not be entirely possible to extrapolate the conclusions of *in vitro* studies to the clinical setting.

Finally, it is suggested that in future studies other techniques, including microradiography and micro-computed tomography scanning, be used for more accurate evaluation of changes in the enamel surface.

CONCLUSION

Within the limitations of the present study it can be concluded that daily toothbrushing with low-abrasive fluoridated toothpaste, carried out immediately after bleaching with 15% carbamide peroxide, can increase enamel surface roughness; however, postponing the procedure for one or two hours after daily bleaching and exposing the teeth to saliva during the delay results in surface roughness comparable to that of the control group.

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Conflict of Interest Declaration

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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Wear Rates of Resin Composites

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TM Wilwerding

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Clinical Relevance

Laboratory wear testing of resin composites provides valuable information for clinicians in selecting materials for clinical use.

SUMMARY

A laboratory study was conducted to examine the wear of resin composite materials using a generalized wear simulation model. Ten specimens each of five resin composites (Esthet•X [EX], Filtek Supreme Plus [SP], Filtek Z250 [Z2], Tetric EvoCeram [EC], and Z100 Restorative [Z1]) were subjected to wear challenges of 100,000, 400,000, 800,000, and 1,200,000 cycles. The materials were placed in cylinder-shaped stainless-steel fixtures, and wear was generated using a flat stainless-steel antagonist in a slurry of polymethylmethacrylate beads. Wear (mean facet depth [μm] and volume loss [mm^3]) was determined using a non-contact profilometer (Proscan 2000) with Proscan and ProForm software. Statistical analysis of the laboratory data using analysis of variance and Tukey's post hoc test showed a significant difference ($p<0.05$) for mean wear facet depth and volume loss for both the number of cycles and resin composite material. Linear regression analysis was used to develop predictive wear rates and volume loss rates. Linear wear was demonstrated with correlation coefficients (R^2) ranging from 0.914 to 0.995. Mean wear values (mean facet depth [μm]) and standard deviations (SD) for 1200K cycles were as follows: Z1 13.9 (2.0), Z2 26.7 (2.7), SP 30.1 (4.1), EC 31.8 (2.3), and EX 67.5 (8.2). Volume loss (mm^3) and SDs for 1200K cycles were as follows: Z1 0.248 (0.036), Z2 0.477 (0.044), SP 0.541 (0.072), EC 0.584 (0.037), and EX 1.162 (0.139). The wear rate (μm) and volume loss rate (mm^3) per 100,000 cycles for the five resin composites were as follows: wear rate Z1 0.58, EC 1.27, Z2 1.49, SP 1.62, and EX 4.35, and volume loss rate Z1 0.009, EC 0.024, Z2 0.028, SP 0.029, and EX 0.075. The generalized wear model appears to be an excellent method for measuring relative wear of resin composite materials.

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INTRODUCTION

Resin composite materials are now routinely used for the restoration of the posterior dentition. In evaluat-

Table 1: *Resin Composite Materials*

Material	Manufacturer	Lot	Shade	Study Code
Esthet•X	DENTSPLY Caulk, Milford, DE, USA	061206	A2	EX
Filtek Supreme Plus	3M ESPE Dental Products, St Paul, MN, USA	8WU	A2 Body Shade	SP
Filtek Z250	3M ESPE Dental Products, St Paul, MN, USA	9JE	A2	Z2
Tetric EvoCeram	Ivoclar Vivadent AG Schaan, Liechtenstein	L56579	A2	EC
Z100 Restorative	3M ESPE Dental Products, St Paul, MN, USA	7PP	A2	Z1

ing the performance of posterior composites, wear characteristics are an important parameter. While resin composites are now generally accepted for use in the posterior region, the materials currently available are very different in formulation and may not exhibit similar clinical performance. There is significant value in knowing how materials perform relative to others available. Clinicians need good scientific data to provide optimal care for their patients.

Clinical and laboratory studies have been used over the years to assess the wear characteristics of resin composite materials. Trying to relate clinical and laboratory wear data is a significant challenge because adequate clinical data are not available. In addition, clinical testing should ideally be done with multiple materials in the same study and with a large number of patients. To gain even more information, multiple-site studies should also be conducted. This approach is very expensive, takes years to complete, and before the clinical trials are over, the materials in the studies may be obsolete.

An alternate approach is to look at the relative wear of a number of materials in a laboratory wear simulation study and compare the rates of wear among the materials. The wear rates could be further compared with a benchmark material with demonstrated low wear in both clinical and laboratory studies. This approach has been used by Barkmeier and others[1] in reporting the generalized clinical wear rates (contact-free area [CFA]) for Z100 and P50 (3M ESPE, St Paul, MN, USA) and comparing the clinical wear rates to simulated wear rates using a laboratory model to simulate generalized wear. Linear regression was employed to predict both clinical and laboratory wear rates of these two materials. Z100 demonstrated minimal clinical and laboratory wear and would certainly qualify as a benchmark material for further studies. This study showed a similar relationship for the ratios of wear rates between the two materials in both laboratory and clinical testing, indicating that this is a promising approach for examining and comparing wear rates of resin composite materials.

Wear simulation provides an efficient means to develop relative wear rates among materials and to compare these results to a benchmark material that has exhibited good laboratory and clinical performance. Because of the void in clinical wear data available, additional wear simulation data are required to expand the information base needed to examine and compare the performance of resin composite materials. The purpose of this laboratory study was to continue developing data related to simulated generalized wear of resin composite materials and provide additional information to clinicians for the selection of materials for clinical practice. A reference (benchmark) resin composite material (Z100), with previously published laboratory and clinical data,[1,2] was selected for comparison to four other composite materials with different formulations that are commonly used for restoration of the posterior dentition.

METHODS AND MATERIALS

Five resin composite materials were evaluated in this study and are listed in Table 1. Ten specimens for each of the five resin composite materials (total of 50 specimens) were prepared for wear challenges of 100,000, 400,000, 800,000, and 1,200,000 cycles using a generalized wear model (CFA wear) in a Leinfelder-Suzuki wear simulation device (Alabama machine). The methodology for sample preparation and the generalized wear model has been previously described by Barkmeier and others.[1] In summary, stainless-steel custom fixtures with cavities 4.5 mm in diameter and 4 mm deep were used to hold the

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Figure 1. *Stainless-steel custom fixture with resin composite material.*

test materials. The resin composites were cured in two increments of approximately 2 mm for 40 seconds with a Spectrum 800 curing unit (DENTSPLY Caulk, Milford, DE, USA) set at 600 mW/cm^2. After 24 hours, the composite surfaces were polished flat to 4000 grit (Figure 1) using a sequence of silicon carbide papers (Struers Inc, Cleveland, OH, USA). The custom fixtures were mounted inside a plastic water bath, and a cylinder was placed around each fixture. A water slurry of polymethyl methacrylate was used as the abrasive media and placed inside the cylinders over the resin composite specimens. Stainless-steel antagonists 6.5 mm diameter (Figure 2), mounted in spring-loaded pistons, were then used to deliver the wear challenges in the wear simulation machine. The pistons rotated approximately 30° as the load was applied (maximum load of 78.5 N) at a rate of 2 Hz.

Prior to wear testing, the specimens for each resin composite material were profiled using a Proscan 2000 noncontact optical profilometer (Scantron Industrial Products Ltd, Taunton, England) with Proscan software. The individual scanned surfaces were used as the pretest digitalized surface (Figure 3) for each individual specimen.

Following each cycling period (100K, 400K, 800K, and 1200K), the specimens were ultrasonically cleaned (L&R Solid State Ultrasonic T-14B, South Orange, NJ, USA) for three minutes in distilled

Figure 2. *Stainless-steel antagonist tip.*

water and then profiled using the Proscan 2000 unit (Figures 4 and 5). The pre- and posttest digitalized surfaces were compared using ProForm and Proscan software (Scantron Industrial Products Ltd).

The individual pretest scan and posttest scan for each material, after each cycling period, were loaded in ProForm. The pretest and posttest scans were manually fitted (X, Y, and Z parameters) using the ProForm software. Following the fitting, a "difference file" was created (saved) and then opened in the Proscan software program for analysis of the differences between the pretest and posttest digitalized surfaces. Two wear measurements were determined using the difference files in Proscan: 1) mean wear depth (μm) and 2) volume loss (mm^3). The wear measurements were determined from differences between the before and after data sets.

Volume loss and mean wear depth data were analyzed using a two-way analysis of variance (ANOVA) and Tukey's post hoc test. Factors for the

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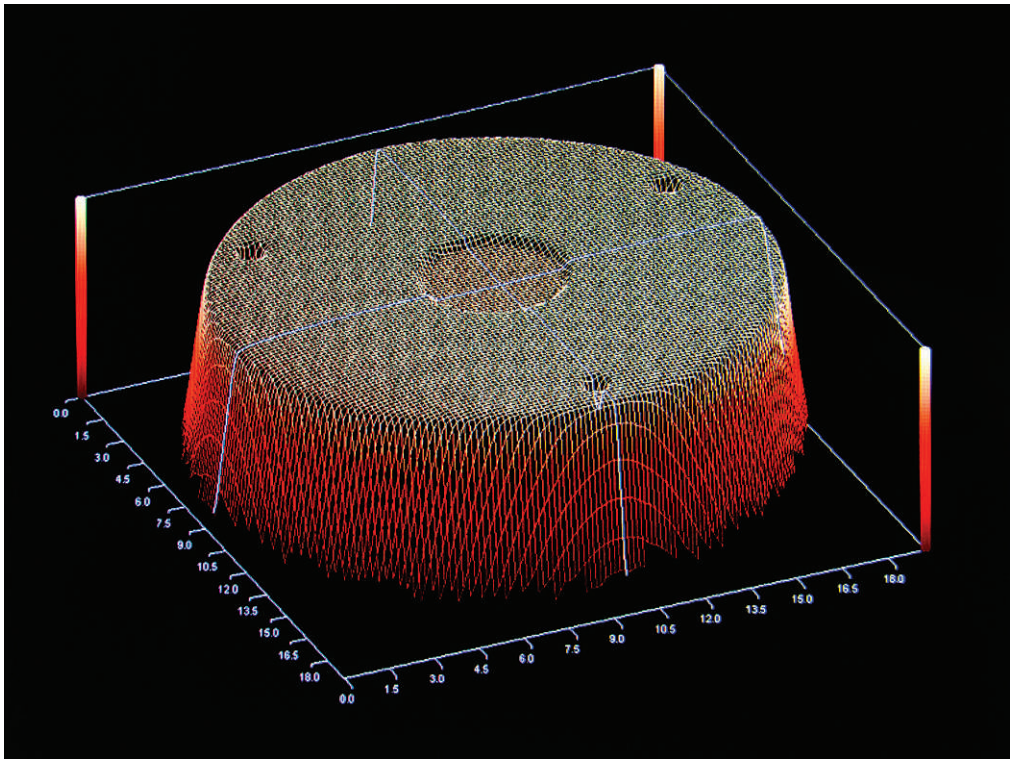

Figure 3. *Scanned surface of custom fixture with polished resin composite material before wear challenge.*

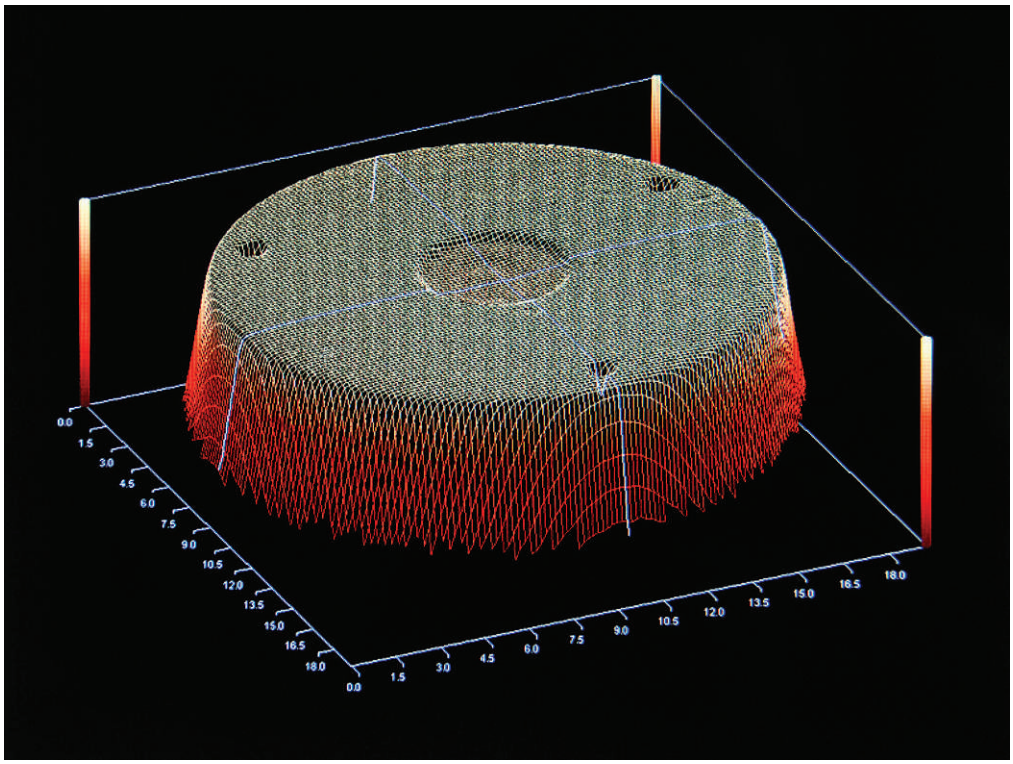

Figure 4. *Scanned surface of resin material with minimal wear after 1200K cycles of generalized wear simulation.*

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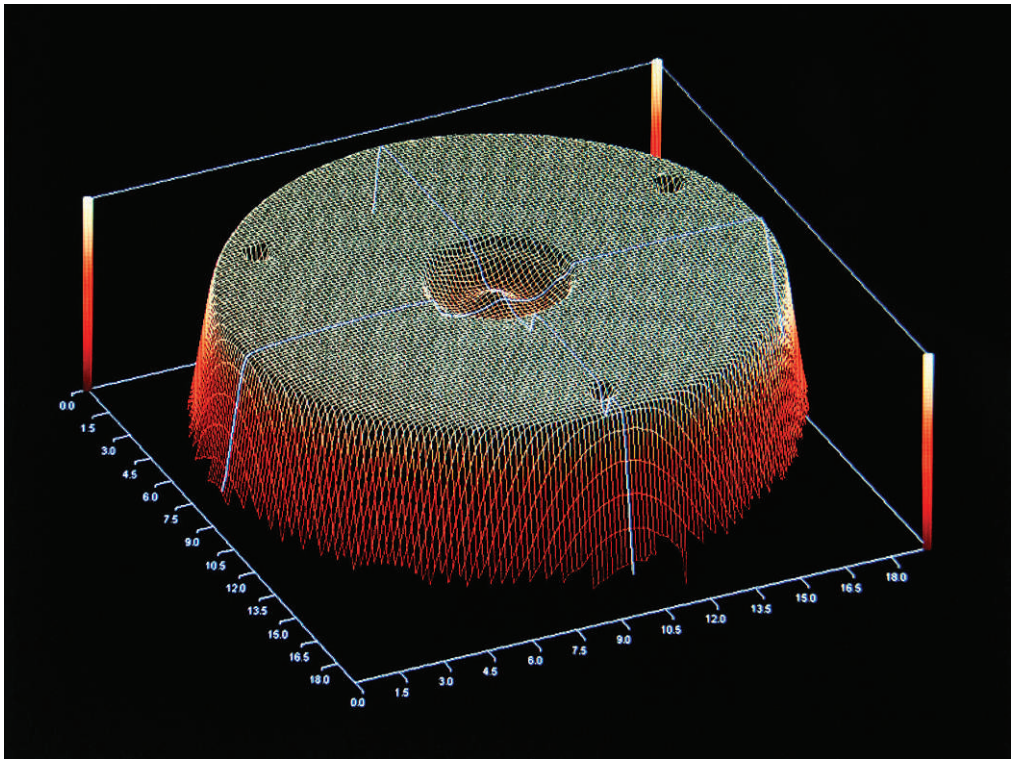

Figure 5. *Scanned surface of resin material with moderate wear after 1200K cycles of generalized wear simulation.*

ANOVA tests were 1) resin composite material and 2) number of cycles. Linear regression analysis of mean wear depth and volume loss data was used to examine the relationship of the variables in this study: 1) resin composite material and 2) number of cycles. The association strength between the variables, R^2 (square of the correlation coefficient), was determined for each resin composite material at the four cycling periods (100K, 400K, 800K, and 1200K). A regression line was also developed to predict wear rates and volume loss rates for the resin composites.

RESULTS

The two-way ANOVA of the laboratory data, for both volume loss and mean wear depth, revealed a significant effect for the factors of resin composite material (p=0.000) and number of cycles (p=0.000), as well as for the interaction of resin composite material and number of cycles (p=0.000). The ANOVA results are presented in Tables 2 and 3.

The generalized wear values (mean wear depth and volume loss) for the five resin composite materials at the four cycling periods (100K, 400K, 800K, and 1200K) are summarized in Tables 4 and 5. The statistical differences (p<0.05) for wear depth and volume loss for each material at the four cycling periods, as well as difference among materials at each cycling period, are also presented in Tables 4 and 5 (multiple pairwise comparison with Tukey's post hoc test). As the number of cycles increased, the occurrence of significant differences (p<0.05) for the individual resin composites tested also increased. The data also showed differences (p<0.05) among materials at the various cycling periods (Table 4 and 5).

Regression lines for wear depth and volume loss vs cycling periods for the five resin composites are presented in Figures 6 and 7. The regression lines for both wear depth and volume loss all had slopes that were significant at the 0.05 level. The strength of association (R^2) between the variables of resin composite material and number of cycles for both wear depth and volume loss are presented in Table 6. A strong association was found between the variables for both wear depth and volume loss. Predicted wear rates and volume loss rates determined by linear regression are also presented in Table 6.

DISCUSSION

Limited clinical data are available in the dental literature for clinicians to assess the performance of resin composite materials. In the 1970s and 1980s, when resin composite materials were first being

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Table 2: *Analysis of Variance—Mean Facet Depth*

Source	Sum-of-Squares	*df*	Mean Square	*F*-Ratio	*p*
Material	23334.071	4	5833.518	438.825	0.000
Cycles	11564.731	3	3854.910	289.985	0.000
Material*Cycles	5837.278	12	486.440	36.592	0.000
Error	2326.361	175	13.293		

Table 3: *Analysis of Variance—Volume Loss*

Source	Sum-of-Squares	*df*	Mean Square	*F*-Ratio	*p*
Material	6.770	4	1.692	366.490	0.000
Cycles	3.774	3	1.258	272.420	0.000
Material*Cycles	1.681	12	0.140	30.329	0.000
Error	0.808	175	0.005		

Table 4: *Generalized Wear—Mean Wear Depth (SD)*[a]

Cycles	Mean Facet Depth, μm				
	Z1	Z2	SP	EC	EX
100K	7.5 (1.3) aA	9.7 (1.2) aA	12.8 (2.2) aAB	17.3 (1.8) aB	18.5 (4.8) aB
400K	9.6 (1.6) abA	15.6 (2.2) bB	17.8 (3.1) aBC	22.8 (2.2) abC	35.8 (6.4) bD
800K	11.9 (1.8) abA	19.6 (2.2) bB	25.9 (2.2) bC	26.5 (2.4) bcC	50.6 (6.7) cD
1200K	13.9 (2.0) bA	26.7 (2.7) cB	30.1 (4.1) bB	31.8 (2.3) cB	67.5 (8.2) dC

[a] *Groups in vertical columns with the same small-case letter are not different at the 5% significance level. Groups in different columns with same number of cycles and same capital case letter are not different at the 5% significance level.*

advocated for the posterior region, early evidence suggested significant wear compared to amalgam.[3] Because of the skepticism surrounding the use of resin composites in the posterior dentition, acceptance guidelines were developed by the American Dental Association,[4] and manufacturers conducted clinical studies to gain product acceptance for the posterior area. As materials improved and resin composites were more widely accepted for the posterior region, manufacturers have been more reluctant to invest in clinical studies. Limited quantitative wear data are available in the dental literature for resin composite restorative materials.

Various approaches have been taken by researchers to fill the void in clinical data by conducting wear simulation studies in the laboratory. Wear simulation studies have been used to develop the wear rates of laboratory specimens and then compare the rates against values determined from reported clinical studies.[1,5] Heintze and others[5] recently published a

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Table 5: *Generalized Wear—Volume Loss (SD)*[a]

Cycles	Volume Loss (mm^3)				
	Z1	Z2	SP	EC	EX
100K	0.135 (0.025) aA	0.173 (0.020) aA	0.226 (0.039) aAB	0.309 (0.032) aB	0.320 (0.085) aB
400K	0.196 (0.030) abA	0.275 (0.035) abAB	0.319 (0.063) aBC	0.411 (0.039) abC	0.618 (0.100) bD
800K	0.212 (0.031) abA	0.347 (0.031) bB	0.464 (0.045) bC	0.480 (0.049) bcC	0.886 (0.117) cD
1200K	0.248 (0.036) bA	0.477 (0.044) cB	0.541 (0.072) bB	0.584 (0.037) cB	1.162 (0.139) dC

[a] *Groups in vertical columns with same small-case letter are not different at the 5% significance level. Groups in different columns with the same number of cycles and same capital case letter are not different at the 5% significance level.*

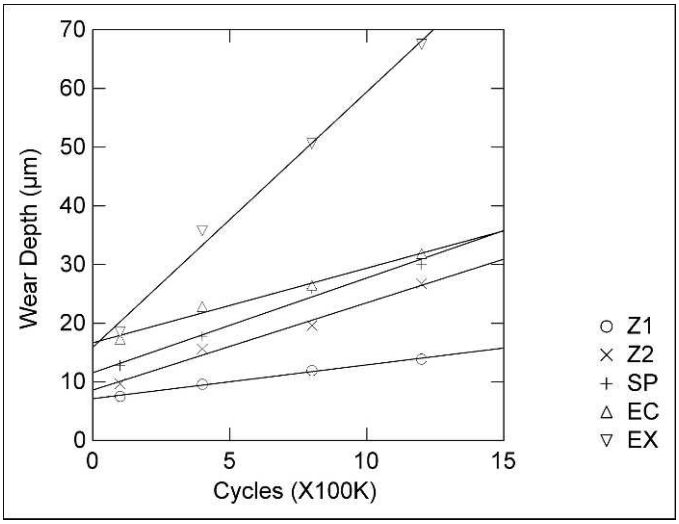

Figure 6. *Wear depth of resin composites (µm) vs cycles (X100K).*

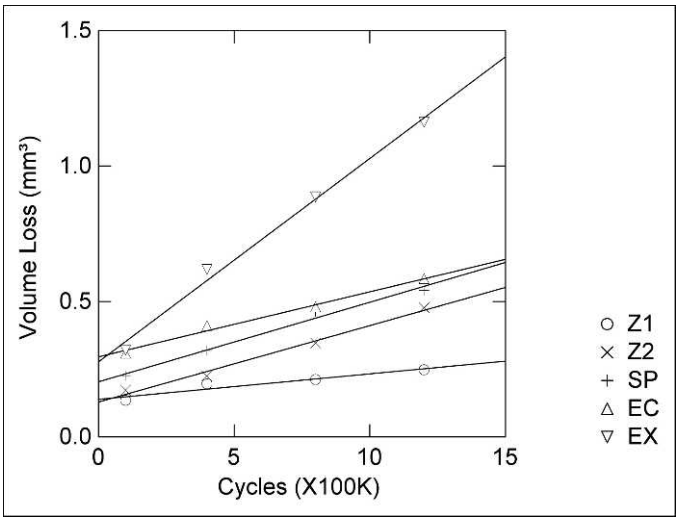

Figure 7. *Volume loss of resin composites (µm) vs cycles (X100K).*

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Table 6: *Regression Analysis—Wear (μm) and Volume Loss (mm^3) per 100K Cycles and* R^2 *Value*

Resin Composite	Wear Rate	R^2	Volume Loss Rate	R^2
Z1	0.58	0.995	0.009	0.914
EC	1.27	0.985	0.024	0.986
Z2	1.49	0.986	0.028	0.988
SP	1.62	0.983	0.029	0.985
EX	4.35	0.993	0.075	0.992

summary of information regarding the use of six different approaches for wear simulation.

In 2008, Barkmeier and other[1] examined the relationship of simulated generalized wear to CFA clinical wear. Clinical wear was estimated using the Moffa-Lugussy (M-L) technique[6,7] (M-L Scale, Joseph P. Moffa, Las Vegas, NV, USA), and cumulative wear after three years for P50 was 29.7 μm and 17.0 μm for Z100. Wear measurements using the M-L scale are estimates of CFA wear. Simulated wear (mean maximum depth and mean depth) was approximately twice as much for P50 when compared with Z100, which paralleled the clinical findings. There was good agreement between the relationship of simulated and clinical wear. Because of the proven clinical performance and paralleled low laboratory simulation wear rates of Z100, this material is an ideal candidate to be selected as a benchmark material when examining the wear characteristics of resin composite materials.

In the present study, simulated generalized (CFA) wear values were developed to help expand the information base related to resin composite materials. Dental manufacturers, as well as clinicians, are in need of information to assess the wear characteristics of resin composite materials. Linear regression was used to provide predicted wear rates for the five materials evaluated in this study (Table 6). The regression lines (Figures 6 and 7) for both wear depth and volume loss show three materials (EC, SP, and Z2) clustered in the middle of the graph. The Z1 line exhibits the lowest wear rate for depth (Figure 6) and volume loss (Figure 7), and the line for EX reveals the greatest wear rate for depth and volume loss. It should be noted that the regression lines do not converge on the origin of the graph. This is because each material initially loses a small but different amount of material. This can cause confusion if wear values are examined and compared instead of wear rates. For example, EC has initial wear that is greater than SP (Tables 4–5) but a lower wear rate (Table 6). Over a long time period, EC would presumably perform better. While wear is just one parameter for consideration in the selection of a restorative material, the predicted rates for wear and volume loss should provide valuable information for both resin composite developers and clinicians.

CONCLUSIONS

Wear simulation was used to develop relative wear rates of five resin composite materials. The results demonstrated significant differences ($p<0.05$) among materials and the number of cycles used. Simulated wear in the laboratory using a benchmark material, with good clinical and simulated wear performance, may provide an avenue for predicting the clinical performance of resin composite materials.

Conflict of Interest Declaration

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

(Accepted 22 May 2012)

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Effect of Resin-Modified Glass Ionomer Containing Bioactive Glass on the Flexural Strength and Morphology of Demineralized Dentin

M Khoroushi • S-M Mousavinasab • F Keshani • Shirin Hashemi

Clinical Relevance:

Flexural strength of the human dentin decreases after it is demineralized in vitro. This in vitro study demonstrates that resin-modified glass ionomer (RMGI) containing bioactive glass (BAG) can compensate for this loss of strength. RMGI without BAG does not restore the strength of such demineralized dentin.

http://dx.doi.org/10.2341/11-325-L

A Five-Year Clinical Evaluation of Direct Nanofilled and Indirect Composite Resin Restorations in Posterior Teeth

AR Cetin • N Unlu • N Cobanoglu

Clinical Relevance:

Under clinical conditions, posterior direct nanofilled composites and indirect composite inlay systems have the potential to present a high success rate and were clinically satisfactory at five years postplacement.

http://dx.doi.irg/10.2341/12-160-C

Effect of Using Silorane-based Resin Composite for Restoring Conservative Cavities on the Changes in Cuspal Deflection

NM Shabayek • FM Hassan • EH Mobarak

Clinical Relevance:

After five minutes of curing, the change in the organic matrix of the resin composite using silorane has a positive effect on controlling the cumulative cuspal deflection.

http://dx.doi.org/10.2341/12-035-L

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