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Clinical Techniques/Case Report

Esthetic Rehabilitation of a Patient With Severe Oligodontia

EG Reston • J Bervian • PF Kramer
MH Spiguel • SH Ferreira • LQ Closs

SUMMARY

Oligodontia, or the congenital absence of teeth, can occur in isolation or as part of a syndrome. This study describes a case of isolated oligodontia associated with conical teeth and large diastemas in the anterior region. The patient was treated using direct composite resin restorations. Recent improvements in adhesive restorative materials allow practitioners to offer this low-cost, conservative esthetic treatment, which has the advantage of preserving sound dental structures, to patients with missing teeth.

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PURPOSE

Oligodontia is defined as the absence of six or more permanent teeth due to hypodevelopment of tooth buds. It may be caused by different gene mutations or polymorphisms or may develop as a manifestation of malformative syndromes. Oligodontia is one of the most prevalent somatic aberrations involving tooth development in the general population.[1-3]

Early diagnosis and management of patients with oligodontia is important to avoid compromising such functions as mastication, phonation, and functional occlusion.[2] Moreover, according to some reports in the literature, patients with oligodontia, especially those with a large number of missing teeth, may develop psychosocial problems related to their facial esthetics.[4-6]

Dental rehabilitation usually involves a multidisciplinary approach. Recent advancements in adhesive restorative materials have allowed practitioners to provide a low-cost, simple procedure for such patients, while preserving sound dental structures and achieving great functional benefits and high rates of patient satisfaction.[7]

The present article describes the case of a patient with congenital absence of several permanent teeth and the esthetic treatment provided through direct composite resin restorations.

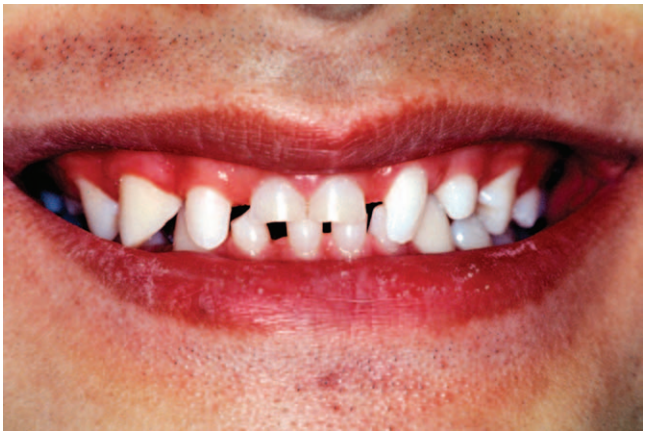

Figure 1. *Clinical appearance at baseline.*

DESCRIPTION OF TECHNIQUE

A 17-year-old male patient visited the Pediatric Dental Clinic with a chief complaint related to the esthetics of his teeth. He had large diastemas in the anterior region and tooth crowns that were not compatible in size with the dental arches (Figures 1 and 2).

The patient's health history data did not suggest the presence of any serious disease or systemic condition. Intraoral examination revealed several teeth missing (Figure 3). The deciduous teeth presented no alterations in size, shape, or structure. Permanent dentition was normal, except for the maxillary lateral conical incisors.

Radiographic examination revealed that the following teeth were missing: maxillary and mandibular central incisors (11, 21, 31, and 41), lateral mandibular incisors (32 and 42), maxillary second premolars (15 and 25), mandibular left second premolar (35), mandibular second molars (37 and 47), maxillary left canine (23), and maxillary and

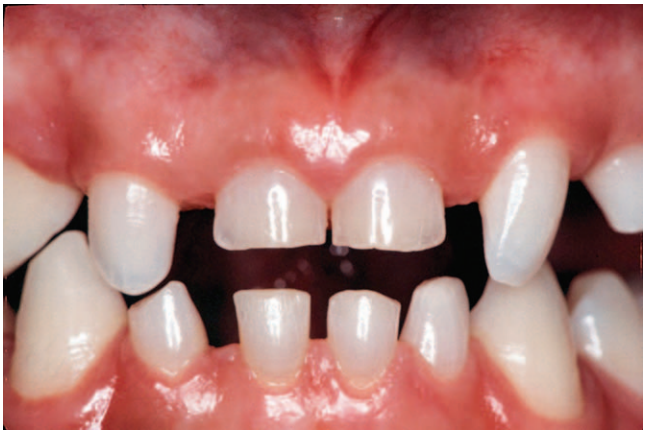

Figure 2. *Pretreatment clinical appearance.*

17 16 55 14 13 12 51	61 22 63 24 65 26 27
46 45 44 43 82 81	71 72 33 34 75 36

Figure 3. *Patient's odontogram.*

mandibular third molars (18, 28, 38, and 48) (Figures 4 and 5).

Based on the patient's history and clinical and radiographic findings, a diagnosis of isolated non-syndromic oligodontia was established. The treatment plan consisted of remodeling the crowns of anterior teeth with composite resin. The patient was given details of the limitations of the material to be used, particularly with regard to longevity and color maintenance.

The materials used for restorative treatment were a total-etch self-priming agent and a nanoreinforced resin. Treatment followed a sequence established in advance. First, prophylaxis with pumice and water was performed using Robinson brushes to remove debris from the dental structure. Occlusion was assessed and shade was determined immediately after prophylaxis. A diamond bur of medium grit was used to produce a wider area of rough surface for adhesive application. The operating field was then topically and locally anesthetized, and procedures were conducted under total rubber-dam isolation. Enamel acid etching (37% phosphoric acid for 15 seconds), thorough rinsing for 30 seconds, drying, application of the adhesive system, and polymerization were carried out according to manufacturer's instructions.

Deciduous teeth are known to have a different chemical composition from permanent teeth, so even though 15 seconds of enamel acid etching is usually enough, longer times can be found in the literature. This is usually because of the absence of prisms in the outer enamel layer, which is considered an obstacle to adequate penetration of adhesive restorative materials and could have a negative impact on esthetic-functional restorations, such as the one here described.[8-10]

In our patient, a chisel was used to remove the primless layer and thus improve acid etching results

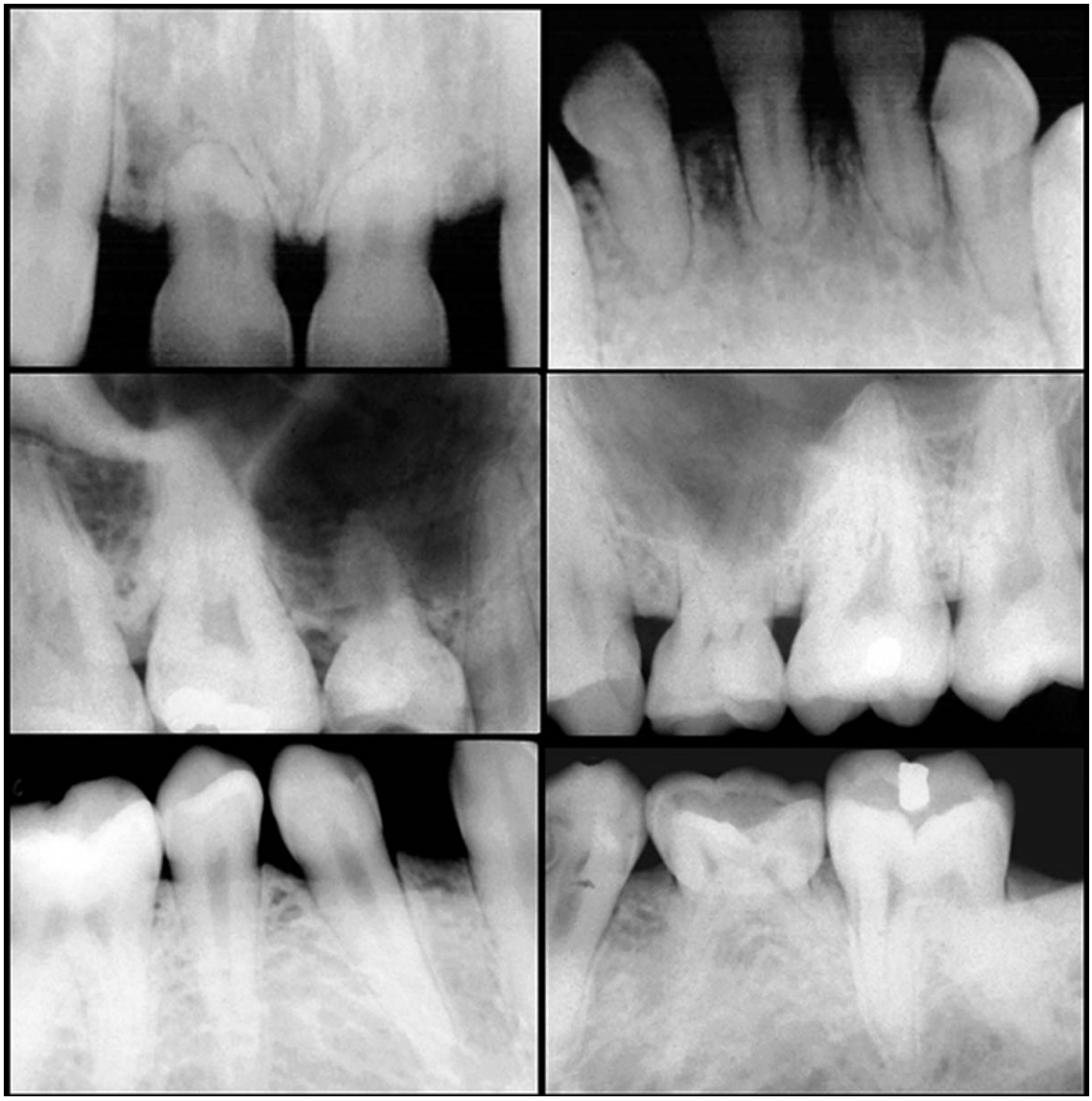

Figure 4. *Pretreatment radiograph.*

on the smooth surfaces of deciduous teeth.[11,12] Moreover, according to previous electron microscopy studies, 35% phosphoric acid produces acid-etch types I and II in Silverstone's classification after different application times, which justifies the use of 37% phosphoric acid for 15 seconds in our study.[8,13]

Because of the spaces between teeth, most of the restoration process was conducted freehand. Tooth shape was carefully contoured using flat dental brushes, which helped obtain smooth surfaces and a harmonic transition between the restorative material and natural tooth structure. Whenever necessary, dead soft matrices were used before resin application for better finishing in the cervical regions. Small layers of resin were placed against the matrices and light-cured, creating a buildup for future increments. As increments were light-cured, respecting the maximum limit of 2-mm-thick layers, they served as the basis for additional layers and shades, all applied freehand, until the buccal surface of the teeth was totally veneered. Each layer was light-cured for 40 seconds using a light-emitting diode device previously calibrated to 900 mW/cm^2; the light tip was held as close to the composite as possible without contact. After final polymerization, finishing and polishing were conducted using 12-bladed burs, finishing disks, aluminum oxide points, and a diamond paste in association with felt disks.

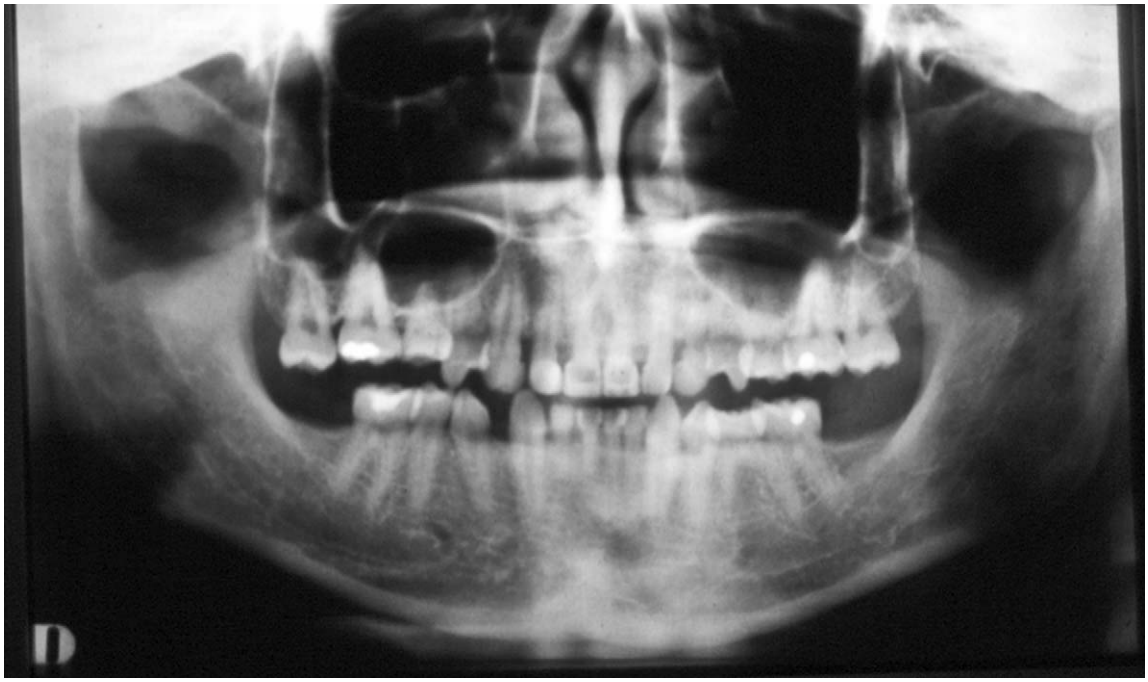

Figure 5. *Panoramic radiograph.*

Final occlusion was adjusted using high-speed diamond burs under water cooling and focused on centric relation as well as anterior and canine guidance, so as to restore the patient's bite and protect both natural tooth structure and restorations.

Treatment results were considered esthetically pleasing by the patient and his family members, both immediately after treatment completion (Figure 6) and six months later (Figures 7 and 8). The patient was scheduled for regular preventive maintenance visits so as to keep an adequate clinical and radiographic follow-up.

LIST OF MATERIALS USED

- 35% phosphoric acid gel (3M ESPE Dental Products, St Paul, MN, USA)
- Scotch Bond Multipurpose adhesive system (3M ESPE Dental Products)
- Filtek Z350, shades P, A2, A3, and B2 (3M ESPE Dental Products)
- Sof-Lex Pop-On polishing discs (3M ESPE Dental Products)
- 12-bladed burs (Sybron Kerr, Orange, CA, USA)
- Aluminum oxide points (Enhance, Dentsply, Konstanz, Germany)
- Diamond paste (Diamond R, FGM, Joinville, Brazil)
- Felt disks (Diamond Flex, FGM)
- Flat dental brushes (X, Hot Spot Design, Curitiba, Brazil)

POTENTIAL PROBLEMS

The ideal treatment approach in cases of oligodontia should take into consideration the esthetic appearance of anterior teeth and provide posterior indirect full-coverage restorations with ceramics in order to restore the vertical dimension of occlusion. However, the hardness of ceramic materials may impose major stress on periodontal tissues, potentially leading to accelerated root resorption in deciduous teeth. Moreover, in our patient, the long time needed for

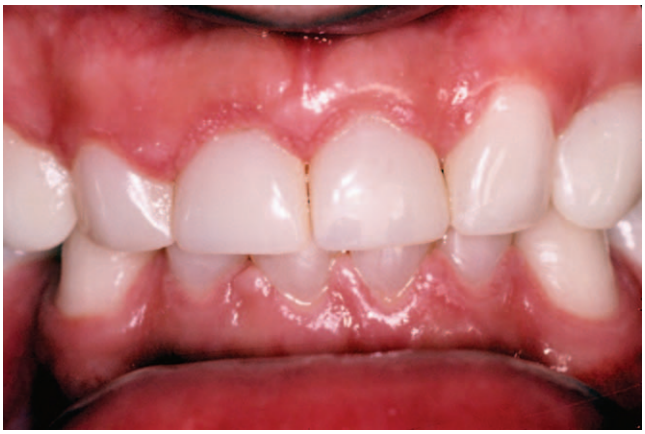

Figure 6. *Final clinical appearance.*

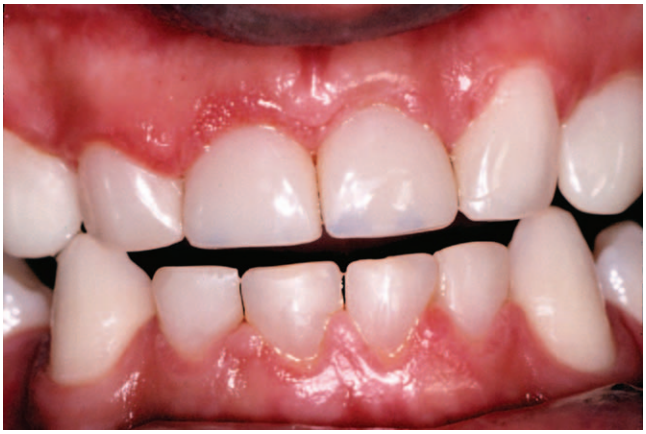

Figure 7. *Clinical appearance six months after treatment completion.*

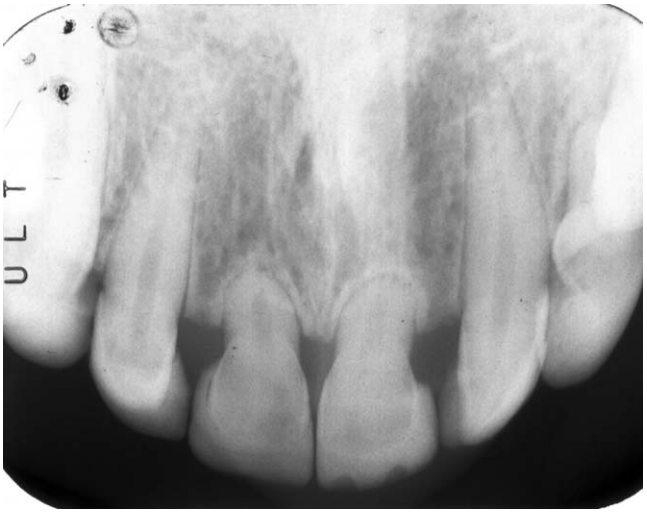

Figure 8. *Radiographic appearance six months after treatment completion.*

a full mouth reconstruction was not acceptable, as he was eager for esthetic improvements. Therefore, as an alternative approach, and with the patient's consent, we decided to remodel the crowns of anterior teeth only, using composite resin and maintaining the original vertical dimension of occlusion.

SUMMARY OF ADVANTAGES AND DISADVANTAGES

According to the literature, oligodontia may present alone or in association with a syndrome.[3,14,15] When associated with syndromes, in addition to a large number of missing teeth, the patient usually presents other abnormalities of ectodermal origin, affecting such structures as the skin, hair, and nails.[15,16] In the present case, the patient presented with congenital absence of several permanent teeth but no involvement of other ectodermal structures. In addition, his medical history did not reveal any previous diagnosis of serious diseases or systemic conditions. As a result, a clinical diagnosis of isolated nonsyndromic oligodontia was established. Previous studies by Stimson and others[14] and Brook[17] have suggested a genetic basis for this abnormality, even though its manifestation may be modified by environmental factors.

Stimson and others[14] emphasized the importance of being aware of the psychological status of patients with congenitally missing teeth. The treatment strategy adopted in this case aimed to preserve the deciduous teeth still present in the oral cavity while at the same time providing functional and esthetic rehabilitation to the dental structures.

The recent improvements in adhesive materials have motivated several authors to recommend similar treatment approaches.[4,14,18,19] According to Erridge,[4] for example, reconstruction of retained deciduous and conical teeth with composite resin is a simple treatment, associated with reduced chair time—a very important aspect of the management of younger patients. According to Prati,[7] the evolution of adhesive restorative materials has made it possible to offer more conservative functional and esthetic rehabilitation therapies.

In summary, the treatment strategy described in this study offers a simple, conservative solution for a major esthetic problem that is often associated with low self-esteem and social withdrawal. The current state of the art of adhesive restorative materials offers a low-cost, simple treatment option for patients with missing teeth while preserving sound dental structures, characteristics that usually attract and please patients. Finally, treatment is effective and repairable in case of fracture. Regular preventive recalls are important for maintaining dental restorations and assessing the stability of retained deciduous teeth.

Conflict of Interest

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

(Accepted 15 July 2013)

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Use of Cast Post-and-cores for the Alteration of Crown Position for the Esthetically Compromised Patient

PA Hansen • J Veurink • M Ambrososio

Clinical Relevance

The use of a cast post-and-core will allow the angle of insertion and the position of the core for a new crown or retainer for a fixed partial denture to be altered. This is done in order to obtain optimal esthetics.

SUMMARY

The authors describe the use of cast post-and-cores, which allow a change in position of the clinical crown to permit the fabrication of two fixed partial dentures. The two fixed partial dentures change the clinical appearance of the patient.

INTRODUCTION

Post-and-cores have traditionally been used to restore teeth when there is not enough tooth structure to retain a crown.[1] Many types of post-and-core systems have been documented in the literature over the years, with an excellent long-term prognosis.[2,3] Hayashi et al[4] reported that when cast-metal post-core systems are subjected to oblique and vertical load, it takes greater loads to fracture teeth. Cast post-and-cores contain one solid material and do not separate under stress or function.[5,6] Cast metal posts are best used for those teeth with canals that are elliptical or irregular in diameter.[7] Excessive tooth reduction does not have to be accomplished in order to accommodate a cast post; it will fill all shapes of canals. A precast post, on the other hand, may require excessive tooth reduction for the canal space, and if the canal is elliptical, the precast post may not fill the post space well.[7] With laboratory support, a cast post can be ready in 30-40 minutes from time of post pattern fabrication.[8-10] Keeping as much tooth structure as possible will aid in the longevity of the endodontically treated tooth.[11-15]

When a tooth is out of position, it cannot be prepared for a veneer or crown without causing possible pulp damage, but a prophylactic root canal and post can be completed. This will allow proper positioning of the new crown for esthetics and retain enough tooth core to support the new crown. Bada[16] stated that there is a limit to how much a precast post can bend and how much buildup material the precast post can retain. Bada also stated that a cast post-and-core is needed in clinical situations where it

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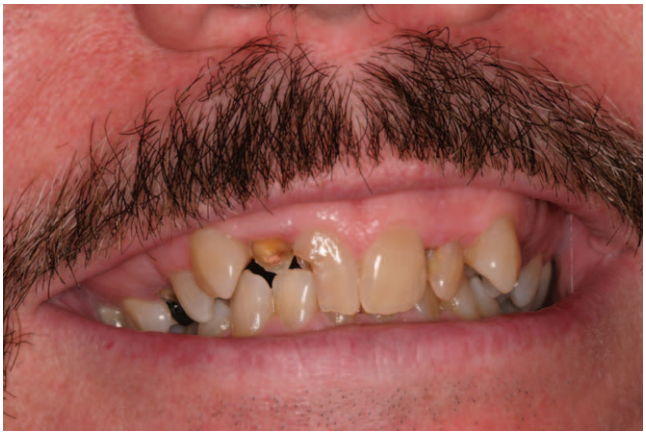

Figure 1. *Preoperative smile.*

is necessary to alter the angle of the core in relation to the tooth.[16]

This article will address the clinical situation where tooth position is not optimal for the placement of fixed prosthodontic work.

CLINICAL TECHNIQUE

A 56-year-old man came into the University of Nebraska College of Dentistry clinic requesting dentures to correct his irregular smile (Figures 1 and 2). A comprehensive dental exam was done, including radiographs and periodontal probing. All teeth were restorable except tooth no. 7, due to caries, and tooth no. 10, which had a vertical root fracture. Caries was also present in tooth no. 9. The patient's chief complaint was that his smile was problematic in the business world, and he wanted a better appearance. He had no missing teeth except

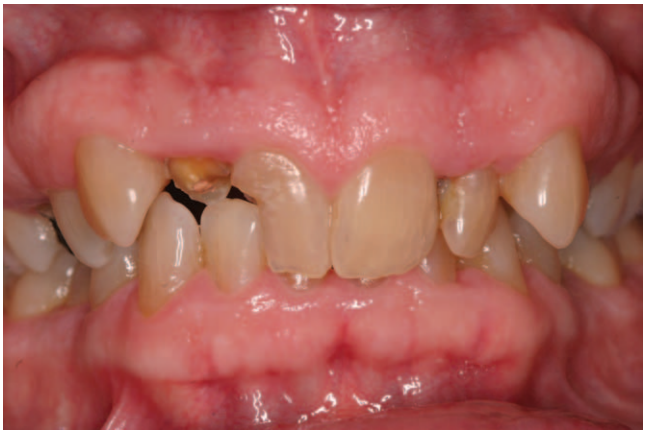

Figure 2. *Close-up of fractured and malpositioned teeth. Teeth nos. 7 and 10 are nonrestorable due to caries and vertical root fracture.*

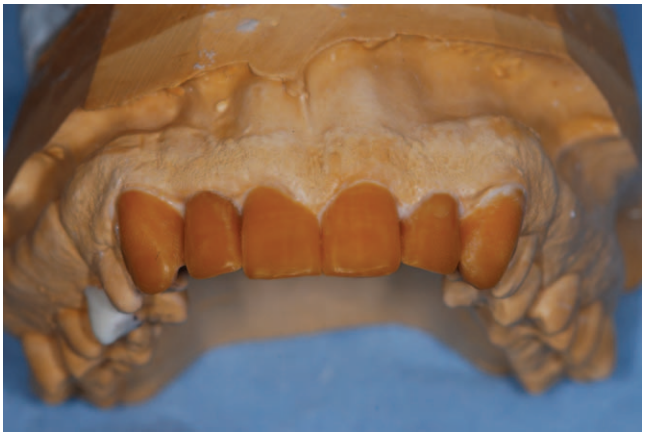

Figure 3. *Diagnostic wax-up completed on mounted casts. Ideal contours were developed for esthetics and function. This wax-up was duplicated as a solid stone cast, and a vacuum-formed matrix was made.*

nos. 1, 16, 17, and 32. He had no medical contraindications to dental treatment.

A referral was made to orthodontics, but the patient refused treatment due to the length of time to correct the esthetics. Diagnostic casts were made and mounted in a semiadjustable articulator. A diagnostic wax-up was completed to determine the optimum position of the retainers and pontics for maximal esthetics for the fixed partial dentures. It was determined by the wax-up that any attempt to prepare the teeth for the retainers and create an esthetic smile would result in pulpal exposure. A proposal was made to the patient to perform prophylactic endodontic treatment, to place cast post-and-cores, and to restore with zirconia-based fixed partial dentures (Figure 3). The wax-up was

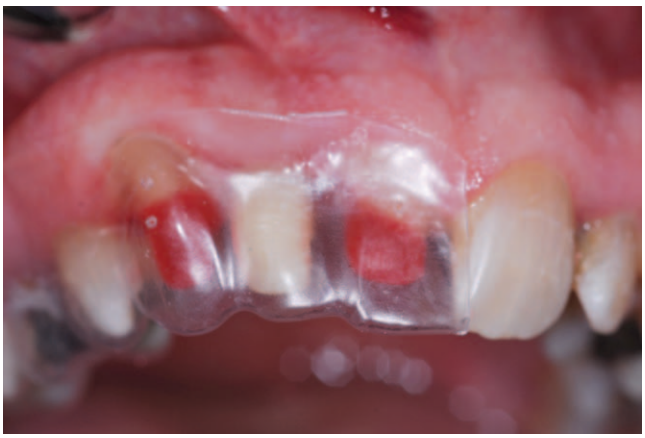

Figure 4. *The vacuum-formed matrix was placed over the endodontically treated teeth to ensure the proper position of the direct post-and-cores.*

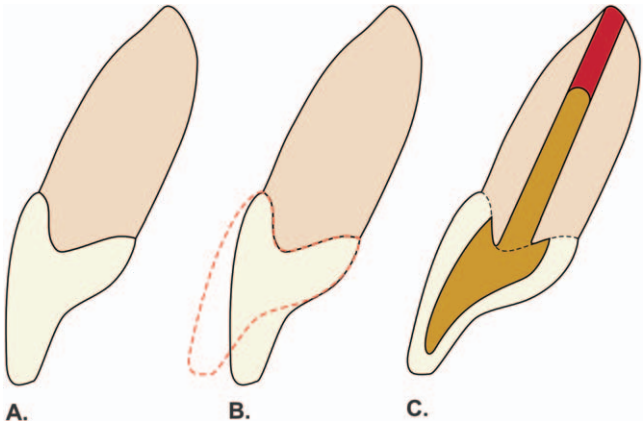

Figure 5. *The use of a cast post and core will allow alteration of the position of the crown of the tooth. A prefabricated post would not be able to accommodate the angle change.*

duplicated and a stone cast made. A vacuum-formed matrix was fabricated to duplicate the desired position of the teeth (Figure 4).

After prophylactic endodontic treatment for teeth nos. 6, 8, 9, and 11, the patient returned to the restorative clinic for tooth preparation and fabrication of the cast post.

Teeth nos. 6, 8, 9, and 11 were prepared for post-and-cores. Ideal crown preps were made using the vacuum-formed matrix to ensure proper position. Every effort was made to conserve tooth structure for the best long-term prognosis for the teeth. Once the tooth preparation was complete and canal space was made, Duralay resin (Reliance Mfg Co, Worth, IL, USA) was used to fabricate direct post-and-cores. The use of the vacuum-formed matrix allowed the preparation of the cores to mimic the ideal position of the prepared tooth for the new fixed partial dentures.

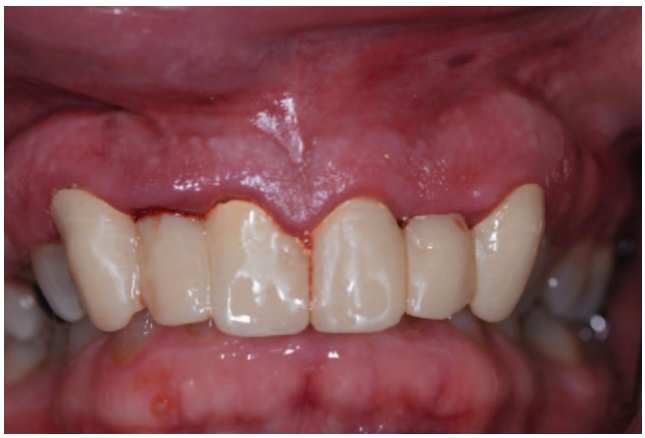

Figure 6. *Immediately placed indirect provisional. A provisional is placed at the time of extractions.*

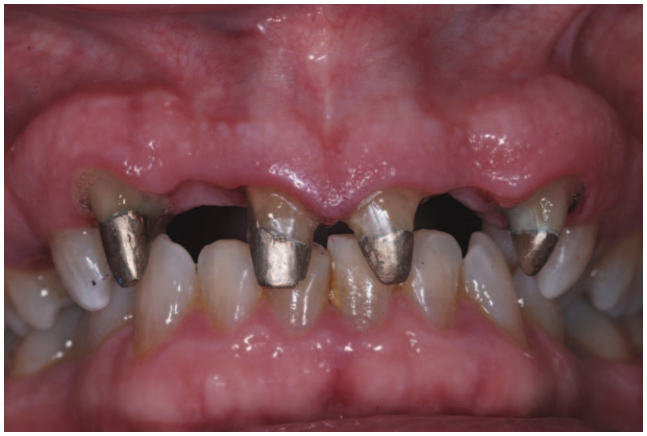

Figure 7. *Intermediate healing 3 months after extractions and the placement of the post-and-cores.*

The use of the cast post-and-core was done due to the significant change of angle from the root structure. A prefabricated post would not have been able to make the angulation change (Figure 5). After the cementation of the cast post-and-cores, an impression was made of the maxillary arch. A cast was made from the impression, teeth nos. 7 and 10 were cut off the cast, and indirect provisional restorations were fabricated (Jet Resin, Lang Manufacturing Co, Wheeling, IL, USA).[17] Teeth nos. 7 and 10 were then extracted and the new indirect provisionals cemented into place (Figure 6). The tissues were allowed to heal for six months with the provisional restorations in place (Figure 7).

The provisional restorations allowed the clinician to predict the outcome of the final restorations. Lingual contours could be altered to ensure there

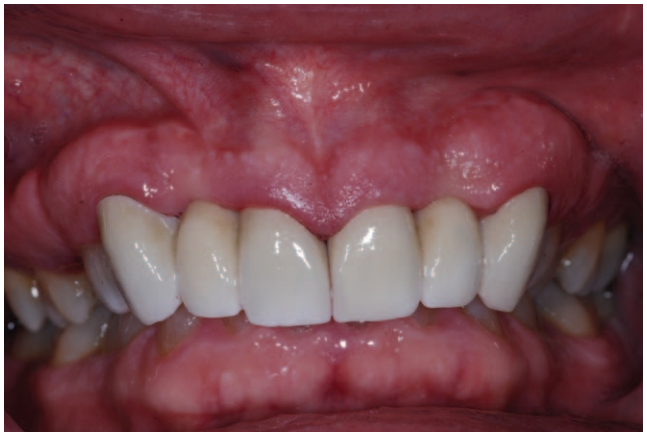

Figure 8. *Close-up of the new three-unit fixed partial dentures. The restorations are made of a zirconia substructure, with feldspathic porcelain layered on the substructure. An extra layer of opaque was placed on the substructure to help block any dark tooth structure.*

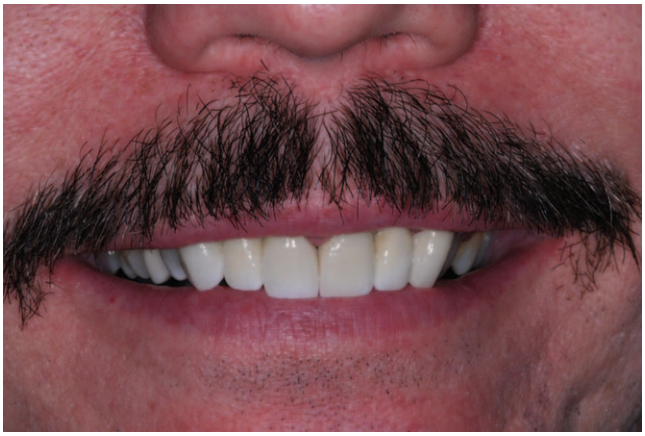

Figure 9. *The new smile.*

were no functional problems for all mandibular movements and to establish anterior guidance. The position of the incisal edges and the labial contour of the provisional could be altered if necessary. After healing, an impression was made of the provisional restorations to provide the dental laboratory technician a reference for the position of the new restorations. A custom incisal-guide table was made to duplicate the lingual contour of the provisional restorations.

Following healing, the tooth preparations were modified to the free gingival margin of the healed tissue. Tissue margins had moved in an apical direction after healing. Final impressions were made and sent to the dental laboratory. Due to the dark color of the cast post and core, a layer of ceramic opaque was placed on the zirconia framework to mask the color of the teeth. Feldspathic porcelain was layered on the zirconia framework and the new fixed partial dentures completed. The fixed partial dentures were tried in and cemented (Figures 8 and 9). The patient was extremely pleased with the final result.

This paper has demonstrated the use of cast post-and-cores to modify the angle of the prepared tooth to allow for acceptable esthetic restorations.

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

In Situ Surface Biodegradation of Restorative Materials

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Clinical Relevance

Understanding the surface characteristics of restorative materials submitted to *in situ* biodegradation is an essential issue toward the assessment of the clinical longevity of restorative materials.

SUMMARY

This study aimed to evaluate the surface characteristics of restorative materials (roughness, hardness, chemical changes by energy-dispersive spectroscopy [EDX], and scanning electron microscopy [SEM]) submitted to *in situ* biodegradation. Fifteen discs of each material (IPS e.max [EM], Filtek Supreme [FS], Vitremer [VI], Ketac Molar Easymix [KM], and Amalgam GS-80 [AM]) were fabricated in a metallic mold (4.0 mm × 1.5 mm). Roughness, hardness, SEM, and EDX were then evaluated. Fifteen healthy volunteers used a palatal device containing one disc of each restorative material for seven days. After the biodegradation, the roughness, hardness, SEM, and EDX were once again evaluated. Data obtained from the roughness and hardness evaluations were submitted to Kolmogorov-Smirnov and Tukey-Kramer tests ($p<0.05$). All esthetic restorative materials showed a significant increase in the roughness after biodegradation. Before biodegradation, significant differences in the hardness among the materials were seen: EM>AM>FS>KM>VI. After biodegradation, the hardness was significantly altered among the materials studied: EM>AM>FS=KM>VI, along with a significant increase in the hardness for AM, KM, and VI. SEM images indicated degradation on the surface of all materials, showing porosities, cracks, and roughness. Furthermore, after biodegradation, FS showed the presence of Cl, K, and Ca on the surface, while F was not present on the VI and KM surfaces. EM and AM did not have alterations in their chemical composition after biodegradation. It was concluded that the dental biofilm accumulation *in situ* on different restorative materials is a material-dependent parameter. Overall, all materials changed after biodegradation: esthetic restorative materials showed increased roughness, confirmed by

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SEM, and the ionomer materials and silver amalgam showed a significantly higher hardness. Finally, the initial chemical composition of the composite resin and ionomer materials evaluated was significantly altered by the action of the biofilm *in situ*.

INTRODUCTION

Biofilms form not only on dental hard and soft tissues, but also on restorative biomaterial surfaces used in the oral cavity; in addition, biofilms are the major cause of caries and periodontal diseases.[1] However, the adhesion and aggregation of microorganisms are different among materials with different compositions and surface properties .[2,3]For most restorative materials, acid metabolites produced by cariogenic biofilm can cause surface damage such as corrosion, softening, and a roughness increase, which is known as biodegradation.[4,5] This is a complex process and includes disintegration and dissolution in saliva and other types of chemical/physical degradation, such as wear and erosion caused by food, chewing, and bacterial activity.[6] Thus, in order to present a satisfactory performance, the priority of restorative materials should be the resistance to that adverse condition.

There is limited knowledge about the influence of cariogenic biofilms on the surface of restorative materials. Long-term *in vitro* studies show an increase in the roughness and morphology damages for resin composites, polyacid-modified composites,[7] and ionomeric materials.[5] An *in situ* study found a lower hardness for Vitremer after 14 days of biodegradation.[8] Metallic materials, such as gold and amalgam, were observed to have thick biofilms covering their surfaces *in vivo*, though their cells were found to be barely viable.[9] This probably caused less deterioration of the materials' properties. Conversely, biofilms on ceramic biomaterial, which is considered the most inert of all dental materials used for restorations,[10] were found to be relatively thin but highly viable (from 34% to 86%).[9] All of these findings reflect the complex environment and biome observed in oral conditions.[8]

It is known that no *in vitro* test is capable of reproducing the complex biodegradation process. Consequently, many studies choose lactic acid as a representative of dental biofilm since this is the most important metabolic product from *Streptococcus mutans* in the biofilm exposed to sucrose.[11] Nevertheless, it is possible that the concentration, pH, and effective contact of this acid solution *in vitro* would differ from oral conditions, thus overestimating the degradation effects. In this context, the *in situ* model is a recognized experimental design that has been successfully used to evaluate the formation of cariogenic dental biofilm.[12] There are few studies on the influence of biofilm on the surface characteristics of restorative materials *in situ*.[8,13,14] Therefore, the aim of this study was to evaluate the effects of the *in situ* biodegradation on the surface characteristics of restorative materials. The hypothesis tested was that restorative materials subjected to seven days of biofilm interaction have significant modifications in regard to their roughness, hardness, and microstructure.

METHODS AND MATERIALS

Specimen Preparation and Storage Groups

Fifteen specimens of each restorative material (described in Table 1) were fabricated according to

Table 1: *Materials Used in This Study*

Materials	Classification	Contents (Manufacturer Information)
IPS e.max (Ivoclar Vivadent, Schaan, Liechtenstein)	Glass ceramic	Powder: 97% SiO_2, Al_2O_3, P_2O_5, K_2O, Na_2O, CaO, F, 3% TiO_2, and pigments
		Liquid: water, alcohol, chloride
Filtek Supreme (3M ESPE, St Paul, MN, USA)	Composite resin	Bis-GMA, Bis-EMA, UDMA, TEGDMA Zirconia/silica cluster filler and a nonagglomerated silica filler
Vitremer (3M ESPE, St Paul, MN, USA)	Resin-modified glass ionomer	Powder: fluoroaluminosilicate glass; redox system
		Liquid: aqueous solution of a modified polyalkenoic acid, HEMA
Ketac Molar Easymix (3M ESPE, St Paul, MN, USA)	Glass ionomer cement	Powder: fluorosilicate glass, strontium and lantanium
		Liquid: polycarbonic and tartaric acids and water
Amalgam GS-80 (SDI, Victoria, Australia)	Silver amalgam	Powder: 40% Ag, 31.3% Sn, 28.7% Cu
		Liquid: mercury

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

the manufacturer's instructions, by using metal rings (4 mm diameter; 1.5 mm depth), at a temperature of 23°C ± 1°C, and a relative humidity of 50% ± 5%.

The specimens, with the exception of the ceramics, were covered with an acetate strip (Probem Ltda, Catanduva, SP, Brazil) and pressed onto a glass slide to compact the material. Filtek Supreme (FS) and Vitremer (VI) were photoactivated for 40 seconds each at the upper and lower surfaces of the matrix by a curing light (Elipar Trilight, 3M ESPE, St Paul, MN, USA), with an intensity of up to 750 mW/cm^2, and checked by a light-curing meter (Hilux Dental Curing Light Meter, Benliglu Dental Inc, Turkey). Ketac Molar Easymix (KM) and Amalgam GS-80 (AM) were allowed to set at room temperature for 15 minutes. After the setting reactions were completed, the ionomeric specimens were superficially protected with petroleum jelly. For IPS e.max (EM), specimens were fabricated in a prosthetic laboratory by using the pressing process in an oven (Programat P500, Ivoclar Vivadent, Schaan, Liechtenstein), which simulates the clinical reality.

All of the restorative materials were stored at 37°C and 100% relative humidity for 24 hours. Afterwards, each specimen surface was polished according to the manufacturer's instructions. For FS, VI, and KM, Sof-Lex discs (3M ESPE, St Paul, MN, USA) were used. AM was polished with a polishing kit (KG Sorensen, Cotia, SP, Brazil). For the finishing of EM, discs were ground flat with an aluminum oxide jet (50 μm, Bio-Art, São Carlos, SP, Brazil) followed by a diamond drill (4138F, KG Sorensen, Barueri, SP, Brazil); the polishing of these specimens was performed with a rubber tip (KG Sorensen), and specimens were then washed in an ultrasonic bath (UNIQUE, São Paulo, SP, Brazil) and glazed.

Roughness Measurements

After the finishing and polishing procedures, all specimens were washed through sonication for 10 minutes, dried, and fitted to a roughness-measuring instrument (Surfcorder SE1700, Kosaka Corp, Tokyo, Japan). The roughness analysis was performed prior to the hardness assessment in order to avoid interference with their results. Moreover, each specimen was divided in the middle, with the left side being used for the roughness analysis and the right side for the hardness assessment. To record the roughness measurements, the needle moved at a constant speed of 0.5 mm/sec with a load of 0.7 mN. The cut-off value was set at 0.25 mm to maximize the filtration of the surface waviness. The measurement of roughness for each specimen was taken across the diameter over a standard length of 0.25 mm. The mean surface roughness values (μm) of the specimens were obtained from three successive in-line measurements from the center to the boundary of each disc at different angles (0°, 45°, and 90°). A calibration was done periodically to check the performance of the roughness-measuring instrument.

Hardness Measurements

Hardness tests were carried out with a hardness tester (Shimatzu, Tokyo, Japan) by using a Vickers indenter, with a load of 500 g for the ceramic and of 200 g for the composite resin, glass ionomer cements, and silver amalgam. All materials had a dwell time of 15 seconds. Five readings were taken for each specimen, which were then used to calculate the mean hardness. The mean hardness was calculated before and after the biodegradation.

Surface Morphology Assessment and Energy-Dispersive X-ray Analysis

Before and after the *in situ* biofilm experiment, three additional representative specimens of each group were rinsed, dried, and mounted on a holder using a double-sided adhesive carbon tape. Carbon was then sputtered on the specimens before the analysis. Energy-dispersive X-ray analysis (EDX) was performed before and after the biodegradation. The EDX measurements were calibrated by a certified engineer, using the standard samples of Cr_2O_3, titanium, silica, and $CaSiO_3$, as described by Statham.[15] Afterwards, these same specimens were examined with a JEOL scanning electron microscope (Model JSM 5600 LV, Tokyo, Japan), operating at a 1000× magnification.

Panelists and Ethical Aspects

Fifteen healthy adults participated in the study (ages 21-30 years). The volunteers were selected according to the following inclusion criteria: good general and oral health, normal salivary flow rate, absence of antibiotic use for two months before the experiment, absence of prosthesis or orthodontic devices, no signs of gingivitis or caries, and ability to comply with the study.[13] Visual oral examinations were carried out by an experienced dentist. All of the volunteers agreed to participate and signed an informed written consent form. The study design was approved by the local Ethics Committee (protocol 136/2009).

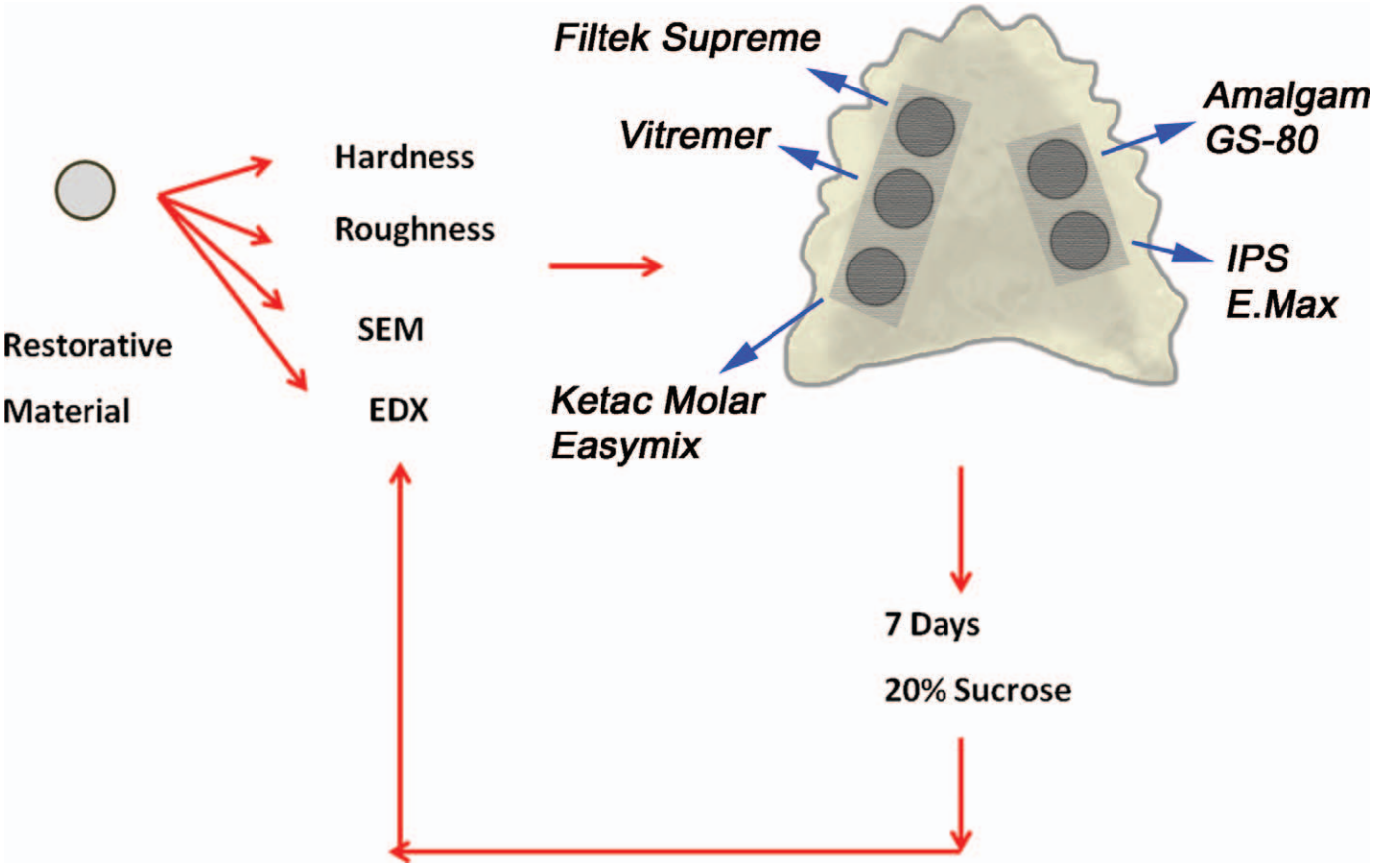

Figure 1. *Experimental design.*

In situ *Phase*

The volunteers' teeth were impressed with alginate (Jeltrate, Dentsply, Petrópolis, RJ, Brazil), and type III gypsum models were obtained. An individual intraoral acrylic resin palatal device, containing five 2.5-mm deep wells (one restorative material per well) was prepared for each volunteer,[9] as shown in Figure 1. A plastic mesh was fixed on two sides of the intraoral device, leaving a 1-mm space for the accumulation of the dental biofilm on the specimens. To assure their acceptability for the study, oral and written instructions of the *in situ* protocol were given to the volunteers before receiving the intraoral devices. There were no restrictions on the volunteers' diet. The only recommendation was to remove the device during meals and before ingesting any beverages or food, and to keep the oral devices moist in plastic boxes provided by the researchers. Volunteers were instructed to perform oral hygiene three times per day with a standardized fluoride dentifrice (1100 mg F/g as NaF). Only the palatal region of the device was extraorally brushed to avoid disturbing the biofilm. The cariogenic challenge was provided by the application of a 20% sucrose solution extraorally on the specimens (10×/day). The volunteers removed the devices from the mouth, excess saliva was cleaned with gauze, and one drop of the solution was dripped onto each specimen at 8:00, 9:30, 11:00, 12:30, 14:00, 15:30, 17:00, 18:30, 20:00, and 21:30 hours.[16] The sucrose was gently dried after 5 minutes and the device was reinserted into the mouth. After seven days, the specimens were carefully removed from the device and washed in an ultrasonic bath for 10 minutes; the final measurements for roughness, hardness, and surface morphology were then performed.

Statistical Analysis

The measurements were analyzed by using the Kolmogorov-Smirnov test at a 5% level of significance to assess the normality of the distribution. A methodology of mixed models for repeated measurements and Tukey-Kramer statistical tests at a 5% level of significance were used with a PROC MIXED SAS statistical software (Cary, North Carolina, USA).

Table 2: *Surface Roughness Means (SD), in μm**

Groups	Biodegradation	
	Baseline	7 Days
Filtek Supreme	0.34 (0.07)Bc	1.74 (1.51)Aab
Vitremer	0.62 (0.17)Bb	1.87 (0.99)Aab
Ketac Molar Easymix	0.57 (0.17)Bb	1.37 (0.83)Ab
Amalgam GS-80	1.70 (0.66)Aa	2.81 (1.13)Aa
IPS e.max	0.86 (0.45)Bb	2.40 (2.19)Aa

** Means followed by different letters (upper-case letters in each row and lower-case letters in each column) differ significantly (p≤0.05). Kolmogorov Smirnov and Tukey-Kramer statistical tests.*

Table 3: *Surface Hardness Means (SD), in VHN**

Groups	Biodegradation	
	Baseline	7 Days
Filtek Supreme	105.47 (2.09)Ac	101.27 (4.93)Ac
Vitremer	62.57 (6.00)Be	73.73 (7.25)Ad
Ketac Molar Easymix	81.59 (3.53)Bd	105.03 (5.95)Ac
Amalgam GS-80	129.45 (5.92)Bb	161.39 (27.13)Ab
IPS e.max	581.05 (37.24)Aa	577.69 (21.41)Aa

** Means followed by different letters (upper-case letters in each row and lower-case letters in each column) differ significantly (p≤0.05). Kolmogorov-Smirnov and Tukey-Kramer statistical tests.*

RESULTS

Tables 2 and 3 show the mean and standard deviations of the roughness and hardness, respectively, for each material before and after biodegradation, *in situ*.

All esthetic restorative materials studied showed a significant increase in the roughness after the biofilm/material interaction. Only AM showed no statistical difference between the periods analyzed. Before the biodegradation, AM presented the highest roughness, followed by VI, KM, and EM, with FS showing the lowest. However, after biodegradation, AM and EM had a higher roughness than KM, while FS and VI presented intermediate roughness and had no statistical difference with the other materials.

It was observed that before the biodegradation, the hardness was statistically different between the materials studied, with the following sequence: EM>AM>FS>KM>VI. VI, KM, and AM presented significant differences between the experimental periods, with higher values after the biodegradation period. However, FS and EM did not show significant differences between the periods. After biodegradation, the hardness was as follows: EM>AM>FS=KM>VI.

By EDX analysis, presented in Figure 2, the initial chemical composition of AM and EM was not altered by the action of the biofilm *in situ*. AM showed the presence of Hg, Sn, Ag, Si, and Cu, while EM showed Si, Al, K, and Na, among others. However, FS, VI, and KM did present alterations in their spectra. EDX results for FS revealed that before and after biodegradation, Si is present in the highest amount, followed by P and C. However, there was an adsorption of ions on the material surface, possibly originating from saliva. In VI and KM, Al, Si, and Ca were present in the highest amount before and after biodegradation; the quantity of F decreased after biodegradation. Furthermore, there was also the incorporation of ions such as Ca^{2+} and Cl^{-} on the surface of VI and KM.

The scanning electron micrographs in Figure 3 show details of the surface morphology of the studied materials. Materials are presented in rows and periods in columns. Regarding the resin-based materials (FS and VI), the polishing of the resin produced an irregular surface with the loss of the organic matrix and the exposure of the filler particles. Furthermore, the biodegradation produced an irregular surface coating, with the displacement of some filler in the organic matrix. KM also presented exposed fillers and cracks on the surface before the biofilm interaction and after the *in situ* experiment; the ionomeric material surface showed cracks and biodegraded areas with filler displacements, as shown by the arrows. For AM, a subtle corroded aspect after the surface degradation was observed, while EM showed an increased amount of surface cracks associated with an increase in the size of the nodules when compared with the specimen before biodegradation.

DISCUSSION

The success of restorative procedures depends on many factors, from treatment planning and a patient's adequacy, to clinical steps and subsequent preservation and maintenance of the restoration performed. Thus, it is important to carefully select a restorative material able to withstand the functional force and chemical environment of the oral cavity. Fundamentally, the factors known to cause surface damage to restorative materials include low pH due to cariogenic biofilm, consumption of acid drinks or foodstuffs, and the action of enzymes, all of which can soften the outermost layers and damage restorative materials.[5,8,17-21]

Most conditions of the oral cavity can be simulated by an *in situ* study, such as saliva properties (salivary flow, buffer capacity, clearance, mineral and protein

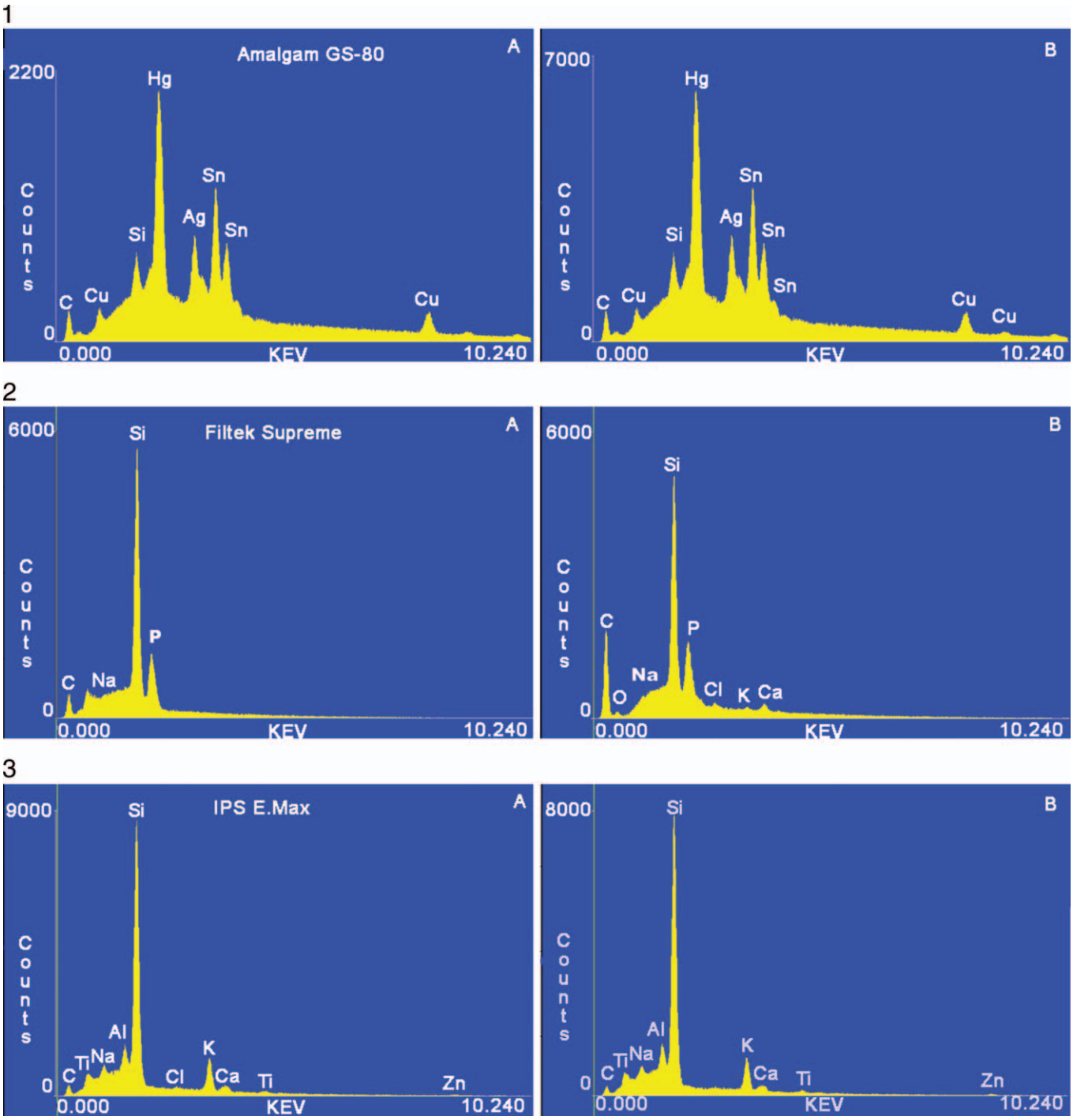

Figure 2. *EDX spectra of different restorative materials compared before (A) and after (B) biodegradation.*

content, and enzymes), biofilm accumulation (diversity of species, microorganism selection, succession, nutrient availability, and competition), temperature fluctuations, and aqueous environment, among others. However, little information is available regarding the surface degradation of restorative materials after the interaction with biofilms *in situ*. Among the available *in situ* studies, a focus was given on the evaluation of either the biofilm characteristics or the restorative material.[8,13,14] In this context, the present study allowed for the development of biofilm on different restorative materials using the *in situ* model

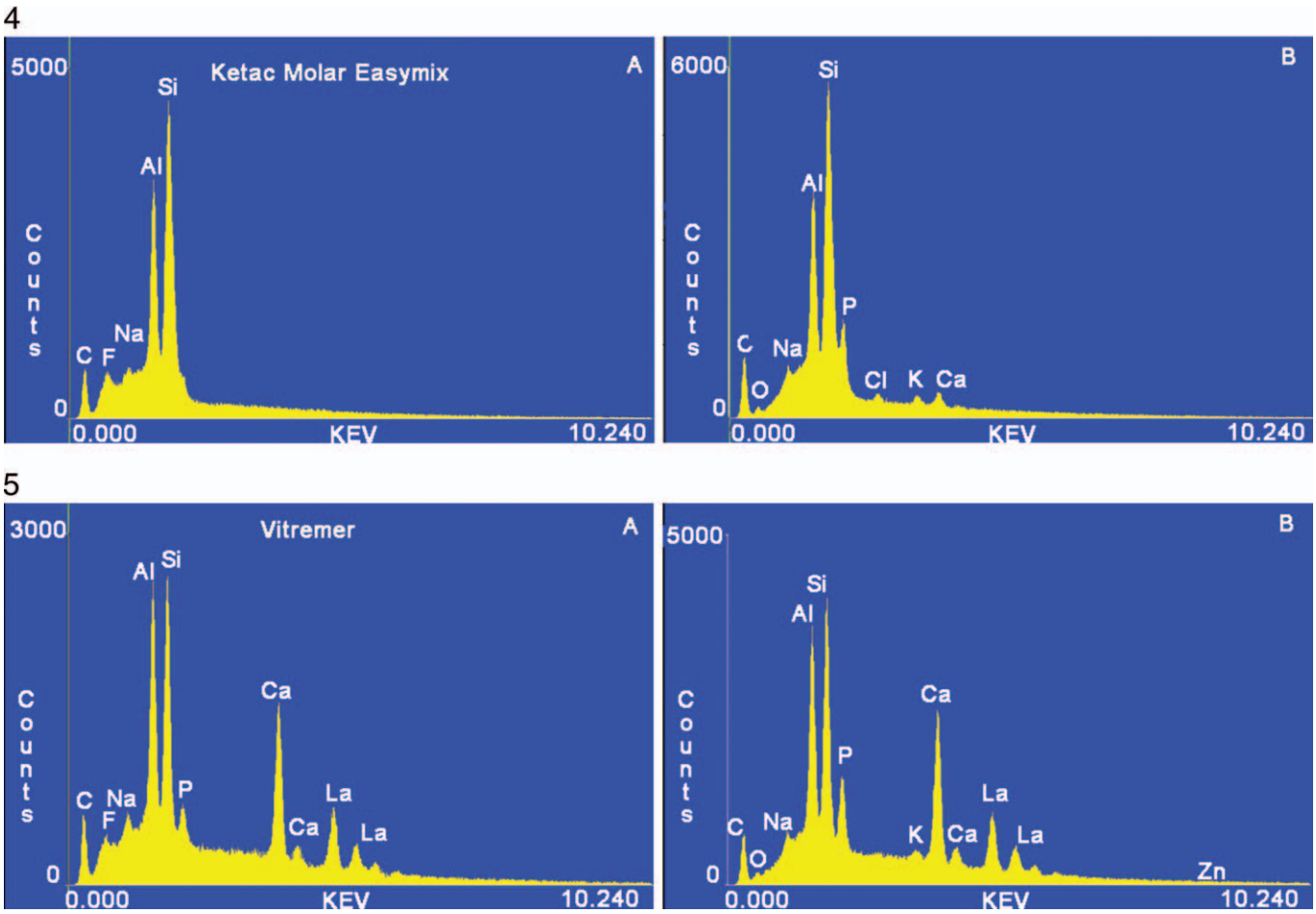

Figure 2. *Continued*

in order to analyze the consequences of this bio-interaction on the materials' surface, under frequent biodegradation.

Different groups of restorative materials (amalgam, composite resin, glass ionomers, and ceramic) were selected due to studies that suggested that the biofilm accumulation and biodegradation intensity are influenced by the surface upon which it is developing, which is directly related to the physical and chemical properties of the restorative materials.[8,14,22,23] Furthermore, the selected materials are representative of the different classes of restorative materials used in odontology, as well as of the classes established in materials science (ie, metals, polymers, ceramics, and composites).[24]

All materials were handled according to their manufacturer's recommendations, including photoactivation, setting time, and polishing procedures. The polishing procedure was performed since it improves the esthetic characteristics and the durability of the restoration, decreases the porosity of the surface, decreases the surface staining, and also improves its mechanical properties.[25] Furthermore, the polishing removes the organic matrix of different restorative materials and exposes the fillers particles, a fact that is confirmed by the scanning electron microscopy (SEM) images taken before biodegradation. These micrographs show that the polishing produced some scratches on the composites and amalgam surfaces, removing the matrix and exposing particle fillers on the direct restorative materials studied (Figure 3).

All esthetic materials in this study showed an increase in the roughness after the biofilm activity. The acid attack by bacterial metabolism can cause biodegradation through different ways for restorative materials. For Filtek Supreme, there is a release of TEGDMA and UDMA monomers from the resin matrix when it is in contact with salivary enzymes and bacterial acids.[26] During biodegradation, Vitremer releases HEMA, a highly hydrophilic cosolvent and the main component released from the

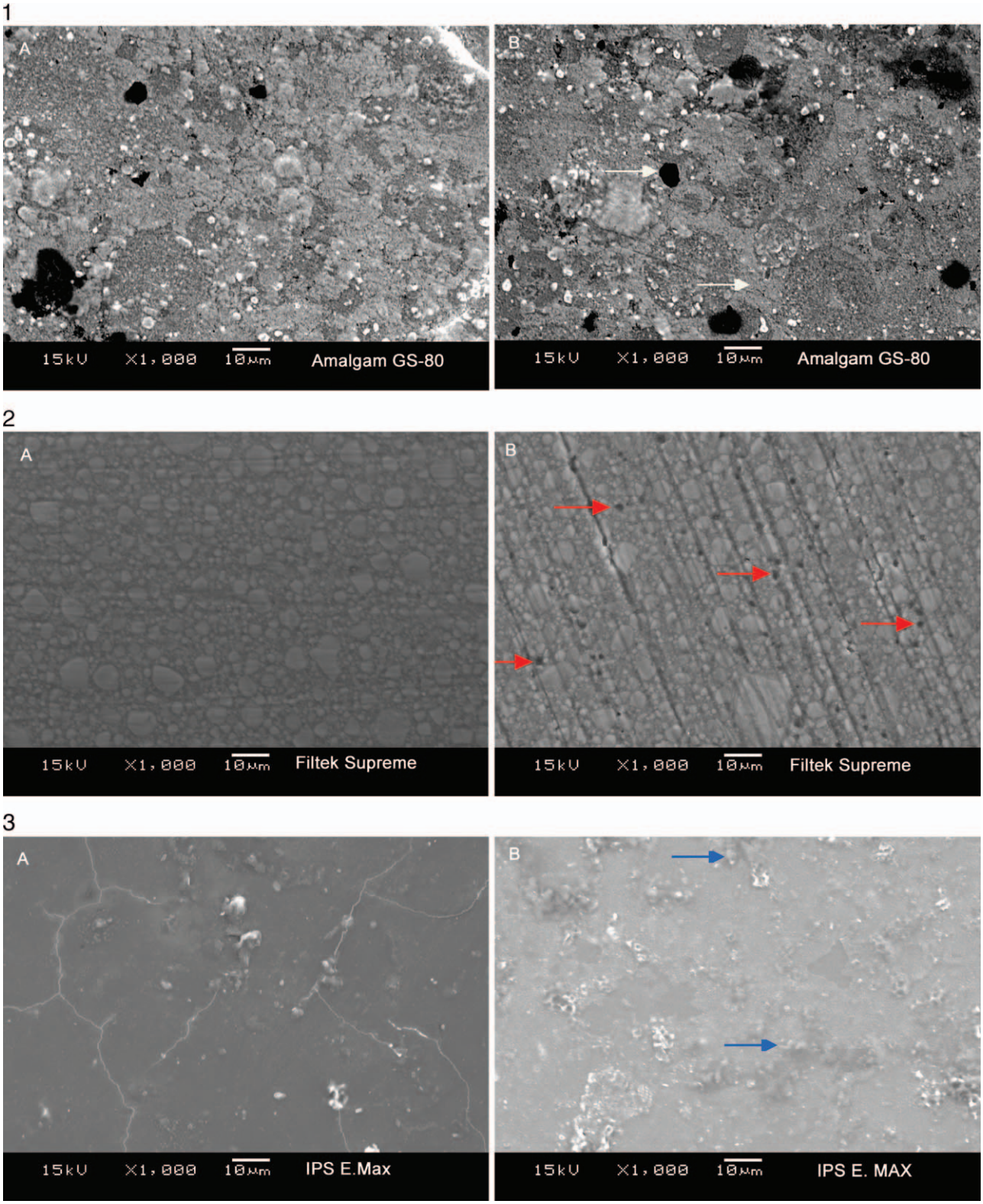

Figure 3. *SEM images of different restorative materials. Left, before biodegradation; right,after biodegradation (1000×). Red arrows show filler particles removed from the organic matrix; black arrows show cracks; white arrows show various phases; and blue arrows show surface nodules.*

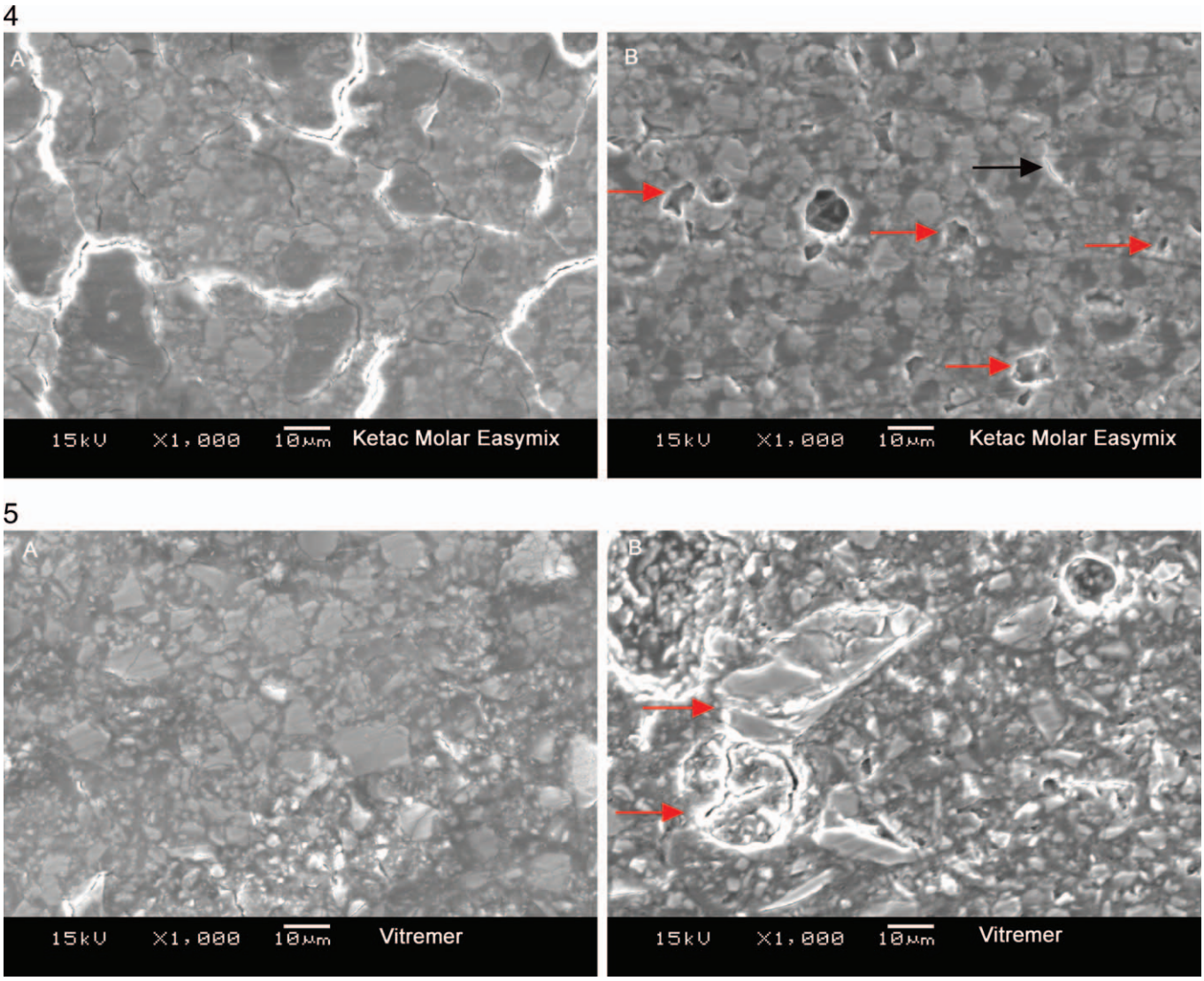

Figure 3. *Continued*

organic phase.[27] Glass ionomer cements withstand a complex process of absorption, disintegration, and outward transportation of ions, with an erosive loss of matrix components and leaching of glass particles. This process (absorption, disintegration) is more intense in acid medium.[6,28,29] As the biodegradation softens the organic matrix and releases ions from ionomeric materials, it is possible that the loss of components from two Vitremer matrixes (polyacrylate-inorganic and polymer-organic) and from the organic matrix of Filtek Supreme leads to changes in the roughness.

For the dental ceramic, the initial increased roughness may be related to the characteristics of the polishing phase and not necessarily to the biodegradation. This result was not expected because the ceramics are considered the most inert of all dental materials used for restoration.[30] There are fundamentally two types of glaze application: the autoglaze and the overglaze. Fahmy and others[31] observed a crack length significantly smaller for the autoglaze group than for the overglaze group, while Atay and others[32] showed greater color stability for the autoglaze specimens than for the overglaze specimens; Zaki and Fahmy[33] showed that bleaching agents did not affect significantly the roughness of the autoglaze group. Thus, the autoglaze appears to have a more resistant surface to biodegradation than the overglaze, which is the one used in this study. In this way, the vitreous ceramic of the glaze possibly suffered biodegradation due to the increase of roughness. Besides, Chang and others[34] observed

an increase in particle grit sizes for ceramics, which form nodules, thus corroborating with the formation of nodules observed in our SEM images. This event possibly occurs due to the poor thermal conductivity of porcelain associated with the formation of large temperature spikes at the point of contact between the diamond bur and the porcelain.

However, the roughness of the amalgam did not alter significantly after the biodegradation, possibly due to some factors related to this material's characteristics: a high copper content, spherical copper particles, polishing, and a passive layer on the surface. The high copper content (>6%) was achieved through the optimization of amalgam alloys by Innes and Youdelis[35]; the introduction of spherical copper particles was further performed by Asgar[36]; the polishing leads to a substantial increase in the corrosion resistance once it removes the tinmercury alloy (gamma-2 phase) and decreases the concentration of electrolytic cells[37]; and, finally, the passive layer formed on the surface also contributes to the improvement of the corrosion resistance.[38] Considering the last aspect, studies on the mercury liberation from dental amalgams suggest the formation of a passive layer that is composed of an oxide film on the material surface, which interferes with the dissolution process of the metal components and substantially diminishes their lixiviation.[38-41] Furthermore, it is important to note that seven days is a relatively short time to promote considerable corrosion on silver amalgams.

Regarding the hardness, it was observed that the amalgam and ionomeric materials presented an increase in their hardness values after the biodegradation experiment, probably related to a posthardening process after the setting time. For ionomeric materials, this process could be explained by the slow rate of the acid-base reaction forming the polyacrylate salts (KM and VI) and the free-radical polymerization reaction (VI), which continued after light-irradiation.[42,43] A maturation over time could also occur with the amalgam. During the trituration process, the mercury dissolves the surface of the alloy particles and a plastic mass is formed by the setting and hardening of the amalgam. The amalgam crystallization can continue for several days,[30] according to our results.

In a different way, the rapid setting reaction of the resin composite is initiated by light exposure, and most parts of the conversion process end immediately after the photoactivation, leading to a reduced postirradiation polymerization.[44,45] Moreover, the presence of Bis-EMA and TEGDMA in the matrix composition possibly contributed to the hardness stability of Filtek Supreme. TEGDMA can decrease the surface softening caused by acids and increase the degree of polymerization of resin-based material,[46] while Bis-EMA showed a lower amount of released products and a higher stability.[47] In the sintering process of ceramics, the compacted particles suffer a coalescence phenomenon that leads to the increase of the solid density. Consequently, the hardness of these materials undergoes a significant increase as the mechanical integrity of the body is favored. However, after the sintering process, the hardness of these materials tends to suffer little alteration after the setting reaction,[31,48,49] corroborating our results.

According to the EDX results, the initial chemical composition of AM and EM was not significantly altered after biodegradation, while FS, VI, and KM did present alterations in their spectra. Thus, it was observed that the fluoride released from KM and VI after seven days possibly occurred due to their intrinsic characteristics, resulting in dissolution and diffusion processes, which occur mainly in an acid medium.[20,50] Furthermore, there was the adsorption of ions such as Ca^{2+} and Cl^{-} on the surface of some of the materials (FS, VI, and KM), probably from saliva, after the *in situ* experiment.

In the oral environment, an established or mature biofilm can accumulate at stagnant sites, such as interproximal surfaces, pits and fissures, and gingival crevices, beyond compatible levels of oral health.[51] This can develop into disease conditions, such as secondary caries, as well as into the demineralization process of marginal enamel and dentin.[52] Therefore, it would be important to assess patients individually regarding their salivary flow, caries risk, buffer capacity of saliva, diet and oral hygiene, among others, and then carefully select the restorative material for intraoral sites where the biofilm would be protected against dynamic shear forces from saliva and tongue and toothbrushing, which stimulates its accumulation and maturation.

The hypothesis that restorative materials subjected to a biofilm interaction have a significant difference on roughness, hardness, and microstructure, after seven days has to be partially accepted since there was a material dependence among the characteristics analyzed.

CONCLUSIONS

In conclusion, within their limits, the present findings show that the influence of dental biofilm

accumulation *in situ* on different restorative materials is a material-dependent event. All of the materials changed after biodegradation *in situ*. Thus, all esthetic restorative materials showed increased roughness, confirmed by SEM, while ionomer materials and the silver amalgam showed significantly higher hardness. The initial chemical composition of the composite resin and ionomer materials evaluated was significantly altered by the action of the biofilm *in situ*.

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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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Efficacy of an Anesthetic Gel in the Reduction of Pain During Impression Making

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Clinical Relevance

This study suggests that the described anesthetic gel can be effective in significantly reducing patient discomfort during the displacement of the gingival tissue.

SUMMARY

Introduction: Gingival tissue displacement can be an uncomfortable procedure, often performed without injectable local anesthesia. The present study evaluated the efficacy of an anesthetic gel in reducing pain during this procedure.

Material and Methods: Thirty patients undergoing definitive dental impression, for fabricating full-coverage restorations, were evaluated for pain perception on displacement of the surrounding gingival tissues. All the participants were randomly divided into two groups. Five minutes before the insertion of the displacement cord, the anesthetic gel, a mixture of 5% lidocaine and 5% prilocaine, was applied in the sulcus of test group patients. Each patient was asked to complete the Visual Analogue Scale (VAS) and the Verbal Rating Scale (VRS).

Results: In the two scales considered, test group patients showed a statistically significant pain reduction (VAS p=0.0002, VRS p=0.01054) compared to control group patients.

Conclusions: Within the limitations of this study, a clinically significant efficacy of the anesthetic gel was noticed during gingival displacement procedures.

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INTRODUCTION

Many patients are afraid of dentistry, and they often associate dental procedures with pain. Even if patients have different abilities to tolerate painful and stressful dental procedures, the World Health Organization estimates the percentage of those who are dentist-phobic between 15% and 20%.[1,2]

The aim of modern dentistry is to provide the patient with maximum comfort during any session and, to achieve this goal, it is important to reduce the pain and to eliminate, as much as possible, the anxiety induced by stimuli such as anesthesia, needles, scalpels, and drills. Although different types of anesthetic agents are available, fear of injections is one of the main reasons patients avoid dental care. A nontraumatic anesthesia would provide patients with greater comfort, avoiding all pre- and post-infiltration problems, such as injection pain and swelling, lip numbness, and tongue paresis.[3] Different alternatives, such as biofeedback, reassurance, hypnosis, distraction, transcutaneous electronic nerve stimulation (TENS), and nitrous oxide, have been utilized to reduce pain associated with dental procedures, but none of them can completely replace anesthesia by local injection.[4]

An anesthetic gel, containing 5% prilocaine and 5% lidocaine (Oraqix, Dentsply Pharmaceutical, York, PA, USA), has been recommended to reduce pain during routine treatments, such as obtaining probing depths, scaling, and root planing. Jeffcoat and others[5] showed that Oraqix gel provides valid pain reduction during scaling and root planing, especially in advanced cases of periodontal disease. Donaldson and others[6] emphasized that Oraqix gel is statistically significantly more effective than placebo in reducing pain caused by periodontal debridement. Furthermore, the favorable anesthetic efficacy of anesthetic gel in pain-sensitive patients was confirmed by Magnusson and others[7]. Van Steenberghe and others[8] indicated that for scaling and root planing, the subgingival application of the Oraqix anesthetic gel offers substantial advantages over injection anesthesia; its efficacy on the gingival sulcus was related to the ability to remain in situ, thanks to a particular mixture that permits a status change from liquid to gel in contact with the body temperature (37°C) and the capacity to become active in 30 seconds and to last 20 minutes.[6,9]

While taking a dental impression, the transfer of an accurate replication of the patient's hard and soft tissue to the dental laboratory is a crucial factor, especially with subgingival margins.[10] Since definitive impressions replicate both the tooth structure and the surrounding gingiva, success is based on creating an accurate preparation, on selecting the appropriate impression material and impression techniques, and on managing properly the periodontal tissues.[11,12] In addition to creating an area free of fluid and debris, gingival tissue should be displaced to expose the tooth finish line when making a definitive impression.[13] In order to achieve this purpose, a displacement cord has been extensively recommended.[14] Unfortunately, such a technique can be an invasive method that often causes patients discomfort.[15] Novel techniques were studied to reduce or to eliminate patient discomfort; a clinical trial by Yang and others[16] demonstrated that the injection-type displacement material, with or without aluminum chloride, ensures adequate tissue retraction and is preferable to retraction cord since the application of this material is painless.[16]

Even if pain is a subjective experience, several methods to describe it have been described with different pain scales. The Visual Analogue Scale (VAS) is represented by a 100-mm horizontal blank ruler where the patient is asked to mark the position that best describes the pain, taking into account the left and the right end point marked, respectively, "no pain" and "worst pain imaginable."[17] The Verbal Rating Scale (VRS) is a six-point scale with the following choices: "no pain," "very mild pain," "mild pain," "moderate pain," "severe pain," and "very severe pain."[18]

The purpose of the present clinical study was to verify the efficacy of Oraqix anesthetic gel during the displacement of gingival tissues to reduce patient pain perception. The null hypothesis was that there is no difference in pain perception during displacement cord insertion with or without the use of Oraqix anesthetic gel.

METHODS AND MATERIALS

Thirty patients (16 females and 14 males) undergoing definitive dental impression were randomly selected for this study and randomly divided into two groups: the test group (TG) was treated with Oraqix, and the control group (CG) was treated with chlorexidine digluconate gel (Corsodyl Dental Gel*30G, GlaxoSmithKline, Verona, Italy).

Treatments were performed in a private dental clinic (Padova, Italy), and the local Internal Ethics Committee (Protocol N.0103#) approved the study protocol. Informed consent was obtained from all the subjects included in the study. In obtaining the informed consent and in conducting the study, the principles outlined in the Declaration of Helsinki on Experimentation Involving Human Subjects were adhered to as revised in 2000. In addition, the sample size was determined considering a power of 90% using a two-tailed test at the 5% level of significance and a minimum detectable difference

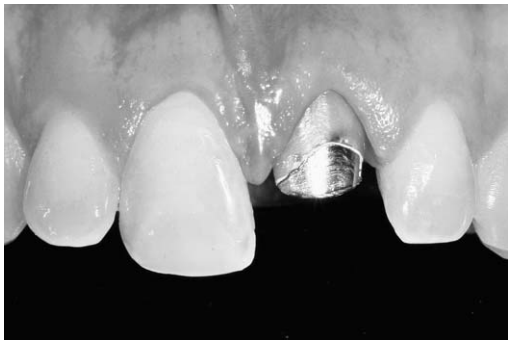

Figure 1. *Tooth prepared for receiving full coverage restoration with vertical finishing line.*

between the groups (test and control) of 1 (VAS unit) with a standard deviation of 0.91.

All patients enrolled were healthy with no systemic diseases or immunodeficiency or neurological/psychiatric handicap. They also were able to comprehend the pain scales and signed an informed consent prior to inclusion in the study. Patients with history of allergy, sensitivity, or any form of reaction to local anesthetics of the amide type, in pregnancy or lactation, with significant cardiovascular, renal, or liver disease, were excluded. All patients presented good oral hygiene, with healthy periodontal tissues (periodontal pocket depth < 4 mm; negative bleeding on probing) and a plaque index lower than 20%.

Every patient had one nonvital tooth with a provisional restoration in place for at least 6 weeks. All the teeth were prepared with vertical tooth preparation the day of the provisional restoration insertion. The day of the definitive impression, gingival displacement procedures were performed in both groups. A double cord technique was used. At

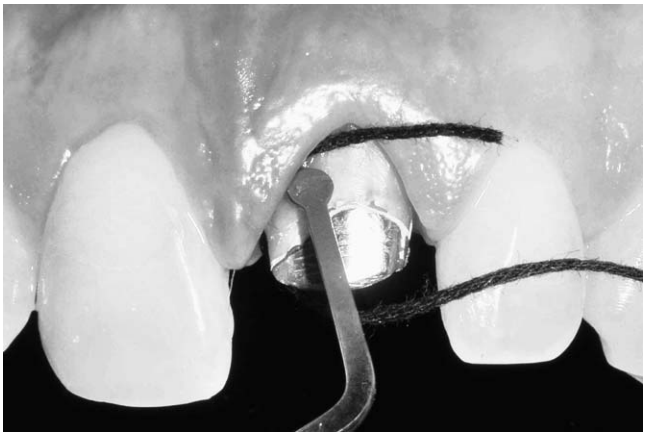

Figure 3. *Insertion of 000 cord in the sulcus.*

first, a "000" cord was positioned at the bottom of the gingival sulcus (Ultrapack Cord, Ultradent Products Inc, South Jordan, UT, USA). Subsequently, a "1" cord was placed over the first one (Ultrapack Cord). Before cord insertion, anesthetic Oraqix gel was applied in the sulcus of TG patients, left in situ for 30 seconds according to the manufacturer's instructions, and then cleaned out with abundant water irrigation. The same procedure was performed for the CG in which a chlorexidine digluconate gel was applied instead of the Oraqix gel. After 10 minutes of gingival displacement, the second cord was removed, and the definitive impression was taken with a polyether material (Impregum Penta Soft, 3M ESPE, St Paul, MN, USA) For each patient, sex, age, use of Oraqix, number of teeth examined, sulcus depth, bleeding during cord insertion, and pain experienced during the procedure were recorded (Figures 1–4).

Patients were assigned to one of the two treatment groups using a computer-generated randomization table. All patients participated in the study with a

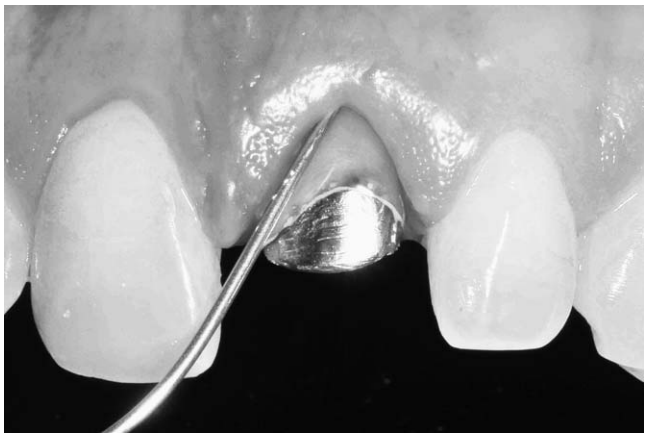

Figure 2. *Application of non injectable anesthetic gel in the sulcus.*

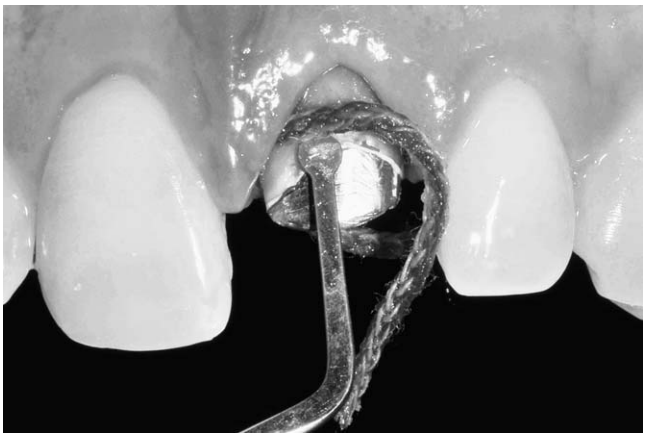

Figure 4. *Insertion of 1 cord in the sulcus.*

Table 1: *Study Sample Data. Mean Values and Standard Deviations Are Reported for Each Variable Examined.*

Variables	Test Group (n=15)		Control Group (n=15)		*p*-Level
	Mean Values	SD	Mean Values	SD	
Age (y)	46.13	8.3	46.66	15.78	NS
Sulcus v (mm)	1.53	0.66	1.26	0.41	NS
Sulcus m (mm)	1.83	0.64	1.63	0.51	NS
Sulcus l/p (mm)	1.63	0.58	1.43	0.37	NS
Sulcus d (mm)	1.7	0.7	1.53	0.48	NS
Mean sulcus (mm)	1.67	0.28	1.46	0.19	NS

Abbreviations: SD, standard deviation; NS, not significant.

single tooth. Fifteen teeth were assigned to the CG and 15 teeth to the TG. In the case of patients presenting with multiple teeth to be treated, the selection was performed by tossing a coin. Allocation concealment was achieved using a sealed coded opaque envelope containing the treatment of the specific subject. The sealed envelope containing treatment assignment was opened during the surgery immediately before the gel positioning.

Immediately after the cord insertion was completed, every patient, blinded to the study groups, was asked to assess their discomfort on two different pain scales: the VAS and the VRS. All statistical data were elaborated with the SAS System, an integrated system of software products (2012 SAS Institute Inc., SAS Campus Drive, Cary, North Carolina 27513, USA), and different variables, including the VAS and VRS scales, were examined. Different VAS values were reported in millimeters (between 0 and 100 mm), as determined by the patients' designation on the scale. The VRS values were divided into six different classes:

- Class 0: no pain
- Class 1: very mild pain
- Class 2: mild pain
- Class 3: moderate pain
- Class 4: severe pain
- Class 5: very severe pain

Analysis of variance for unpaired populations was used to compare the two groups according to the VAS scale. Nonparametric data on VRS scales were analyzed with the Pearson chi-square test.

RESULTS

Data of all 30 patients are reported in Table 1. The mean age for both groups was approximately 46 years, while sulcus depth appeared deeper in TG (1.67 mm) than in CG (1.46 mm). The pain experienced by patients during the displacement of the gingival tissues was evaluated with the VAS scale of pain (Table 2).

The results on the VRS scale are shown in Table 3. The percentage of patients graded in each class of value was utilized as a criterion of reference. The worst pain perceived was classified as moderate, corresponding to class 3. Most of the TG patients were distributed in the first three classes, and their pain was considered mild, while most of the CG patients were in class 3 (73%).

Table 2: *Pain Experienced by Patients and Evaluated with VAS Scale of Pain.*

Variables	0: Oraqix (n=15)		1: Control (n=15)		*p*-Level
	Mean Values	SD	Mean Values	SD	
Age (y)	46.13	8.3	46.66	15.78	NS
Sulcus v (mm)	1.53	0.66	1.26	0.41	NS
Sulcus m (mm)	1.83	0.64	1.63	0.51	NS
Sulcus l/p (mm)	1.63	0.58	1.43	0.37	NS
Sulcus d (mm)	1.7	0.7	1.53	0.48	NS
Mean sulcus (mm)	1.67	0.28	1.46	0.19	NS
VAS values (mm)[a]	19.26	12.48	43.6	17.67	0.0002

Abbreviations: SD, standard deviation; NS, not significant.
[a] *VAS mean values have been compared using an analysis of variance F-test.*

Table 3: *VRS Values. Pearson Chi-Square: 11.23077 (*p*=0.01054).*

VRS Values	Test Group		Control Group	
	n	%	n	%
Class 0: no pain	2	13.33	1	6.67
Class 1: very mild pain	5	33.33	1	6.67
Class 2: mild pain	6	40.00	2	13.33
Class 3: moderate pain	2	13.33	11	73.33
Class 4: severe pain	0	0.00	0	0.00
Class 5: very severe pain	0	0.00	0	0.00
Total	15		15	
Median	Class 1: very mild pain		Class 3: moderate pain	
Mode	Class 2: mild pain		Class 3: moderate pain	

DISCUSSION

This clinical study was designed to reveal whether the anesthetic gel Oraqix is effective in pain reduction during gingival tissue displacement for restorative needs. The data support rejecting the null hypothesis that no pain differences would be present with or without the utilization of the anesthetic gel. These results agree with those demonstrating its effectiveness as a valid alternative to the traditional injection technique during periodontal procedures such as scaling and root planing.[5–7]

Also, as in the findings of Gracely and Dubner[17] and Paice and others,[18] the three scales utilized in the present study for pain assessment reported clinical differences between TG and CG groups. As the utilization of displacement cord is common for definitive impression, it is also an uncomfortable procedure; hence, this gel represents a valid alternative to traditional anesthesia for reducing patient discomfort on gingival retraction, as reported by Azzi[15] and Yang and others.[16]

By analyzing the examined group of 30 patients, the efficacy of Oraqix gel was not influenced by sex, age, tooth position, and bleeding during cord insertion. Instead, the main variable of pain experienced, examined through the three different scales, demonstrated that the discomfort perceived in patients treated with Oraqix gel was significantly lower than the control group. As shown in Table 2, VAS values for TG patients were lower than CG patients. It is interesting to note that none of the patients treated with Oraqix gel reported a result >60 mm. For the VRS scale (Table 3), similar results are confirmed. Most of the CG patients belonged to class 1 and 2 (very mild pain = 33.44% and mild pain = 40.00%), while most of the CG patients belonged to class 3 (moderate pain = 73.33%). A deeper analysis of the table shows that about 86.67% of TG patients perceived mild pain (classes 0, 1, and 2), while most of the CG patients (73.33%) perceived moderate pain (class 3). Significant results are present in the analysis of sulcus depth, which was higher in TG (1.67 mm) compared to CG (1.46 mm). Even if sulcus depths are similar, it is interesting to note that Oraqix gel was effective with an average sulcus depth of 1.67 mm.

Due to the reduced sample size, further studies are necessary for the analysis of sulcus depth and anesthetic gel efficacy, but the clinical feeling reported improved results related to the presence of deeper sulcus. A shallow sulcus represents a difficult site to properly contain the applied gel. In fact, this anesthetic gel was originally created for application in periodontal pockets able to contain the gel in situ during its modification from liquid to gel. For this reason, the utilization of the provisional restoration to apply the liquid could be an interesting method to insert the material inside the sulcus, reducing its migration. Even if the results of this clinical study are encouraging, the limited sample size did not allow us to relate the efficacy of this anesthesia to different types of tooth preparation or to gingival biotypes, and the several limitations should be considered. With regard to the dimensions of the peri-implant soft tissues, an interesting analysis could be carried out on the utilization of Oraqix gel in the clinical procedures related to implant-supported restorations.

CONCLUSIONS

Within the limitations of this study, the anesthetic gel Oraqix, a mixture of 5% lidocaine and 5% prilocaine, was clinically effective in reducing pain experienced during gingival tissue displacement with cord insertion. However, further studies with increased sample sizes are required in order to better

evaluate the efficacy in relation to different periodontal biotypes and preparation design.

Conflict of Interest

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

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Laboratory Research

Predictable Casting for Dimensional Shrinkage of Fast-cast Post-and-cores

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Clinical Relevance

A cast post-and-core needs to be slightly smaller than the space created for it. This paper looks at three methods to cast post-and-cores and evaluates the predictability of dimensional shrinkage for the three methods. The use of a phosphate investment with an accelerated technique can produce shrinkage with consistency in dimensional stability.

SUMMARY

Statement of problem: Prior investigations into dimensional shrinkage of fast-cast post-and-cores resulted in variable dimensions. There is a need for consistent shrinkage of cast post-and-cores to reduce or eliminate lateral stresses on the remaining tooth structure.

Purpose: The purpose of this research was to find a method of casting post-and-cores that would result in consistent shrinkage.

Material and Methods: A total of 45 methyl methacrylate post-and-core patterns, 10 mm long, were fabricated from a standardized steel block. Three methods were used to cast the post-and-core. Investment materials used were Fast Fire 15 (Whip Mix Corp, Louisville, KY, USA) and Beauty Cast gypsum (Whip Mix Corp). Three groups of sample post and core patterns were cast, varying the investment material used and the burnout time.

Results: Results show a statistical significance between the investment materials ($p<0.05$). The accelerated technique produced the most consistent results, and all samples had shrinkage of the casting.

Conclusions: The use of a phosphate investment with an accelerated technique can produce consistent and predictable shrinkage of cast post-and-cores.

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INTRODUCTION

The development of the casting process in dentistry undoubtedly represents the greatest single forward step in the science and art of the profession that has been made. The casting process, which Dr. Taggart gave the profession in November 1907, is almost identical to the one used today. Although others conceived the idea of the cast restoration prior to Taggart, they did nothing to bring the technique into a state of perfection whereby it could be used by the

profession.[1] The ability to cast metals into an investment mold with a minimum for bench set time and oven time will allow the technician or dentist to work faster and produce more. This is especially important in the casting of custom post-and-cores. Post-and-cores are fabricated to provide retention and resistance for crowns and fixed partial dentures.[2] Rosenstiel et al[2] and Baba[3] recommended the use of a cast post-and-core for anterior teeth with flared or elliptical canals. The conventional method of casting a post-and-core is time consuming and requires the patient to make two appointments. This process involves a minimum of 2¼ hours of laboratory time: sprueing and investing, 10 to 15 minutes; investment setting, 45 to 60 minutes; and staged burnout and casting, a minimum of 60 to 75 minutes.[4] The process can be frustrating and time consuming for the dentist as well as the patient. The dentist must reschedule the patient and fabricate a provisional restoration. Rescheduling the patient is expensive and inconvenient.

In 1991, W.V. Campagni and M. Majchrowicz[5] were the first to introduce an accelerated technique for custom post and cores. The accelerated casting procedure may reduce casting time to 30 to 40 minutes. Initially suggested as a way to make cast post-and-core restorations a one-visit procedure, the procedure has been found to produce castings with accuracy and surface roughness similar to traditional methods.[6] The accelerated technique of pattern elimination has received increased attention as a method of improving productivity. Hansen et al[6] showed the quality of the fast-cast castings to be identical to that of a slow, conventional technique. Although quality is the same, all techniques showed shrinkage or expansion. There is a need for a technique that would give predictable shrinkage for all castings.[6] The cast post-and-core has been recommended, as opposed to a prefabricated post, because it requires less tooth instrumentation in correlation with a lesser degree of tooth perforation.[7] The cast post-and-core restoration also has the ability to resist rotational forces, which is not possible with a prefabricated post system.[8,9] An undersized cast post may also limit the stress placed on the radicular tooth structure, possibly decreasing the likelihood of root fracture.[4,10] Figure 1 shows the effect of a casting that may be too large for the prepared space. A post should rest on a flat area 90° from the post space, with a small amount of space between the post and the tooth root for the luting agent.[2,3]

The fast-cast technique shortens certain time intervals of the conventional method. The first

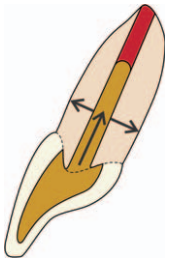

Figure 1. *If the post-and-core is larger than the corresponding post space, lateral pressure will be placed on the root of the tooth and can possibly lead to fracture of the remaining tooth.*

reduction is the mold's bench-setting time. As soon as the mold for the fast-cast technique has reached adequate wet strength, the mold is placed into a preheated furnace. The second reduction is the pattern's elimination time in the furnace. The elimination-time reduction is a combination of placing the mold in a preheated furnace and limiting its time at maximum temperature to 15 minutes.[5,10,11]

A cast post-and-core should fit somewhat loosely in the canal because a tight fit may promote root fracture. The casting should be slightly undersized, which can be accomplished by restricting expansion of the investment (ie, by omitting the usual ring liner) or changing the direction of the expansion.[10,12,13] Post-and-core restorations must be carefully placed into the tooth during trial insertion because resistance to full seating of the casting can cause tooth fracture as the casting binds and attempts to expand the tooth (Figure 1). The post-and-core should seat passively with little discernible movement or rotation. If significant movement occurs, a new post-and-core must be made that has better adaptation.[14] An undersized cast post is easier to fit and cement in the prepared root canal, which reduces chair time.[10]

In order to consistently shrink post-and-cores, an investment material and technique that is reproducible and capable of tolerating the accelerated techniques of fast casting must be used. It is essential to avoid using a ring liner when investing resin patterns because the liner compensates for investment expansion.[4] By using an unlined stainless steel ring, the investment cannot expand outward and is forced toward the center of the mold, resulting in a smaller mold cavity (Figure 2). Gypsum-bonded investment is capable of expansion inward, resulting in post shrinkage. The problem with gypsum investment is that it is not capable of tolerating the reduced bench set, the preheated oven

temperature of 1300°F, and the accelerated burnout in the oven.[4] A phosphate-bonded investment must be used for this accelerated technique. Phosphate-bonded investment materials offer certain advantages over gypsum-bonded investments. They are more stable at high temperatures, they expand rapidly at the temperatures used for casting alloys, and their size can be conveniently controlled. The increased expansion that they exhibit results from a combination of the following factors:

1. The high heat from the oven quickly vaporizes the resin pattern, allowing the expansion of the phosphate-bonded investment to compress the void where the pattern had been.[4]
2. The increased strength of the material at high temperatures restricts shrinkage of the alloy as it cools.[4]
3. The powder mixed with colloidal silica reduces the surface roughness of the castings and also increases expansion.[4]

Expansion can be conveniently controlled by slightly diluting the colloidal silica with distilled water due to the higher casting temperature compared with gypsum-bonded investment.[12,13] The principal difference between gypsum-bonded and phosphate-bonded investments is the composition of the binder and the relatively high concentration of silica refractory material in the latter. The binder consists of magnesium oxide and an ammonium phosphate compound. Contrary to gypsum-bonded products, this material is stable at burnout temperatures above 650°C (1200°F), which allows for additional thermal expansion. Most phosphate-bonded investments are mixed with a specially prepared suspension of colloidal silica in water. Increasing the proportion of special liquid (colloidal silica) also increases expansion.[14,15]

The purpose of this study was to develop an accelerated casting technique that consistently produces post shrinkage. There is shrinkage with gypsum-bonded investments, but they cannot tolerate the stress of fast casting.[5] The focus was on Fast Fire 15 (Whip Mix Corp, Louisville, Ky), a phosphate-bonded investment.

MATERIALS AND METHODS

A total of 45 Duralay resin (methyl methacrylate, Reliance Mfg Co, Alsip, IL, USA) post patterns were used in this study. These patterns were fabricated by inserting the resin into a stainless steel block that contained a 2.5 × 2.0-mm tapered hole (Figure 3). The taper on the steel die was fabricated to allow the resin pattern to be made, cast, and measured for expansion or shrinkage of the casting. A plastic sprue (Whip Mix Corp) was placed into the resin and allowed to set for five minutes. The resin post was removed from the block and was placed in a crucible former (Whip Mix Corp); the length of each resin post was measured for consistency. Each pattern was inspected for defects and discarded if any were found.

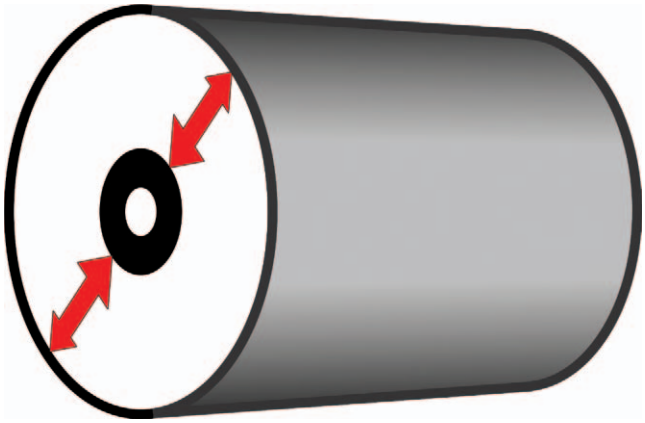

Figure 2. *Expansion of the investment material will press against the walls of the casting ring. Because the casting ring becomes a restricting force to prevent outward expansion, the investment will actually compress the void left from the resin pattern. The result will be a slightly smaller casting than the original pattern.*

The resin posts were divided into three groups of 15. Each investment type has variable bench set and burnout times (Table 1). The Beauty Cast (Whip Mix Corp) gypsum investment (group 1) and the Fast Fire 15 (Whip Mix Corp) (group 2) were prepared according to the manufacturer's instructions. The Fast Fire 15-A (group 3) was the accelerated investment. For group 1, each pattern was lightly sprayed with a surfactant agent (Smoothedex debubbilizer, Whip Mix Corp). Each resin post was invested with a gypsum-bonded investment (Beauty-Cast, Whip Mix Corp) using the manufacturer's recommendation. According to Shillingburg,[13] adding 1.0 to 2.0 cm^3 of extra water to 50 g of investment and not using a ring liner in the casting ring will result in a slightly smaller dowel core that should have less tendency to bind in the canal. Therefore, to ensure shrinkage, and to follow Shillingburg's protocol, 1.0 cm^3 of extra water was added to the manufacturer's recommendation. Following the manufacturer's recommendations, the distilled water was measured at 72°F, the Beauty Cast (Whip Mix Corp) was hand mixed for 15 seconds, vacuum mixed on slow speed for 40 seconds, vibrated into the stainless steel casting ring measuring 1¼ inches

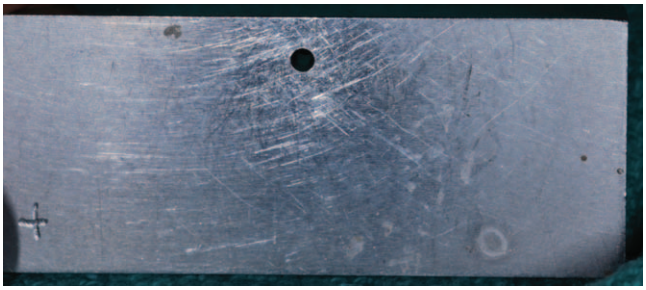

Figure 3. *The stainless steel block with the tapered space to form the resin pattern to be cast.*

diameter × 1⅞ inches high (Whip Mix Corp), and bench set for 30 minutes. The casting ring was placed in the Accu-therm oven (Jelenko, San Diego, CA, USA) at room temperature and allowed to slowly heat from room temperature (approximately 75°F) to 1200°F, at which point the investment was allowed to heat soak for one hour.

For group 2, the phosphate-bonded investment (Fast Fire 15, Whip Mix Corp) was prepared using the manufacturer's recommendation. For group 3, the accelerated casting technique, a phosphate-bonded investment (Fast Fire 15-A, Whip Mix Corp) was used. A 20:80 distilled water-special liquid (colloidal silica, Whip Mix Corp) solution was incorporated with the investment powder. Shillingburg stated that a high silica and low water mixture will result in more expansion of the investment.[13] The total liquid volume was reduced by 2 mL to give a more viscous mix. Some latitude in expansion can be gained by altering certain of the variables found in the investment procedure. Decreasing the powder-liquid ratio reduces the setting expansion slightly, and vice versa. That is, if 50 g of investment is used with 17 mL of water to produce an average degree of setting expansion, the use of 18 mL of water would decrease expansion and 16 mL would increase expansion.[15] The Whip Mix Corp also recommends using more special liquid and less water to achieve maximal expansion.[14] The investment was hand mixed for 125 seconds, vacuum mixed on slow speed for 15 seconds, bench set for 15 minutes, and put in a 1300°F preheated oven for 15 minutes, following the manufacturer's recommendations.

A single operator performed the investing and casting procedures. All castings were performed with a natural gas torch (National Blowpipes, Keystone Industries, Cherry Hill, NJ, USA) using 4 dwt of new Ney-Oro B-20 type III gold (Dentsply, York, PA, USA). The arm of the casting machine (Kerr Centrifico Casting Machine, Orange, CA) was totally unwound prior to each casting and was then rotated clockwise until the first stiff resistance was met; from that point, three full turns were applied. Once the gold was melted, it was dusted with Ney casting flux (Degussa-Ney Dental, Yacaipa, CA, USA) to dissolve any oxides formed. The gold was then centrifuged into the mold upon release of the casting machine arm. The casting ring was quenched in cold water before devesting. The castings were soaked in Stripping Acid (American Dental Supply Inc, Allentown, PA, USA) and placed in the ultrasonic for five minutes to remove any residual investment material. The posts were steam cleaned and then inspected under a 6×-magnification microscope (Swift Instruments, Schertz, TX, USA) (Figure 4). All nodules were removed from the post using a ¾-inch medium sandpaper disk (EC Moore Company, Buffalo Grove, IL, USA). Small nodules on the cast post can cause wedging stresses that precipitate root fracture. A single air bubble entrapped when the pattern is invested can produce a positive nodule on the metal post.[13]

The measurement of the cast post was performed by placing the casting back into the steel block, giving a half-twist clockwise and applying a 70-g weight to completely seat the post (Figure 5). Post-and-core restorations must be carefully placed into the tooth during the trial insertion. No attempt should be made to force the casting into position in the tooth. A steady but modest force should be applied with the fingers or a hand instrument until complete seating is achieved. It was evident that shrinkage had occurred if the post was able to pass through the tapered hole in the steel block. If the casting was unable to fit back into the block and have the end of the post reach the bottom of the steel block, the casting had expanded. A traveling 32×-magnification microscope (Gaertner Scientific Cor-

Table 1: *Investing and Casting Variables*

Investment	Investment Properties	Casting	Burnout (°F)	Bench Set	Burnout
Beauty Cast	Gypsum bonded	Manufacturer recommended	75–1200	30 min	60 min
Fast Fire 15	Phosphate bonded	Manufacturer recommended	1300	15 min	30 min
Fast Fire 15-A	Phosphate bonded	Accelerated	1300	15 min	15 min

Figure 4. *The casting made from type III gold. The casting is sprued, then cast.*

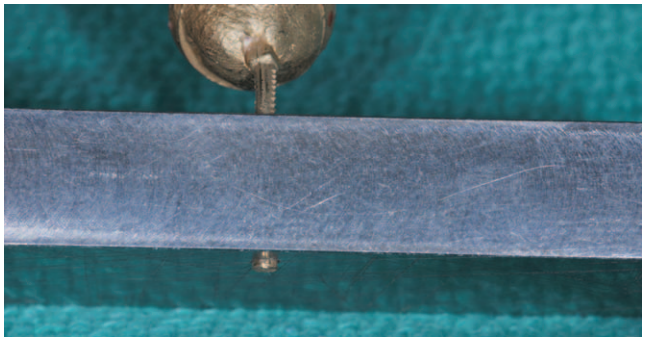

Figure 5. *The casting tried back in the steel block after examination to ensure no nodules are present on the casting. If the casting is smaller, it will pass through the channel, and the tip will protrude out the inferior border of the steel block. The length of the post is measured from the tip of the post to the border of the steel block.*

poration, Chicago, IL, USA) was used to measure (in millimeters) the protrusion of the post from the tapered hole. Measurements were made from the apex of the post to the flat bottom of the steel block. For the posts that expanded, an impression was taken of the space left in the tapered hole using polyvinylsiloxane (President, Coltene Whaledent, Cuyahoga Falls, OH, USA); the impression was measured with the traveling microscope to determine how much it expanded. Measurements were made from the apex of the polyvinylsiloxane impression material to the image of the bottom of the steel block.

RESULTS

Means and standard deviations were calculated for each group. This study was a one-factor, completely randomized design. A one-factor analysis of variance with Fisher least significant difference post hoc test was used to determine which pairwise contrasts were significant (Table 2).

The Beauty Cast investment (group 1) resulted in shrinkage of all 15 castings, with the largest mean shrinkage of −0.6914 mm. The Fast Fire 15 (group 2) resulted in only eight castings that shrank, with a mean of −0.0281 mm; four that expanded; and three that fit the steel die perfectly, meaning there was neither shrinkage nor expansion. The Fast Fire 15-A (group 3) resulted in shrinkage of all 15 castings, with a mean shrinkage of −0.3348 mm and a standard deviation of 0.1782. Refer to Table 3 for individual casting results. The results show a statistical significance between the different investments when testing for dimensional stability ($p<0.05$). Even though the Beauty Cast produced the most shrinkage, it also had the most variance in its results, with a standard deviation of 0.3946. The investment with the least amount of variance was the Fast Fire 15-A, with a standard deviation of 0.1782. This shows that the accelerated technique produced much more consistent results than the other investments.

In order to examine the reliability of the measurements, the author blindly remeasured 10 castings using the same microscope. A Pearson correlation coefficient was computed between replicated measurements. A correlation coefficient of $r = 0.999$ was obtained. This coefficient confirmed that the measurements were accurate and reproducible.

DISCUSSION

Each investment considered for the accelerated technique requires individual evaluation. The use of a standardized accelerated investment procedure for all types of investments is not advisable.[5]

The results of this study show that the accelerated casting technique using Fast Fire 15 produced post shrinkage with no significant standard deviation. The Beauty Cast has been shown[6] to produce post shrinkage and did so in this study as well, but with a significant standard deviation. It is important to point out that the Beauty Cast investment took a considerable amount of time to produce a cast post: a 30-minute bench set, slow oven heating from 75°-1200°F, a one-hour heat soak, and, to produce

Table 2: *Mean and SD for Each Group (mm)*

Investment	Mean	Standard Deviation
Beauty Cast	−0.6914(a)	0.3946
Fast Fire 15	−0.0281(b)	0.2818
Fast Fire 15-A	−0.3348(c)	0.1782

Table 3: *Dimensional Stability—Individual Results*

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
BC	−1.729	−0.423	−0.425	−0.786	1.44	−0.442	−0.344	−0.79	−0.767	−0.663	−0.361	−0.572	−0.575	−0.515	−0.539
FF	−1.66	−0.192	0.628	0	−0.062	−0.28	0	−0.12	−0.315	0.086	−0.334	−0.371	0.361	0	−0.286
FF-A	−0.66	−0.16	−0.372	−0.319	−0.374	−0.172	−0.241	−0.428	−0.593	−0.637	−0.34	−0.261	−0.174	−0.158	−0.133

Abbreviations: BC, Beauty Cast; FF, Fast Fire 15; FF-A, Fast Fire 15-A.

another casting, the oven must be cooled back down to room temperature. This entire process took 5 hours 25 minutes to complete. In comparison, a cast post was produced in 30-40 minutes using the accelerated technique. It is important to note that both Fast Fire 15 investment groups consistently produced smoother castings, which decreased the time required to sand the nodules from the cast. Several of these did not require sanding at all. This is another positive factor that may reduce chair time.

It is necessary to use a casting ring with no liner. The absence of the ring liner and the direct contact of the investment material with the metal casting ring constitute a physical restraint to the investment's expansion.[2,17] It is important to use a rigid casting ring to ensure that it can contain the thermal expansion of the investment and redirect the expansion inward to give a smaller mold cavity, resulting in smaller castings (Figure 2). Stainless steel, although relatively expensive, produced the hardest, strongest, and most acceptable casting ring.[16] Investment expansion directed inward by the rigid casting ring caused a reduction in the volume of the mold cavity.[16] During the thermal expansion of the investment, the mold is empty after the elimination of the resin pattern. The outside walls of the mold are pressed inward by the expansion of the investment, which is supported by the ring.[17] By using a 20:80 distilled water-special liquid solution and decreasing the total liquid mixture by 2 mL, a thicker and more expansive investment was produced, which aided in the dimensional shrinkage results.

CONCLUSIONS

A slightly undersized post can be produced using a conventional gypsum investment technique, but this procedure would require more than one office visit. The use of a phosphate investment with an accelerated technique can produce shrinkage with more consistency in dimensional stability. By choosing a casting technique that can achieve the same results in one day instead of a two-office visit procedure will ultimately save the dentist chair time and, therefore, allow for more productivity.

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

Polymerization Shrinkage Stress Kinetics and Related Properties of Bulk-fill Resin Composites

HM El-Damanhoury • JA Platt

Clinical Relevance

While achieving adequate curing at 4-mm thicknesses, not all bulk-fill composites are characterized by low-shrinkage stress. Clinicians should be cautious with these material selections for high C-factor applications.

SUMMARY

The present study assessed the polymerization shrinkage stress kinetics of five low-shrinkage light-cured bulk-fill resin composites: Surefil SDR flow (SF, Dentsply), Tetric EvoCeram Bulkfil (TE, Ivoclar Vivadent), Venus Bulk Fill (VB, Heraeus Kulzer), x-tra fil (XF, Voco), and experimental bulk fill (FB, 3M ESPE). Filtek Z250 (FZ, 3M ESPE) was used as a control. Real-time shrinkage stress of investigated composites was measured using a tensometer; maximum shrinkage stress, stress rate (R_{max}), and time to reach maximum stress rate (t_{max}) were recorded. Flexural strength and modulus were measured using a standard procedure, and curing efficiency of 4-mm long specimens was determined using bottom/top percentage Knoop microhardness. Data were analyzed using one-way analysis of variance and Bonferroni multiple range tests at a significance level of α=0.05. Results of shrinkage stress, R_{max}, and t_{max} of all bulk-fill materials were significantly lower ($p<0.05$) than those of the control except for XF. All tested bulk-fill materials were able to achieve acceptable curing efficiency (≥80% bottom/top percentage) at 4-mm depth. In conclusion, this study reports a significant reduction in polymerization shrinkage stress while maintaining comparable curing efficiency at 4 mm for some bulk-fill composites and supports their potential use in posterior clinical situations.

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INTRODUCTION

A significant concern for dental clinicians when placing direct resin-based posterior composite restorations is reducing polymerization shrinkage stress. Polymerization of dimethacrylate-based composites is accompanied by substantial volumetric shrinkage ranging from 1%-3% in packable composite and up to 6% in flowable composite.[1] Shrinkage develops during polymerization due to reduction in distance between monomer molecules as a result of short

covalent bond formation, reducing the overall free volume within the monomer structure and producing densely packed polymer molecules.[2]

During polymerization, the viscosity of the resin increases as the gel point is approached, accompanied by a rapid increase in stiffness. As the lengthening chains become entangled and a covalently bonded cross-linked network is formed, contraction stress occurs within the material.[3]

Shrinkage stress may be relieved in the early plastic stage (before the polymerization gel point) through flow. Subsequent shrinkage is obstructed, and the material becomes rigid enough to resist significant plastic flow, dramatically decreasing stress relief.[4] In addition, shrinkage manifests itself as stress when the material is confined within a prepared cavity and bonded to preparation walls. Thus, the material is restrained from changing its shape, except at the free surfaces, and further internal stresses will result.[5]

Previous studies have shown that the magnitude of generated stress depends on a combination of material properties and characteristics of the prepared cavity. In particular, confinement conditions imposed on the composite, the restoration's volume, utilized restorative techniques, and the compliance of the bonding substrate have been identified as contributing factors.[6,7] The cavity configuration factor (C-factor) is the ratio of the bonded surface area to the unbonded or free surface area. When restoring cavities with high C-factor, the resultant stresses put resin-tooth interfaces under increased tension as there is less chance for relaxation of shrinkage stress.[8] It has been reported by several investigators[9-13] that the increase in C-factor is associated with a progressive decrease in bond strength and a potential deleterious effect on marginal integrity and gap formation.[14] Alternatively, high bond strengths may cause cusp deflection and enamel cracking.[15,16]

The amount of induced polymerization stress is influenced by many characteristics of the composite formulation, such as matrix type, filler content, polymerization kinetics, degree of conversion, and modulus of elasticity.[17,18] Therefore, several attempts have been made to minimize the amount of generated polymerization stress through changing this formulation. Strategies to reduce polymerization shrinkage have included reduction of reactive sites per unit volume by increasing the filler load with nanometer size fillers,[19,20] increasing the molecular weight per reactive group through the replacement of some of the lower-molecular-weight triethylene glycol dimethacrylate (TEGDMA) with a blend of higher-molecular-weight urethane dimethacrylate (UDMA) and bisphenol A polyetheylene glycol diether dimethacrylate (Bis-EMA),[21,22] or using ring opening polymerizations based on siloranes.[23-26]

A different approach to minimizing shrinkage stress is placing composite in small increments toward fewer bonded surfaces to minimize material constraint and allow flow of the shrinking composite from available free surfaces.[22] Although incremental placement techniques may have the advantage of maximizing polymerization of each increment due to less light attenuation through smaller increments of material and increased adaptation of the composite to cavity walls, the value of incremental placement in reducing shrinkage stress has been questioned.[27-29]

Recently, many products have been marketed as low-shrinkage stress materials that have modified initiation systems, potentially allowing placement of these materials in one layer (bulk-fill) of up to 4 mm in thickness with adequate polymerization and short activation times. These materials vary in their rheologic properties and are indicated for use as flowable base materials to be veneered with 2 mm of posterior hybrid composite or as a final filling that does not require veneering.[30-32] Insufficient data are available regarding the shrinkage stress behavior of these bulk-fill low-shrinkage composites. Consequently, the objective of this *in vitro* study was to investigate the shrinkage stress kinetics and some related properties of this new class of materials. The null hypotheses tested were 1) there is no difference in polymerization shrinkage stress, stress kinetics, flexural strength, or flexural modulus between bulk-fill and a microhybrid resin-composite control; and 2) placing this class of material in 4-mm bulk will not compromise its curing efficiency.

METHODS AND MATERIALS

Shrinkage Stress Test

A tensometer (American Dental Association Foundation, Paffenbarger Research Center, Gaithersburg, MD, USA)[33] was used to measure shrinkage stress of five bulk-fill materials: Surefil SDR flow (SF, Dentsply), Tetric EvoCeram Bulkfil (TE, Ivoclar Vivadent), Venus Bulk Fill (VB, Heraeus Kulzer), x-tra fil (XF, Voco), and experimental bulk fill (FB, 3M ESPE). The methacrylate-based microhybrid composite Filtek Z250 (FZ, 3M ESPE) was used as a control material (Table 1).

Table 1: *Materials Used in This Study*

Code	Product	Type	Manufacturer	Lot No.	Composition	Filler Type	Filler Loading
SF	Surefil SDR flow	Bulk-fill base	Dentsply, Konstanz, Germany	11281	UDMA, TEGDMA, EBPDMA	Barium/strontium-aluminofluoro-borosilicate glass	68% by wt and 44% by vol
VB	Venus Bulk Fill	Bulk-fill base	Heraeus Kulzer, GmbH, Hanau, Germany	010031	UDMA, EBPDMA	Barium alumino-fluorosilicate glass, YbF_3, and SiO_2	65% by wt and 38% by vol
FB	Experimental flowable	Bulk-fill restorative	3M ESPE, St Paul, MN, USA	N310870 (EXD-784)	Bis-GMA, Bis-EMA, Procrylat, UDMA	zirconia/silica ytterbium trifluoride	64.5% by wt and 42.5% by vol
TE	Tetric EvoCeram bulk fill	Bulk-fill restorative	Ivoclar Vivadent, Schaan, Liechtenstein	P72398	Dimethacrylate co-monomers	Barium glass, YbF3, oxides, and prepolymers	80% by wt and 60% by vol
XF	x-tra fil	Bulk-fill restorative	Voco, Cuxhaven, Germany	1147096	MMA, Bis-EMA	Inorganic fillers	75% by wt and 58% by vol
FZ	Filtek Z250	Microhybrid restorative	3M ESPE, St Paul, MN, USA	N222162	Bis-GMA, Bis-EMA, UDMA, and TEGDMA	Zirconia/silica	82% by wt and 60% by vol

MMA: Methyl methacrylate, Bis-GMA: Bisphenolglycidyl dimethacrylate, Bis-EMA: Bisphenol A polyetheylene glycol diether dimethacrylate, TEGDMA: Triethyleneglycol dimethacrylate, UEDMA: Urethane dimethacrylate, EBPDMA: Ethoxylated bisphenol A dimethacrylate.

A 4-mm, prefabricated, nylon gauge block was used to adjust the space between two quartz rods attached to the base and cantilever beam of the machine. The C-factor was calculated to be 0.75 for all specimens. The specimens were cured for 20 seconds through the lower rod using a light-emitting diode curing unit (Demetron A.1, Kerr/Sybron, Orange, CA, USA) in standard mode. Output intensity was confirmed to be 1000 ± 50 mW/cm^2 following every five specimens using a hand-held dental curing radiometer (Demetron LED radiometer Model 100, Demetron Research, Orange, CA, USA). As the composite shrinks, it pulls the two ends of the quartz rods closer together; and as the upper rod moves, the frame and beam bend and the amount of movement is measured with a linear variable differential transformer (LVDT). This measurement was used to calculate a corresponding load from a previous calibration curve. The corresponding shrinkage stress was then obtained by dividing the measured tensile force by the cross-sectional area of the sample. The stiffness of the beam was controlled by the location of the upper collet that holds the quartz rods. The location of the beam and the resulting stiffness was determined from the calibration curve provided by the manufacturer.

The shrinkage stress was monitored continuously for 30 minutes from the start of light activation to yield real-time shrinkage stress during polymerization. The mean maximum shrinkage stress was calculated from five samples for each material (n=5). Using the tensometer software, an average stress curve was generated for each material, and stress rate (first derivative of real-time stress), maximum stress rate R_{max} (peak value in first derivative), and time to achieve maximum stress rate (t_{max}) were calculated.

Flexural Strength and Modulus Test

Flexural strength and modulus were determined using standard procedures in accordance with ISO 4049.[34] Five identical bar-shaped specimens (25 mm in length, 2 mm in width, and 2 mm in height) of each tested material were fabricated using a stainless steel mold. Specimens were light-cured using a light-emitting diode curing unit (Demetron A.1, Kerr/Sybron) at the top and bottom surfaces with a 12-mm diameter curing light tip in standard mode. The central portion of each bar-shaped specimen was subjected to an initial irradiation of 20 seconds. Two subsequent overlapping positions (by half the diameter of the curing light tip) were then irradiated for 20 seconds to cure the entire length of the bar-shaped specimen. Output intensity confirmation was done every five specimens. Specimens were stored for 24 hours in double distilled deionized water at 37°C ± 1°C. Flexural strength and modulus of the material were determined using a universal testing machine (Sintech Renew 1123, MTS, Eden Prairie, MN, USA, with TestWorks 4.08 software) with a 2.5-kg load cell at a crosshead speed of 1 mm/min and a support span length of 20 mm. Flexural modulus (E) was determined using the initial slope of the load displacement curve. Flexural strength (σ) and E

Table 2: *Mean Values and SD for Shrinkage Stress, Maximum Stress Rate (R_{max}), Time to Achieve Maximum Stress Rate (t_{max}), Flexural Strength, Flexural Modulus, Top Hardness, Bottom Hardness, and Bottom/Top Hardness % of the Investigated Materials*[a]

Code	Shrinkage Stress, MPa	R_{max}, MPa/s	t_{max}, s	Flexural Strength, MPa	Flexural Modulus, GPa	Top Hardness, KHN	Bottom Hardness, KHN	Bottom/top, %
VB	1.607 (0.04)^{A}	0.06 (0.01)^{A}	13.30 (1.72)^{A}	107.4 (3.8)^{A}	4.48 (1.08)^{A}	36.60 (0.97)^{A}	35.53 (1.32)^{A}	97.08 (2.31)^{A}
FB	1.649 (0.06)^{B}	0.07 (0.02)^{A}	9.82 (1.87)^{B}	109.8 (9.2)^{A}	5.47 (2.02)^{AB}	39.37 (3.20)^{A}	34.31 (2.60)^{A}	87.21 (3.01)^{BC}
SF	1.710 (0.03)^{BC}	0.08 (0.01)^{A}	8.91 (1.60)^{B}	125.5 (9.4)^{B}	6.76 (1.26)^{B}	48.53 (2.38)^{B}	44.27 (2.78)^{B}	91.31 (5.93)^{AB}
TE	1.883 (0.09)^{C}	0.09 (0.01)^{AB}	9.67 (2.00)^{B}	85.28 (12.8)^{C}	8.98 (3.87)^{C}	55.40 (3.27)^{C}	44.13 (2.26)^{B}	79.73 (2.59)^{CD}
XF	2.135 (0.07)^{D}	0.11 (0.02)^{B}	9.96 (1.35)^{B}	143.7 (8.04)^{D}	16.03 (8.20)^{D}	59.07 (6.37)^{C}	51.20 (7.61)^{C}	86.43 (5.17)^{C}
FZ	2.364 (0.04)^{D}	0.15 (0.02)^{C}	6.54 (1.73)^{C}	143.8 (2.6)^{D}	11.65 (8.32)^{D}	57.40 (1.51)^{C}	43.10 (3.18)^{B}	75.17 (6.57)^{D}

[a] *Within a column, different superscript capital letters indicate significant differences by pair-wise Bonferroni* post-hoc *multiple comparison test (α=0.05).*

were calculated according to equations 1 and 2, respectively:

$$\sigma = \frac{3PL}{2bd^2} \quad (1)$$

$$E = \frac{PL^3}{4bdh^3} \times 10^3 \quad (2)$$

where L is the support span length (mm), b the specimen width (mm), h the specimen height (mm), P the load (N) at a convenient point on the straight line portion of the curve, and d the deflection (mm) corresponding to the load P.

Knoop Hardness Measurement

A stainless steel cylindrical split-mold, with height of 4 mm and internal diameter of 4 mm, was placed on a glass slide covered by a Mylar strip (Hawe Stopstrip Straight, KerrHawe, Bioggio, Switzerland). The mold was then filled in bulk with one of the six resin composites. The top side of the mold was covered with a second Mylar strip, and the resin material made flush with the mold by use of a second glass slide. A standardized 50-g stainless steel weight was placed over the top glass slide for 30 seconds to ensure proper packing of the composite. The second glass slide was removed, and the resin composite was light-cured for 20 seconds using the same curing unit and protocol that was used for the shrinkage stress test. The curing light tip was kept centered and in contact with the second Mylar strip. After light curing, the specimens were removed from the mold and stored for 24 hours in double distilled deionized water at 37°C ± 1°C.

The top and bottom surfaces of each specimen were polished with a diamond suspension (mean grain size: 5 μm) with an automatic polishing device (Buehler, Lake Bluff, IL, USA) under a 50-g load. Cured composite thickness of each specimen was verified with a digital caliper of 0.01 mm precision (Mitutoyo IP 65, Kawasaki, Japan). Knoop microhardness (KHN) of each top and bottom surface was measured (LECO, M-400 Hardness Tester, St Joseph, MI, USA) at three different points on each surface, with a 10 N load for 15 seconds. The average of three readings was calculated for both top and bottom of each specimen, and the bottom to top percentage was calculated from the average of five specimens (n=5).

Statistical Analysis

Multiple comparisons of group means of shrinkage stress, flexural strength, and modulus and microhardness were made utilizing one-way analysis of variance (ANOVA) and Bonferroni multiple range tests at a significance level of α=0.05. Correlation between the shrinkage stress and flexural modulus results was analyzed using regression statistics.

RESULTS

Descriptive statistics are presented in Table 2. Statistical analysis of the shrinkage stress results revealed that the FZ group recorded the highest ($p<0.05$) mean shrinkage stress value (2.364 ± 0.04 MPa) followed by the XF group, then the TE, SF, and FB groups, respectively, while the VB group recorded the lowest shrinkage stress mean value (1.607 ± 0.04 MPa). The difference between groups was statistically significant ($p<0.05$) as indicated with ANOVA. The Bonferroni *post-hoc* test showed that there was a nonsignificant ($p>0.05$) difference in the shrinkage stress results between the VB and FB, VB and SF, and FB and SF groups. Also, there was nonsignificant ($p>0.05$) difference between the SF and TE groups. Figure 1 compares the shrinkage

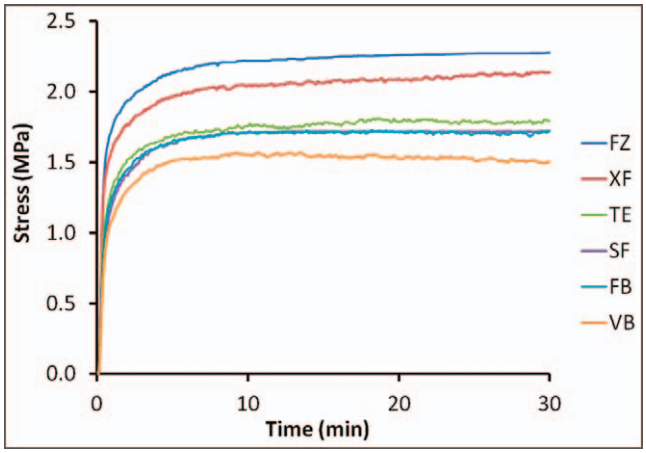

Figure 1. *Comparison of the shrinkage stress development (averaged curves, n=10) as a function of time for the five measured bulk-fill composites and control.*

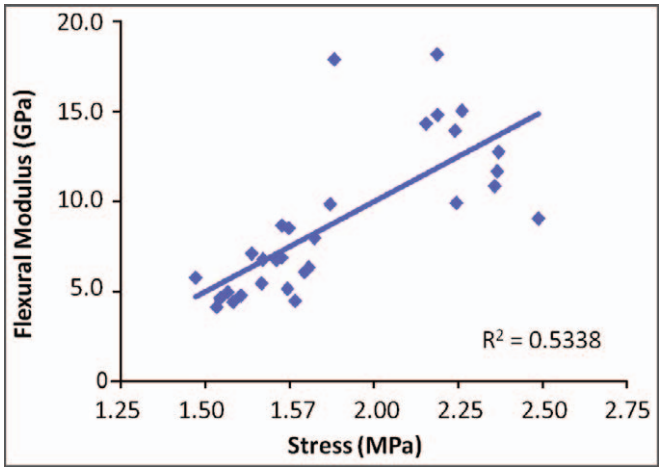

Figure 3. *Linear regression of shrinkage stress vs flexural moduli of the investigated materials.*

stress development as a function of time for the five measured bulk-fill composites and control.

Presentation of the rate of shrinkage stress development of the tested materials is shown in Figure 2. As indicated by ANOVA, the control material FZ showed the highest ($p<0.05$) R_{max} (0.15 ± 0.02 MPa/s) and the shortest t_{max} (6.54 ± 1.73 s), VB exhibited the lowest R_{max} (0.06 ± 0.01 MPa/s) and the longest t_{max} (13.30 ± 1.72 s), which was not significantly different ($p>0.05$) than the recorded R_{max} for FB, SF, and TE (0.07±0.02, 0.08±0.01 and 0.09±0.01 MPa/s respectively). No significant difference was found between R_{max} of TE and XF(0.11±0.02 MPa/s). The recorded t_{max} of FB, SF, TE, and XF were not significantly different ($p>0.05$).

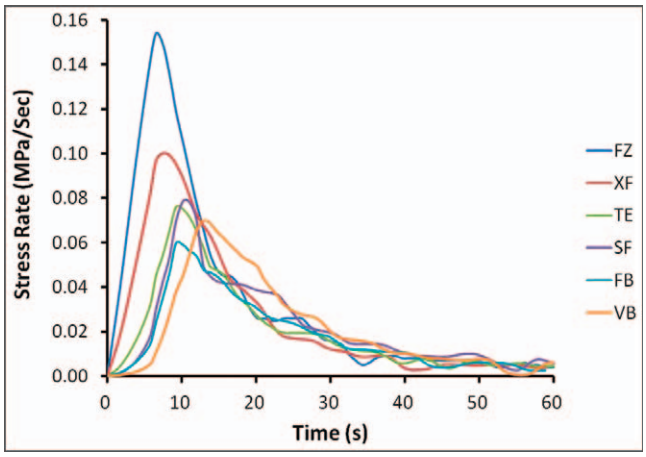

Figure 2. *Shrinkage stress rate development in time within the 60 seconds from composite irradiation, mean curves (n=10) of the investigated materials.*

XF recorded the highest flexural modulus mean value followed by FZ then TE, SF, and FB, respectively, while VB recorded the lowest mean value. A significant difference ($p<0.05$) was found between all groups except between VB and FB, FB and SF, and XF and FZ. Regression analysis revealed a statistically significant ($p=0.0001$) direct correlation between stress and flexural modulus ($r^2=0.5338$; Figure 3).

The top and bottom hardness results varied widely between the tested materials. The highest mean top hardness value was recorded for XF (59.07 ± 6.37 KHN), which was not statistically different ($p>0.05$) from that for TE (55.40 ± 3.27 KHN) and the control material FZ (57.40 ± 1.51 KHN). The lowest mean top hardness value was recorded for VB (36.60 ± 0.97 KHN), which was not statistically different ($p>0.05$) from that for FB (39.37 ± 3.20 KHN).

The results of the polymerization efficiency test at 4-mm depth revealed that VB exhibited the highest bottom/top hardness percentage value (97.08% ± 2.31%) and was significantly higher ($p<0.05$) than all other tested materials except SF (91.31% ± 5.93%). As indicated by the pair-wise Bonferroni *post-hoc* test, no significant difference ($p>0.05$) was found between the bottom/top hardness percentage of FB and SF (87.21% ± 3.01% and 91.31% ± 5.93%, respectively) and between TE and FZ (79.73% ± 2.59% and 75.17% ± 6.57%, respectively); the latter group exhibited the lowest ($p<0.05$) bottom/top hardness percentage. Regression analysis revealed a significant ($p=0.0001$) positive linear correlation between top hardness and stress ($R^2=0.7135$; Figure 4).

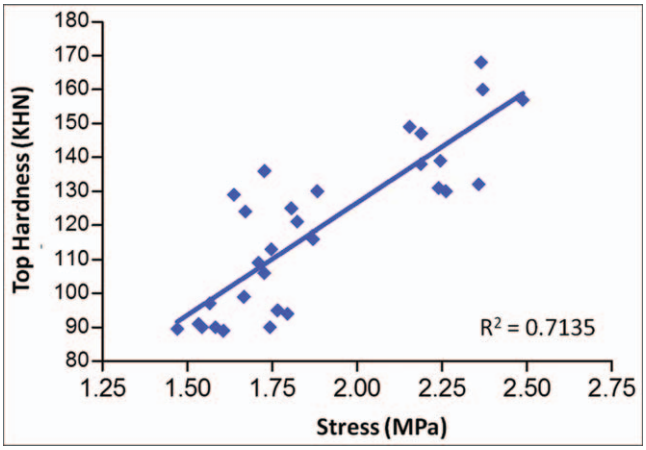

Figure 4. *Linear regression of shrinkage stress vs top hardness of the investigated materials.*

DISCUSSION

This study investigated the shrinkage stress kinetics and related properties of five recently introduced bulk-fill materials and compared them to a hybrid resin composite. The C-factor used in the tensometer was comparatively low, but was standardized for all materials and was the result of placing and curing tested materials in a 4-mm thickness, which might be a significant influencing factor in the shrinkage stress of the bulk-fill material.

Statistically significant differences ($p<0.05$) were found between the shrinkage stress of the studied materials, and accordingly, the first null hypothesis was rejected. On the other hand, there was no statistically significant difference ($p<0.05$) between VB, experimental FB, and SF, which exhibited the lowest shrinkage stress; meanwhile, these materials were significantly different from the bulk-fill materials TE, XF, and control FZ. It is worth mentioning that the setting of this study provided a controlled environment that allows comparing the behavior of materials under such circumstances, but the results may vary under different testing conditions, especially with increasing the C-factor.

VB, FB, and SF bulk-fill materials have the lowest filler volume fraction (between 38% and 44%), and even though their volumetric polymerization shrinkage remains similar to other flowable resin composites (around 3-6 vol%),[35] they were designed to remarkably reduce shrinkage stress.[36] It was suggested that addition of high filler levels to reduce resin volume is not an efficient approach to reduce post-gel shrinkage and polymerization stress;[37] therefore, another approach was used, which was a modified chemistry that slows the rate of polymerization to reduce shrinkage stress. That was supported in the results of this study, as these composites exhibited longer time to achieve maximum stress rate t_{max} (13.30, 9.82 and 8.91 seconds for VB, FB, and SF respectively) in comparison to 6.54 seconds for the control FZ.

For many years, there was a general understanding among clinicians and researchers that placing resin composite in small increments significantly reduced shrinkage stresses.[21,22] This concept was recently questioned by several researchers,[28-30] who recommended the use of larger increments or even bulk filling of the entire cavity with one increment. One main concern raised with using the bulk filling technique, is that the material may suffer from reduced polymerization at the deeper layer of the increment due to light attenuation. Recently introduced bulk-fill materials were claimed to have more potent initiator systems,[38] and most of the products in this class of material are characterized by low viscosity and high translucency.

It has been reported that resin-based filling materials should exhibit a minimum of 80% bottom/top hardness percentage when cured in a 2-mm increment in order to be considered as adequately polymerized.[39] Accordingly, in the current study, a similar percentage at 4-mm depth was considered acceptable curing , and above 90% was considered high curing efficiency. The results revealed that all investigated bulk-fill materials exhibited acceptable to high curing at the deepest portion of a 4-mm increment (bottom/top% $\geq$ 80%). This is in agreement with comparable studies that showed that bulk-fill materials met the requirements stipulated in the ISO 4049 specification, even with a light-curing time as short as 20 seconds.[40,41] Therefore, the second null hypothesis was accepted, and the reduction in polymerization shrinkage stress of the tested bulk-fill materials cannot be attributed to inadequate curing.[42,43]

A significant difference was found between the flexural modulus of the investigated materials ($p<0.05$), which ranged between 4.0 and 9.0 GPa, with VB exhibiting the lowest value (4.48 $\pm$ 1.08 GPa). Meanwhile, the regression analysis revealed a statistically significant correlation between stress and flexural modulus ($r^2=0.5338$) and between stress and filler loading by volume ($R^2=0.7333$). This may confirm that the main contributing factors to the reduced shrinkage of bulk-fill materials are their low flexural modulus and low filler loading. This finding is in agreement with many previous studies.[22] Flexural modulus is a function of many

factors such as filler content, monomer chemistry, monomer structure, filler/matrix interactions, and additives. SF, VB, and FB have similar monomer structures based on UDMA, which is characterized by a significant reduction in polymerization shrinkage and higher molecular weight that contributes to reduced shrinkage stress.

It is important to note that the flexural strength and flexural modulus testing in the current study was done with optimum polymerization settings and did not simulate a gradual decrease of polymerization that might be expected if the molds were 4 mm in thickness. Although flexural properties of resin-based materials would vary according to their curing efficiency when placed in large-increment thicknesses, the tested bulk-fill materials in this study maintained acceptable to high curing efficiency at a 4-mm thickness. Further investigation is required to determine if the modulus decreases dramatically in these materials as has been reported for a nano-hybrid composite.[44]

Regression analysis has also revealed a significant ($p=0.0001$) direct relation between top hardness and stress ($R^2=0.7135$; Figure 4); therefore, it can be noted that shrinkage stress, flexural modulus, and hardness correlate positively and that bulk-fill materials which exhibit lower shrinkage stress are characterized by low hardness and flexural moduli. Accordingly, the intended use of bulk-fill materials may vary from one material to another consistent with their mechanical properties. The results of TE and XF were significantly different ($p<0.05$) from those of VB, FB, and SF. These results were expected due to the difference in clinical application, filler loading (60 and 58 % by volume for TE and XF respectively), and rheologic properties of the two groups of materials; TE and XF are designed to be used as final posterior restorative materials in comparison to the recommended use of VB, FB, and SF as base materials, requiring a covering of universal or posterior resin composite.[45] According to the reported differences in top hardness of the bulk-fill materials in this study, further investigation of the wear behavior of these materials should be conducted and would be of high clinical value.

Although its mechanical properties are close to those of control composites, TE exhibited significantly lower stress values than XF and the control material FZ, and stress values that were not statistically different from those of VB, FB, and SF. Moreover, the top hardness results of TE were significantly higher ($p<0.05$) than all of the tested bulk-fill materials except for XF and the control material FZ. These results indicate that among the investigated materials, TE is the only material that can be considered for use as a posterior final restoration while maintaining good mechanical properties and reduced shrinkage stress.

CONCLUSIONS

The current study showed that in comparison with a conventional posterior resin composite, the distinctive shrinkage stress kinetics, flexural properties, and enhanced initiation systems of only some of the investigated bulk-fill materials resulted in significant reduction in shrinkage stress while maintaining adequate curing at a 4-mm thickness. This may support the intended use of these materials for bulk filling high C-factor and deep cavities.

Acknowledgement

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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. JA Platt was redacted from all review and publication decisions concerning this manuscript.

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Bond Strength of Adhesive Luting Agents to Caries-affected Dentin

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Clinical Relevance

Caries-affected dentin could remain during conservative preparation for indirect restorations. However, the reduced bond strength of adhesive luting agents to this altered substrate might affect the longevity of restorations.

SUMMARY

Introduction: The aim of this study was to evaluate the bond strength of adhesive luting agents to caries-affected dentin (CAD). Methods: Forty human molars were sectioned to create dental slices presenting exposed occlusal dentin. Half of the samples were submitted to eight caries-induction demineralizing/mineralizing cycles. The pH-cycling model consisted of three hours in a demineralizing solution followed by 45 hours of immersion in a mineralizing solution. Dentin hardness was measured before and after the pH cycling. Resin cement cylinders were built up over the dentin surface using RelyX Unicem or RelyX ARC/ Scotchbond Multipurpose Plus. The cement cylinders were submitted to shear load, and the data were analyzed using two-way analysis of variance (ANOVA) and Tukey test ($p<0.05$). Hardness data were also submitted to two-way ANOVA and Tukey test ($p<0.05$). The relationship of hardness vs bond strength was assessed via nonlinear regression analysis. Results: Sound dentin (tested and used in caries induction) showed similar values of hardness and were superior to CAD. Both resin cements showed higher bond strength to sound dentin than to CAD. Independent of substrate, RelyX ARC showed the highest values of bond strength. A positive linear relationship between dentin hardness and bond strength was observed for both cements evaluated. Conclusions: The adhesive luting agents evaluated showed lower bond strength to CAD.

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Introduction

Improvement of adhesive luting systems and ceramic materials has resulted in more conservative preparations for indirect restorations, while the cavity preparation can be limited only to caries removal and eliminating retentive areas. In this approach, the restoration is retained mainly by bonding procedures. The goal of caries excavation is to eliminate the caries-infected dentin, which presents as an irreversibly demineralized and denatured substrate.[1,2] To preserve the dental tissue, the underlying partially preserved and remineralizable dentin (caries-affected dentin

[CAD]) is maintained.[3-5] Significant alterations of mineral content have been described for CAD, where the intertubular dentin presents lower mineral content than normal dentin, and where acid-resistant minerals are observed obliterating the dentin tubules.[6-8] The obliteration of tubules can interfere with resin infiltration, thus preventing tags during bonding procedures.[9] Conversely, the lower mineral content of intertubular dentin in CAD permits deeper etching of this substrate.[10]

Considering the maintenance of CAD during conservative cavity preparations that ensure the receiving of indirect restorations, proper bonding between the restorative material and CAD is fundamental to the success of the procedure. For many years, resin cements were used with adhesive systems to lute metal-free indirect restorations. However, multiple-step protocols of cementation increase the technical sensitivity and clinical time required for the procedure.[11] Furthermore, incompatibility between simplified adhesives and resin cements has been an issue when the chemical activation of cement polymerization is predominant.[12] Thus, simplified luting agents are gaining in popularity in such a context.

Self-adhesive resin cements (SARCs) have been developed to simplify clinical procedures and to overcome the technique sensitivity of multiple-step systems. According to manufacturers, SARCs require no pretreatment of the dental surfaces and are applied in a single clinical step.[13,14] The bonding mechanism of SARCS is attributed to a chemical reaction between phosphate methacrylates and hydroxyapatite as well as to the infiltration of these materials into the tooth tissues.[15,16] Reduced bond strength of adhesive systems to CAD has been demonstrated,[6,8,17] but little information is available about the bonding of SARCs to this clinically relevant substrate. One of the difficulties in evaluating adhesive systems' bonding to CAD involves obtaining a standardized substrate for the bond-strength studies. Thus, the artificial induction of caries has been used to permit the standardization of the bonding substrate.

The aim of this study was to evaluate the bond strengths of a conventional resin cement and a SARC to artificially created CAD. The null hypotheses tested were as follows: 1) no difference exists between the shear bond strengths of the evaluated luting agents to sound and artificially created CAD; 2) no significant correlation exists between the hardness of the substrate and bond strength; and 3) the bond strength of the evaluated resin cements to both substrates is similar.

METHODS AND MATERIALS

Noncarious human third molars stored in 0.05% thymol saline solution for no more than six months were used in this study. The occlusal surfaces were ground flat using 180-, 320-, and 600-grit silicon carbide paper under running water to remove the enamel and expose a flat dentin surface. A section was performed parallel to the occlusal surface and 2 mm below the cement-enamel junction using a water-cooled slow-speed diamond saw (#7020, KG Sorensen, Barueri, Brazil), while the roots were discarded. An adhesive tape (4×5 mm) was bonded over the occlusal dentin followed by the application of two coats of an acid-resistant, fast-drying nail varnish (Colorama Maybelline, São Paulo, Brazil) on all specimen surfaces. After the tape removal, a window of 20 mm^2 was obtained on the occlusal dentin surface.

Ten Vickers hardness indentations were made on the ground surface under a load of 50 g for a 10-second dwell time (HMV-2, Shimadzu, Tokyo, Japan). The Vickers hardness number (VHN, kgf/mm^2) for each specimen was recorded as the average of the 10 readings. Specimens that presented VHNs that differed by more than 5% from the mean of all specimens were discarded. Forty specimens were selected, and half were submitted to caries-induction demineralization/mineralization cycles. The demineralization/mineralization cycle was characterized by a three-hour immersion of specimens in a demineralizing solution (156.25 mL/tooth) followed by a 45-hour immersion in a mineralizing solution (78.125 mL/tooth). Specimens were submitted to eight cycles of 48 hours; the demineralizing solution was renewed after the fourth cycle, and the mineralizing solution was renewed before the beginning of each new cycle.[17] The compositions of the solutions are listed in Table 1. After demineralization/ mineralization cycling, new Vickers hardness readings were performed on the dentin surfaces of the cycled specimens. Hardness data were analyzed by two-way analysis of variance (ANOVA) and Tukey test ($p<0.05$).

A conventional RelyX ARC (3M ESPE, St Paul, MN, USA) and a self-adhesive RelyX Unicem (3M ESPE) were used in this study. For the RelyX ARC, the adhesive system (Scotchbond Multipurpose Plus, 3M ESPE) was applied over the dentin surface (sound and CAD) before insertion of cement. Thus, the substrate was etched with 35% phosphoric acid

Table 1: *Composition of Solutions Used to Induce Caries*

Solution	Composition
Demineralizing (pH = 4.5)	2.2 mM calcium ($CaCl_2$)
	2.2 mM phosphate (NaH_2PO_4)
	0.05M sodium acetate
	0.05M acetic acid
	1 ppm fluoride (NaF)
Mineralizing (pH = 7.0)	1.5 mM calcium ($CaCl_2$)
	0.9 mM phosphate (NaH_2PO_4)
	0.15M KCl
	0.1M Tris buffer
	10 ppm fluoride (NaF)

(Scotchbond etchant, 3M ESPE) for 15 seconds followed by rinsing of acid. Excess water was removed with absorbent paper, and the adhesive system primer was applied over the wet dentin. After five seconds, the primer solvent was volatilized with a gentle air stream. The adhesive was applied and light-cured for 25 seconds. No previous treatment of the substrate was performed for RelyX Unicem.

Polyvinyl siloxane molds (Aquasil Extra Low Viscosity, Dentsply DeTrey, Konstanz, Germany) containing four cylindrical cavities (1 mm in diameter, 2 mm high) were placed on the dentin surface. The mold cavities were filled with one of the cements, which were light-cured for 20 seconds. All light curings were performed using a light-emitting diode unit (Radii Cal, SDI, Bayswater, Victoria, Australia) with an irradiance of 800 mW/cm^2.

After 24 hours of storage (37°C at 100% humidity), shear bond tests were conducted using a universal testing machine (Instron 5565, Instron, Canton, MA, USA). A thin steel wire (0.2 mm in diameter) was looped around each cylinder, and a shear load was applied to the base of the cylinder at a crosshead speed of 0.5 mm/minute until failure. The average load at failure of the four cylinders was recorded as the microshear bond strength (MPa) for that specimen. Bond strength data were analyzed using two-way ANOVA and Tukey test. The factors evaluated were "cement" and "dentin." The relationship between hardness and bond strength was assessed by nonlinear regression analyses, with bond strength as a dependent variable. All analyses were conducted at a significance level of $p<0.05$.

RESULTS

For the hardness data, ANOVA did not show a significant effect for the factor "cement" ($p=0.232$) and for the interaction between the factors ($p=0.429$). These results showed that both cements evaluated were used in similar dentin. Only the factor "dentin" showed a significant effect ($p<0.001$). The dentin used for caries induction and those tested as sound dentin showed similar hardness values that were higher than those observed for CAD. The results are displayed in Table 2.

Table 2: *Means (SD) of Hardness in VHN*[a]

Resin Cement	Dentin		
	Sound	Moment of CAD Induction	
		Before	After
RelyX ARC	49.0 (1.2)	51.7 (3.3)	15.5 (3.5)
RelyX Unicem	49.6 (3.0)	49.5 (3.4)	13.7 (3.1)
Pooled average	49.3 (2.2)^A	50.6 (3.4)^A	14.6 (3.3)^B

[a] *For pooled average, distinct letters indicate statistical difference (p<0.05).*

For the bond strength data, ANOVA showed a significant effect for the factors "cement" ($p=0.002$) and "dentin" ($p<0.001$) but not for the interaction between the factors ($p=0.209$). The cement RelyX ARC showed higher bond strength than RelyX Unicem did for both substrates. Independent of cement, the highest bond strength values were observed for sound dentin. The results are displayed in Table 3. The relationship between hardness and bond strength followed a significant positive linear behavior for both RelyX ARC ($R^2 = 0.80$; $p<0.001$) and RelyX Unicem ($R^2 = 0.89$; $p<0.001$). A predominance of adhesive failures was evident.

DISCUSSION

Despite the importance of CAD in restorative procedures, most studies that evaluate the bond strength of adhesive materials to dental substrates are performed using sound dentin. The standardization of CAD *in vitro* studies is difficult to determine, and differences in the caries removal and/or caries detection methods can compromise the comparison of results obtained in different studies.[18-20] Even when using proper methods for detecting and removing caries, a heterogeneous substrate can remain after removal of the caries-infected dentin.[21] In some cases, the presence of small sites of CAD surrounded by sound dentin has been observed. Because the sizes and shapes of these sites are often irregular,[22] standardizing the sample preparation for bonding tests is too difficult.

This study artificially induced CAD through demineralizing/mineralizing cycling. This method has been previously described and results in a substrate with similar mineral content to that

Table 3: *Means (SD) for Bond Strength in MPa*[a]

Resin Cement	Dentin		Pooled Average
	Sound	CAD	
RelyX ARC	23.6 (6.8)	8.5 (2.9)	16.0 (9.3) A
RelyX Unicem	16.0 (3.7)	5.0 (1.8)	10.5 (6.4) B
Pooled average	19.8 (6.6) a	6.7 (2.9) b	

[a] *For pooled average, distinct letters (uppercase for cement, lowercase for dentin) indicate statistical difference ($p<0.05$).*

observed in natural CAD.[2,8,17] Natural CAD presents as a hypomineralized tissue because of the cycles of demineralization and remineralization over a long period.[8] This process results in dissolution of mineral content, which precipitates into dentinal tubules, thus creating crystal logs.[7,23] In contrast, it has been demonstrated that short periods of artificial caries induction do not permit the formation of crystallites in the dentinal tubules.[8] Despite this difference, similar values of bond strength between natural and artificial CAD have been reported, though a lower variability of bond strength was observed for artificially induced CAD.[8]

In the present study, the hardness of the evaluated dentin substrate was measured before the induction of CAD. The sound dentin and the dentin used for caries induction showed similar dentin hardness values, demonstrating homogeneity in mineral content between the samples used. Meanwhile, the samples submitted to demineralization/remineralization cycling presented the lowest values of bond strength. These results were expected based on the longer time the samples spent in the demineralization solution. Reduction of dentin hardness also resulted in lower bond-strength values for both adhesive luting agents, and a positive significant correlation was observed between these two measurements. Thus, the first and second hypotheses of the study were rejected.

RelyX ARC was used in the present study, associated with the three-step etch-and-rinse adhesive Scotchbond Multipurpose Plus. Previous studies have also demonstrated reduced bond strength of etch-and-rinse adhesives to CAD when compared with their bond strength to sound dentin.[6,8,17] The presence of crystals in the dentin tubules has explained this reduction, as they reduce the extension of the resin tags.[6] Despite the fact that the artificially induced CAD does not present these crystals, the presence of tags in the dentinal tubules is weakly related to bond strength. Thus, the deeper demineralization that phosphoric acid promotes on more porous CAD can explain the results.[10] A higher discrepancy is expected between the depth of dentin demineralization and adhesive resin infiltration for CAD, resulting in a thicker layer of an unprotected mineral-depleted collagen at the base of the hybrid layer.[24,25] This mineral-depleted layer can act as weak link during shear testing and therefore reduce the bond strength.[25]

Previous studies showed the absence of resin tags and evident dentin demineralization for the self-adhesive resin cement RelyX Unicem when applied on sound dentin.[26,27] The high viscosity and neutralization effect during the setting of this material have been related to this limited capacity to effectively diffuse and decalcify the dentin.[26] Thus, the main bonding mechanism of RelyX Unicem to dentin is probably due to a chemical reaction between the phosphate methacrylates and hydroxyapatite.[15] Considering the importance of the presence of calcium for the proper boding of Unicem to dentin, the lower mineral content of CAD can explain the results. Independent of the substrate evaluated, ARC showed higher bond strength did than Unicem, rejecting the study's third hypothesis. The formation of an effective hybrid layer for ARC, different from that of Unicem, can explain these results.

This study showed that the reduction in hardness that artificial caries induction promotes resulted in the lowest bond strength values for a conventional resin cement and a SARC. The pH-cycling method used in this study simulates *in vivo* physicochemical variations involved in the caries progression. However, the model of artificial caries induction used in this study presents some limitations. In addition to the absence of crystals in dentinal tubules when this method of caries induction is used, the proteinase releases of the microorganisms have an important role in collagen degradation.[28,29] Considering the importance of these collagen alterations and the standardization of the bonding substrate, the use of a microbiologic model for caries induction can produce an ideal model for evaluating bonding to CAD. Thus, further studies for evaluating the bond strength of a SARC to CAD must be conducted to confirm the present study's outcomes.

CONCLUSIONS

Within the current study's limitations, the following conclusions can be made:

- The artificial CAD showed lower hardness than did sound dentin.
- Both evaluated adhesive luting agents showed lower bond strength to CAD than did sound dentin.
- Independent of the substrate, RelyX ARC showed higher values of bond strength than did RelyX Unicem.

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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Light-curing Time and Aging Effects on the Nanomechanical Properties of Methacrylate- and Silorane-based Restorations

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Clinical Relevance

The methacrylate-based restorative system showed higher nanomechanical properties over a silorane-based system, predicting better clinical longevity.

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SUMMARY

The aim of this study was to assess the influence of light-curing time on the nanohardness (H) and reduced elastic modulus (E_r) of components (underlying dentin, hybrid layer, adhesive, and composite) of methacrylate- and silorane-based restorations after 24 hours and six months of storage. Class II slot preparations were carried out in human molars (n=3) and restored with methacrylate (Clearfil SE Bond [Kuraray] + Filtek Z250 [3M ESPE]) or silorane (LS restorative system [3M ESPE]) restorative systems and light-cured using light-emitting diode at 1390 mW/cm^2 for the recommended manufacturers' time or double time. Restorations were sectioned, and bonded dentin-resin interfaces were embedded in epoxy resin and polished for evaluation with a Berkovich fluid cell tip (TI 700 Ubi-1 nanoindenter, Hysitron). Data were statistically analyzed by analysis of variance and Tukey's test (alpha=0.05). Overall, the H and E_r values

were higher for methacrylate-based restorations than for silorane materials ($p \leq 0.05$), an increase in curing time did not improve the H and E_r of the bonded interface components of either material ($p > 0.05$), and aging significantly decreased the mechanical properties of interface components of both resin-based restorative systems ($p \leq 0.05$). In general, nanomechanical properties decreased after six months of storage, the methacrylate restorative system exhibited higher H and E_r than silorane, and light-curing time did not influence the properties tested.

INTRODUCTION

Most current dental composite resins are methacrylate-based monomers, such as bisphenol-A glycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA), and triethylene glycol dimethacrylate (TEGDMA), with well-known high volumetric shrinkage[1] ranging from 1.9 to 3.5 vol%.[2] Light-cured restorative materials exhibit a significant percentage of unreacted methacrylate groups due to incomplete conversion of the C=C bonds.[3] However, a higher monomer conversion rate results in greater shrinkage strain.[4] Polymerization stress can result in many adverse effects, such as cuspal deflection,[5] debonding at the adhesive interface, postoperative sensitivity,[6,7] microleakage,[6] secondary caries, marginal discoloration, and restoration and dental fractures.[7]

A low-shrinkage monomer, silorane, was developed from the reaction of oxirane and siloxane molecules.[5,8,9] Methacrylate polymerizes by the free-radical cure, while silorane chemistry presents a cationic ring-opening polymerization mechanism,[5,8] and more light-curing time is needed to initiate the polymerization reaction by cation formation.[1,5,10] Silorane exhibits low polymerization shrinkage,[5,8,11] less than 1 vol%,[2] and the mechanical properties are comparable to those of conventional methacrylate dental composites.[9] Silorane composite is used with a dedicated two-step self-etch adhesive system; both primer and bond agents are light-cured, creating distinct layers.[1]

During restorative procedures, the distance between the guide tip of the light-curing unit and the resinous material surface in deep cavities reduces the irradiance that reaches the restorative material and decreases the monomer conversion and physical properties.[12] The increase in curing time improves monomer conversion into polymers, thereby improving the physical properties of the material.[4,13,14]

Nanoindentation is a reliable technique to obtain site specific nanohardness (H) and reduced elastic modulus (*Er*) of the resin-dentin interface components.[15] This technique would allow users to determine how extended light-curing time affects the properties of individual components of the resin-dentin bonded interface of different resin-based dental restorative materials.

The objective of this study was to evaluate the influence of different monomers and curing time on the H and *Er* of resin-dentin bonded interface components after 24 hours and six months of water storage. The null hypotheses tested were that (1) there would be no difference between the two distinct resin-based materials, (2) there would be no difference in how the light-curing times affect H and E_r, and (3) the aging would not affect the mechanical properties tested.

METHODS AND MATERIALS

The local Institutional Review Board approved the study protocol (#031/2010) for use of extracted human molars. Twelve freshly noncarious, unrestored human molars were collected and stored in 0.1% thymol solution at 4°C. The teeth were cleaned, stored in distilled water at 4°C, and used within three months after extraction.

The roots were embedded in polystyrene resin (Piraglass, Piracicaba, Brazil), and the occlusal surfaces were ground with 320-grit silicon carbide (SiC) grinding paper (CarbiMet 2 Abrasive Discs, Buehler Inc, Lake Bluff, IL, USA) under running water (APL-4, Arotec, São Paulo, SP, Brazil) until the distance between the occlusal surface and the cementum-enamel junction was 5 mm. Standardized Class II slot preparations with cervical margins in root dentin/cementum were performed in one of the interproximal surfaces of each molar using a high-speed diamond bur (No. 3100, KG Sorensen, Barueri, SP, Brazil) under water spray to final dimensions of 4 mm width, 6 mm height (1 mm below the cementum-enamel junction), and 2 mm axial depth. A custom-made preparation device allowed the standardization of the cavity dimensions. Each bur was used to cut three preparations.

The composition of the restorative materials is shown in Table 1. Two composite resins (Filtek Z250 and Filtek LS) and two self-etching adhesive systems (Clearfil SE Bond and Filtek LS Adhesive) were used to restore the cavities.

Table 1: *Materials Used*

Material	Batch Number	Composition (According to Manufacturer)
Clearfil SE Bond (Kuraray Medical Inc, Okayama, Japan)	Lot 00955A Primer	MDP, HEMA, water, CQ, hydrophilic dimethacrylate.
	Lot 01416A Bond	MDP, Bis-GMA, HEMA, CQ, hydrophobic dimethacrylate, N,N-diethanol p-toluidine, colloidal silica.
Filtek LS adhesive (3M ESPE, Seefeld, Germany)	Lot 9BN Primer	Bis-GMA, HEMA, water, ethanol, silica treated silica filler, CQ, phosphoric acid-methacryloxy-hexylesters mixture, phosphorylated methacrylates, copolymer of acryl and itaconic acid, phosphine oxide.
	Lot 9BK Bond	Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane treated silica, CQ, stabilizers.
Filtek Z250 (A2 shade; 3M ESPE, St Paul)	Lot N144001BR	Filler: 60 vol%, aluminum oxide, silica, and zirconium oxide (0.01-3.5 μm).
		Resin: Bis-GMA, Bis-EMA, and UDMA.
Filtek LS composite (A2 shade; 3M ESPE, St Paul)	Lot N183458	Filler: 55 vol%, silica, and yttrium trifluoride (0.04-1.7 μm).
		Resin: Bis-3,4-Epoxycyclohexylethyl-Phenyl-Methylsilane and 3,4-Epoxycyclohexylcyclopolymethylsiloxane.

Abbreviations: Bis-EMA, ethoxylated bisphenol-A dimethacrylate; Bis-GMA: bisphenol-A glycidyl dimethacrylate; CQ, camphorquinone; HEMA, 2-hydroxyethylmethacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

The preparations were randomized into four treatments, and nanoindentation measurements were performed after 24 hours and six months of storage on the same specimen, for a total of eight experimental groups (n=3) (Table 2). Restoration procedures were carried out using the following protocols. For the methacrylate groups, Clearfil SE Bond primer (bottle A) was first vigorously scrubbed with applicator brushes for 20 seconds; a mild air stream was applied for solvent volatilization; then a coat of adhesive resin (bottle B) was applied, followed by a gentle air stream, and light-curing for 10 seconds (G1 and G5) or 20 seconds (G2 and G6). For the silorane groups, Filtek LS Adhesive primer (bottle 1) was actively applied for 15 seconds, a mild air stream was applied, and then it was cured for 10 seconds (G3 and G7) or 20 seconds (G4 and G8). Afterward, adhesive resin (bottle 2) was applied, followed by a gentle air stream, and light-curing for 10 seconds or 20 seconds. The composite resins were incrementally placed using three horizontal layers (each approximately 2 mm) and light-cured for 20 seconds or 40 seconds, according to experimental groups (Table 2).

Resin-based materials were light-cured from the occlusal surface using a second-generation light-emitting diode (LED) unit (Bluephase 16i, Vivadent, Bürs, Austria) at 1390 mW/cm^2 of irradiance (at 0 mm). The output light power (mW) was measured with a power meter (Ophir Optronics, Har-Hotzvim, Jerusalem, Israel). The tip diameter was measured with a digital caliper (Mitutoyo Sul Americana, Suzano, SP, Brazil) and recorded as 7 mm; the tip area was determined in centimeters squared. Irradiance (mW/cm^2) was calculated by dividing the output light power by the tip area. Irradiance was also calculated by positioning a spacer device (with heights of 4 mm and 6 mm) between the light guide tip of the curing unit and the surface of the power meter as well as beneath the resin disks for both composites (2 mm thick, simulating the first increment) and 4 mm from the top surface of the resin disk. The distance between the tip of the light-curing unit and the bottom of the cavity was 6 mm, resulting in 610 mW/cm^2 of irradiance. The increments of composite were approximately 2 mm thick, the total irradiance of the top surface of the first composite increment was 990 mW/cm^2 (4 mm distance between the tip of the light-curing device and the top surface of the first composite increment). The irradiance at the bottom surface at 6 mm (beneath the composite resin at 2 mm thickness) was 380 ± 5 mW/cm^2.

The restoration was cut in half, and one of the halves was embedded in epoxy resin (Buehler Inc) and polished manually with 800-, 1000-, and 1200-grit SiC grinding paper (CarbiMet 2 Abrasive Discs, Buehler Inc) under running water. A standardized metallographic polishing technique was used, the specimens were polished to a mirrorlike finish with polycrystalline diamond suspensions of grades 9, 6, 3, and 1 μm (MetaDi Supreme, Buehler Inc), and 0.05 μm alumina suspension polish (MasterPrep, Buehler Inc) on soft polishing pads (MicroCloth, Buehler Inc). Between each polishing stage, specimens were cleaned using an ultrasonic cleaner (CD-

Table 2: *Experimental Groups*

Group	Restorative System	Light-curing Time[a]	Water Storage
G1	Methacrylate	As recommended by the manufacturers	24 h
G2	Methacrylate	Double time	24 h
G3	Silorane	As recommended by the manufacturer	24 h
G4	Silorane	Double time	24 h
G5	Methacrylate	As recommended by the manufacturers	6 mo
G6	Methacrylate	Double time	6 mo
G7	Silorane	As recommended by the manufacturer	6 mo
G8	Silorane	Double time	6 mo

[a] *Double time was 10 s or 20 s for the adhesive system and 20 s or 40 s for the composite resin.*

4800, Practical Systems Inc., Odessa, FL, USA) with distilled water for five minutes.

Nanoindentation measurements were performed in the restoration components with a Berkovich fluid tip attached to a TI 700 Ubi-1 nanoindenter (Hysitron Inc, Minneapolis, MN, USA). The bottom of the block of epoxy resin was fixed on a metal disc and stabilized on the equipment platform with magnets. Wax was placed around the boundaries of the block and filled with Hanks' balanced salt solution (BioWhittaker, Lonza Walkersville Inc, Walkersville, MD, USA) to keep the specimen hydrated throughout the testing procedure.

Three regions were selected visually using an optical microscope coupled to the equipment. A three-axis piezo scanner (TriboScan, Hysitron Inc) was attached to the microscope to control the tip positioning and the *in situ* scanning probe imaging; the load-displacement transducer with a probe attached was used to indent the specimen while collecting the load-displacement data. Figure 1 shows the four aggregated images, two topography forward images of the silorane and two gradient forward imaging modes of the methacrylate restorative system. The specimens were stored in Hank's solution for six months at 37°C, changed weekly,[16] and remeasured.

An elongated pyramidal Berkovich fluid diamond tip (curvature radius ≈ 100 nm, Hysitron Inc) was used for imaging and testing on the hydrated samples. At each of the three regions previously selected with the optical microscope, three indentations were made with maximum load values of 100 μN for the hybrid layer and 1200 μN for the other components of the restoration (dentin, layer of the adhesive, and composite) under a standard trapezoidal load function of 10-40-10 seconds. Nine indentations were made in each component per specimen (n=3). The indentation load-displacement data collected were used to calculate the H and E_r by the TriboScope software (version 8.2.0.14, Hysitron Inc), using the Oliver-Pharr method.[17] The H and E_r averages of the nine measurements were used to determine the individual properties of the dentin, hybrid layer, adhesive, and composite resin of each specimen.

Nanomechanical properties readings after 24 hours and six months were performed in the same specimen, and proc-mixed analysis of variance (ANOVA) was used for repeated measures. The H and E_r data for dentin, hybrid layer, adhesive, and composite were analyzed by three-way ANOVA (factors were material, curing time, and aging) and Tukey's test (alpha=0.05). The H and E_r of the bond layer of the Filtek LS System Adhesive were subjected to two-way ANOVA (factors were curing time and aging) and Tukey's test at a preset alpha of 0.05; because this layer was only for the silorane adhesive system, silorane- and methacrylate-based materials were not compared.

RESULTS

ANOVA showed no interaction for any factor studied ($p>0.05$). The H and *Er* data are exhibited in Tables 3 and 4, respectively.

The H and E_r values of the methacrylate resin systems were higher than those for the silorane-based materials ($p\leq0.05$), except for the *Er* of the adhesive at 24 hours for both curing times ($p>0.05$). Mechanical properties of the underlying intertubular dentin were not influenced by the material or curing time ($p>0.05$), and it decreased over time ($p\leq0.05$).

The greater light-curing time did not improve the H and E_r of the materials ($p>0.05$). The six months of storage aging decreased the H and E_r values of all dentin-resin interface components ($p\leq0.05$), except for the E_r of the adhesive ($p>0.05$). The H and E_r of

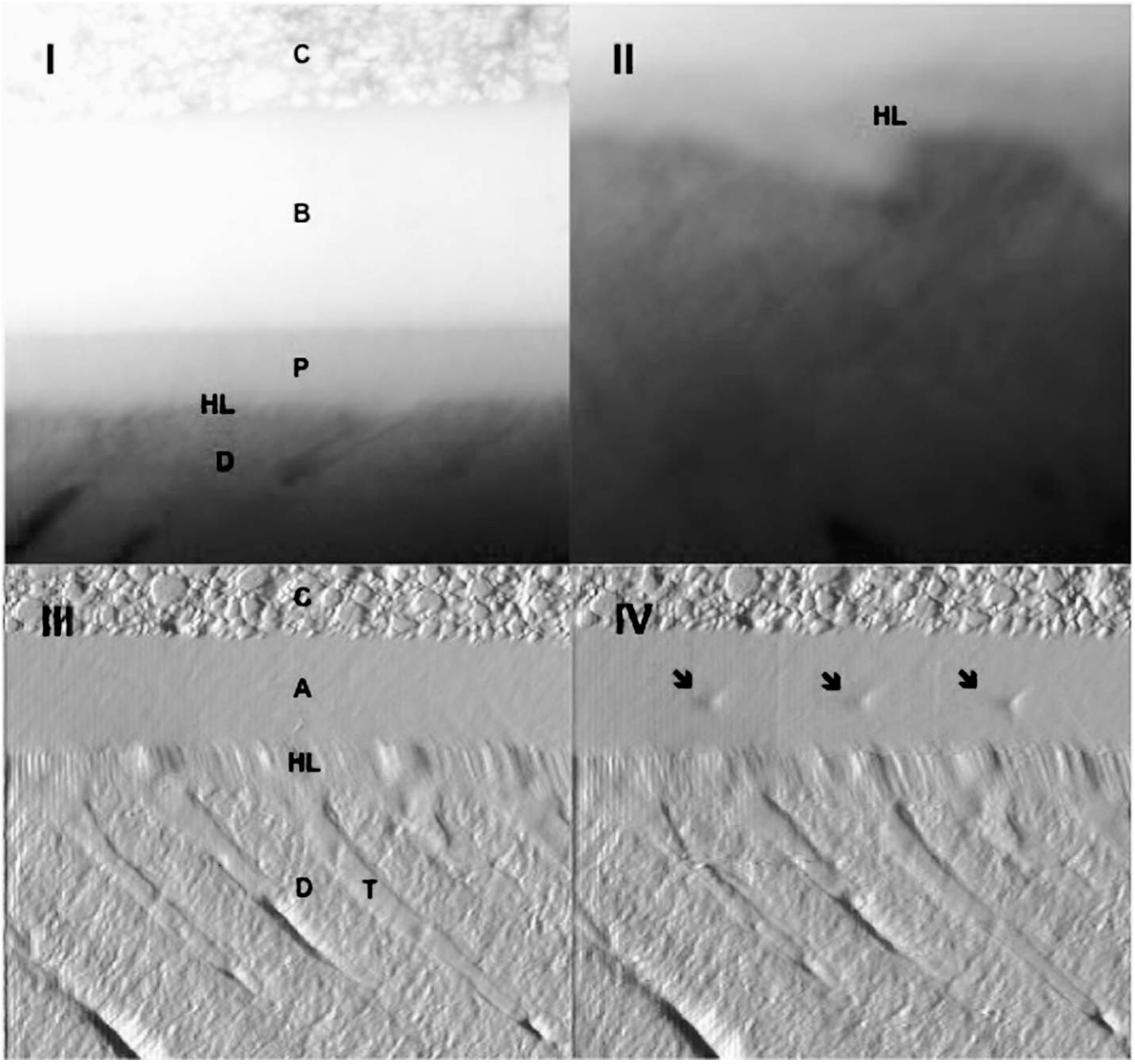

Figure 1. *Representative scanning probe microscopy images obtained by the TriboScanner. (C) Composite resin, (B) Layer of bond agent of the silorane adhesive, (P) Layer of primer of the silorane adhesive, (A) Layer of adhesive of the Clearfil SE Bond, (HL) Hybrid layer, (D) Dentin, (T) Resin tag. (I) Silorane group: topography image mode, scan size 40 × 40 μm. (II) Scan size 20 × 20 μm. (III) Methacrylate group: gradient image mode, scan size 40 × 40 μm. (IV) Indentations performed at the layer of adhesive (arrows).*

the resin composites were not affected by the aging factor ($p>0.05$).

DISCUSSION

Longevity of adhesive restorations is dependent on adequate and stable adhesion of the restorative materials to dental hard tissues over time. The first null hypothesis tested was rejected; the methacrylate restorative system showed higher nanomechanical properties than the silorane system, except for the E_r of the adhesive at 24 hours. Self-etch adhesives are less technique sensitive because there are no rinsing and drying steps; this maintains the ideal dentin humidity and reduces the risk of inaccuracies during application.[18] Two-step self-etch

Table 3: *Nanohardness (H [MPa]) of the Restoration Components According to Light-curing Time, Restorative System, and Aging*

Restoration Component	Light-curing Time	Restorative System	Aging[a]	
			24 h	6 mo
Dentin	Recommended	Methacrylate	1289.63 (125.56) A	959.96 (48.78) B
		Silorane	1390.26 (122.44) A	1002.69 (51.63) B
	Double time	Methacrylate	1301.85 (104.63) A	1030.12 (138.79) B
		Silorane	1290.56 (112.23) A	993.80 (100.44) B
Hybrid layer	Recommended	Methacrylate	379.18 (106.38) Aa	126.56 (47.62) Ba
		Silorane	252.94 (97.36) Ab	89.02 (13.71) Bb
	Double time	Methacrylate	383.15 (40.25) Aa	292.96 (33.23) Ba
		Silorane	327.26 (124.05) Ab	97.75 (23.67) Bb
Adhesive	Recommended	Methacrylate	258.41 (82.29) Aa	186.74 (19.12) Ba
		Silorane	191.62 (34.85) Ab	108.38 (14.32) Bb
	Double time	Methacrylate	282.55 (43.85) Aa	211.21 (26.91) Ba
		Silorane	204.95 (32.06) Ab	137.65 (10.67) Bb
Bond agent	Recommended	Silorane	338.25 (27.41) A	270.45 (4.21) B
	Double time	Silorane	332.23 (25.41) A	296.73 (19.17) B
Composite resin	Recommended	Methacrylate	1108.90 (231.02) a	977.41 (23.95) a
		Silorane	777.16 (62.87) b	638.25 (65.45) b
	Double time	Methacrylate	1123.62 (155.86) a	1089.33 (31.00) a
		Silorane	793.06 (50.74) b	750.52 (74.76) b

[a] *Distinct letters (capital in the row and lowercase in the column) for each restoration component are statistically different at* $p \leq 0.05$.

adhesive contains an acid primer that demineralizes and penetrates monomers into the dentin subsurface simultaneously; this is followed by application of a solvent-free hydrophobic resin to improve the mechanical properties.[19] One-step self-etch adhesives contain a mixture of acid, organic solvents, water, and hydrophilic and hydrophobic monomers in a single bottle.[19]

Clearfil SE Bond consists of a weakly acidic and hydrophilic self-etch primer and a solvent-free viscous hydrophobic resin coating layer that can increase the mechanical properties due to highly cross-linking monomers.[19] A specific two-step self-etch adhesive (Filtek LS System Adhesive) was formulated for silorane low-shrinkage composite. The hydrophilic and solvated self-etch LS primer is applied and light-cured, creating the hybrid layer (Fig. 1-II).[1,20] Then, LS bond is applied as a low-viscosity bifunctional hydrophobic monomer (phosphorylated methacrylate) and also light-cured, creating distinct layers (primer and bond, Fig. 1-I). It reacts with methacrylate by the acrylate groups and with oxirane by the phosphate groups.[11] So LS primer represents a one-step self-etch adhesive.[1,20] The solvated adhesives, such as silorane adhesive primer, have been related to poor mechanical properties, despite the improvement in degree of conversion (DC) from the increased mobility of the molecules over solvent-free adhesives.[21] Thus, the resin bond component of the methacrylate adhesive applied after the primer may exhibit better mechanical behavior, as demonstrated by the higher H and E_r of the hybrid layer and adhesive, compared with the silorane bonding system.

Sufficient cations are necessary to initiate the cationic ring-opening polymerization of the silorane composite; the onset of this reaction is slow, and additional light-curing is required compared with the free-radical cure of the methacrylate monomers into a polymer network.[1,5,10] Higher DC, Knoop microhardness, and depth of cure were found for methacrylate compared with the silorane composite.[22] Thus, the superior physical properties of the methacrylate-based composite probably resulted in higher H and E_r values than silorane.

The second null hypothesis was accepted as the light-curing time did not influence the H and E_r values of the restorative systems. Improvements in the physical properties of resin-based materials have been related to increased curing times because of the higher DC.[4,13,14,18] Light intensity is reduced approximately 10% by interposing 1 mm of air between the guide tip of the light-curing unit and the

Table 4: *Reduced Elastic Modulus (E_r [GPa]) of the Restoration Components According to Light-curing Time, Restorative System, and Aging*

Restoration Component	Light-curing Time	Restorative System	Aging[a]	
			24 h	6 mo
Dentin	Recommended	Methacrylate	23.73 (2.86) A	20.28 (1.61) B
		Silorane	23.83 (1.71) A	20.70 (0.82) B
	Double time	Methacrylate	24.37 (1.88) A	20.34 (2.67) B
		Silorane	24.11 (1.54) A	20.78 (0.90) B
Hybrid layer	Recommended	Methacrylate	6.12 (1.49) Aa	2.40 (0.72) Ba
		Silorane	5.01 (1.51) Ab	1.54 (0.15) Bb
	Double time	Methacrylate	6.08 (0.19) Aa	4.90 (0.67) Ba
		Silorane	5.33 (1.57) Ab	1.90 (0.48) Bb
Adhesive	Recommended	Methacrylate	5.65 (2.07) a	6.18 (0.91) a
		Silorane	4.21 (0.65) a	2.36 (0.20) b
	Double time	Methacrylate	5.89 (0.92) a	5.35 (0.58) a
		Silorane	3.90 (0.70) a	2.80 (0.14) b
Bond agent	Recommended	Silorane	5.29 (0.36) A	4.55 (0.04) B
	Double time	Silorane	5.56 (0.19) A	4.89 (0.30) B
Composite resin	Recommended	Methacrylate	16.79 (2.93) a	15.59 (0.80) a
		Silorane	13.16 (1.33) b	12.53 (1.41) b
	Double time	Methacrylate	16.63 (1.67) a	17.72 (0.98) a
		Silorane	13.80 (0.95) b	12.45 (1.48) b

[a] *Distinct letters (capital in the row and lowercase in the column) for each restoration component are statistically different at* $p \leq 0.05$.

material surface irradiated.[23] Special care should be taken in deep cavities and when the curing unit is at low light power during the polymerization of resinous materials. A light-curing time of 40 seconds is recommended for silorane composite resin using quartz-tungsten-halogen with irradiance between 500 and 1400 mW/cm^2 and for LEDs with output between 500 and 1000 mW/cm^2. For LEDs with irradiance between 1000 and 1500 mW/cm^2, a light-curing time of 20 seconds is recommended. An irradiation of 10 seconds is recommended to cure the primer and bond of silorane system adhesive, without a specific recommendation of minimum irradiance. In this study, a high light power LED of 1390 mW/cm^2 was used, indicating 20 seconds and 10 seconds of light polymerization for composite and adhesive, respectively. However, the irradiance achieved on the surface of the first composite increment was 990 mW/cm^2 at 4 mm from the guide tip and 610 mW/cm^2 at 6 mm for the adhesive system.

The second-generation LED unit used exhibits a narrow spectrum (between 410 and 530 nm, with a peak on the curve at 454 nm), which includes the maximum energy absorption peak of camphorquinone at 468 nm, which absorbs wavelengths from 360 to 510 nm.[24] However, the extended curing time available for light polymerization did not improve the H and E_r of the materials, perhaps because the high light power was sufficient to form more cross-linked polymers, which are less susceptible to degradation than linear polymers,[3] but results in the deceleration of the polymerization reaction and in limits on the conversion rate.[25]

The third null hypothesis was rejected because the long-term storage decreased the mechanical properties of most resin-dentin interface components. Bonding interface components can be degraded by hydrolysis; the water sorption results in polymer plasticization by swelling and reducing the frictional forces between the polymer chains, thereby decreasing the mechanical properties.[18]

The 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer contained in the methacrylate adhesive tested in contact with the dental tissues form the MDP-calcium salt hardly dissolved in water; therefore the bond between MDP and hydroxyapatite should be stable.[26] Thus, the chemical interaction improves the resistance to hydrolytic breakdown and debonding stress, keeping the restoration margins sealed for a longer period.[27] Moreover, application of the hydrophobic bonding agent after the hydrophilic primer improves the mechanical properties by the presence of the cross-linking

monomers,[19] which may contribute to reducing the adhesive interface degradation over time.

All-in-one adhesives, such as the one-step self-etch silorane adhesive primer, act as permeable membranes and can be more susceptible to aging.[28] These adhesives are strongly influenced by the light intensity of the curing unit.[19] The bond of the silorane adhesive is also solvent-free and contains more monomers with more cross-linking ability.[19,21] The nanomechanical properties of the components of the adhesive interface (hybrid layer, adhesive, and bond layer of the silorane adhesive) were reduced after storage, except for the E_r of the adhesive. Although the solvated silorane adhesive primer showed similar E_r as the methacrylate adhesive at 24 hours, after aging it exhibited significant lower properties, likely because of greater susceptibility to plasticization by the greater amount of hydrophilic monomers[28] and possible residual solvent.

Siloxane species present in the silorane composite exhibit high hydrophobicity,[8,9] and the H and E_r of this material was not affected after six months of storage, probably because of the hydrophobic nature of the siloxane species.[8,9] The higher conversion rate of the methacrylate composite compared with silorane[22] may have compensated for its lower hydrophobicity and increased the plasticization resistance of the Filtek Z250, for which properties also did not decrease with aging. The mechanical properties of the underlying intertubular dentin decreased over time regardless of the restorative material. Dentin tissue contains collagenolytic enzymes, such as matrix metalloproteinases and cysteine cathepsins, which are responsible for the hydrolytic degradation of the collagen matrix.[29]

The conversion of the monomers into structured polymers is related to the increase of the physical properties of the resinous material; this polymerization reaction is dependent on various factors, such as design and size of the tip guide, distance of the light guide tip from the material surface, power density, exposure duration, shade and opacity of the composite, increment thickness, and composition of the materials, resulting in clinical performance improvement of light-cured materials and more durability of the adhesive restorations.[12]

CONCLUSIONS

Overall, methacrylate restorative systems exhibited higher H and E_r than silorane; increased light-curing time did not influence the nanomechanical properties, which were significantly reduced after long-term storage.

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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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Early Hardness and Shear Bond Strength of Dual-cure Resin Cement Light Cured Through Resin Overlays With Different Dentin-layer Thicknesses

H-S Chang • J-W Kim

Clinical Relevance

Reducing the dentin-layer thickness while increasing the translucent-layer thickness of resin inlays increases the photopolymerization of dual-cure resin cement, thereby increasing the early bond strength of resin inlays to dentin.

SUMMARY

The purpose of this study was to investigate whether dentin-layer thickness of resin overlays could affect the early hardness and shear bond strength of dual-cure resin cement (DCRC, RelyX ARC) after light curing with light curing units (LCUs) of various power densities: Optilux 360 (360), Elipar Freelight 2 (FL2), and Elipar S10 (S10). Resin overlays were fabricated using an indirect composite resin (Sinfony) with a dentin layer, an enamel layer, and a translucent layer of 0.5 mm thickness each (0.5-0.5-0.5) or of 0.2 mm, 0.5 mm, and 0.8 mm thickness (0.2-0.5-0.8), respectively. The DCRC was light cured for 40 seconds through the overlays, and surface hardness and shear bond strength to bovine dentin were tested 10 minutes after the start of light curing. Surface hardness was higher when the DCRC was light cured through the 0.2-0.5-0.8 combination than when the DCRC was light cured through the 0.5-0.5-0.5 combination with all LCUs. The ratio of upper surface hardness of DCRC light cured through resin overlays relative to the upper surface hardness of DCRC light cured directly was more than 90% only when the DCRC was light cured with S10 through the 0.2-0.5-0.8 combination. The shear bond strength value was higher when the DCRC was light cured with S10 through the 0.2-0.5-0.8 combination than when light cured with S10 through the 0.5-0.5-0.5 combination. This study indicates that reducing the dentin-layer thickness while increasing the translucent-layer thickness of

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resin inlays can increase the photopolymerization of DCRC, thereby increasing the early bond strength of resin inlays to dentin.

INTRODUCTION

Resin cements are the materials of choice for the adhesive luting of resin inlays. Resin cements provide a strong and durable resin bond with high retention,[1] marginal adaptation, and microleakage prevention.[2] Resin cements also increase the fracture resistance of the restored tooth and the restoration itself.[3] The compositions and characteristics of resin cements are similar to those of conventional composite resins,[4] and they are available in chemical-cured, light-cured, and dual-cured formulations. Many of the available resin cements are dual-cured resin cements (DCRCs), and they polymerize when the base and catalyst components are mixed and when they are subjected to the curing light from a light source.[5] DCRCs offer the advantages of extended working time and controlled polymerization,[6] and the chemical activators ensure a high degree of polymerization. However, most DCRCs still require light curing and demonstrate inferior hardness when light curing is omitted.[5,7-9]

During adhesive luting of resin inlays, only the external walls of the cement interface can benefit from direct light curing because they are readily accessible to the dental curing light. Polymerization of the DCRC at the internal walls, for example, the pulpal floor or the axial wall, relies more extensively on the chemical component of the curing mechanism[5] because the curing light is attenuated by the tooth structure or the resin inlay itself.[7] Chan and Boyer[10] and Barghi and McAlister[11] investigated the hardening of light-cured resin cements through porcelain and found that the thickness and shade of porcelain could affect the hardness of the cement. Blackman and others[12] found that the polymerization of resin cements beneath ceramic inlays was proper up to 3 mm distance from the tip of a standard curing light. Hasegawa and others[7] studied the hardening of three DCRCs under resin inlays and reported that chemical curing alone did not completely harden the DCRCs when the curing light was attenuated by the tooth structure and the restoration material. Park and others[13] reported that as much as 120 seconds of curing time using a high-power density halogen light-curing unit (LCU) was needed for proper curing of the DCRCs under the 1.5 mm Targis overlay.

Light intensity decreases as a function of depth, because the translucency decreases as the thickness of the composite resin increases, and the refractive index of the composite resin limits the speed of light transmission.[14] Thorough light curing of DCRCs, therefore, depends on light penetration to a desired depth through resin inlays that may prevent such a penetration.[15] LCUs must emit radiations between 410 and 500 nm, and the power density must be at least 300 mW/cm^2 for proper photopolymerization of the composite resin.[16] To achieve fracture resistance of resin inlays, the minimum thickness of resin inlay should be 1.5 mm,[13] and at least 300 mW/cm^2 of curing light is needed for proper light curing of the DCRCs under the resin inlays. Furthermore, resin inlays are fabricated with more than one layer; the dentin layer, the enamel layer, and the translucent layer are used simultaneously. Unfortunately, there are no studies evaluating how the thickness of each layer affects indirect composite resins, although the thickness of each layer affects the curing light penetration through resin inlays[17,18] and thereby the photopolymerization of DCRCs under resin inlays.

Some studies have evaluated the hardness of DCRCs light cured through various indirect restorations, such as ceramics, zirconia, and composite resins of a single shade.[12,15,19-23] However, there are no studies on the shade combinations of a resin inlay simulating the clinical situation including all the dentin, enamel, and translucent layers. In our previous study with the same indirect composite resin used in this study, we measured the power density of LCUs through resin overlays with various layer thickness combinations. The thickness of the dentin layer was decreased from 0.5 mm to 0.1 mm, the thickness of the enamel layer was kept unchanged at 0.5 mm, and the thickness of the translucent layer was increased from 0.5 mm to 0.9 mm and vice versa to maintain the resin overlay thickness of 1.5 mm. Higher-power density was measured through resin overlays with dentin-layer thickness of less than 0.2 mm and increased translucent-layer thickness in all LCUs.[17]

Therefore the purpose of this study was to investigate whether 1.5-mm-thick resin overlays with dentin-, enamel-, and translucent-layer thicknesses of 0.5 mm each, or 0.2, 0.5, and 0.8 mm could affect the photopolymerization of the DCRC. At first, the DCRC was light cured through two layer thickness combinations of the resin overlays and microhardness was measured. Then, to simulate the clinical situation of resin inlay cementation, the DCRC was light cured through the resin overlays

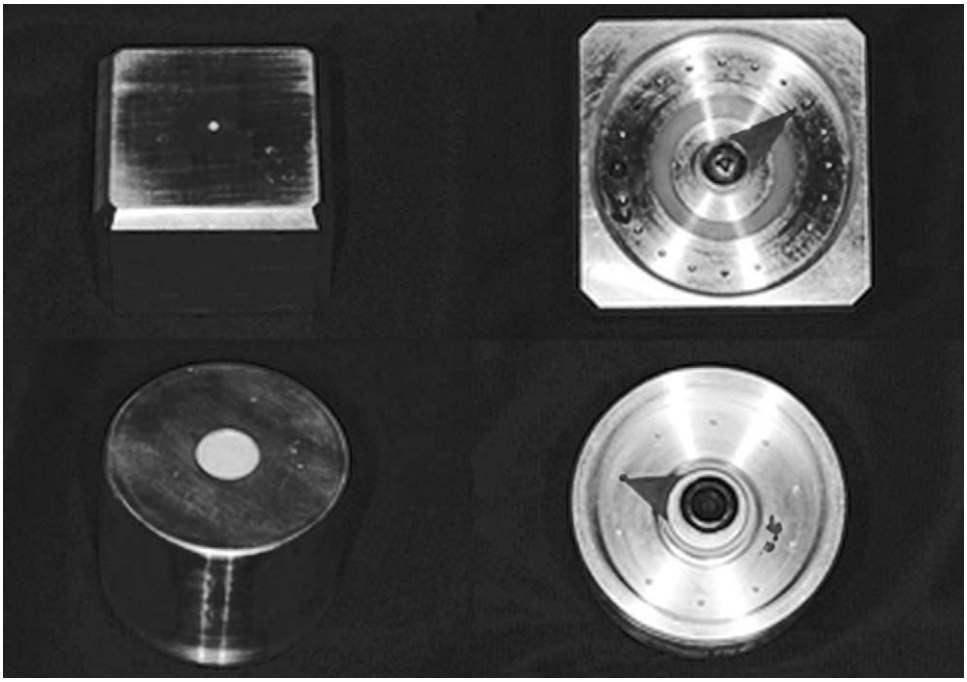

Figure 1. *Photograph of custom-made metallic molds with a movable Teflon plate and a bolt attached to the opposite side of the Teflon plate.*

and the shear bond strength (SBS) to bovine dentin was investigated.

METHODS AND MATERIALS

Resin Overlay Fabrication

We fabricated 1.5-mm-thick resin overlays in 15-mm diameter for the microhardness test and 3-mm diameter for the SBS test. Two layer combinations of resin overlays were fabricated according to our previous study:[17] resin overlays with dentin-layer thickness of 0.5 mm, enamel-layer thickness of 0.5 mm, and translucent-layer thickness of 0.5 mm (0.5-0.5-0.5 combination); and resin overlays with dentin-layer thickness of 0.2 mm, enamel-layer thickness of 0.5 mm, and translucent-layer thickness of 0.8 mm (0.2-0.5-0.8 combination).

Two custom-made cylindrical metallic molds with a hole (15 mm or 3 mm in diameter and 30 mm in depth) at the center were used to fabricate the resin overlays (Figure 1). A flat Teflon plate was inserted into the hole, and a bolt was attached to the opposite side of the Teflon plate, so that the empty space in the hole could be adjusted by rotating the bolt. By rotating the bolt 360° counterclockwise, the Teflon plate could be moved in a downward direction thereby rendering a 1-mm deep empty space in the metallic mold. The rotation of the bolt was marked in 10 steps such that one step corresponded to a downward movement of the Teflon plate by 0.1 mm (Figure 1).

Three layers of Sinfony indirect lab composite (3M ESPE, Seefeld, Germany) were used to fabricate the resin overlays: A2 dentin layer, E2 enamel layer, and T1 translucent layer. To fabricate resin overlays with each layer of 0.5 mm thickness, the Teflon plate in the mold was lowered by 0.5 mm, and the empty space was filled with the A2 dentin layer. Then, the upper surface of the mold was covered with a transparent polyester film and a glass slab to press the surface to remove the excess composite resin. The dentin layer was light cured with a light-emitting diode (LED) LCU (Elipar FreeLight 2 [FL2], 3M ESPE, St Paul, MN, USA) for 5 seconds. After removing the glass slab and the polyester film, the dentin layer was light cured for 20 seconds using an overlapping curing procedure to ensure that every part of the dentin layer was light cured. The Teflon plate with light-cured dentin layer was lowered by another 0.5 mm and the E2 enamel layer was filled into the empty space and light cured as described earlier. The same procedure was repeated with the T1 translucent layer to fabricate the resin overlay of 1.5-mm thickness.

The resin overlays with dentin-layer thickness of 0.2 mm, enamel-layer thickness of 0.5 mm, and translucent-layer thickness of 0.8 mm were fabricated as described previously by controlling the empty

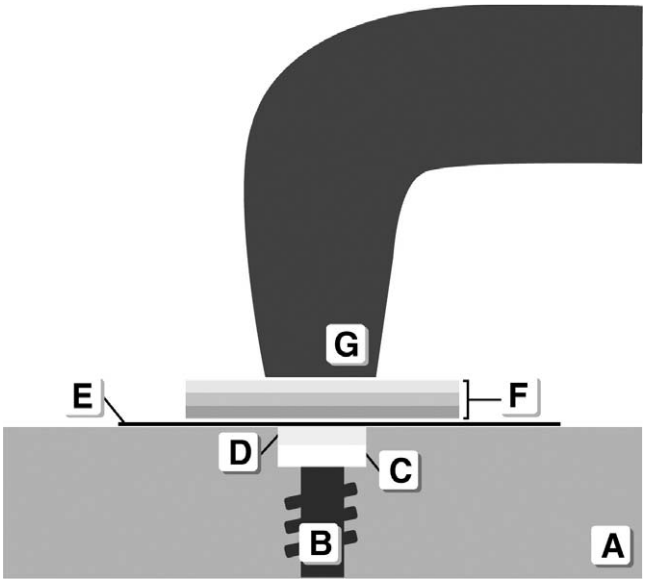

Figure 2. *Schematic diagram of the DCRC specimen fabrication for the microhardness measurement. (A): Metallic mold. (B): Bolt. (C): Teflon plate of 3-mm diameter. (D): DCRC. (E): Polyester film. (F): Layered resin overlay of 15-mm diameter. (G): Light guide of the LCU.*

space made by the Teflon base. Seven resin overlay specimens of each layer thickness combination were fabricated in 15-mm diameter and in 3-mm diameter (n=7).

Microhardness of DCRC Light Cured Through Resin Overlays

To light cure the DCRC in one curing procedure, the custom-made metallic mold with a 3-mm diameter hole was selected as the smallest tip diameter of the LCU was 7.5 mm. The Teflon plate was lowered by 1 mm, and the DCRC (RelyX ARC; 3M ESPE, Seefeld, Germany) was mixed according to manufacturer's instructions and inserted into the empty space. The upper surface of the mold was covered with a polyester film to separate the DCRC and the resin overlay. Previously fabricated resin overlays of 15 mm in diameter were used to cover the DCRC and pressed with a glass slab to control the resin cement thickness to 1 mm. After removing the glass slab, the DCRC was light activated through the resin overlays with LCUs for 40 seconds according to the manufacturer's instructions (Figure 2). Three different LCUs were used to light cure the DCRC: a halogen LCU (Optilux 360 [360]; Demetron, Danbury, CT, USA) with a power density of 530 mW/cm^2, and two LED LCUs (FL2 and Elipar S10 [S10], 3M ESPE) with a power density of 1040 mW/cm^2 and 1340 mW/cm^2, respectively. The power density of the LCUs was measured with a handheld dental radiometer (Cure

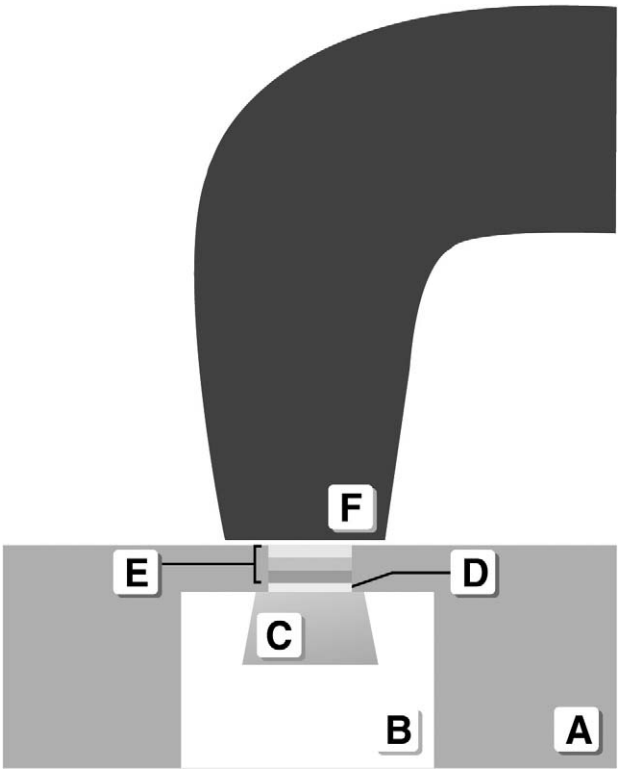

Figure 3. *Schematic diagram of specimen fabrication for the SBS test of DCRC to bovine dentin. (A): Metallic mold. (B): Acrylic resin. (C): Bovine dentin. (D): DCRC. (E): Layered resin overlay of 3-mm diameter. (F): Light guide of the LCU.*

Rite, Kerr, Milford, OH, USA). Ten minutes after the start of light curing, the DCRC was removed from the mold and Vickers microhardness was tested with a microhardness tester (MHT-10, Anton Paar, Graz, Austria) with 100 g load for a 10-second dwell time at three points forming a small triangle in the center of the upper surface of the resin cement; the mean Vickers hardness number (VHN) was recorded. As a control, the DCRC was light cured directly without resin overlays for 40 seconds and the microhardness was measured as described previously. Therefore, nine experimental groups (two resin overlays with different layer combinations and the control group and three LCUs) were tested with seven DCRC specimens each (n=7). The ratio of the upper surface hardness of the DCRC light cured through composite resin overlays relative to the upper surface hardness of the DCRC light cured directly (control) was calculated for the hardness ratio.

SBS of DCRC to Bovine Dentin Light Cured Through Resin Overlays

Freshly extracted bovine incisors were stored in distilled water and the water was changed every two

Table 1: *Summary of Two-way ANOVA for Main Factors (Layer and LCU) and Their Interactions for the VHN*

Source of Variation	Sum of Squares	*df*	Mean Square	F	Significance
Layer combination of resin overlay	1529.391	2	764.695	1012.803	<.001
LCU	772.831	2	386.416	511.789	<.001
Layer combination of resin overlay * LCU	168.689	4	42.172	55.855	<.001
Error	40.772	54	0.755		
Total	108,088.574	63			

days according to ISO/TS 11405:2003(E).[24] The roots were removed with a low-speed disc, leaving only the crown portion. Each crown portion was cut parallel to the labial surface to expose the dentin and then ground flat with wet 600-grit silicon carbide paper. The exposed dentin was sectioned with a low-speed microsaw (Topmet, Norderstedt, Germany) under water spray to a size of 5 × 5 mm. Placing the exposed dentin surface facing the bottom of the mold, bovine dentin was inserted into a metallic mold with a 22-mm inner diameter and a 15-mm depth and chemical-cure acrylic resin (Ortho-jet acrylic, Lang Dental Manufacturing, Wheeling, IL, USA) was poured into the mold. The embedded dentin specimen was removed after curing, and the exposed dentin was wet bonded using 37% phosphoric acid and SingleBond (3M ESPE) application followed by light curing for 20 seconds.

The embedded dentin specimen was inserted into another metallic mold with a 22-mm inner diameter and a 15-mm depth. In the center of the mold, a perforating hole with a 3-mm diameter and 1.7-mm thickness was prepared from the inner side to the outer surface of the mold. With the dentin surface exposed through the 3-mm diameter hole, the DCRC was applied to the dentin surface in 0.2-mm thickness into the hole according to the manufacturer's instructions. Previously fabricated resin overlays of 1.5-mm thickness and 3-mm diameter were inserted into the hole with the dentin layer facing the DCRC and pressed with a glass slab to control the cement layer to 0.2-mm thickness. The glass slab was removed and the DCRC was light cured for 40 seconds according to the manufacturer's instructions (Figure 3). Ten minutes after the start of light curing, the specimens underwent SBS testing with a universal testing machine (Z020, Zwickl, Ulm, Germany) with 500 N load cell at 1 mm/min crosshead speed until fracture; the acquired bond strengths were converted to megapascals (MPa). Therefore, six experimental groups (two resin overlays with different layer combinations and three LCUs) were tested with seven DCRC specimens each (n=7). The VHN and the SBS of the DCRC to bovine dentin were analyzed with two-way analysis of variance (ANOVA) at a significance level of 5%, followed by post-hoc comparisons with Duncan test.

RESULTS

Surface Hardness of DCRC Light Cured Through Resin Overlays

Two-way ANOVA showed a significant effect for the main factors (layer thickness, $p<0.001$; LCU, $p<0.001$) and for their interaction ($p<0.001$, Table 1). A higher VHN was observed when the DCRC was light cured through the 0.2-0.5-0.8 combination than when the DCRC was light cured through the 0.5-0.5-0.5 combination. The VHN of the DCRC was highest when light cured with S10, followed by FL2 and 360 (Table 2). The hardness ratio of upper surface hardness of the DCRC light cured through composite resin overlays relative to the upper surface hardness of the DCRC light cured directly is shown in Table 3. The ratio was 92.5% for the DCRC light cured with S10 through the 0.2-0.5-0.8 combination.

Table 2: *Mean (Standard Deviation) VHN of DCRC Light Cured Through Resin Overlays with Dentin-Enamel-Translucent Layer Combinations of 0.5-0.5-0.5 and 0.2-0.5-0.8*[a]

Layer Combination of Resin Overlay	LCU		
	360	FL2	S10
0.5-0.5-0.5	31.21 (0.65) Aa	36.29 (1.00) Ab	40.99 (0.94) Ac
0.2-0.5-0.8	32.67 (0.87) Ba	38.70 (0.62) Bb	45.41 (1.33) Bc
Without resin overlay	45.88 (0.95) Ca	48.19 (0.58) Cb	49.09 (0.58) Cc

[a] *Mean values followed by different capital letters (column) or small letters (row) are significantly different by Duncan test (p<0.05).*

Table 3: *Hardness Ratio of Upper Surface Hardness of DCRC Light Cured Through Resin Overlays Relative to Upper Surface Hardness of DCRC Light Cured Directly*

Layer Combination of Resin Overlay	LCU		
	360	FL2	S10
0.5-0.5-0.5	68.0	75.3	83.5
0.2-0.5-0.8	71.2	80.3	92.5

SBS of DCRC to Bovine Dentin Light Cured Through Resin Overlays

Two-way ANOVA showed a significant effect only for the main factors (layer thickness, $p=0.007$; LCU, $p<0.001$) and not for their interaction ($p=0.445$, Table 4). The DCRC light cured with S10 showed a higher SBS than the DCRCs light cured with FL2 and 360 (Table 5). The DCRC light cured with S10 through the 0.2-0.5-0.8 combination showed a significantly higher SBS than the DCRC light cured with S10 through the 0.5-0.5-0.5 combination (Figure 4).

DISCUSSION

The retention of resin inlays can be increased through photopolymerization of the DCRC in the internal wall of prepared tooth when there is insufficient time for chemical curing. In this study, the surface hardness and SBS tests were performed 10 minutes after the start of light curing because occlusal adjustment and finishing and polishing of the composite resin inlays are performed immediately after the light-curing procedure. Stress created by these procedures can have a detrimental effect on the quality of the bonding between the dentinal walls and the composite resin inlays.[25,26]

The results demonstrated that VHN and SBS of the DCRC to bovine dentin were dependent on the layer combinations of resin overlays and LCUs. A higher VHN was observed when the DCRC was light cured through the 0.2-0.5-0.8 combination than when the DCRC was light cured through the 0.5-0.5-0.5 combination. Also, a higher VHN was observed when the DCRC was light cured with S10 than when the DCRC was light cured with FL2 and 360, regardless of the layer combination of resin overlays. These results could be explained by the access of the curing light to the DCRC.

In our previous study with the same indirect composite resin,[17] the power density of 360, FL2, and S10 through the 0.5-0.5-0.5 combination was measured as 103 mW/cm^2, 239 mW/cm^2, and 347 mW/cm^2, respectively, whereas the power density of 360, FL2, and S10 through the 0.2-0.5-0.8 combination was measured as 141 mW/cm^2, 307 mW/cm^2, and 447 mW/cm^2, respectively. Therefore, the power density of LCUs through resin overlays could be enhanced by reducing the thickness of dentin layer while increasing the thickness of the translucent layer, since the dentin layer attenuated the curing light more than the enamel and translucent layers.[17] The VHN values from this study were in accordance with the power density of LCUs through both combinations of resin overlays.

Usually, the degree of conversion of a composite resin is assessed by the hardness[27,28] and the hardness ratio,[29] the ratio between the upper and lower surface hardness values of a 2-mm-thick composite resin specimen.[8,30] However, in this study, the hardness of the lower surface of the DCRC was not measured because the thickness of the DCRC was controlled to 1.0 mm. In clinical situations, the thickness of resin cement was reported to be less than 300 μm.[31] In our pilot study, however, hardness testing of a 300-μm-thick DCRC resulted in perforation of the resin cement specimens. Therefore, the thickness of the DCRC had to be increased to 1 mm.

According to previous studies, the relative hardness of a composite resin was used to determine the extent of polymerization. The surface hardness of a

Table 4: *Summary of Two-way ANOVA for Main Factors (Layer and LCU) and Their Interactions for SBS*

Source of Variation	Sum of Squares	*df*	Mean Square	F	Significance
Layer combination of resin overlay	7.451	1	7.451	8.319	.007
LCU	209.952	2	104.976	117.207	<.001
Layer combination of resin overlay * LCU	1.483	2	0.742	0.828	0.445
Error	32.243	36	0.896		
Total	3044.299	42			

Table 5: *Mean (Standard Deviation) SBS (in MPa) of DCRC Light Cured Through Resin Overlays to Bovine Dentin*[a]

Layer Combination of Resin Overlay	LCU		
	360	FL2	S10
0.5-0.5-0.5	4.97 (0.86) Aa	8.35 (0.81) Ab	9.88 (0.88) Ac
0.2-0.5-0.8	5.30 (0.97) Aa	9.33 (1.01) Ab	11.09 (1.12) Bc

[a] *Mean values followed by different capital letters (column) or small letters (row) are significantly different by Duncan test ($p<0.05$).*

test group was compared with that of a control group with maximum polymerization. A ratio of 90% was suggested to be acceptable for a clinical situation.[32] In a study by Arrais and others,[25] the monomer conversion in auto- and dual-polymerizing modes of a DCRC was investigated and the ratio of conversion in auto-polymerized relative to dual-polymerized modes was calculated and termed the "potential of cure." This concept was applied to our study, and the ratio of upper surface hardness of the DCRC light cured through composite resin overlays relative to upper surface hardness of the DCRC light cured directly was calculated. The hardness ratio was more than 90% only when the DCRC was light cured with S10 through the 0.2-0.5-0.8 combination.

To simulate the clinical situation of resin inlay cementation with a DCRC, the SBS test was performed 10 minutes after the start of photopolymerization. The SBS of the DCRC light cured with S10 through the 0.2-0.5-0.8 combination was significantly higher than that of the DCRC light cured with S10 through the 0.5-0.5-0.5 combination. This is in accordance with the hardness ratio of the DCRC light cured through resin overlays and the control. The DCRC light cured with S10 through the 0.2-0.5-0.8 combination was the only group with a hardness ratio greater than 90%. Therefore, it can be assumed that only the DCRC light cured with S10 through resin overlays in a 0.2-0.5-0.8 combination showed proper early photopolymerization. The SBS of the DCRC light cured with 360 and FL2 through the 0.2-0.5-0.8 combination was higher than the SBS of the DCRCs light cured with 360 and FL2 through the 0.5-0.5-0.5 combination; however, the difference was not significant. It can be assumed that the LCUs with a lower power density resulted in insufficient photopolymerization of the DCRC through either combination. Therefore, LCUs with a higher power density should be recommended for photopolymerization of the DCRC under resin inlays, since there are limitations in increasing the amount of curing light through resin inlays with reduced dentin-layer thickness.

This study focused on light curing of the DCRC through resin inlays, and the possible shade alteration of the resin inlays due to decreased dentin layer thickness and increased translucent layer thickness was not taken into account. Therefore,

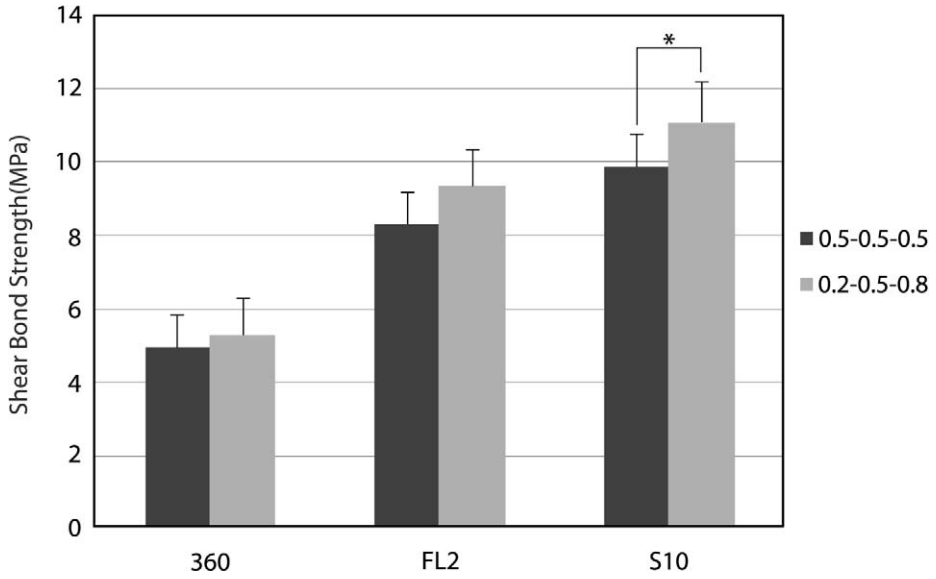

Figure 4. *SBSs (in MPa) of DCRC light cured through resin overlays to bovine dentin. Asterisk (*) indicates significant difference between groups ($p<0.05$).*

additional studies are needed to achieve maximal light curing of the DCRC through resin inlays with minimal shade alteration from the designated shade as the resin inlays are an esthetic treatment option.

CONCLUSIONS

Within the limitations of this study, the VHN and the SBS of the DCRC to bovine dentin under resin inlays could be increased by reducing the dentin-layer thickness while increasing the translucent-layer thickness. Also, LCUs with a higher power density are recommended for light curing the DCRC, since there are limitations in increasing the amount of curing light through resin inlays with reduced dentin-layer thickness.

Acknowledgements

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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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Direct Spectrometry: A New Alternative for Measuring the Fluorescence of Composite Resins and Dental Tissues

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Clinical Relevance

The study presents a new tool to establish a future fluorescence table of dental tissues and composites, similar to the color tables that are currently commercially available.

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SUMMARY

The aim of this study was to evaluate the fluorescence intensity of different composite resins and compare those values with the fluorescence intensity of dental tissues. Different composite resins were used to make 10 discs (2 mm in depth and 4 mm in diameter) of each brand, divided into groups: 1) Z (Filtek Z350, 3M ESPE), 2) ES (Esthet-X, Dentsply), 3) A (Amelogen Plus, Ultradent), 4) DVS (Durafill-VS, Heraeus Kulzer) with 2 mm composite resin for enamel (A2), 5) OES ([Esthet-X] opaque-OA [1 mm] + enamel-A2 [1 mm]); 6) ODVSI ([Charisma-Opal/Durafill-VSI], opaque-OM (1 mm) + translucent [1mm]), and 7) DVSI ([Durafill- VSI] translucent [2 mm]). Dental tissue specimens were obtained from human anterior teeth cut in a mesiodistal direction to obtain enamel, dentin, and enamel/dentin samples (2 mm). The fluorescence intensity of specimens was directly measured using an optic fiber associated with a spectrometer

(Ocean Optics USB 4000) and recorded in graphic form (Origin 8.0 program). Data were submitted to statistical analysis using Dunnet, Tukey, and Kruskall-Wallis tests. Light absorption of the composite resins was obtained in a spectral range from 250 to 450 nm, and that of dental tissues was between 250 and 300 nm. All composite resins were excited at 398 nm and exhibited maximum emissions of around 485 nm. Fluorescence intensity values for all of the resins showed statistically significant differences (measured in arbitrary units [AUs]), with the exception of groups Z and DVS. Group DVSI had the highest fluorescence intensity values (13539 AU), followed by ODVS (10440 AU), DVS (10146 AU), ES (3946 AU), OES (3841 AU), A (3540 AU), and Z (1146 AU). The fluorescence intensity values for the composite resins differed statistically from those of dental tissues (E=1380 AU; D=6262 AU; E/D=3251 AU). The opacity interfered with fluorescence intensity, and group Z demonstrated fluorescence intensity values closest to that of tooth enamel. It is concluded that the fluorescence intensity values were significantly different among the composite resins and compared with dental tissues. The direct spectrofluorimetric method represents a tool for evaluating the fluorescence of composite resins.

INTRODUCTION

Personal satisfaction with one's smile may provide physical and mental well-being, primordial facts for attaining the state of health defined by the World Health Organization. The search for excellence in dentistry has favored the development of resin composites. Consequently, various types and commercial brands of composites have appeared on the market with the promise of optical properties, including fluorescence, opacity, opalescence, and translucence, similar to those of dental tissue. However, the chemical substances responsible for these properties and their concentrations in the different modalities of opaque and translucent resins and enamel are not detailed by the manufacturers.

Studies on the fluorescent phenomena involving teeth and restorative materials have shown that during the day ultraviolet (UV) radiation makes teeth appear whiter and shinier.[1-4] This occurs because of the state of excitation of the atoms of the tooth structure, which, when they return to a state of less excitation, emit light in the visible spectrum between 400 and 450 nm, a range characteristic of blue light.[5-7]

The greater the quantity of UV light falling on the tooth surface, the greater the emission of fluorescence. Therefore, dental fluorescence becomes more evident at sea level, in the mountains, or in rooms with artificial UV light (black light). Thus, artificial materials, such as composite resins and ceramics, that do not have adequate fluorescence appear as black holes or voids in these environments.

Dental fluorescence intensity is attributed to the organic components that are photosensitive to the UV spectrum, which is why dentin presents greater fluorescence intensity than enamel. Dentin fluorescence is attributed to tryptophan and hydroxypyridine.[4,8]

Vanini[9] also demonstrated that a greater extent of mineralization provides a lower level of fluorescence, this being another reason why dentin would be more fluorescent than enamel. According to Dickson and others,[10] dentin fluorescence is four times greater than that of enamel, and the amelodentinal limit shows no fluorescence.

Apparently, the fluorescence of composites does not follow the same model as that of dental tissues.[11] For composite resins, the superficial layers would be more relevant indicators of fluorescence properties that do not occur in the tooth, in which the more fluorescent dentin has a lower chroma.[12] The final balance of dental fluorescence would be the sum of the enamel and dentin fluorescence.[13]

The natural fluorescence of teeth is an important characteristic that must be reproduced in composite resin restorations to provide vitality and luminosity; it is dependent on the tooth, the restorative material, and the duration of exposure to UV light, which may occur under natural daylight or artificial light, such as that of fluorescent lamps, flashes, or the black light of nightclubs.[1,7,14-16] The behavior of dental tissues exposed to light has always been a complicating factor for adequate esthetic restorations, as the dental structure is polychromatic and exhibits different tonalities when light falls on it in different ways.[11]

A composite resin restoration must replace the lost dental structure so that it blends with the surrounding structures. Ideal restorative materials must have fluorescent properties similar to those of natural teeth.[17] If there is an absence of fluorescence, the esthetic qualities of a restoration will suffer, predominantly under UV lighting conditions. However, little is known about the extent to which base

Table 1: *Materials, Equipment, Lot Numbers, and Manufacturers*

Material/Equipment	Composition	Lot Number	Manufacturer
Esthet-X	BisGMA, modified urethane, BisEMA,TEGDMA, aluminum borosilicate fluoride glass, silanized barium	Enamel (A2): 893479 Dentin (A2-0): 064644B	Dentsply
Durafill VS	BisGMA, UDMA, TEGDMA, highly dispersed silicon dioxide, splinter polymer	Enamel (A2): 010213 Enamel (I): 010140	Heraeus Kulzer
Filtek Z-350	BisGMA, UDMA, BisEMA, TEGDMA, nanosilica filler, agglomerates of zirconia/silica particles	Enamel (A2): N125240	3M ESPE
Amelogen Plus	BisGMA, barium boron aluminosilicate glass particles	Enamel (A2): B3SH8	Ultradent
Charisma Opal	BisGMA, TEGDMA, barium aluminum fluoride glass, dispersive silicon dioxide	Dentin (OM): 010022	Heraeus Kulzer
Light Polymerizer	Light-emitting diode		Emitter- Schuster
Spectrometer Ocean Optics USB 4000			Toshiba

Abbreviations: BisEMA, bisphenol A ethoxylate dimethacrylate; BisGMA, bisphenol A glycidyl methacrylate; TEGDMA,triethylene dimethacrylate; UDMA, urethane dimethylacrylate.

composites affect the final fluorescence of restorations and their relationships with the neighboring dental tissues. Therefore, the authors were motivated to seek details about the real properties of the materials available on the market with regards to fluorescence and to gain further knowledge about the phenomenon of fluorescence.

The aim of the present study was to evaluate the difference in fluorescence between several brands of composite resins, and combinations of those brands, on opacity and translucence, and to compare those results with the fluorescence of isolated dental tissues (enamel, dentin, and enamel/dentin) as measured by direct spectrophotometry. The hypotheses tested were as follows: 1) there would be no difference between the fluorescence of dental tissues and the tested composite resins, 2) the composites would not exhibit differences in fluorescence intensity, and 3) direct spectrophotometry would not be effective for measuring the fluorescence of composite resins and dental tissues.

METHODS AND MATERIALS

The study was approved by the Research Committee at the Sao Jose dos Campos School of Dentistry, UNESP- Univ. Estadual Paulista (Protocol 038/2009-PH/CEP).

The materials and equipment used to conduct this study are listed in Table 1, along with the respective lot numbers and manufacturers. The following brands of composite resin were used to compare the levels of fluorescence with those of a human tooth: Filtek Z350 (3M ESPE, St Paul, MN, USA); Esthet-X (Dentsply International, York, PA, USA); Durafill VS (Heraeus Kulzer, Heraeus GmbH, Hanau, Germany), Amelogen Plus (Ultradent, South Jordan, UT, USA), and Charisma Opal (Heraeus Kulzer, Heraeus GmbH, Hanau, Germany).

A total of 70 specimens were made, divided into seven groups (n=10 each). The first four groups (n=40) were fabricated using only composite resins for enamel, in shade A2: 1) Z (Filtek Z350, 3M ESPE); 2) ES (Esthet-X, Dentsply); 3) A (Amelogen Plus, Ultradent); 4) DVS (Durafill-VS, Heraeus Kulzer). The other three groups (n=30) were fabricated using combinations of opaque, enamel, and translucent composites: 5) OES ([Esthet-X] opaque-OA [1 mm] + [Esthet-X] enamel-A2 [1 mm]); 6) ODVSI ([Charisma-Opal] opaque-OM [1 mm] + [Durafill-VSI] translucent [1 mm]); and 7) DVSI ([Durafill- VSI] translucent [2 mm]).

The specimens were obtained using a nonstick metal matrix and were standardized at 2 mm in depth and 4 mm in diameter. A polyester matrix strip was placed over the composite resin and pressed with a glass slide to provide smooth, compact, standardized specimens. The composite resin was inserted in a 1-mm increment and was polymerized with a light-emitting diode (LED) emitter (Schuster, Santa Maria, Brazil) that presented 750 mW/cm^2 of power for 40 seconds in contact with the polyester matrix strip.

All composite resin specimens were attached to glass slides per group of resin with the polymerized surface up, using an ethyl cyanoacrylate adhesive (Super Bonder, Henkel, Düsseldorf, Germany), to keep groups in individual supports and to have the fluorescence intensity measured in the same surface

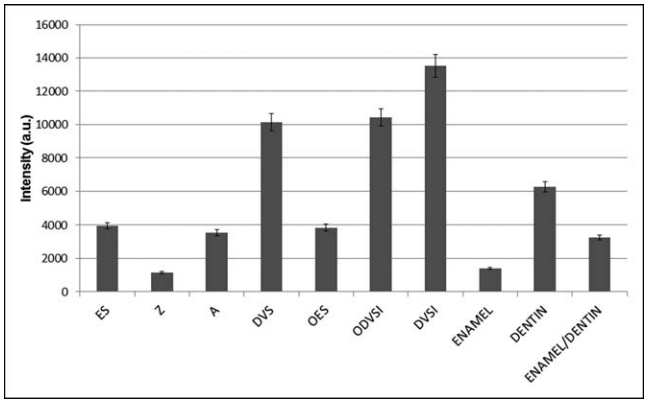

Figure 1. *Column graph (mean ± standard deviation) of the fluorescence intensity values (AU) according to the different types of composite resins versus* control groups (dentin, enamel and enamel/dentin).

of each specimen. These glass slides were immersed in artificial saliva at 37°C for 24 hours. The artificial saliva was prepared according to the method of Gohring et al. [18] using 4.8 g HCl, 3.4 g NaCl, 0.2 g $MgCl_2$, 0.4 g $CaCl_2$, 0.4 g KSCN, 1.4 g H_2KPO_4, 0.2 g H_3BO_3, and 0.4 g $CHNaO_3$.

Ten sound anterior human teeth, extracted for periodontal reasons, were used for comparison of the fluorescence levels of enamel, dentin, and enamel/dentin and for comparison of the fluorescence of the composite resins. The teeth were obtained following a protocol approved by the university's ethical research committee.

The initial fluorescence was recorded directly on the surface of the whole tooth using fiber optics associated with a USB 4000 spectrometer (Ocean Optics, Dunedin , FL, USA). After this initial measurement, enamel and dentin cylinders were obtained using a trephine bur (4 mm in internal diameter). To obtain 1-mm dentin specimens, the enamel of the cylinders was removed. The same was done to obtain 1-mm enamel specimens, where the dentin was removed. For that, the enamel or dentin surfaces were polished in a polishing device (DP-10, Panambra Industrial e Técnica, São Paulo, Brazil) using a sequence of 600 and 1200 grit aluminum oxide abrasive disks (Extec, Enfield, CT, USA). All specimens were stored in artificial saliva[18] at 37°C, up to the time of fluorescence measurement.

The composite resin specimens were excited using an ultraviolet LED appliance with a peak centered at 398 nm. A xenon ion source (Model PX- 2), coupled to a bifurcated optical fiber connected to the spectrometer, was used to measure the fluorescence absorption and detection. The values obtained were reproduced in graphs on a computer using the Origin 8.0 program (OriginLab Corporation, Northampton, MA, USA). The fluorescence intensity values were located in the visible light spectrum between 450 nm and 700 nm.

Statistical Analysis

Dunnet, Tukey, and Kruskall-Wallis tests were performed at a level of significance of 5%.

RESULTS

The mean fluorescence intensity values of the composites, the combinations with opaque and translucent composites, and dental tissues are shown in Figure 1.

Regarding absorption measurements, there were some differences among the analyzed composites. From a general aspect, the composites had absorptions between 250 and 450 nm (Figure 2), and there was a significant difference among the composites. In the Esthet-X group, composites with different degrees of opacity had a small difference from 250 to 300 nm, probably due to the composition of Esthet-X OA2 compared with Esthet-X A2. For the Durafill VS group, when combined with Charisma Opal there was no difference in absorption, whereas the Durafill VSI group only showed a difference between 250 and 350 nm, exhibiting greater absorption. These differences may be attributed to the compositions of the composite resins, which varied according to their brand.

Based on the maximum absorption peaks, the emission spectra of all of the composite resins studied had maximum emissions at approximately 485 nm (Figure 3).

In the Z-350 group, there was a plateau during emission between 488 nm and 517 nm, probably due to the structural characteristics of this resin. In the Esthet-X group, in addition to a maximum emission at 485 nm, the appearance of a shoulder was noted at 520 nm, possibly for the same reason as the previous group. The composition of Esthet-X OA2 (opaque) did not alter the spectral profile; it only favored an increase in emission.

In the case of the Durafill VS group, there was an increase of approximately 35% in its emission compared with the Durafill VSI group. Moreover, Durafill VSI resin alone presented the highest emission, with a peak at 484 nm. In the Amelogen Plus group, in addition to the maximum emission at

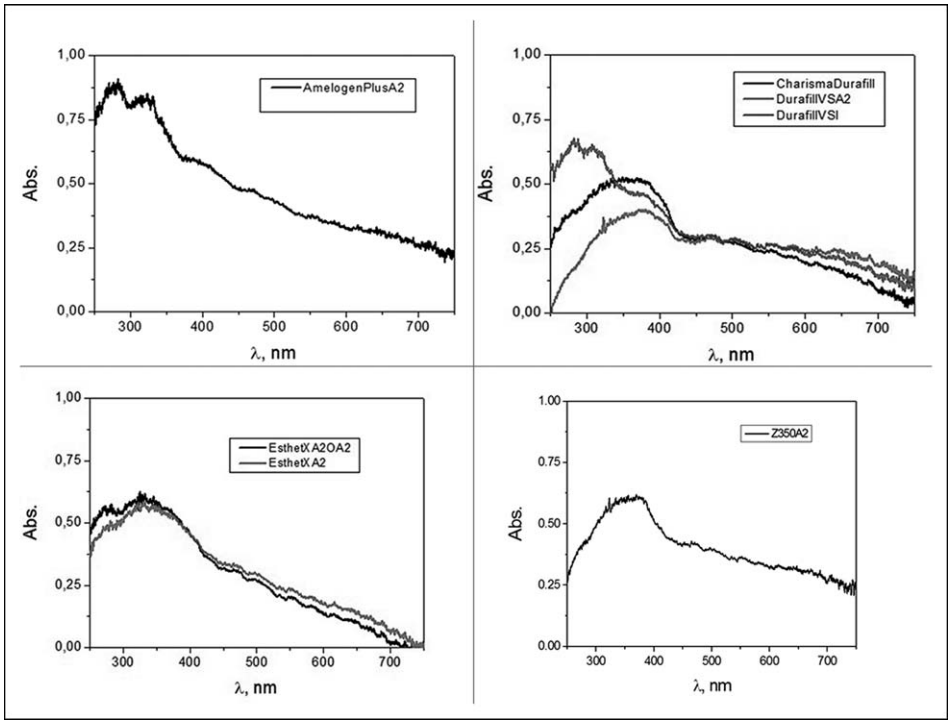

Figure 2. *Absorption spectra of the analyzed composites.*

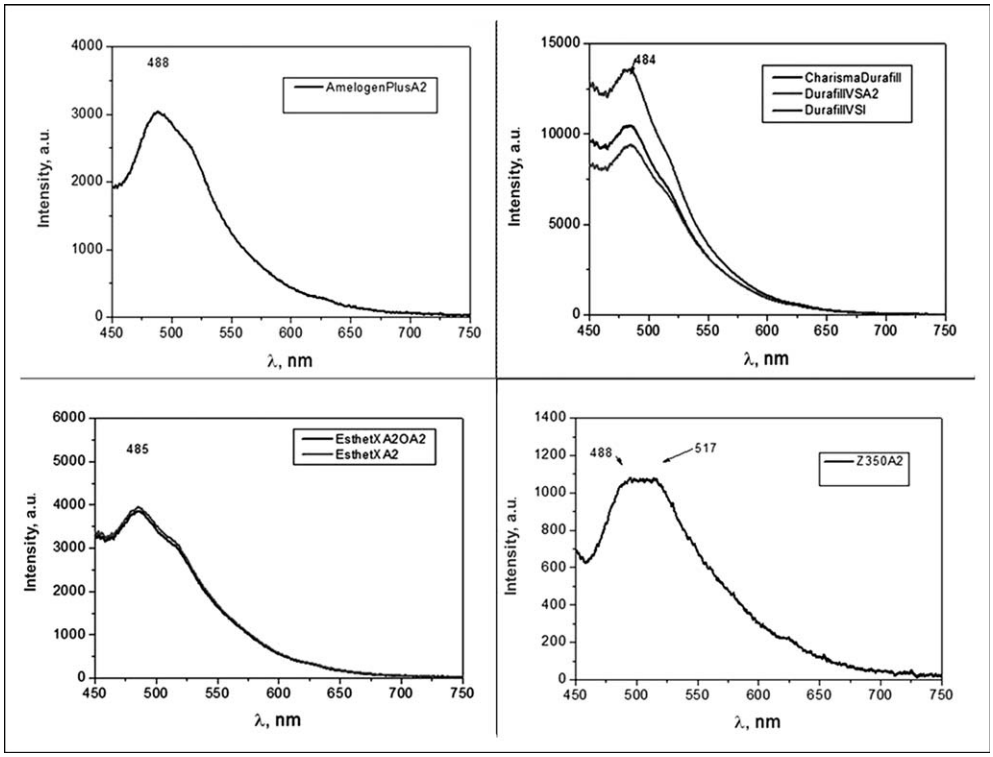

Figure 3. *Emission spectra of the composites analyzed.*

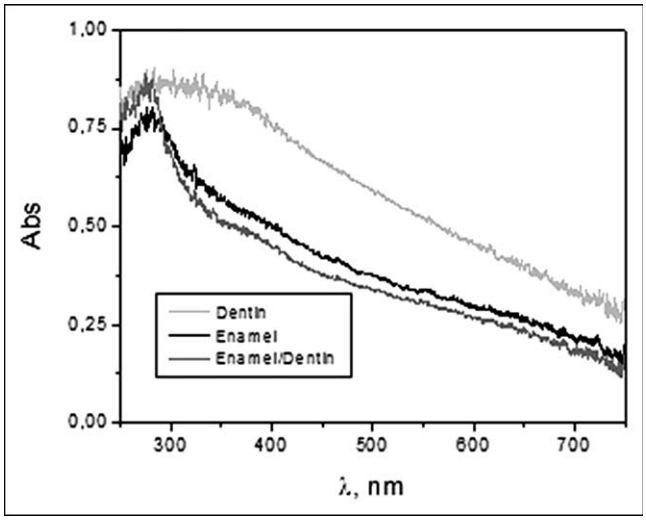

Figure 4. *Absorption spectra of dental tissues.*

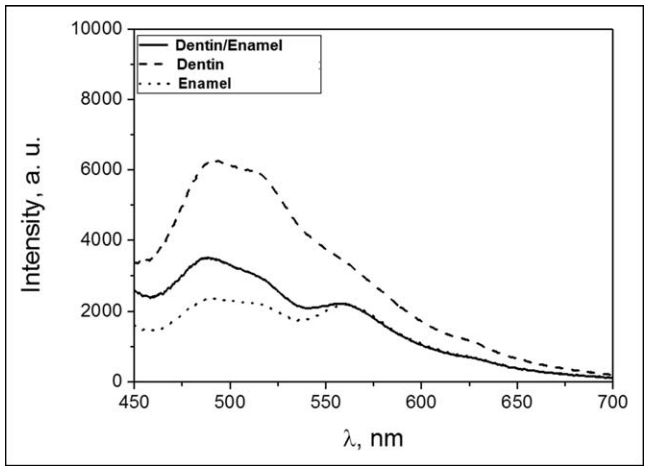

Figure 5. *Emission spectra of enamel, dentin, and enamel/dentin.*

488 nm, the beginning of a shoulder is observed at 520 nm, possibly due to its composition.

With regards to dental tissues, the light absorption spectrum was between 250 nm and 300 nm (Figure 4). Dentin presented the broadest and highest spectrum compared with tooth enamel. In the emission spectrum, the peaks were higher than 450 nm, as is shown in Figure 5, which means that the highest emission values (peak) are above 450 nm or are in the visible light spectrum area, more precisely, among the values close to 490 nm. This area (including the peak) is a result of the emission process (fluorescence) of the whole tooth due to the absorbed energy (during the light absorption process by the tooth or the resin). The absorption process implicates the transition of electrons (from the tooth or resin components) from the ground state to the excited state (higher energy level), while the emission process is implicated in the transition from the excited stated to the ground state.

Therefore, the absorption peaks of the composite resins are between 250 nm and 450 nm, and those of dental tissues are between 250 and 300 nm. All of the composites had maximum emissions close to 485 nm. There was a statistically significant difference between composites with regards to the fluorescence intensity, with the exception of the group Z-350. The translucent DVSI group exhibited the highest fluorescence intensity value (13539 AU), followed by ODVSI (10440 AU), DVS (10146.2 AU), ES (3946.2 AU), OES (3840.8 AU), A (3540.1 AU), and Z (1146.2 AU).

All of the studied composite resins exhibited fluorescence intensities that differed statistically from those of the dental tissues (enamel=1380 AU; dentin=6262 AU; enamel/dentin=3251 AU). The Z group was the one that presented a fluorescence intensity that approximated dental enamel.

In this current study, the opaque composites did interfere in the final fluorescence analysis in mixed specimens, indicating that the subsuperficial layer may interfere in the fluorescence of composites.

DISCUSSION

The method used in this study is innovative because it is a direct fluorescence measurement method using an optical fiber. There are many reports on fluorescence measurement through lab spectrophotometers that are not suitable for clinical direct measurements.[1,8,19-21] Therefore, no studies were found in the literature about the direct measurement of fluorescence in dental tissues and composite resins samples. When the fluorescence intensity of composites and dental tissues is established, the direct method—using a spectrometer coupled with an optical fiber—will be an important clinical tool for selecting composites not only based on color shades but also on degree of fluorescence. Therefore, it will be possible to produce a new scale that involves color and fluorescence.

There is a wide variety of composite resins on the market that are true direct restoration systems presenting opaque composites for dentin and translucent composites for enamel, each with a different degree of translucence. However, fluorescence varies according to the resin brand and not according to the characteristics of its particles or opacity and trans-

lucence.[13] The results of the present study are in disagreement with those of other studies because composites of the same brand but with distinct qualities also exhibited distinct fluorescence values.

In this current study, statistically different fluorescence intensity values were observed among the composite resins and between the dental tissues, probably because of the different compositions of each substrate.[1,13,15,22] For all of the composites, one may infer that the same chromophore is responsible for the phenomenon because of the similarities of the spectral profiles. However, the emission intensity might vary based on the composition of each of the composites (Filtek Z-350, 3M ESPE; Esthet-X, Dentsply; Durafill VS, Heraeus Kulzer; Amelogen Plus, Ultradent; Charisma Opal, Heraeus Kulzer). Nevertheless, manufacturers do not indicate which chemical substances are responsible for the fluorescence of their products, which encourages investigation into how the chemical composition may influence fluorescence.

A study Studies has shown lower emission of fluorescence from composite resins than from dental structure.[19] Those authors observed that dental tissues showed a greater intensity of excitation than did composite resins at a wavelength higher than 430 nm. Nevertheless, the present study showed that samples of translucent and opaque/translucent composites obtained higher fluorescence intensity values than did dental structure. Moreover, it could be perceived that, when analyzing the mixed opaque/translucent samples or translucent samples, the composites of the same commercial brand presented different fluorescence values, which occurs within natural teeth, for enamel and dentin values. This result is in disagreement with the study of Macedo and others,[13] in which the same brand of composites indicated for reproducing dentin, enamel, or the incisal edge presented equal fluorescence values.

Another relevant finding in the literature is that the fluorescence of composite resins does not follow the same model as that of dental tissues; that is to say that only the superficial layer of composite would be relevant in the fluorescence indices.[23] In this study, composite resin for enamel samples did not obtain lower fluorescence intensities than the mixed opaque/translucent samples. The present study revealed the interference of the subsuperficial layer in the measurement of the fluorescence of composite resins, which contradicts other findings in the literature. Furthermore, other studies agree that the application of sealants and accumulation of pigments may alter the fluorescence of a composite, both in the transmission of light on the surface of the material and in the absorption of the fluorescence emitted.[6,16,21,24]

In some studies, the results indicated that there is a considerable variation in fluorescence between restorative materials and dental structure.[16] Some authors found that the transmission of light was lower than that of dental tissues.[8,13,20,25] Other authors found that the fluorescence intensity of the restorative material was higher, which compromised the quality of the restoration, affecting the esthetic success or failure of restorative treatment.[8,16]

The behavior of the dental structure exposed to light has always been a complicating factor in adequate esthetic restorations, as the dental structure is polychromatic and exhibits different tonalities when exposed to different types of light.[11,25] In the current study, the dental structure could be analyzed in specimens of enamel/dentin, enamel, and dentin, which showed differences in their intensities, proving the polychromatic structure of dental tissues. Dentin exhibited greater fluorescence intensity than enamel because of a higher collagen content, which contains the amino acids responsible for fluorescence, such as tryptophan and hydroxypyridine.[8,9] However, many studies did not include separate test specimens of enamel and dentin for their analyses, obtaining only the combined results of the fluorescence of the two tissues.

The results of this present study showed that the composite resin Filtek Z-350 (A2) most approximated the fluorescence intensity of tooth enamel. Whereas Esthet-X (A2), Amelogen Plus (A2), and Durafill VS (A2) obtained higher values than those of pure enamel, which would compromise the result of the restoration. However, these composite resins presented values closer to the values obtained with the enamel/dentin combination. These results differed from those of previous studies, in which no brand of composite resin for enamel or translucent composite had fluorescence intensities similar to that of enamel, and only Esthet-X OA2 had fluorescence similar to that of human dentin.[8]

All of the combinations of opaque, enamel, and translucent composite resins may vary in terms of fluorescence for different brands. This study demonstrated that different combinations of composite resins/shades interfere with the final fluorescence

result. Fluorescence varies from tooth to tooth and between dental tissues. Therefore, it is of great importance to develop a method capable of directly measuring tooth fluorescence in vivo for developing a future composite resins fluorescence guide. Further studies should establish the fluorescence of the available composite resins and their combinations so that this information can be used in clinical practice in the same way the shade guide is used.

CONCLUSIONS

According to the methodology used, it can be inferred that

- The null hypotheses were refuted, as there were significant differences in fluorescence among the analyzed composite resins and the dental tissues;
- The direct method of measuring fluorescence using a spectrophotometer is efficient, in addition to being a promising tool for selecting composite resins by fluorescence.

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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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Effect of Double-layer Application on Dentin Bond Durability of One-step Self-etch Adhesives

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Clinical Relevance

The double-application technique could be recommended for most one-step self-etch adhesives to improve their bonding capability and to improve the stability of the bond strength to dentin over time.

SUMMARY

Purpose: **The aim of this *in vitro* study was 1) to analyze the influence of a double-layer application technique of four one-step self-etch adhesive systems on dentin and 2) to determine its effect on the stability of the adhesive interfaces stored under different conditions.**

Materials and Methods: **Four different one-step self-etch adhesives were selected for the study (iBondSE, Clearfil S^3 Bond, $XenoV^+$, and Scotchbond Universal). Adhesives were applied according to manufacturers' instructions or with a double-layer application technique (without light curing of the first layer). After bonding, resin-dentin specimens were sectioned for microtensile bond strength testing in accordance with the nontrimming technique and divided into 3 subgroups of storage: a) 24 hours (immediate bond strength, T_0), b) six months (T_6) in artificial saliva at 37°C, or c) five hours in 10 % NaOCl at room temperature. After storage, specimens were stressed to failure. Fracture mode was assessed under a light microscope.**

Results: **At T_0, iBond SE showed a significant increase in microtensile bond strength when the double-application technique was applied. All adhesive systems showed reduced bond strengths after six months of storage in artificial saliva and after storage in 10% NaOCl for five hours; however at T_6, iBond SE, Clearfil S^3 Bond, and $XenoV^+$ showed significantly higher microtensile bond strength results for the**

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double-application technique compared with the single-application technique. Scotchbond Universal showed no difference between single- or double-application, irrespective of the storage conditions.

Conclusion: The results of this study show that improvements in bond strength of one-step self-etch adhesives by using the double-application technique are adhesive dependent.

INTRODUCTION

Simplification and reduced technique sensitivity of adhesive systems are a continuous trend in adhesive dentistry. One approach among simpler and faster innovations is the use of one-step self-etch adhesives, which have been marketed for their ability to achieve standardized applications and durable bond strengths over time.[1] Initially, self-etch adhesives required two separate application steps: application of an acidic primer followed by a layer of a relatively hydrophobic and unfilled bonding agent. To achieve faster application times, manufacturers have incorporated all components of the adhesive system into one solution.[2,3]

Compared with the etch-and-rinse approach, one-step self-etch adhesives are supposed to etch and infiltrate dentin simultaneously, at least from a theoretical point of view.[3] Apart from the success of immediate bonding and sealing, initially the durability of the adhesive interface could not be ensured, especially for the first formulation adhesives in this class.[4,5] Indeed, water is needed to dissociate acidic methacrylates and allow one-step self-etch adhesives to permeate the smear layer and the underlying mineralized dentin.[2] The intrinsic hydrophilic nature of one-step adhesives leads to hydrolytic degradation,[4] suboptimal polymerization,[6] and phase separation,[7] which have been reported to be important factors for degradation of the simplified adhesive interface over time. In particular, because of suboptimal polymerization, the interface created by one-step self-etch adhesives may result in a semipermeable adhesive layer.[8]

Infiltration of adhesives into the dentin and the thickness of the adhesive layer are directly correlated to rheological and chemical characteristics,[9,10] but they could also be influenced by the mode of application.[11] Different clinical approaches have been proposed to improve monomer infiltration: use of an additional layer of hydrophobic resin agent,[12] multiple-layer application,[13-17] enhanced solvent evaporation,[18] and prolonged curing-time intervals[6,17,19] are some of the modifications to the clinical protocol that showed bonding improvements.

Some authors have indicated that an active application of self-etch adhesives on dentin could expedite solvent evaporation, resulting in a higher rate of monomer impregnation into the smear layer.[20] The active application could carry fresh acidic monomers to the underlying dentin, causing more aggressive demineralization and allowing better diffusion of the monomers[20] and increased concentration of comonomers, which could finally improve the quality of the hybrid layer.[9]

Enhanced microtensile bond strength and reduced interfacial nanoleakage were found when the application time of one-step adhesives was prolonged as well as when a hydrophobic coating was applied after a one-step adhesive system.[21,22] It has been reported that double application of one-step self-etch adhesives may result in a more uniform infiltration of the adhesive into smear layer–covered dentin if a one-step self-etch adhesive is applied in two layers.[15,23]

Thus, the aim of the present study was to assess the effect of a double-application technique on recently formulated (Scotchbond Universal, 3M ESPE, Seefeld, Germany), storage-improved (iBond SE, Heraeus Kulzer, Hanau, Germany; XenoV$^+$, Dentsply DeTrey, Konstanz, Germany) or well evaluated (Clearfil S^3 Bond, Kuraray Medical Inc, Tokyo, Japan) one-step self-etch adhesives (Table 1) on immediate and long-term stability of the adhesive interface. The null hypotheses tested in this study were that 1) a double-application technique does not improve the immediate bond strength of the four tested one-step self-etch adhesives and 2) that the double-application technique would not influence bond strength after different aging protocols.

METHODS AND MATERIALS

Four different one-step self-etch adhesives were selected for the study: iBond SE, Scotchbond Universal, Clearfil S^3 Bond, and XenoV$^+$. A total of 56 sound, recently extracted, human third molars were disinfected in 0.5% aqueous chloramine-T solution after written informed consent was obtained from the patients, as required from the local ethics committee, and stored for at least four weeks at 4°C in distilled water. All of them were cut using a low-speed diamond saw (Isomet Low Speed Saw, Buehler, Lake Bluff, IL, USA) under water irrigation until middle/deep dentin was exposed. A standardized smear layer was created on dentin by using 180-

Table 1: *Components, Compositions, and Application Procedure of the Tested Self-etch Adhesives*

Adhesive/Manufacturer	Batch Number	Composition[a]	Application Procedure	Storage
iBond SE (Heraeus-Kulzer, Hanau, Germany) pH 2.0	010110	Acetone, UDMA, TEGDMA, 4-methacryloxyethyltrimellitic anhydride, glutaraldehyde, photoinitiator	Manufacturer's instructions: 1. Scrub adhesive for 20 s on dentin 2. Air thin 3. Light cure for 20 s	Group 1: 24h in distilled water Group 9: 6 mo in artificial saliva at 37°C Group 17: 5 h in NaOCl
			Double application: 1. Scrub adhesive for 20 s on dentin 2. Air thin 3. Scrub adhesive for 20 s on dentin 4. Air thin 5. Light cure for 20 s	Group 2: 24 h in distilled water Group 10: 6 mo in artificial saliva at 37°C Group 18: 5 h in NaOCl
Xeno V^+ (Dentsply DeTrey, Konstanz, Germany) pH 1.3	1101000851	Bifunctional acrylic amides, acrylamidoalkylsulfonic acid, "inverse"functionalized phosphoric acid ester, acrylic acid, ter-butanol, butylatedbenzenediol, water, camphorquinone	Manufacturer's instructions: 1. Apply adhesive agitated for 20 s on dentin 2. Air thin for 5 s 3. Light cure for 10 s	Group 3: 24 h in distilled water Group 11: 6 mo in artificial saliva at 37°C Group 19: 5 h in NaOCl
			Double application: 1. Apply adhesive agitated for 20 s on dentin 2. Air thin for 5 s 3. Apply adhesive agitated for 20 s on dentin 4. Air thin for 5 s 5. Light cure for 10 s	Group 4: 24 h in distilled water Group 12: 6 mo in artificial saliva at 37°C Group 20: 5 h in NaOCl
Scotchbond Universal (3M Espe, Seefeld, Germany) pH 2.7	Uno-VT-Bulk-0001	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond Copolymer, filler, ethanol, water, initiators, silane	Manufacturer's instructions: 1. Scrub adhesive for 20 s on dentin 2. Gently air thin for 5 s 3. Light cure for 10 s	Group 5: 24 h in distilled water Group 13: 6 mo in artificial saliva at 37°C Group 21: 5 h in NaOCl
			Double application: 1. Scrub adhesive for 20 s on dentin 2. Gently air thin for 5 s 3. Scrub adhesive for 20 s on dentin 4. Gently air thin for 5 s 5. Light cure for 10 s	Group 6: 24 h in distilled water Group 14: 6 mo in artificial saliva at 37°C Group 22: 5 h in NaOCl
Clearfil S^3 Bond (Kuraray Medical Inc., Tokyo, Japan) pH 2.6	0157 BA	MDP, bis-GMA, HEMA hydrophobic dimethacrylate, DL-camphorquinone, ethyl alcohol, water, silanated colloidal silica	Manufacturer's instructions: 1. Apply adhesive and leave undisturbed for 20 s on dentin 2. Air thin for 5 s 3. Light cure for 10 s	Group 7: 24 h in distilled water Group 15: 6 mo in artificial saliva at 37°C Group 23: 5 h in NaOCl
			Double application: 1. Apply adhesive and leave undisturbed for 20 s on dentin 2. Air thin for 5 s 3. Apply adhesive and leave undisturbed for 20 s on dentin 4. Air thin for 5 s 5. Light cure for 10 s	Group 8: 24 h in distilled water Group 16: 6 mo in artificial saliva at 37°C Group 24: 5 h in NaOCl

[a] *Composition of the materials as provided by the manufacturers: bis-GMA, bisphenol-glycidyl methacrylate; HEMA, hydroxyethylmethacrylate; MDP, methacryloyl-oxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.*

grit wet silicon carbide papers.[24] Smear layer–covered dentin substrates were then equally randomly assigned to the different treatment groups as shown in Table 1 (N=7). Each dentin bonding system was applied as recommended by manufacturers' instructions or with a double-application step without light curing of the first layer (Table 1).

Dentin bonding systems were light cured according to the manufacturer's instructions (Table 1) with a dental halogen-curing unit (Elipar Trilight, 3M ESPE) with at least 750 mW/cm^2 (checked periodically with a radiometer; Radiometer 100, Demetron Research Corp, Danbury, CT, USA). Four 1-mm thick layers of Filtek Z250 (3M ESPE) were incrementally applied on the bonded dentin surface.[25] Each increment was light cured separately. To facilitate microtensile bond-strength testing (4 mm dentin/4 mm resin composite), the pulp chamber was bonded with Adper Scotchbond Multi-Purpose (3M ESPE, three-step etch-and-rinse adhesive) and filled with Filtek Z250, in accordance with the manufacturer's instructions.

Resin-dentin bonded specimens (0.9 mm × 0.9 mm) were sectioned for microtensile bond strength testing with a low-speed diamond saw under water irrigation in accordance with the nontrimming technique.[26] To convert data into megapascals (MPa), the dimension of each beam was measured using a digital caliper (accurate to ±0.01 mm). Specimens of each tooth were divided into three subgroups of storage: a) 24 hours (T_0) in artificial saliva (5 mM $C_8H_{18}N_2O_4S$, 2.5 mM $CaCl_2$, 0.05 mM $ZnCl_2$, and 0.3 mM NaN_3, pH 7.4) at 37°C,[27] b) six months (T_6) in artificial saliva at 37°C, or c) five hours in 10% NaOCl and then one hour in distilled water at room temperature.

After aging the specimens were fixed on a modified jig for microtensile testing (Zwicki Z 2.5, Zwick Roell, Ulm, Germany) with cyanoacrylate adhesive (Loctite 401 and Aktivator 7455, Loctite, Munich, Germany) and stressed until failure under tension at a crosshead speed of 1 mm/min. All debonded beams during specimen preparation (pretesting failure) were recorded but not included in statistical evaluation. Only values of beams that failed during storage or microtensile testing were noted and analyzed statistically.

Failures were classified as adhesive, cohesive in dentin, cohesive in composite, or mixed and were examined by a single observer using a stereomicroscope (SV11, Zeiss, Oberkochen, Germany) at 50× magnification.

Four representative specimens of fractured microtensile beams of each group that were classified as adhesive or mixed under light microscopy were evaluated under scanning electron miscroscopy (SEM; Leitz ISI-SR-50, Tokyo, Japan) at 50× to 500× magnification.

Bond-strength data were statistically analyzed by using SPSS 19.0 for Windows (SPSS, Chicago, IL, USA). Because groups exhibited non-normally distributed data (Kolmogorov–Smirnov test), nonparametric tests were used. Statistical differences between the groups were analyzed pairwise using the Mann-Whitney U test at a level of significance of $p < 0.05$.

RESULTS

Microtensile bond-strength mean values and standard deviations are shown in Table 2. At T_0 (24 hours of storage in artificial saliva), the double-application technique resulted in statistically significant increased bond strength for iBond SE compared with the control application in accordance with manufacturers' instructions ($p = 0.008$; Table 2); no differences were found for all other tested adhesives (Clearfil S^3 Bond, $p = 0.439$; Scotchbond Universal, $p = 0.593$; and Xeno V$^+$, $p = 0.902$; Table 2). Differences within the adhesive systems were also found at T_0 as XenoV$^+$ showed the statistically significant lowest bond strength among the tested adhesives irrespective of the application mode ($p<0.05$; Table 2).

At T_6 (after six months of storage in artificial saliva), iBond SE ($p = 0.046$), Clearfil S^3 Bond ($p = 0.006$), and XenoV$^+$ ($p = 0.026$) showed statistically significant higher microtensile bond strength when applied with the double-application technique compared with application in accordance with the manufacturer's instructions (Table 2); no differences were found for Scotchbond Universal ($p = 0.578$; Table 2). The effect of aging on the bond strength compared with that of the T_0 specimens was statistically significant for iBond SE and Xeno V$^+$ ($p<0.05$) regardless of the application technique and for Clearfil S^3 Bond if applied in accordance with manufacturer's instructions ($p=0.0015$). Conversely, no reduction in bond strength between T_0 and T_6 was found for Scotchbond Universal (stable bond strength regardless of the application technique, $p>0.05$; Table 2) and for Clearfil S^3 Bond if the double-application technique was used ($p=0.229$; Table 2).

After storage in 10% NaOCl for five hours, all adhesives showed statistically significant reduced

Table 2: *Mean ± Standard Deviation (Number of Intact Sticks Tested/Number of Debonded Specimens During Cutting) of Microtensile Bond Strengths in MPa*[a]

Adhesive System	iBond SE			Clearfil S^3 Bond			Scotchbond Universal			Xeno V$^+$		
Application Procedure	Double Application	Increase of Bond Strength	Manufacturer's Instructions	Double Application	Increase of Bond Strength	Manufacturer's Instructions	Double Application	Increase of Bond Strength	Manufacturer's Instructions	Double Application	Increase of Bond Strength	Manufacturer's Instructions
24 h artificial saliva (T_0)	53.6 ± 15.4^b (49/1)	+18%	44.2 ± 15.0^{a,c} (49/2)	51.0 ± 14.0^b (41/1)	+6%	48.0 ± 14.2^{a,b} (50/1)	53.0 ± 16.8^b (45/1)	+1%	52.3 ± 16.7^b (43/1)	22.1 ± 11.7^e (64/4)	+14%	19.0 ± 12.8^e (66/5)
6 mo artificial saliva (T_6)	42.7 ± 9.5^c (47/2)	+13%	37.1 ± 11.3^d (48/1)	47.6 ± 10.5^{a,b} (52/1)	+18%	39.0 ± 11.1^d (52/3)	48.5 ± 11.4^{a,b} (50/2)	+2%	47.6 ± 11.7^{a,b} (52/2)	9.0 ± 5.4^g (53/3)	+30%	6.3 ± 3.8^{f,j} (52/2)
5 h in 10% sodium hypochlorite	7.6 ± 2.9^{f,g} (37/1)	+70%	2.3 ± 2.3^h (27/1)	7.2 ± 4.2^{f,g} (40/2)	+12%	6.4 ± 3.6^{f,j} (40/0)	12.0 ± 4.4^i (33/2)	+8%	11.0 ± 4.8^i (32/0)	5.5 ± 2.1^j (32/1)	+60%	2.2 ± 1.4^h (25/0)

[a] *Groups identified with same superscripted letters are not significantly different ($p<0.05$). Premature failures due to preparation procedures were not included in the statistical evaluation.*

bond strength ($p<0.05$; Table 2), though Scotchbond Universal showed the highest results regardless of the application mode ($p<0.05$; Table 2). Only Xeno V$^+$ showed failures during storage in 10% NaOCl; these five failures were recorded as 0 MPa in statistical evaluation.

Results of failure mode distribution are shown in Table 3. Analysis of the failure mode exhibited nearly 100% adhesive failures for Xeno V$^+$ regardless of the application mode or storage conditions. When stored in 10% NaOCl, Xeno V$^+$, iBond SE, and Clearfil S^3 Bond showed 100% adhesive failure in all groups, and Scotchbond Universal showed 90% adhesive failures. For Scotchbond Universal and Clearfil S^3 Bond, a significant increase in percentage of adhesive failure was found at T_6 compared with T_0.

The SEM examinations of fractured microtensile sticks from the dentin side are shown in Figures 1 through 4. Adhesive and mixed fracture modes are shown.

DISCUSSION

The results of the present investigation led to the partial rejection of the null hypotheses as the use of a double-application technique increased bond strength or reduced degradation over time for some of the tested adhesives (iBond SE, Clearfil S^3 Bond, and Xeno V$^+$), though no difference was found for Scotchbond Universal. The most pronounced improvement of microtensile bond strength after double application in the present study was achieved for iBond SE, which confirmed previous findings.[13,14,15,21] We can speculate that this unfilled adhesive profits from a thicker adhesive layer

Table 3: *Failure Mode Distribution in Percent*[a]

Adhesive System	Application Procedure	24 h in Artificial Saliva (T_0)				6 mo in Artificial Saliva (T_6)				5 h in 10% Sodium Hypochlorite			
		A	CC	CD	M	A	CC	CD	M	A	CC	CD	M
iBond SE	Double application	46.9	30.6	12.2	10.2	38.3	44.7	12.8	4.3	97.3	0	0	2.7
	Manufacturer's instructions	51.0	22.4	16.3	10.2	72.9	18.8	6.3	2.1	100	0	0	0
Clearfil S^{3}Bond	Double application	70.7	24.4	2.4	2.4	86.3	11.8	2.0	0	100	0	0	0
	Manufacturer's instructions	68.0	26.0	2.0	4.0	84.6	13.5	1.9	2.4	100	0	0	0
Scotchbond Universal	Double application	22.2	60.0	8.9	8.9	52.0	42.0	6.0	0	90.9	0	9.1	0
	Manufacturer's instructions	14.7	67.6	8.8	8.8	38.5	42.3	11.5	7.7	90.6	3.1	3.1	3.1
Xeno V$^+$	Double application	98.4	0	0	1.6	100	0	0	0	100	0	0	0
	Manufacturer's instructions	95.5	1.5	1.5	1.5	100	0	0	0	100	0	0	0

[a] *Abbreviations: A, adhesive failure; CC, cohesive failure in composite; CD, cohesive failure in dentin; M, mixed failure.*

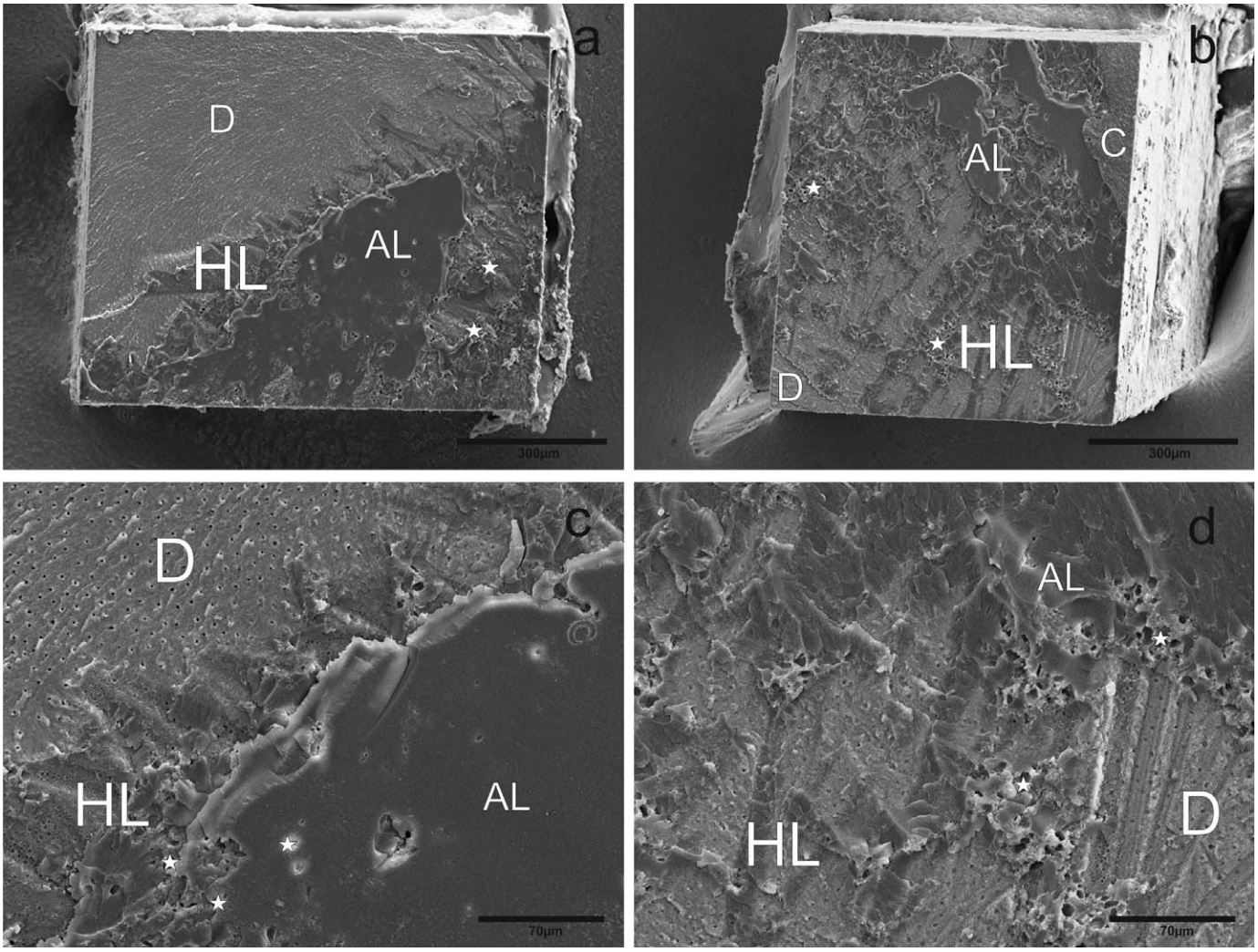

Figure 1. *SEM images of debonded iBond SE specimens after six months (a, c = manufacturer's instructions; b, d = double application) from the dentin side. Abbreviations: AL, adhesive layer; HL, hybrid layer; D, dentin; C, composite. The overall area of adhesive covering the surface (AL) is considerably larger with single application (a, c). In both groups, some osmotic blistering is detectable (asterisks). Mixed-type failures with exposed intertubular dentin are evident under the SEM at larger magnifications.*

resulting from the second application step. Moreover, a second application step of unpolymerized acidic monomers could improve the etching performance of iBond SE by increasing the amount of acidic reagents in direct contact with dentin and reducing the buffer capability of hydroxyapatite. This may synergistically combine with the increased adhesive thickness to explain the increase in microtensile bond strength.[28]

Clearfil S^3 Bond and Scotchbond Universal incorporate the functional monomer methacryloyl-oxydecyl dihydrogen phosphate (MDP). MDP is known for its primary chemical interaction with hydroxyapatite, which occurs within a clinically relevant time span of 20 seconds.[29] This kind of chemical interaction did not increase the immediate microtensile bond strength, but investigations evaluating the biodegradation resistance of adhesive interfaces have shown that it could enhance long-term stability.[30,31]

Clearfil S^3 Bond showed a higher decrease in microtensile bond strength than Scotchbond Universal when used according to manufacturer's instructions. This may be related to the fact that Scotchbond Universal contains Vitrebond Copolymer (3M ESPE), which creates an additional bond to hydroxyapatite,[32] and furthermore, Scotchbond Universal maybe result in better bonding of the filler particles inside the adhesive.

Clearfil S^3 Bond is also known to exhibit debonding of silica filler particles after six months of storage in water, as described by Van Landuyt and others,[33]

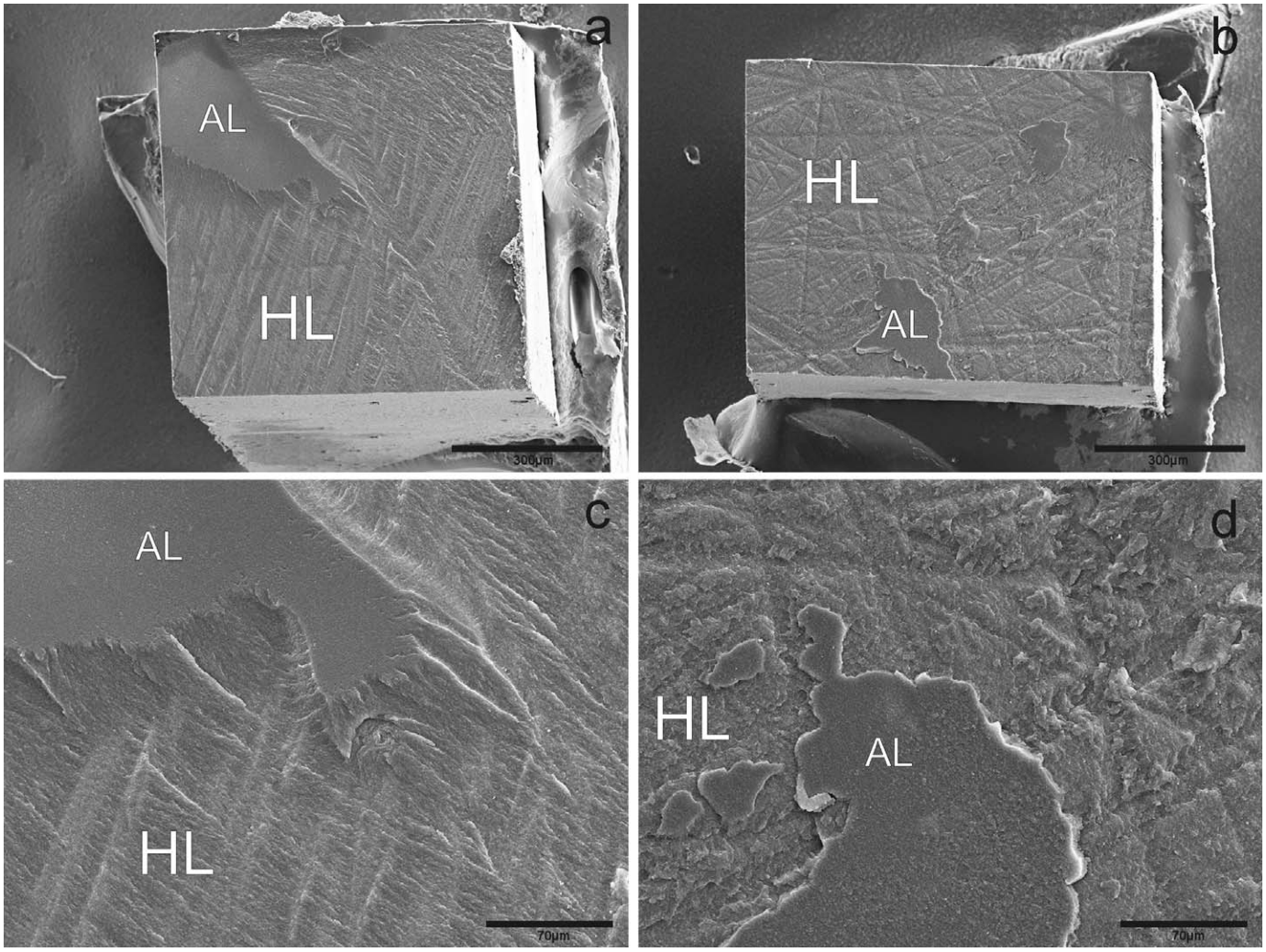

Figure 2. *SEM images of debonded Clearfil S^3 Bond specimens after six months (a, c = manufacturer's instructions; b, d = double application) from the dentin side. Abbreviations: AL, adhesive layer; D, dentin. Dentin areas show a continuous view of the top of the hybrid layer with only few areas still covered with adhesive (AL). Mixed failures with exposed intertubular dentin structures are not found.*

who suggested that water uptake into the adhesive layer leads to hydrolysis of the coupling agent, resulting in filler detachment from the resin matrix. In the present study we may speculate that this phenomenon is responsible for the better performance of Clearfil S^3 Bond when used with the double-application technique. We could also speculate that the observed improvement is due to a thicker adhesive layer or better impregnation of the collagen fibers when Clearfil S^3 Bond is applied twice.

Scotchbond Universal was the only adhesive tested in the present investigation that did not benefit from double application regardless of the aging protocol. Moreover, Scotchbond Universal also remained stable over time if applied in accordance with manufacturer's instructions. This is probably due to its improved curing capability, which allows a high degree of polymer cross-linking, even in the thin adhesive layer created by the single-application technique.[34] So perhaps the filled adhesives form a sufficiently thick adhesive layer in one application step[35,36] resulting in its being less prone to oxygen inhibition.[37]

The double-application technique of self-etch adhesives also influences the etching ability due to increased application time and continuous refreshment of new acidic monomers because of agitated application. This can be of particular importance if a thick smear layer is present because it is known that the smear layer can affect the bonding ability of self-etch adhesives.[38-41] Clinically, the smear layer

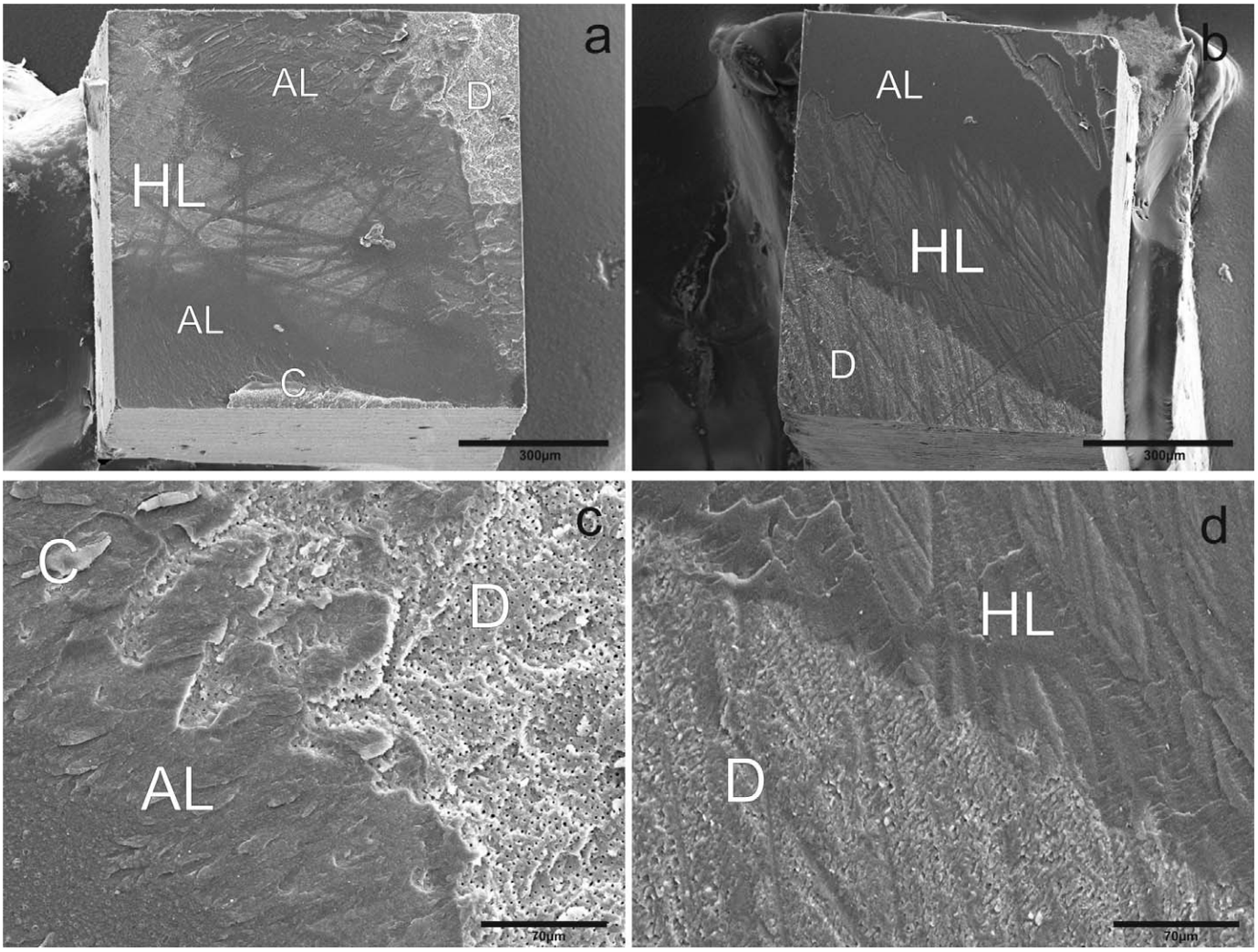

Figure 3. *SEM images of debonded Scotchbond Universal specimens after six months (a, c = manufacturer's instructions; b, d = double application) from the dentin side. Abbreviations: AL, adhesive layer; D, dentin; C, composite. The failure patterns extend more into the dentin (D) when a single application was performed (a, c). The failure site distribution is more uniform when the adhesive was applied twice (b, d).*

created using a medium-grit diamond bur (average particle size of 70 µm) is rougher than the smear layer produced by using 600-grit wet paper used in *in vitro* studies. Because increased smear-layer thickness and greater surface roughness correspond to lower self-etch adhesive impregnation, bond strength obtained after using a 600-grit paper (average particle size of 14.5 µm) may be overestimated due to enhanced adhesive penetration.[42,43] Therefore, in this study 180-grit wet paper (average particle size of 63 µm) was used to prepare a clinically relevant smear layer.[44]

The double-application technique may also result in overetched dentin substrates, which leads to the formation of dentin-unprotected collagen fibrils within the hybrid layer, and this can be responsible for the degradation of the bond over time due to enzymatic degradation.[45,46] An easy way to challenge the durability of this unprotected collagen is to store bonded interfaces in 10% NaOCl at room temperature for five hours, according to Yamauti and others.[47] The potent proteolytic agent NaOCl degrades unprotected collagen due to the presence of superoxide radicals in aqueous solution. This aging method simulates aging within a short time and allows the collection of data on the ability of a dentin bonding system to infiltrate and penetrate the exposed dentin matrix appropriately. This aging protocol was used in previous studies to accelerate aging of the adhesive interface[27] and as an indicator of complete infiltration into the hybrid layer.[48] Additionally, Toledano and others[49] showed that beside the dentin matrix suboptimally polymerized

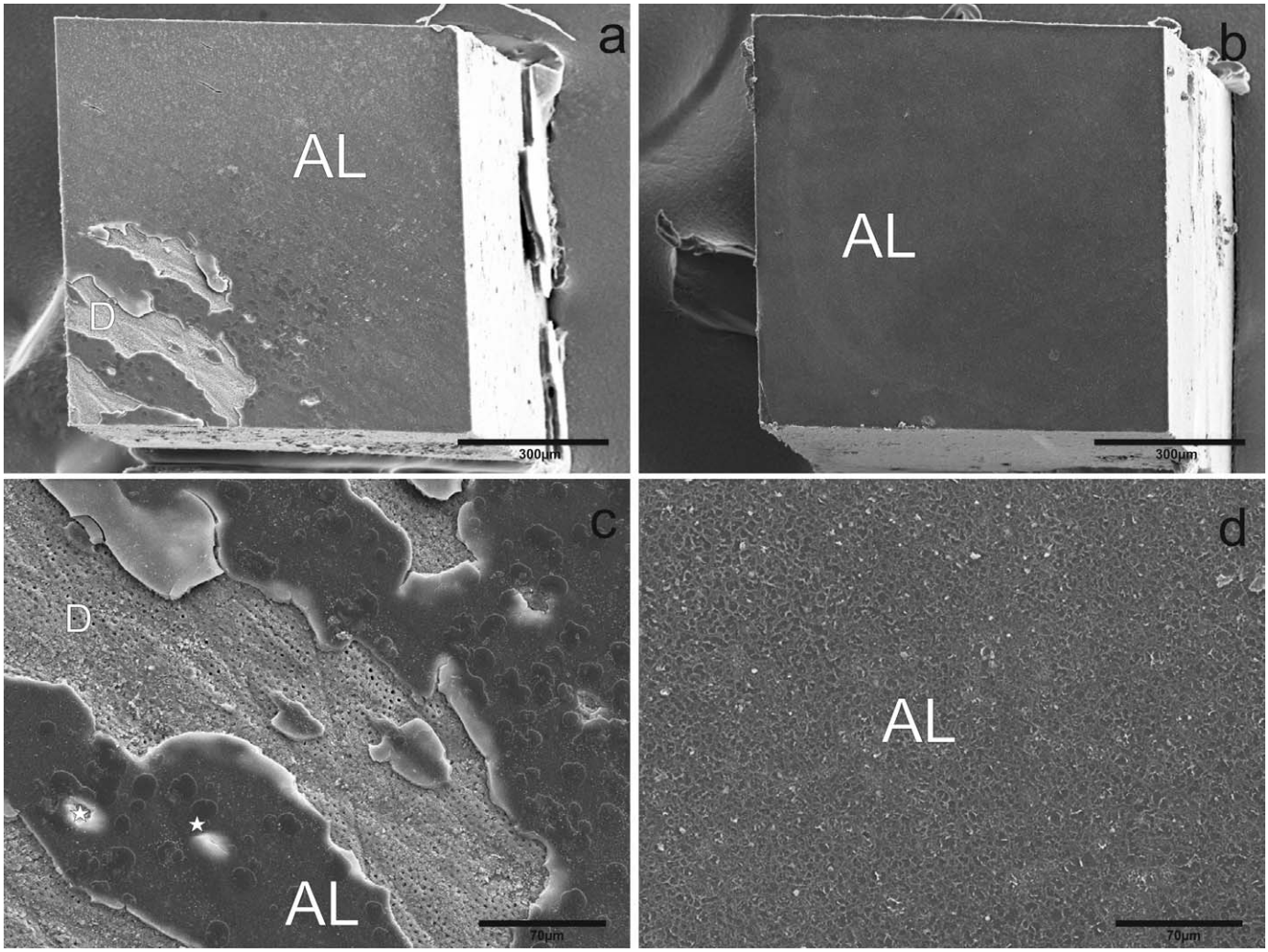

Figure 4. *SEM images of debonded Xeno V⁺ specimens after six months (a, c = manufacturer's instructions; b, d = double application) from the dentin side. Abbreviations: AL, adhesive layer; D, dentin. Some mixed-type failure patterns with exposed dentin areas (D) were detected only after single application. When applied twice, a more uniform failure pattern was evident (d). Also some distinct osmotic blistering areas can be identified (asterisks).*

resin could also be degraded, depending on the adhesive system tested.

The results of this present study show that immersion in 10% NaOCl for five hours is a useful method to challenge the adhesive interface. Whereas previous findings obtained with different adhesive systems showed bond strength reductions comparable to six-month storage in artificial saliva,[27,50] in the present study microtensile bond strength values were lower than the those obtained after storage in artificial saliva for six months.

Overall, in the present study there is a higher tendency for adhesive failures in adhesives with lower microtensile bond strength results and for cohesive failures mainly in composite (CC) for those with higher microtensile bond strength. This phenomenon is in accordance with results from Toledano and others,[51] where more adhesive failures were also observed for one-step self-etch adhesives with lower microtensile bond strength results, either immediately or after artificial ageing. Further clinical trials should investigate the influence of double application of one-step self-etch adhesives on the durability of these bonds over time.

CONCLUSION

In conclusion, the results of this study show that a double application of one-step self-etch adhesives improves immediate bond strength and increases bond stability for some one-step self-etch adhesives.

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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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Comparative *In Vitro* Effect of TiF_4 to NaF and Potassium Oxalate on Reduction of Dentin Hydraulic Conductance

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Clinical Relevance

This study showed that titanium tetrafluoride (TiF_4) varnish and solution were less effective than sodium fluoride (NaF) varnish and potassium oxalate gel in reducing dentin hydraulic conductance. Therefore, TiF_4 might be not successful against dentin hypersensitivity (DH), whereas NaF varnish or potassium oxalate gel could benefit patients with DH.

SUMMARY

Dentin hypersensitivity (DH) is related to an increase in dentin permeability. This study tested the effect of titanium tetrafluoride (TiF_4) compared with sodium fluoride (NaF) and potassium oxalate gel on reducing hydraulic conductance (Lp) from the perspective of diminishing dentin permeability. The Lp of the dentin disks (1.0 ± 0.2 mm) was evaluated using Flodec. The maximum Lp values of each disk were taken after phosphoric acid etching (15 seconds) and randomly allocated to seven groups (n=8) according to the treatments. The minimum (smear layer) and the maximum (after acid etching) Lp values were recorded. Treatments were performed for 4 minutes as follows: 1) NaF varnish 2) and solution (2.45% F, pH 5.0), 3) TiF_4 varnish and 4) solution (2.45% F, pH 1.0), 5) 3% potassium oxalate gel, 6) free fluoride varnish (placebo, pH 5.0), 7) and no treatment (control). The Lp after each treatment was assessed. Samples were exposed to an erosive challenge (6% citric acid, pH 2.1, 1 minute), and the final Lp was recorded. The data were statistically analyzed using repeat-

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ed measures two-way analysis of variance ($p<0.05$). All treatments were effective in reducing dentin Lp compared with the control immediately after the application. However, only potassium oxalate and NaF varnish significantly differed from placebo varnish ($p<0.0001$). The same results were found after the erosive challenge. Therefore, the TiF_4 was less effective than the NaF varnish and potassium oxalate gel in reducing dentin permeability. Using this experimental model, both NaF varnish and potassium oxalate gel reduced the Lp similarly to the presence of smear layer.

INTRODUCTION

Dentin hypersensitivity (DH) afflicts many people.[1] This painful clinical condition often occurs because of dentin exposure resulting from continuous loss of tooth substance as a result of such wear processes as erosion, abrasion, attrition, and abfraction as well as gingival recession. The DH mechanism is still uncertain, although the most accepted hypothesis is the hydrodynamic theory.[2] According to this theory, any decrease in dentinal fluid movement (conductance) should result in reduced DH.[3] Many products have been developed to alleviate this clinical condition, such as potassium oxalate, fluoride, laser, and other agents with dentin occlusive effects.[4,5]

The action of potassium oxalate in diminishing DH is well known. This desensitizing agent obliterates the dentinal tubules by the precipitation of calcium oxalate crystals.[4,6] However, it seems that the crystals formed by the reaction between potassium oxalate and hydroxyapatite are dissolved over time.[7] Additionally, the potassium oxalate does not have any effect against continual dentin erosive wear.

Sodium fluoride (NaF) is one of the most commonly used agents in the treatment of DH.[8,9] Its mechanism of action is attributed to the precipitation of calcium fluoride (CaF_2) on the tooth surface.[4] This layer acts as a mechanical barrier obliterating the opening of the dentinal tubules,[9] minimizing the contact between liquids and dental structure and, consequently, reducing DH.[8,9] Topical fluoride application has also been investigated as a way to decrease the tooth erosive wear,[10] which might be related to the development and progression of DH.[11]

Other fluoride salts besides NaF (eg, titanium tetrafluoride [TiF_4]) have been tested. *In vitro* and *in situ* studies showed that TiF_4 was efficient in minimizing the enamel and dentin erosion compared with other fluoride salts.[12-16] The protective effect of TiF_4 has been attributed not only to the fluoride but also to the titanium that can be immobilized near the surface region of apatite and bonded to the phosphate group, resulting in an acid-resistant metallic layer on the tooth surface.[10,17-19]

Despite the favorable results TiF_4 shows in reducing dentin erosion, its effect in diminishing DH is unknown because only a few studies have been conducted. It has been demonstrated that 0.1% to 1 % TiF_4 solutions have acid-resistant properties to reduce dentin permeability compared with NaF.[20] It also achieved a high level of dentin desensitization (in 75% of patients) when applied *in vivo*.[21]

Therefore, this study compared the effect of TiF_4 varnish and solution, NaF varnish and solution, and potassium oxalate gel on dentin permeability by using hydraulic conductance (Lp) measurements. The tested null hypotheses were that there are no significant differences among the treatment groups and that the control/placebo varnish on the Lp changes 1) after the treatment and 2) after the erosive challenge.

METHODS AND MATERIALS

Experimental Design

This *in vitro* study involved the analysis of two factors: treatment (in seven levels) and dentin condition (in four levels). The response variable was the measurement of Lp.

Tooth Selection and Specimen Preparation

After the Ethics Committee from Bauru School of Dentistry – University of São Paulo, Brazil (Protocol 58/2010) approved the study and the patients signed the informed consent for teeth donation, 220 nonerupted human third molars were obtained. Immediately after the extraction, teeth were stored in 0.02% NaN_3 (sodium azide) solution (Merck, Darmastadt, Germany) at 4°C for a maximum of 6 months. The teeth were sectioned with a diamond disk (XL-12205, Extec Corporation, Enfield, CT, USA) in an Isomet machine (Buehler Ltd, Lake Bluff, IL, USA) at low speed (300 rpm) and under deionized water irrigation. The crown of each tooth was sectioned transversely above the projection of the pulp horns and below the occlusal amelodentinal junction. Only one dentin disk, 1.0 ± 0.2 mm thick, was obtained from the middle third of each molar's crown. The thickness of the disks was controlled using a digital micrometer (Starrett Indústria &

Comércio Ltda, São Paulo, Brazil). A stereoscopic magnifying lens (Meji Techno Co Ltda, Tokyo, Japan) was used to verify whether the disks' surfaces were free of enamel and pulp horn. Thus, 98 dentin disks were obtained in ideal conditions and were stored in deionized water at 10°C until the experiment.

Fluid Flow and Lp Measurements

The rate of fluid flow was obtained using a Flodec machine (DeMarco Engineering, Geneva, Switzerland), as described earlier,[5] for the specimen's selection and distribution proposal and for measuring Lp on the different experimental conditions. A plexiglass split-chamber device was used to connect the dentin disk to the Flodec, allowing the standardization of the dentin surface area (0.282 mm^2) defined by the O-ring through which the fluid passes, under a constant deionized water pressure of 140 cm (2 psi).

The movement of the bubble inside a capillary glass (inside diameter, 0.83 mm; outside diameter, 4 mm; detectable volume by step, 2.71 nL) in the Flodec system was recorded for 5 minutes. The last 3 minutes was used to obtain the Lp for each experimental condition described in the following sections.

Selection of the Specimens

Both sides (occlusal and pulpal) of the 98 dentin disks were first etched by immersion in a 37% phosphoric acid solution for 15 seconds and rinsed in deionized water to ensure maximum permeability. After that, the selected disks were drafted using SPSS program version 13.0 (SPSS Inc, Tulsa, OK, USA). Next, 56 disks were randomly distributed to seven groups (n=8) corresponding to the experimental treatments and controls, so that the mean baseline permeability values were not statistically significant different among the groups (analysis of variance [ANOVA]; $p>0.05$).

Experimental Procedures and the Treatments

Four measures of the Lp were performed for each dentin disk, followed by these conditions: 1) in the presence of the smear layer created by polishing the occlusal side of the dentin disks with 600-grit SiC abrasive paper (Buehler Ltd), which represented minimum permeability; 2) after the removal of the occlusal smear layer with 37% phosphoric acid gel for 15 seconds to obtain maximum permeability; 3) after application of the tested materials (Table 1) (treatment permeability); and 4) after an erosive challenge with 6% citric acid (Merck), pH 2.1, for 1 minute (final permeability).

Details about the treatments are displayed in Table 1. Before application of the materials on the surface of each dentin disk, the disk was gently dried with an absorbent paper. All materials were applied for 4 minutes, and the dentin disk surfaces were washed with deionized water for 30 seconds. Fluoride solutions were applied using a pipette (v=0.2 mL/sample). The varnishes were applied with a microbrush and removed with a probe, taking care not to touch the dentin surface. Potassium oxalate was applied with a cotton swab.

Statistical Analysis

GraphPad InStat version 2.0 for Windows and GraphPad Prism software version 4.0 for Windows (Graph Pad Software, San Diego, CA, USA) were used to perform the statistical analysis. The assumptions of equality of variances and normal distribution of errors were checked for all the variables tested using the Bartlett and Kolmogorov-Smirnov tests, respectively. Because the assumptions were satisfied, repeated measure two-way ANOVA (variables = treatment and dentin condition) followed by Bonferroni post hoc test were used. The significance level was set at $\alpha=0.05$.

RESULTS

Mean values and standard deviations of Lp measurements (percent of Lp reduction compared with the maximum Lp) of the dentin disks treated with the tested materials are displayed in Table 2. Repeated measures two-way ANOVA revealed significant intragroup (criteria = dentin conditions; $p<0.0001$) and intergroups (criteria = the treatments; $p<0.001$) differences and interactions between them ($p<0.001$).

Mean minimum and maximum Lp values within groups differed significantly from each other. Treatment Lp means were similar to minimum Lp values for NaF varnish and potassium oxalate gel only. The same results were found after the erosive challenge: the final Lp means for NaF varnish and potassium oxalate gel were still similar to the minimum values. For the other treatments, the treatment Lp and final Lp values were significantly higher than the minimum Lp values.

All tested materials were effective in reducing the dentin Lp compared with the control immediately after treatment (treatment Lp). After the erosive

Table 1: *Experimental Materials Applied on the Occlusal Surface of Dentin Disks*

Material	Manufacturing	Components	pH
Control	—	—	—
Potassium oxalate gel	Ativus Farmacêutica, Valinhos, SP, Brazil	3% Potassium oxalate monohydrate carboxymethylcelulose	4.1
NaF solution	Sigma-Aldrich, St Louis, MO, USA	5.42 % NaF (2.45% F, pH adjusted with 5 M H_3PO_4 solution)	5.0
TiF_4 solution	Sigma-Aldrich	4% TiF_4 (2.45% F)	1.0
NaF varnish	FGM-Dentscare, Joinvile, SC, Brazil	5.42% NaF (2.45% F, synthetic resin, pH adjusted with 5 M H_3PO_4 solution)	5.0
TiF_4 varnish	FGM-Dentscare	4% TiF_4 (2.45% F, synthetic resin)	1.0
Placebo varnish	FGM-Dentscare	Synthetic resin without fluoride	5.0

challenge (final Lp), all treatments were also found to be effective in maintaining the reduced dentin Lp compared with the control. However, only potassium oxalate and NaF varnish significantly differed from placebo varnish in both conditions. No differences were found among NaF, TiF_4, and potassium oxalate.

DISCUSSION

Almost all desensitizing agents occlude tubules by salt precipitation or resin deposition. Therefore, partial or total tubule obstruction could reduce fluid movement and consequently, reduce the DH.[4] Accordingly, this study applied a method to measure fluid movement *in vitro*.

A method to register dentin fluid movement is the assessment of Lp by using Flodec.[4,5,22] One limitation of the method is that the etched dentin disks, when kept in deionized water for more than 1 week, partially lose their maximum permeability. Because of this, a large number of teeth must be collected (in our case, 220 teeth) to achieve the expected number of samples for a limited time of storage (n=8 per group; 56 disks). Other aspects can also influence the selection of the samples, such as the size and thickness of the dentin disk and the absence of enamel and pulp horn.

Although the maximum Lp represents the opened dentin tubules, the minimum Lp is the permeability of the smear layer (eg, nearly no permeability). The desensitizing agent is considered effective when the Lp values achieved are similar to those found in the presence of smear layer.

This study mainly focused on TiF_4 treatment, as it is considered a potential anticariogenic and antierosive agent.[10] Because of its mechanism of action,[17-19] it was thought that this metallic fluoride could occlude dentin tubules and reduce DH.[20,21] However, only potassium oxalate and NaF varnish were able to significantly reduce the dentin Lp compared with the control and placebo varnish. Both treatments were potentially effective as occlusive agents, as they achieved Lp values (treatment and final Lp means) similar to the minimum values. Therefore, the null hypotheses of this study were rejected.

Table 2: *Mean (Standard Deviation) of the Lp Values ($\mu l\ c^{-2}\ min^{-1}\ cm\ H_2O$) of the Different Treatment Groups Under Four Experimental Dentin Conditions (n=8)*[a]

	Minimum (With Smear Layer)*	Maximum (After Acid etching)	Treatment*	Final (After Erosive Challenge)*
Control	1.4 (1.8)^{Aa} (93.9%)	22.8 (14.6)^{Ba}	22.0 (9.7)^{Ba} (3.5%)	24.2 (7.2)^{Ba} (−6.0%)
Potassium oxalate	1.4 (1.8)^{Aa} (94.5%)	25.5 (12.2)^{Ba}	3.9 (3.0)^{Ac} (84.7%)	5.6 (4.0)^{Ac} (79.1%)
NaF solution	0.6 (0.6)^{Aa} (96.9%)	19.7 (8.7)^{Ca}	10.0 (6.1)^{Bc} (49.2%)	13.6 (6.1)^{BCbc} (31.0%)
TiF_4 solution	1.8 (0.9)^{Aa} (89.7%)	17.5 (7.9)^{Ba}	11.3 (10.1)^{Bbc} (35.4%)	11.3 (7.4)^{Bbc} (35.4%)
NaF varnish	1.4 (0.8)^{Aa} (93.0%)	19.9 (8.6)^{Ba}	6.9 (3.5)^{Ac} (65.3%)	7.6 (3.8)^{Ac} (61.8%)
TiF_4 varnish	0.7 (0.5)^{Aa} (96.0%)	17.6 (8.1)^{Ba}	11.0 (4.0)^{Bbc} (37.5%)	3.6 (5.8)^{Bbc} (22.7%)
Placebo varnish	1.1 (0.9)^{Aa} (94.5%)	20.1 (4.4)^{Ba}	18.7 (7.8)^{Bab} (7.0%)	17.6 (7.8)^{Bab} (12.4%)

[a] *Mean percent of Lp reduction compared with the maximum Lp values. In the same row (intragroup comparison), different uppercase superscript letters indicate significant difference among the experimental conditions for each treatment separately ($p<0.05$). In the same column (intergroups comparison), different lowercase superscript letters indicate significant differences among the treatments for each condition individually ($p<0.05$) (repeated measures two-way ANOVA: the experimental conditions, $p<0.0001$; the treatments, $p<0.001$; and the interaction between them $p<0.001$).*

The positive effect of potassium oxalate is attributed to the formation of a insoluble salt of calcium oxalate that can precipitate and penetrate the dentin tubules, partial or totally obliterating them and reducing the dentin permeability[23] and consequently the DH.[4,24] The gel is able to penetrate the tubule and react with calcium ions from the dentin fluid to form insoluble calcium oxalate crystals.[4]

On the other hand, the mechanism of action of the conventional fluoride (NaF) occurs by the precipitation of CaF_2 on the dental surface, which apparently acts as a mechanical barrier,[9] which could also affect dentin permeability and DH.[8] However, clinical studies have shown that its antihyperesthesia effect is of short duration.[25] Among the NaF agents, only the varnish was able to maintain an Lp similar to the minimum Lp after an erosive acid attack. It might be speculated that NaF varnish created more stable crystallization retention inside the dentinal tubules than the NaF solution.

Corroborating the findings, previous studies have shown that TiF_4 solution created a precipitated layer on peri- and intertubular dentin with partial or no tubule covering. Nothing has been seen inside the tubules.[10,15] The present study was developed on dentin without a smear layer, and TiF_4 was unable to reduce Lp as the smear layer. Kazemi and others[20] showed that TiF_4 applied on dentin containing smear layer reduced dentin permeability after an erosive attack. Therefore, it might be speculated that the formation of the TiF_4 glaze layer on a dentin surface is modulated by the presence of a smear layer. Because of the low pH of the TiF_4, its interaction with the smear layer might allow the precipitation of the layer throughout the dentin surface, which would prevent not only erosion, as shown by previous studies,[10] but probably the DH as well.[21] Futures studies should be done to test the effect of TiF_4 on Lp values when applied on dentin in the presence of a smear layer, as happens in a clinical situation.[21]

Despite the ineffectiveness of TiF_4, the samples treated with this fluoride salt did not show an increase in Lp values after erosive challenge. Preservation of Lp may be attributed to some superficial precipitation, which does not behave like the smear layer at all.

One important point to be considered in interpreting the data is that the varnish usually remains for a longer time on the tooth surface than the solution. Therefore, the results of this *in vitro* study might not be directly extrapolated to clinical conditions, where the contact of the varnish with the tooth lasts longer, and the saliva might have interplay on the DH process and the reaction between the fluoride salt and the tooth. Accordingly, future studies should be done to test the performance of the experimental agents against DH in a clinical trial.

The longevity of the precipitates or resins on or inside dentinal tubules and their ability to resist acid challenge over time are also unknown. It has been shown that oxalate precipitates appear to wash out under challenge in the clinical environment, and the effect of NaF is reduced after 3 and 6 months of application.[25] Considering that the clinical effectiveness of these materials depends in part on the dissolution resistance or solubility level of precipitates or resins, new studies should be done, including repetitive erosive challenges.

CONCLUSIONS

In conclusion, TiF_4 was less effective than NaF varnish and potassium oxalate gel in reducing dentin permeability using this experimental model. The NaF varnish and potassium oxalate gel seem to be good materials to reduce dentin permeability and, consequently, DH.

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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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Color Stability of Resin Used for Caries Infiltration After Exposure to Different Staining Solutions

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Clinical Relevance

Patients should be aware of minimizing the intake of colored staining solutions when submitted to resin infiltration treatment of white spot lesions in esthetically compromised areas.

SUMMARY

Purpose: **The aim of this study was to investigate the staining behavior of demineralized enamel infiltrated by low-viscosity resin.**

Methods and Materials: Bovine enamel/dentin cylindrical samples (3 × 2 mm) were assigned into four groups (n=45) according to the enamel treatment: sound enamel (control), demineralization + artificial saliva, demineralization + daily application of 0.05% NaF, demineralization + resin infiltration (Icon, DMG). Artificial white spot lesions were produced in groups with demineralization. After the treatments, color was assessed by spectrophotometry, using the CIE L*a*b* system. The specimens (n=15) were then immersed in deionized water, red wine, or coffee for 10 minutes daily for eight days. Color was measured again, and the specimens were repolished with sandpaper discs. The final color was assessed. Data were analyzed by two-way analysis of variance and Tukey tests (α=0.05). A paired *t*-test was used for comparison between staining and repolishing conditions.

Results: **There were significant differences for surface treatment and dye after staining and repolishing. Immersion in wine and coffee resulted in significantly increased color alteration (ΔE) compared with water (p=0.001). The resin-infiltrated group exhibited the highest staining values (p=0.001). The repolishing procedures resulted in significantly decreased color change.**

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Conclusion: **The exposure of specimens to colored solutions resulted in significant color alteration. The demineralized enamel treated with resin infiltration showed significantly higher staining than all other tested groups; however, the repolishing of the specimens minimized the staining effect.**

INTRODUCTION

The caries lesion is a product of the dynamic process comprising several disorders of mineral balance between the tooth and the fluid of adjacent dental plaque, resulting in mineral loss. This loss can be reflected clinically in different ways from its first manifestation as enamel opacity (white spot), to large cavities that can extend up to the dental pulp.[1]

The active white spot is a consequence of an optical effect related to light scattering, due to differences in the refractive index of the involved components. A greater scattering is obtained when the porosities of demineralized enamel are filled with air, since its refractive index is 1.00, lower than the refractive index of hydroxyapatite (1.62-1.65). This results in a whitish and opaque appearance of the enamel. If the enamel porosities of an early-stage lesion are filled with an aqueous solution showing the same refraction index of hydroxyapatite, the existing spaces are full and the enamel keeps its translucency.[2]

Since the stage of white spot does not involve enamel cavitation, noninvasive treatment with topical fluorides associated with diet and hygiene procedure orientation are recommended. However, the whitish appearance may remain, even if the lesion is arrested. This is because the remineralization of deeper lesions occurs only superficially, so that the body of the lesion remains porous and therefore still whitish, as a permanent scar.[3,4]

The presence of white spots in the anterior teeth can compromise esthetics, and it is frequent in orthodontic patients that do not perform oral hygiene adequately, as the excess of the material that promotes the adhesion between the orthodontic bracket and the tooth represents a critical factor for plaque accumulation. In addition, the cleaning action promoted by saliva, and chewing is reduced around the brackets.[3] The cosmetic treatment for white spot lesions that remain as scars generally involves removal of affected tissue, by microabrasion of enamel[5,6] or conventional restoration.[7]

The resin infiltration technique was recently introduced in dentistry to prevent further progression of enamel lesions. The objective is to fill the pores within the lesion body by capillary action, with a low-viscosity light-cured resin. This prevents further diffusion of bacteria, and consequently, lesion development, and establishes a barrier within the caries lesion, which can reinforce the enamel structure, avoiding or delaying cavitation and disruption of the surface.[8,9] This technique requires no preparation or anesthesia and does not alter the anatomical shape of the tooth.[10]

Although this treatment was first introduced to arrest caries lesions, an additional positive effect was the esthetic improvement of anterior teeth when white spot lesions were present. This occurs due to the infiltration of the enamel porosities with the low-viscosity resin, which alters the refractive index of the light and consequently the final tooth appearance. Since the refractive index of the infiltrant resin (1.51) is close to hydroxyapatite, its ability to mask white spot lesions was observed.[11-13]

The resin-based materials are widely used in esthetic restorations, but they are subject to color alteration over time. The most common causes of discoloration are adsorption of dyes as a result of exposure to exogenous sources.[14] The extent of discoloration varies according to the patient's habits, such as oral hygiene and diet.[14-18]

Whereas the resin infiltration technique seems promising for dentistry (and as previously mentioned, resins may suffer discoloration), this study investigated the staining behavior of low-viscosity resin used for infiltration. It also investigated the effect of repolishing in the color alteration of the stained specimens.

The null hypotheses tested were that the color of the demineralized enamel surface treated with topical fluoride and resin infiltration is not changed after exposure to dyes and that the polishing of the specimens after staining does not interfere with their color.

METHODS AND MATERIALS

Specimen Preparation

The methods described by Wiegand and others[19] were used to prepare the specimens. For that, 90 extracted, nondamaged bovine incisors were stored in 0.1% thymol solution at room temperature until required. Enamel-dentin specimens, 3 mm in diameter and 2.2 mm in height, were prepared from the labial surface with a trephine mill.

The specimens were positioned in a silicon mold with a cavity 6 mm in diameter and 2 mm in depth. On the bottom of the mold, there was a second-level cavity 3 mm in diameter and 0.1 mm depth, as described in a previous study.[20] The specimens were placed inside the internal cavity with the enamel surface to the bottom of the mold. The mold was filled with low-viscosity composite resin (Opallis Flow, FGM, Joinville, Brazil) and light cured for 40 seconds. On the side of the mold, there was a projection in the shape of a line that produced a lateral groove on the specimen that helped to achieve the correct position at the time of the color reading. The specimens were attached to a metal holder, and 0.1 mm of enamel was removed by polishing with sequential aluminum oxide abrasive papers (1200, 2400, and 4000 grit, FEPA-P, Struers, Ballerup, Denmark) in a polishing device (DP- 10, Panambra Industrial e Técnica SA, São Paulo, SP, Brazil) for 20 seconds each. The dentin side of specimens was abraded with a 1200-grit abrasive paper, removing 0.1 mm of dentin and resulting in specimens of 1 mm of enamel and 1 mm of dentin. The prepared specimens were examined under the stereomicroscope to certify the absence of cracks or other surface defects. After preparation, the specimens were stored in 0.1% thymol solution to avoid dehydration.

Prior to treatment, the baseline L* value of each specimen was assessed (M1) under standardized ambient conditions according to the Commission Internationale de l'Eclariage (CIE) L*a*b* system, using a spectrophotometer (CM2600d, Konica Minolta, Osaka, Japan). The device was adjusted to use the D65 standard light source with 100% UV and specular component included. The observer angle was set at 2°, and the device was adjusted to a small reading area (SAV). The color of each sample was measured three times and averaged. The results of color measurements were quantified in terms of three coordinate values (L*, a*, b*), as established by CIE, which locates the color of an object in a three-dimensional color space. The L* axis represents the degree of lightness within a sample and ranges from 0 (black) to 100 (white). The a* plane represents the degree of green/red color, while the b* plane represents the degree of blue/yellow color within the sample.

According to the L* value, a stratified allocation was performed among 12 groups (n=15). In three groups, the enamel was kept sound and stored in daily changed artificial saliva throughout the study period (groups SE/n=45). In the other groups, artificial enamel caries lesions were created, as described below (groups DE/n=135).

Specimen Demineralization

Following the proposal of Queiroz and others,[21] artificial enamel subsurface lesions were produced by individually immersing and storing the specimens in a buffer solution. The demineralizing solution was composed of 50 mM acetate buffer solution containing 1.28 mM $Ca(NO_3)_2{*}4H_2O$, 0.74 mM $NaH_2PO_4{*}2H_2O$, and 0.03 ppm F at pH 5.0 for 16 hours. The specimens were immersed separately in the solution at 37°C. The total volume of solution used was calculated using 2 mL/mm^2 of the enamel area. This method produces subsurface lesions with a mean depth of 43 μm.[22]

Experimental Design

The demineralized groups were divided according to treatment of white spot lesions (n=45): AS (artificial saliva), specimens were stored in 5 mL of artificial saliva for four weeks, changed every day, and DF (0.05% fluoride solution), specimens were immersed daily for one minute in 1 mL of 0.05% NaF solution for four weeks. The fluoride solution was manipulated in our laboratory. After the daily fluoride immersion, the specimens were rinsed with deionized water and stored in artificial saliva; RI (resin infiltration), specimens were resin infiltrated (Icon, DMG, Hamburg, Germany) and stored in artificial saliva for four weeks. The infiltration procedure was performed according to the manufacturer's instructions. A 15% hydrochloric acid gel (Icon-Etch) was applied on the demineralized enamel surface for two minutes and then water rinsed and air dried for 30 seconds, followed by the application of ethanol (Icon-Dry) during 30 seconds and additional air drying. The low-viscosity resin infiltrant (Icon-Infiltrant) was applied on the surface two times, the first time for three minutes and the second time for one minute. Both applications were light cured for 40 seconds. Specimens were polished with aluminium oxide abrasive papers (4000 grit, FEPA-P; Struers) for 20 seconds for removal of the excess resin. A pilot study was conducted to verify the thickness of the specimens before and after the resin infiltration using a micrometer, and it was determined that the application of the 4000-grit paper for 20 seconds was adequate to remove the excess layer of infiltration resin.

Artificial saliva was prepared according to the formulation of Gohring and others.[23] The pH was

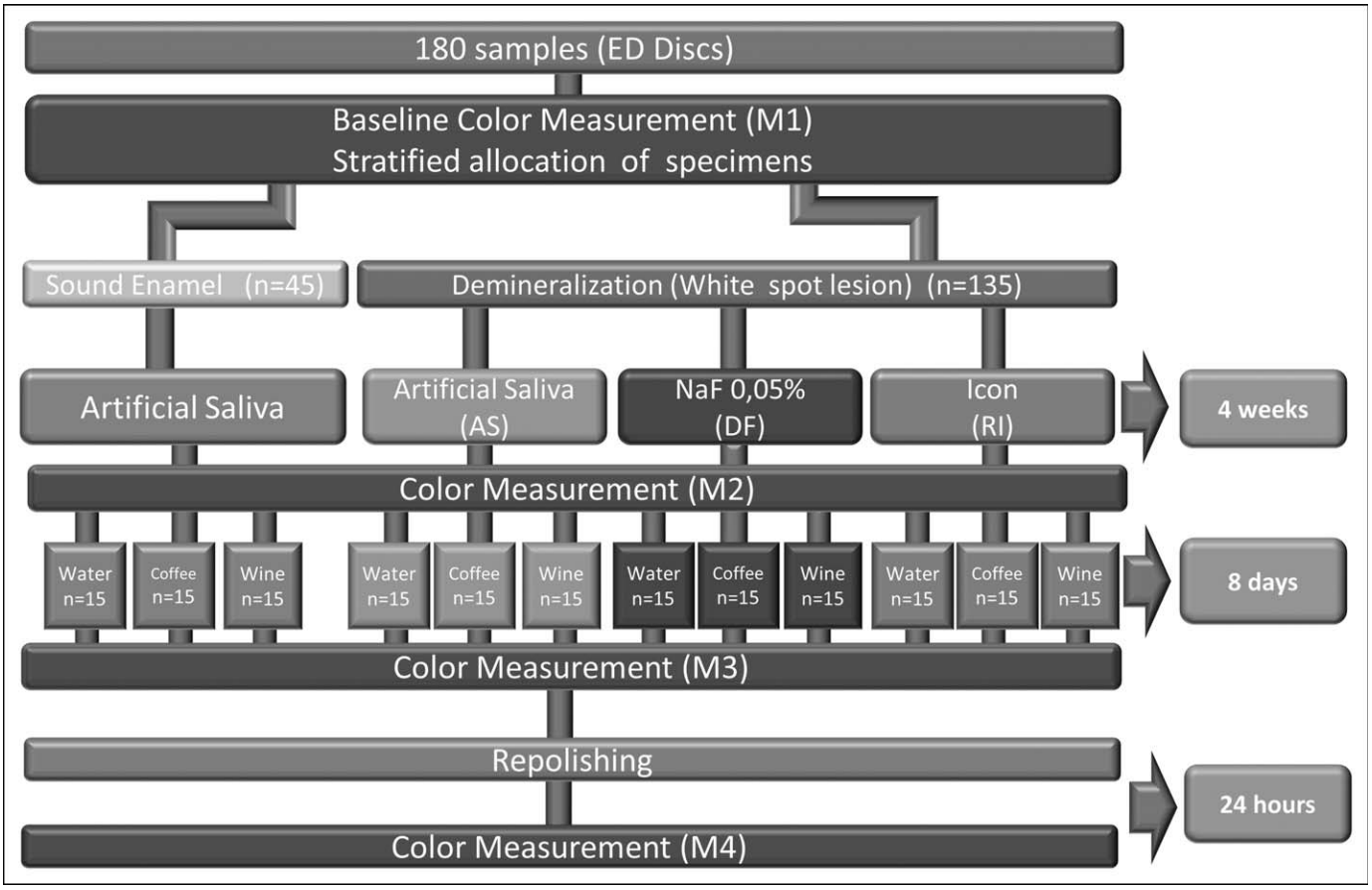

Figure 1. *Experimental Design*

adjusted to 7.0. After four weeks, the color was measured again (M2) for all samples, and then each group was divided into subgroups (n=15) according to exposure to 250 mL of the following solutions: deionized water, soluble coffee solution prepared using 25 g of powder to 250 mL of water (Tradition Nescafé, Nestlé, Araras, SP, Brazil, pH = 5.0), and red wine (Santa Carolina, Merlot, Chile, 2010, 12.3°G.L., pH = 3.4).

The specimens remained immersed in the solutions mentioned above for 10 minutes daily during eight days and stored in artificial saliva in the intermediate periods.[24] The solutions were changed at each exposure. After this period, the specimens were rinsed with deionized water and color measurement was performed (M3).

The surface of the specimens was then polished using fine-grained abrasive discs (FGM) for five seconds and immersion in deionized water for rehydration for 24 hours. After this period, the color was measured again (M4).

The differences in the values of L*(ΔL), a*(Δa), and b*(Δb) were determined and the overall change in color (ΔE) was calculated using the following formula:

$$\Delta E_1 = [(L_{M3*} - L_{M2*})^2 + (a_{M3*} - a_{M2*})^2 + (b_{M3*} - b_{M2*})^2]^{0,5}$$

$$\Delta E_2 = [(L_{M4*} - L_{M2*})^2 + (a_{M4*} - a_{M2*})^2 + (b_{M4*} - b_{M2*})^2]^{0,5}$$

Figure 1 shows the experimental design.

Statistical Analysis

Data were statistically analyzed using two-way analysis of variance and Tukey's tests for color measurement after staining and after repolishing. A paired *t*-test was conducted to evaluate if the repolishing procedures influenced the specimens

Table 1: *Mean and Standard Deviation of Color Changes After Dye Immersion (ΔE_1) and Repolishing (ΔE_2)*

Group	Sound Enamel		White Spot Lesion + AS		White Spot Lesion + DF		White Spot Lesion + RI	
	ΔE1[a]	ΔE2[b]	ΔE1[a]	ΔE2[b]	ΔE1[a]	ΔE2[b]	ΔE1[a]	ΔE2[b]
Water	2.4 (1.3)a	2.2 (0.9)AB	2.9 (1.6)a	2.0 (1.3)AB	2.7 (2.2)a	1.6 (1.8)A	2.3 (1.7)a	2.1 (0.9)AB
Wine	14.4* (4.2)cd	10.1* (2.2)C	11.1* (5.1)bc	9.3* (5.4)C	14.2* (2.7)cd	8.4* (3.2)C	17.3* (2.7)de	14.7* (4.7)D
Coffee	14.5* (4.4)cd	8.1* (4.5)C	9.5* (5.9)b	8.4* (5.7)C	7.3* (2.3)b	6.0* (1.4)BC	21.3* (4.3)e	16.6* (4.3)D

** Significant difference between ΔE1 and ΔE2 for each substrate condition, using a paired t-test (p < 0.05).*
[a] Different capital letters mean significant differences among the groups for ΔE1 (p < 0.05).
[b] Different lowercase letters mean significant differences among the groups for ΔE2 (p < 0.05).

color, for each experimental condition. The significance level used was $p<0.05$.

RESULTS

The overall color change of the specimens after dye immersion (ΔE_1) and after repolishing procedures (ΔE_2) for all experimental conditions is shown in Table 1. The cross-product surface treatment vs dye as well as the factors surface treatment and dye were statistically significant ($p=0.001$).

The paired *t*-test compared color changes after dye immersion (ΔE1) and after repolishing (ΔE2), group by group. For all experimental conditions with coffee or wine immersion, the repolishing procedures resulted in significantly decreased color change (Table 1).

Table 2 shows the results of the surface treatment as a factor. The color change after dye immersion and after repolishing was significantly higher for the white spot lesion + resin infiltration group.

The results of the Tukey test for the dye factor are shown in Table 3. The immersion in coffee and wine resulted in a significant color alteration compared with water exposure. Analysis of L*a*b* values showed decreasing lightness values (ΔL) for coffee and wine, whereas Δa remained stable. The specimens immersed in the coffee solution exhibited an increase in Δb.

DISCUSSION

Several methods can be used to evaluate tooth color with visual comparison with tooth color shade guides, image analysis, colorimeters, or spectrophotometers.[18,25] Since spectrophotometers allow an objective color assessment and provide precise quantitative data, this was the method used in the present study.[26,27] The spectrophotometer presents L*, a*, and b* values; therefore, as the staining solutions alter the specimens color in all axis, the parameter ΔE was used as a response variable, because it indicates the color change of specimens at two different moments, allowing one to evaluate the overall color change.[12,27] In the present study, the most affected parameter was L*, indicating a reduced lightness of stained specimens.

Because of the difficulties in obtaining human teeth with the ideal characteristics for the experiment, bovine incisors were used, providing discs with standardized enamel/dentin thickness. The chemical and physical properties of bovine substrate, such as composition, density, and microhardness, are very similar to human enamel.[28] Bovine and human substrates are also found to have a similar behavior regarding staining effects.[29]

Tooth color perception is associated with scattering of incident light on the tooth structure and its absorption along this way.[30] The initial enamel caries lesions is characterized by a whitish appearance, due to changes of the refractive index of the light in the demineralized tissue compared with the surrounding sound enamel,[31] causing an undesirable esthetic appearance when present in the anterior teeth.

When properly treated with preventive measures, the white spot lesions may become arrested and exhibit a smooth and shiny appearance. In the remineralization process, saliva plays an important role, providing minerals that will partially replace the mineral lost during the demineralization process.[31] Nevertheless, during this remineralization phase, dyes can be incorporated into the lesions,

Table 2: *Mean and Standard Deviation (SD) Data and Results of Tukey Test for Surface Treatment for ΔE_1 and ΔE_2*

Surface Treatment	After Staining (ΔE_1)*	After Repolishing (ΔE_2)*
Sound enamel (control)	10.43 (6.70)b	6.80 (4.45)a
White spot lesion + AS	7.85 (5.77)a	6.57 (5.58)a
White spot lesion + DF	8.07 (5.32)a	5.33 (3.59)a
White spot lesion + RI	13.63 (8.83)c	11.13 (7.43)b

** Different letters imply significant difference (p=0.001) among the groups.*

Table 3: *Mean and Standard Deviation (SD) Data of ΔL, Δa, Δb, and ΔE After Staining and After Repolishing*

Dye	After Staining				After Repolishing			
	ΔL	Δa	Δb	ΔE_1 [a]	ΔL	Δa	Δb	ΔE_2 [b]
Water	1.67 (1.65)	−0.16 (0.30)	−1.19 (1.61)	2.58 (1.73)a	−0.31 (1.51)	−0.16 (0.25)	−0.91 (1.53)	1.99 (1.27)A
Coffee	−9.94 (5.67)	1.16 (1.17)	8.34 (4.25)	13.16 (6.92)b	−8.06 (4.90)	0.49 (0.86)	5.18 (3.59)	9.78 (5.83)B
Wine	−13.22 (4.62)	1.36 (0.81)	−4.19 (2.40)	14.25 (4.33)b	−10.27 (4.77)	0.66 (0.67)	−1.60 (1.65)	10.59 (4.69)B

[a] *Different lowercase letters imply significant difference among the groups for ΔE_1 (p=0.001).*
[b] *Different capital letters imply significant difference among the groups for ΔE_2 (p=0.001).*

resulting in the formation of unesthetic brown spots. In this study, the staining of the demineralized enamel groups after exposure to saliva and fluoride as remineralizing agents and immersion in dyes was observed. In general, immersion in coffee and wine promoted significant color change of specimens, regardless of the type of treatment of white spot lesion. Therefore, the first null hypothesis was rejected.

Coffee and red wine were chosen as dye testing substances because they are frequently consumed.[32] Coffee exhibits a strong potential for staining both tooth structure and resinous materials.[17,33] The compatibility between the brown dye of the coffee and the resin polymer chain has been suggested, facilitating the adsorption and penetration of the dye in the resin.[33] The specimens immersed in coffee exhibited a yellow appearance, besides reduced lightness. On the other hand, the specimens immersed in wine presented a decrease in b* values. The complex combination of pigments present in the wine solution makes the prediction of changes on a* and b* parameters difficult. Perhaps the purple pigments interfere with the b* coordinate, directing it toward blue. Moreover, the presence of alcohol and low pH could promote softening of the polymeric material,[34] which could make the adsorption of pigments on the resin surface easier, such as tannins present in red wine.[35,36]

The susceptibility to extrinsic staining of a resinous material may also be related to the type of resin matrix used, due to its rate of water sorption and solubility. The absorption of water and other fluids by the organic component of resin can result in discoloration and degradation of the polymer matrix.[37]

A high penetration coefficient and low viscosity of resin monomer are desirable features to allow infiltration of resin into the subsurface of carious lesions. The combination of experimental TEGDMA, HEMA, and ethanol showed satisfactory characteristics for an infiltrating resin.[38] However, HEMA has a hydroxyl functional group, making it hydrophilic.[37] Thus, although the composition of the infiltrating resin (ICON) is not detailed by the manufacturer, it may favor the absorption of water-based dyes and increase the susceptibility of the material to staining. In addition, the inhibition of polymerization by oxygen and polymerization shrinkage of the infiltrating resin may also result in nonhomogeneous areas,[37] which could facilitate the penetration or adsorption of the dyes. Nevertheless, a recent study reported that the polishing of infiltrated lesions increases their resistance to staining challenges.[12]

The second null hypothesis was also rejected, since repolishing with fine-grain abrasive discs led to a significant reduction in ΔE values for all groups. Similar results were also observed in a previous study with composite resins,[32] and this is due to the fact that the dyes can remain adsorbed on the surface, with little penetration into the resinous materials or the tooth substrate.[17]

However, even after repolishing, the stained specimens still presented color alteration significantly higher than those immersed in deionized water. Overall, those subjected to the resin infiltration technique exhibited ΔE values significantly higher than all other groups. Nevertheless, it should be pointed out that the combination of immersion time in the dyes and absence of tooth brushing probably increased the staining susceptibility.[37] In clinical conditions, the dilution of the dye solutions by saliva and the action of brushing associated with the use of toothpastes can result in a greater resistance of the infiltrating resin to staining. Nonetheless, patients should be warned about the potential for staining by food dyes and avoid or minimize the consumption of such colored foods to increase the longevity of the esthetic outcomes promoted by the treatment of white spot lesions with infiltration resin in anterior teeth.

CONCLUSIONS

Based on the methodology used and considering the limitations of this study, it can be concluded that the

immersion of the specimens in staining solutions (wine and coffee) resulted in significant color alteration. The demineralized enamel treated with resin infiltration showed significantly higher staining than all other tested groups; however, repolishing of the specimens can minimize the staining effect.

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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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Polymerization Shrinkage and Depth of Cure of Bulk Fill Flowable Composite Resins

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Clinical Relevance

Flowable composites had higher shrinkage than the nanohybrid restorative composite that was modified to have a flowable consistency during ultrasonic insertion. All materials tested had significantly less depth of cure than either manufacturers' claims or ISO 4049 scrape test results.

SUMMARY

Objective: **To evaluate polymerization shrinkage and depth of cure of two bulk fill flowable composites, one nanohybrid composite modified to a flowable consistency, and one standard flowable composite, comparing the scraping method to the Knoop hardness test.**

Methods: **Two bulk fill flowable composites, SureFil SDR *flow* (SSF) (Dentsply) and Venus Bulk Fill (VBF) (Heraeus Kulzer), one standard flowable, Filtek Supreme Ultra Flowable (FSUF) (3M/ESPE) (control), and one regular bulk composite that can be made flowable, SonicFill (SF) (Kerr), were used in this study. For polymerization shrinkage (PS), ten 2-mm samples were made for each composite and cured for 20 seconds and shrinkage was measured with a Kaman linometer. For hardness, ten specimens of each composite were made in a 10 × 10-mm mold and cured for 20 seconds; the bottom surface was scraped according to ISO 4049 specification, and the remaining thickness was measured with a micrometer. Hardness samples were prepared at 2-, 3-, 4-, and 5-mm thick ×14-mm diameter, cured for 20 seconds, and polished. After 24 hours of dry storage, a Knoop indenter was applied at 100 g load for 11 seconds. Three readings were made on the top and bottom of each specimen and averaged for each surface to calculate a Knoop hardness value and a bottom/top hardness ratio. One-way analysis of variance and Tukey tests were used to determine significant differences between thicknesses and between test methods for each material.**

Results: **PS values were 3.43 ± 0.51%, 3.57 ± 0.63%, 4.4 ± 0.79%, and 1.76 ± 0.53% for FSUF, SSF, VBF, and SF, respectively. VBF showed**

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significantly greater shrinkage (4.4 ± 0.79%), followed by FSUF (3.43 ± 0.51%) and SSF (3.57 ± 0.63%), which were similar, and SF (1.76 ± 0.53%), which had significantly less shrinkage ($p<0.05$). Values for the scraping method for depth of cure were significantly greater for SSF and VBF (>5.0 mm), followed by SF (3.46 ± 0.16 mm) and FSU (2.98 ± 0.22 mm). Knoop top hardness values (KHN) were: VBF 21.55 ± 2.39, FSUF 44.62 ± 1.93, SSF 29.17 ± 0.76, and SF 72.56 ± 2.4 at 2 mm and were not significantly different at 3-, 4-, and 5-mm thick within each material. Ratios for bottom/top values (depth of cure) for 2, 3, 4, and 5 mm were: VBF 0.80 ± 0.1, 0.78 ± 0.03, 0.67 ± 0.10, and 0.59 ± 0.07, respectively; SSF 0.74 ± 0.08, 0.72 ± 0.08, 0.69 ± 0.18, and 0.62 ± 0.08, respectively; SF 0.82 ± 0.05, 0.68 ± 0.05, 0.47 ± 0.04, and 0.21 ± 0.02, respectively; and FSUF 0.56 ± 0.08 at 2 mm and 0.40 ± 0.08 at 3 mm. The bottom/top ratio was .80 or less at all depths and decreased below 0.70 at 4-mm depth for VBF and SSF, at 3 mm for SF and at 2 mm for FSUF.

INTRODUCTION

Innovative bulk fill composite resins with reported significant flow and low polymerization shrinkage have been marketed. Clinical recommendations suggest that they have greater depth of cure and can be placed in a 4-mm bulk increment and will have adequate polymerization. The stress-decreasing resin technology was created as a new resin system that would minimize shrinkage stress and allow bulk placement. Clinically, this would eliminate the need for incremental placement and curing and reduce the need for material manipulation during insertion. To do this, it was necessary to regulate the overall modulus development while maintaining its polymerization rate and conversion.[1-3]

This new and innovate technology is based on changes in monomer chemistry. Manufacturers introduced this new technology by modifying the Bowen monomer (Bis-GMA: 2,2-bis [4-(2-hydroxy-3-methacryloxypropoxy) phenyl]propane) to create monomers with lower viscosity.[1-5] This new modification could be achieved by incorporating hydroxyl-free Bis-GMA, aliphatic urethane dimethacrylates, partially aromatic urethane dimethacrylate, or highly branched methacrylates.[6] The outcomes of these changes in monomer and composite organic matrix have been shown to reduce polymerization shrinkage stresses over 70%.[1,2,7,8]

Polymerization shrinkage is related to the organic and inorganic content of the composite resins. Flowable composites generally contain more organic matrix in order to gain increased flow. Thus, they have greater shrinkage compared to hybrid composites, which have less organic matrix.[9] As a result of the increased resin matrix, flowable composites reduce internal stresses during polymerization shrinkage due to their lower Young's modulus compared to regular packable composites.[10]

As composites are light cured, they convert from a viscous phase into a rigid or solid material and undergo shrinkage. When the composite is placed inside a preparation and subsequently bonds to the tooth surfaces, the polymerized composite develops internal mechanical stress, which is transmitted to the tooth structure-bond interface[11]; if the contraction stress exceeds the bond strength, debonding can occur, resulting in possible marginal leakage, secondary caries, and cohesive tooth fractures at the margins.[12] The cavity configuration or C-factor described by Feilzer and others[13] claims that the stress inside a restoration after it was polymerized is proportional to the ratio of its bonded and nonbonded surfaces, and this C-factor is responsible for the development of the contraction stress in composite resin restorations.

Many laboratory methods such as infrared spectrometry,[14-17] resonance imaging, and Knoop and Vickers hardness[18] have been used to determine the depth of cure of restorative materials. The standard and most common test, the scraping method ISO 4049, has been researched and has been shown to overestimate depth of cure values.[5,14,16,19] When hardness values are obtained, a mean bottom/top ratio (B/T) hardness value is usually determined to establish the depth of cure. This reflects the relative extent of conversion of the deeper surfaces in relation to the top surface. As an accepted minimum standard, many authors have claimed that a ratio of 0.80 is clinically acceptable.[19-22] It has been shown that the value is mostly dependent on filler content and filler size.[22] This is an accurate method to compare the relative extent of cure between different composites at specified depths. Undercured composites can lead to early failure of a restoration and should be avoided in any clinical technique.

The purpose of the present study was to evaluate polymerization shrinkage and depth of cure of three bulk fill flowable composites. Both the scraping method (ISO 4049) and Knoop hardness test were conducted on each material and compared in order to verify the accuracy of the test methodology and to

Table 1: *Materials Used in the Study*

Material	Type	% by Weight	Filler Composition	Manufacturer
Venus Bulk Fill	Nanohybrid	65%	Ba-Al-F silica glass, YbF_3, and SiO_2	Heraeus Kulzer (South Bend, IN, USA)
SureFil SDR *flow*	Urethane dimethacrylate with hybrid glass filler	68%	Barium and strontium, alumino-fluoro-silicate glasses	DENTSPLY Caulk (Milford, DE, USA)
SonicFill	Nanohybrid	83.5%	Barium, aluminum boron, silicate	Kerr Corporation (Orange, CA, USA)
Filtek Supreme Ultra Flowable (control)	Nanocomposite	65%	Ytterbium trifluoride, silica, zirconia/silica clusters	3M/ESPE (St Paul, MN, USA)

better evaluate the suggested depth of cure in the marketing literature of this group of materials.

The two null hypotheses tested were:

- There is no significant difference in polymerization shrinkage and depth of cure between the different bulk fill composite resins.
- There is no significant difference in depth of cure measurements between the ISO 4049 scraping test and the bottom/top hardness ratio test.

METHODS AND MATERIALS

The composites tested in this study are listed in Table 1. SureFil SDR *flow* and Venus Bulk Fill are bulk fill flowable composites. SonicFill is a nanohybrid bulk fill composite that can be placed ultrasonically in a flowable state, and Filtek Supreme Ultra Flowable is a standard flowable used as the control. A light shade was selected for each material (shade A2, except for SureFil SDR *flow*, which was a universal shade) so that light penetration would be optimal and the effect of shading pigments would not be a confounding variable.

Polymerization Shrinkage

Ten disk-shaped samples of each composite material approximately 5 mm in diameter and 1.5 to 2 mm thick were fabricated. The uncured composite was dispensed in a Teflon ring with a glass slide on the bottom which was coated with a separating medium to allow the composite to shrink free of surface adhesion. A lubricated flat aluminum target parallel to the glass slide covered the open side of the sample. Once in position, the composite specimens were polymerized for 20 seconds using the SmartLite iQ2 (Model No. 200, DENTSPLY Caulk) curing unit with a wavelength range between 450 and 475 nm and a power of 800 mW/cm^2. This value was verified every five samples to corroborate adequate polymerization. This analysis was performed with a Kaman linometer (KADA, Kaman Instrumentation, Colorado Springs, CO, USA),[23] where the arrangement consists of a noncontact displacement transducer with the sensor placed in a vertically oriented quartz tube. The holder allows the placement of the curing light approximately 1 mm away from the composite. The linear shrinkage data were recorded as change in length (ΔL), and the percentage of shrinkage was calculated using the following formula: $Lin\% = \Delta L/(L + \Delta L) \times 100$. Here, ΔL *i*s the displacement recorded in microns, and L is the thickness of the specimen in microns after polymerization. The $Lin\%$ is then converted mathematically to a volumetric value using the formula:

$$Vol\% = 3Lin\% - 0.03(Lin\%)^2 + 0.0001(Lin\%)^3$$

Depth of Cure

For the ISO 4049 scraping method, depth of cure was calculated by fabricating ten specimens of each composite in a 10 × 10 mm mold and light curing for 20 seconds using the SmartLite iQ2 curing unit. After the specimen was removed from the mold, the bottom surface was scraped to remove soft unpoly-

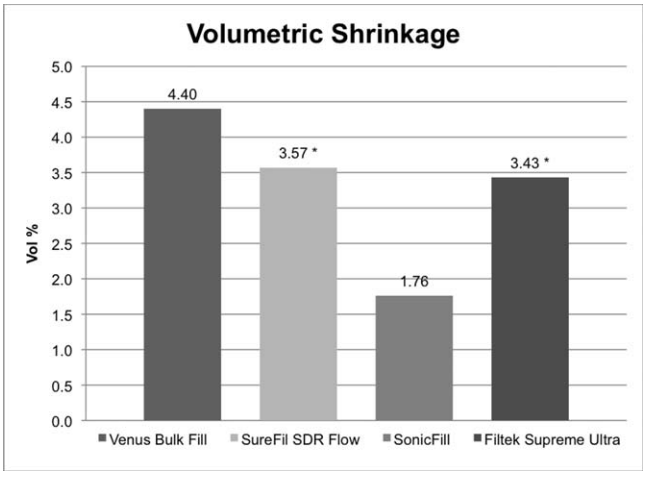

Figure 1. *Mean volumetric shrinkage values for each material tested. Values with an (*) were not significantly different.*

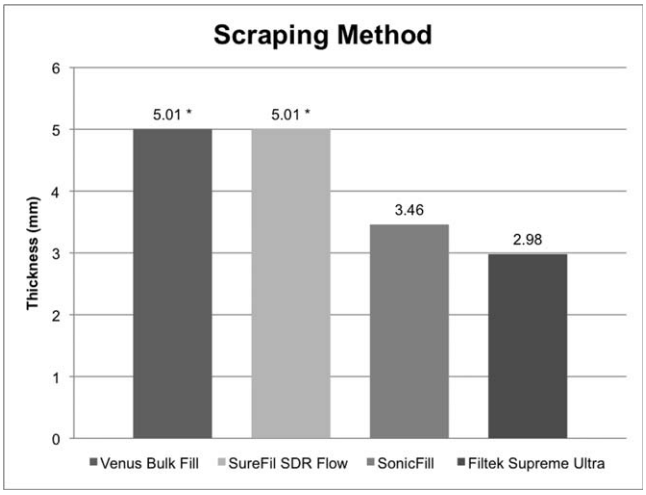

Figure 2. *Mean scraping method values for each material tested. Values with an (*) were not significantly different.*

merized material using a plastic spatula according to ISO 4049, and the remaining thickness of composite was measured three times with a micrometer (Mitutoyo America, Aurora, IL, USA), averaged and divided by two.

For Knoop hardness, a split brass mold was used to prepare composite samples 14 mm in diameter and 2, 3, 4, and 5 mm in thickness. Ten specimens of each thickness were fabricated for each composite. Under low light conditions, composite was applied to fill each hole in the brass mold. A thin layer of Al-Cote Septarating Agent (Dentsply, York, PA) was placed on a clean glass slide (1 mm thick), which was then placed on top of the composite to create a flat surface. Samples were light cured for 20 seconds through the glass slide using the SmartLite iQ2. Each sample was then removed from the mold and polished at the top and at the bottom surface with 2400-grit silicon carbide paper (Mager Scientific, Dexter, MI, USA), followed by a 9-μm polycrystalline diamond suspension (Micro Star 2000, Inc, Ontario, Canada) to remove the resin-rich layer and develop a polished surface to make the indentation more visible in the microscope. After a dark storage period of 24 hours, a Tukon 2100B testing machine (Wilson Instruments, Northwood, MA, USA) was calibrated, and a Knoop diamond indenting tool was applied three times with a 100 g load to the top and the bottom of each sample surface with a dwell time of 11 seconds.

The following formula was used to calculate the Knoop hardness number, which is the ratio of the load applied to the area of the indentation:$KHN = L/(I^2Cp)$. In this formula, L is the applied load in kilograms, I is the length of the long diagonal of indentation (mm), and Cp is the constant related to the area of the indentation (0.070).

One-way analysis of variance (ANOVA) was used to determine a significant difference among materials for all three variables. Tukey multiple comparison test was then used to determine significant differences between the means ($p<0.001$).

RESULTS

Polymerization Shrinkage

The mean values for volumetric shrinkage (Figure 1) for Venus Bulk Fill, SureFil SDR *flow*, Filtek Supreme Ultra Flowable, and SonicFill were 4.40% (±0.79), 3.57% (±0.63), 3.43% (±0.51), and 1.76% (±0.53), respectively. Tukey test revealed that SonicFill had the lowest shrinkage value ($p<0.001$), and Venus Bulk the highest shrinkage value. SureFil SDR *flow* and Filtek Supreme Ultra Flowable were not significantly different.

Scraping Method

Figure 2 represents the mean depth of cure values using the scraping method: Venus Bulk Fill 5.01 mm (±0.02) and SureFil SDR *flow* 5.01 mm (±0.03) both demonstrated full depth of cure at 5 mm, while both Filtek Supreme Ultra Flowable 2.98 mm (±0.11),

Table 2: *Means and Standard Deviations for Top and Bottom Surface Knoop Hardness Values*[†]

Material	Top Hardness					Bottom Hardness				
	2 mm	3 mm	4 mm	5 mm	*p*-value	2 mm	3 mm	4 mm	5 mm	*p*-value
Venus Bulk Fill	21.6 (2.40)*	21.3 (1.02)*	23.5 (3.25)*	23.0 (2.34)*	0.13	17.0 (2.35)*	16.6 (0.70) [a*]	15.6 (2.09)*	13.5 (0.74) [a]	<0.001
SureFil SDR *flow*	29.1 (0.77)*	29.4 (1.16)*	29.7 (3.97)*	31.5 (1.42)*	0.11	21.5 (2.53) [a*]	21.1 (1.98)*	19.8 (2.782)*	19.4 (2.74)*	0.21
SonicFill)	72.6 (2.40)*	72.3 (3.20)*	72.4 (2.11)*	71.1 (2.64)*	0.60	59.4 (2.85)	48.9 (2.06)	34.0 (1.88)	15.1 (1.29) [a]	<0.001
Filtek Supreme Ultra Flowable	44.6 (1.93)*	42.8 (3.07)*	—	—	0.13	24.7 (3.32) [a]	16.9 (3.13) [a]	—	—	<0.001

[†] *Column values with the same letter are not significantly different at* $p<0.001$*; row values with the same symbol (*) are not significantly different.*

Table 3: *Means and Standard Deviations for Hardness Ratio Values (B/T)*†

Material	2 mm	3 mm	4 mm	5 mm	*p*-value
Venus Bulk Fill	0.80 (0.10) a*	0.78 (0.03) a*	0.67 (0.10) a	0.59 (0.07) a	<0.001
SureFil SDR *flow*	0.74 (0.08) a*	0.72 (0.08) a*	0.69 (0.18) a*	0.62 (0.08) a*	0.09 (NS)
SonicFill	0.82 (0.05) a	0.68 (0.05) a	0.47 (0.04)	0.21 (0.02)	<0.001
Filtek Supreme Ultra Flowable	0.56 (0.08)	0.40 (0.08)	—	—	<0.001

Abbreviations: NS, not significant.
† *Column values with the same letter are not significantly different at* p<*0.001; row values with the same symbol (*) are not significantly different.*

and SonicFill 3.46 mm (±0.08) were significantly lower.

Surface Hardness: Knoop Hardness

The means and standard deviations for top and bottom hardness using the Knoop hardness indentation for each material at 2-, 3-, 4-, and 5-mm thickness are represented in Table 2. Top hardness values for each material revealed no significant differences at all four thicknesses. For bottom hardness values, Venus Bulk Fill decreased significantly between 4- and 5-mm thickness. SureFil SDR *flow* was the only material that showed statistically similar values at all four thicknesses tested. SonicFill had a significant decrease at 2, 3, 4, and 5 mm. Filtek Supreme Ultra Flowable showed a significant difference between 2 and 3 mm and was too soft to test at both 4 and 5 mm.

Bottom/Top Hardness Ratio (Depth of Cure)

Table 3 represents the means and standard deviations for B/T ratio at different thicknesses. For reference, a perfectly cured material would have a ratio of 1.0. For Venus Bulk Fill, B/T ratio was .80 or less at all depths, with a significant decrease between 3 and 4 mm. B/T ratio values for SureFil SDR *flow* were 0.7 or less, and the decreases at each depth were not statistically significant. For SonicFill, B/T ratio was 0.80 or less with a significant decrease at all depths. For Filtek Supreme Ultra Flowable, B/T ratio values were significantly lower compared to the other materials (<0.6) at both 2- and 3-mm thicknesses, and the material was too soft to measure at 3 and 4 mm.

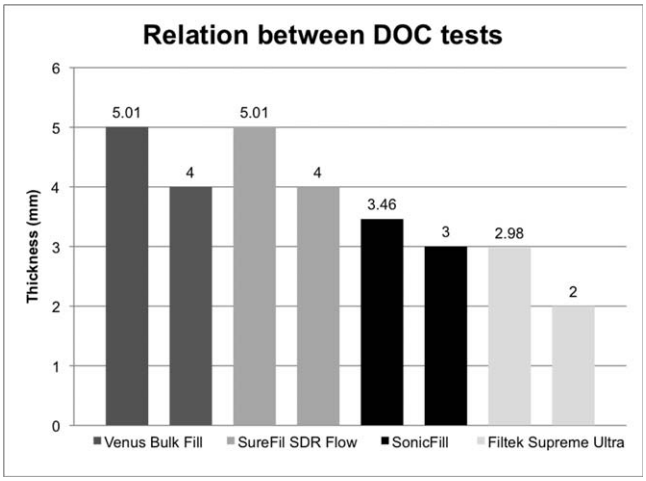

Figure 3. *Graphic representation of relation between depth of cure tests. The left bar for each material represents the scraping method value in mm. The right bar represents the critical depth in mm where the bottom to top hardness ratio decreases below 0.70.*

Relation Between Depth of Cure Tests

Depth of cure values from the scraping method and Knoop hardness were compared to determine if an association existed between the two tests (Figure 3). Venus Bulk Fill and SureFil SDR *flow* both cured to 5 mm depth using the scraping test, but were at least 1 mm less when tested using hardness tests and only a 70% cure. SonicFill and Filtek Supreme Ultra Flowable cured to lesser depths but also were about 1 mm less using the hardness tests. All B/T hardness values were significantly lower than those obtained by the scraping method.

DISCUSSION

The results for this study document that the three flowable materials show greater shrinkage (3.3-4.4 vol%) than the nanohybrid material, SonicFill (1.8%). A recent study reported volumetric shrinkage of 3.1% for SureFil SDR *flow*,[1] which is in agreement with this study. SonicFill is actually a full body composite resin with an estimated filler content of 83.5% by weight and 69% by volume and thus is expected to have less shrinkage than a flowable material with higher resin content.[7] Shrinkage values have a direct relationship with the amount of organic matrix in the material.[24] A study by Herrero and others[23] in 2005 found that not only the size and amount of filler content were important in determining the shrinkage, but the shape of the particles can also generate a different outcome. The distinct advantage of a material like SonicFill is that

it has a consistency that is similar to a flowable when it is being placed, and then it has the properties of a hybrid after it polymerizes.

Even though SonicFill had the lowest volumetric shrinkage, further investigations are needed to determine shrinkage stress. An assumption can be made that even though SureFil SDR *flow* had higher volumetric shrinkage compared to SonicFill, stress can be lower due to the new modulator incorporated in the chemistry, which slows the rate of polymerization and reduces stress.[1] It is important to understand that a significant reduction in reaction rate does not necessarily correspond to reduction in contraction stress.[25]

In the present study, depth of cure values using the scraping method were greater for the two modified flowable materials that are marketed with depth of cure values that support bulk fill techniques than the nanohybrid or the standard flowable materials. These results are in accordance with a recent study where scraping method values for the same bulk fill materials were found to be higher than the control flowable composite resins.[5] The higher depth of cure of the bulk fill materials may be due to the incorporation of more efficient initiator systems and higher translucency of composites.[5] Even though SonicFill is considered to be a bulk fill composite, the true depth of cure was less than 4 mm in this study. It has been demonstrated that filler size and content in dental composites may reduce light penetration and is directly related to depth of cure.[14,17] The presence of pigments in shaded composite materials should also have an effect on depth of cure because pigments are opaque particles that will limit light penetration and reduce the degree of polymerization at greater depths within a cavity preparation.

Knoop hardness indentation is an actual measurement of surface hardness compared to the estimated values from the scraping method in ISO 4049. For each of the materials tested, top hardness values revealed no significant differences at the four thicknesses, as seen in Table 2. This result is in accordance with a previous study,[26] and this outcome is expected since the light source is being applied at the top surfaces of all composites. For bottom hardness values, all materials decreased as the thickness of the samples increased, which can be expected. Venus Bulk Fill showed a statistically significant decrease at 4- and 5-mm thickness. Numerous studies have defined depth of cure based on hardness measurements performed on the top and bottom surface of a light-cured resin composite specimen and agree that a ratio of 0.80 may be used as a critical minimum acceptable threshold value.[16,19,22] All B/T values for the materials in this study were less than the 80% critical value for depth of cure, and their use in bulk fill situations greater than 3-mm depth should be questioned.

As seen in Table 3, top and bottom hardness values revealed that SureFil SDR *flow* had statistically similar results at 2, 3, 4, and 5 mm. In comparison, Venus Bulk Fill showed a B/T ratio of 0.80 at 2 and 3 mm but had a significant decrease at 4 and 5 mm. These results are in accordance with a study done by Czasch and Ilie[3] in 2012 where it was shown that SureFil SDR *flow* was significantly harder than Venus Bulk Fill. It is important to state that in the present study even though SureFil SDR *flow* had similar results at the different depths, the B/T ratio at 2 mm was less than 0.80. Composition in the chemistry of SureFil SDR *flow* is based on a modification of triethylene glycol dimethacrylate, which is found to have more flexible side groups, reducing and forming a more homogeneous polymer network.[8]

The difference in hardness values for the materials tested can also be a result of filler content. It has been reported that decreasing filler size can affect depth of cure, while increasing filler volume can improve the hardness.[16] Overall, if the filler content of a composite resin is increased, the mechanical properties will improve.[16] SonicFill had the highest filler content of the materials tested (83.5%), but the depth of cure significantly decreased at 3, 4, and 5 mm. A possible reason might be the lack of light penetration through the composite at increasing depths because a high percentage of the wavelengths are absorbed near the top surface of the composite and not available to excite co-initiators at greater depths. Further research is needed to identify the role of the polymerization modulator on mechanical properties of these new bulk fill materials.

CONCLUSIONS

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

1. SonicFill had significantly lower shrinkage values ($p<0.05$) than all of the other materials. Filtek Supreme Ultra Flowable and SureFil SDR *flow* were higher but not significantly different. Venus Bulk Fill showed significantly higher shrinkage values than the other materials tested.
2. Using the scraping method (ISO 4049 specification), Venus Bulk Fill and SureFil SDR *flow*

showed significantly higher depth of cure values than Filtek Supreme Ultra Flowable and SonicFill.
3. For top hardness measurements, all materials exhibited similar values, regardless of thickness.
4. For bottom hardness measurements, all materials showed a decrease in hardness with increasing depth.
5. The bottom/top ratio was 0.80 or above for only SonicFill at 2-mm depth. It decreased below 0.70 at 4-mm depth for Venus Bulk Fill and SureFil SDR *flow*, at 3-mm depth for Sonic Fill, and at 2-mm depth for Filtek Supreme Ultra Flowable.
6. The scraping method significantly overestimated depth of cure values for all materials, and this could influence clinical outcomes.

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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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Shear Bond Strength of Four Different Repair Materials Applied to Bis-acryl Resin Provisional Materials Measured 10 Minutes, One Hour, and Two Days After Bonding

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Clinical Relevance

Strong bonding can be achieved between bis-acryl base and bis-acryl repair material. Time after bonding is an important factor for bond strength of repaired bis-acryl provisional restorations.

SUMMARY

This study investigated the shear bond strength of repaired provisional restoration materials 1) to compare the bond strengths between bis-acryl resin and four different materials and 2) to investigate the effect of the amount of time elapsed after bonding on the bond strength. The self-cured bis-acryl resin (Luxatemp) was used as the base material, and four different types of resins (Luxatemp, Protemp, Z350 flowable, and Z350) were used as the repair materials. Specimens were divided into three groups depending on the point of time of shear bond strength measurement: 10 minutes, one hour, and 48 hours. Shear bond strengths were measured with a universal testing machine, and the fracture surface was examined with a video measuring system. Two-way analysis of variance revealed that the repair materials ($p<0.001$) and the amount of time elapsed after bonding ($p<0.001$) significantly affected the repair strength. All of the repaired materials showed increasing bond strength with longer storage time. The highest bond strength and cohesive

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Table 1: *Materials Used in the Experiment*

Product	Major Components	Polymerization Mode	Manufacturer	Lot
Luxatemp-Plus	Bis-acryl	Self-cured	DMG	681163
Protemp 4	Bis-acryl	Self-cured	3M ESPE	B 483558
				C 482519
Filtek Z350 XT Flowable	Bis-GMA	Light-cured	3M ESPE	N 420221
Filtek Z350 XT	Bis-GMA	Light-cured	3M ESPE	N 453720

Abbreviation: bis-GMA, bisphenol A diglycidyl ether dimethacrylate.

failure were observed for bonding between Luxatemp base and Luxatemp at 48 hours after bonding.

INTRODUCTION

During the past decade, bis-acryl resin composites have become popular as provisional restoration materials as a result of their ease of use, minimal polymerization shrinkage, and low exothermic reaction.[1-3]

A vacuum-formed acrylic splint or polyvinyl siloxane impression is frequently used as a direct matrix for provisional formation with bis-acryl resins.[4] In the process of fabricating provisional restorations, relining or repair is inevitable because of trapped air,[5] shortened margin,[6,7] and fracture.[8] There have been clinical attempts to use bis-acryl resin for self-repair, but that strategy proved ineffective.[9]

Light-cured flowable resins have been suggested[10,11] for the repair of bis-acryl resin provisional restorations. They are easily manipulated and bonding with bis-acryl resin is effective and durable.[12,13] Light-cured packable composite resin has the same components as light-cured flowable resin. However, to date there has been no published study on the bond strength between a bis-acryl composite and light-cured packable composite resin.

Shear bond strength tests are commonly used to evaluate the bonding between experimental substances and bis-acryl resins, and most laboratory bond strength tests are performed 24 hours after the bonding process and water storage of the specimens. However, in most clinical situations, the repaired interface of a provisional restoration is subjected to various kinds of stress immediately after bonding. These stresses result from adjustments and polishing procedures of the repaired provisional restorations as well as physical loading in the oral environment because it is necessary for provisional restorations to replace removed tooth structure as soon as possible after preparation as an interim treatment of the fixed prosthodontic treatment. Therefore, the translation of previous laboratory results to the clinical setting is difficult, and time is considered an important factor in the design of experiments on shear bond strength of repaired provisional restorative materials.

The purposes of this *in vitro* study were 1) to compare the bond strengths between bis-acryl resin and four different materials, including light-cured packable composite resin, and 2) to investigate the effect of the storage time on the bond strength at the repaired interface of provisional restorations.

METHODS AND MATERIALS

Materials

The self-curing bis-acryl resin (Luxatemp AM Plus, DMG, Hamburg, Germany; shade A2, Lot 681163) was used as the base material. Four different types of resins (Luxatemp, Protemp, Z350 flowable, and Z350) were used as repair materials, and Table 1 provides an overview of the materials tested. All commercial materials were used according to the manufacturers' recommendations.

Preparation of Specimens

The shear bond strength test required the use of a sample holder to fix the specimen in the universal testing machine perpendicular to the orientation of the shear force. Transparent acrylic-glass rods (30 mm in diameter; Polymicar, Tae-guang, Korea) were fabricated for this purpose. The mixed self-curing bis-acryl resin (Luxatemp AM Plus) was dispensed into a 12-mm-wide hole in the acrylic-glass rod and was allowed to set for 20 minutes. Procedures for fabricating each specimen, including preparing base materials and bonding with repair resin, were finished within 30 minutes.

Prior to repair, the surfaces of the specimens were ground with silica-carbide (SiC) paper (grit 600), rinsed with water for 10 seconds, and dried for 30 seconds with an air syringe. Hard transparent gelatin capsules (4.0 mm in diameter) were used as

Table 2: *Study Design in the Experiment*

	Luxatemp + Luxatemp	Luxatemp + Protemp	Luxatemp + Z350 (Flowable)	Luxatemp + Z350
10 min	Group 1 (LT-LT/10 min)	Group 2 (LT-PT/10 min)	Group 3 (LT-F/10 min)	Group 4 (LT-P/10 min)
1 h	Group 5 (LT-LT/1 h)	Group 6 (LT-PT/1 h)	Group 7 (LT-F/1 h)	Group 8 (LT-P/1 h)
48 h	Group 9 (LT-LT/48 h)	Group 10 (LT-PT/48 h)	Group 11 (LT-F/48 h)	Group 12 (LT-P/48 h)

matrices for the production of columns of relining materials bonded to the bis-acrylic provisional resin surface. The capsule was partially filled with one of four repair materials to limit the thickness of the bonded material to 2 mm. Light-cured flowable resins (Filtek TM Z350 XT Flowable, 3M ESPE, St. Paul, MN, USA) and light-cured packable composite resins (Filtek TM Z350 XT, 3M ESPE) were light-cured with a commercial light-curing unit (Elipar freelight 2, 3M ESPE) for 40 seconds. After polymerization, specimens were removed from the mold and stored in a dry oven (WiseVen, Daihan Scientific Co, Seoul, Korea) at 37°C. Table 2 describes the groups tested. Bonding strength of repaired specimens was measured 10 minutes, one hour, and 48 hours after bonding. Each group included 10 specimens.

Determination of Shear Bond Strength

After the storage time, the shear bond strength was measured with a universal testing machine (AG-10KNX, Shimadzu Co, Japan). A knife-edge shearing rod at a crosshead speed of 2 mm/min was used to place a load on the specimens until fracture occurred. The shear bond strength in MPa was calculated from the peak load of failure. The aspect of each bonding failure was determined using a video measuring system (Optical video measuring system, Seven Ocean, Korea) at 10× magnification and was recorded.

Statistical Analysis

The mean and standard deviation of the shear bond strengths were calculated for each group. Data were evaluated for homogeneity of variance based on the Levene test ($p=0.05$). The influence of independent variables, including materials and storage times, was analyzed by a two-way analysis of variance (ANOVA) ($p=0.05$). Comparison of bond strength between repaired specimens was conducted by one-way ANOVA and Tukey multiple comparison tests ($p=0.05$). All statistical analyses were carried out with SPSS for Windows (release 12.01; SPSS Inc, Chicago, IL, USA).

RESULTS

The results of shear bond strength testing for each experimental group are summarized in Table 3 and Figure 1. Tables 4 and 5 show comparisons among repaired specimens evaluated by one-way ANOVA ($p<0.05$). Two-way ANOVA revealed that there were significant differences in shear bond strength because of the repair resin variables and the storage times ($p<0.001$; Table 6).

The results for detachment at 10 minutes after bonding indicated that group 4 (LT-P/10 minutes) had the highest shear bond strength, followed by group 3 (LT-F/10 minutes), group 1 (LT-LT/10 minutes), and group 2 (LT-PT/10 minutes). However, there were no statistically significant differences between groups 4 and 3 and between groups 1 and 2 (Table 4). Thus, the shear bond strengths of group 3 (LT-F/10 minutes) and group 4 (LT-P/10 minutes) were statistically higher than those of group 1 (LT-LT/10 minutes) and group 2 (LT-PT/10 minutes) ($p<0.05$).

Group 8 (LT-P/one hour) showed the highest shear strength at one hour after bonding, which was significantly higher than that of group 5 (LT-LT/one hour) and group 6 (LT-PT/one hour) ($p<0.05$; Table 4).

The shear bond strengths of group 9 (LT-LT/48 hours) were the highest measured in this study, which significantly differed with the values of the

Table 3: *Mean Shear Bond Strength Value (Standard Deviation [SD]) in MPa for Repaired Specimens*

	Luxatemp + Luxatemp	Luxatemp + Protemp	Luxatemp + Z350 (Flowable)	Luxatemp + Z350 (Packable)
10 min	3.6 (0.7)	3.3 (1.3)	7.4 (2.5)	8.0 (1.6)
1 h	7.1 (1.7)	6.8 (1.9)	9.0 (1.6)	11.1 (1.7)
48 h	18.7 (4.0)	11.7 (2.4)	14.0 (2.7)	12.7 (1.5)

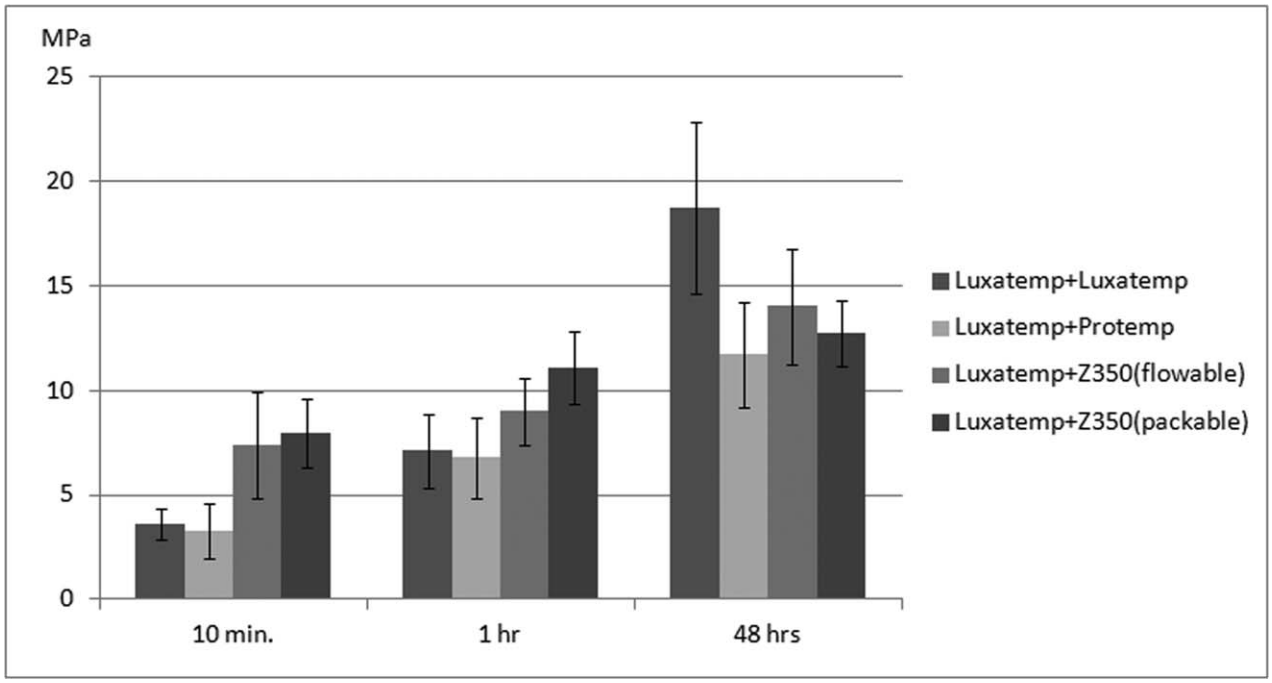

Figure 1. *The bond strengths at 10 minutes, one hour, and 24 hours of each of the repaired materials.*

other groups that were measured after 48 hours ($p<0.05$; Table 4).

All of the repaired materials showed increasing bond strengths with longer storage time. LT-LT and LT-PT revealed significantly increasing bond strengths at all period-of-time factors ($p<0.05$), and the largest differences were found with the LT-LT groups with respect to the storage time (Table 5).

Specimens showed three types of failure: adhesive (interface), cohesive (only at the provisional resin base material), and mixed (interface and base material). The distribution of fracture aspects on the debonded specimen surface is shown in Table 7. All of the groups measured after 10 minutes or one hour showed adhesive failure. While the groups measured after 48 hours showed adhesive, mixed,

Table 4: *Comparison Among Repaired Specimens with Measuring Times*[a]

	LT + PT	LT + F	LT + P
10 min			
LT + LT	n.s.	0.001	0.001
LT + PT	—	0.001	0.001
LT + F	—	—	n.s.
1 h			
LT + LT	n.s.	n.s.	0.006
LT + PT	—	n.s.	0.003
LT + F	—	—	n.s.
48 h			
LT + LT	0.002	0.048	0.025
LT + PT	—	n.s.	n.s.
LT + F	—	—	n.s.

Abbreviations: F, Z350 (flowable); LT, Luxatemp; P, Z350; PT, Protemp..
[a] *One-way analysis of variance (ANOVA) test (p values given in Table); n.s., not significant (p>0.05).*

Table 5: *Comparison Among Repaired Specimens with the Sorts of Materials*[a]

	1 h	48 h
LT + LT		
10 min	0.011	0.000
1 h	—	0.000
LT + PT		
10 min	0.004	0.000
1 h	—	0.000
LT + F		
10 min	n.s.	0.001
1 h	—	0.013
LT + P		
10 min	0.022	0.001
1 h	—	n.s.

Abbreviations: F, Z350 (flowable); LT, Luxatemp; P, Z350; PT, Protemp..
[a] *One-way analysis of variance (ANOVA) test (p values given in Table); n.s., not significant (p>0.05).*

Table 6: *Results of the Two-way Analysis of Variance (ANOVA) for All Test Groups*

Independent Variable	Significance
Material	$p < 0.001$
Storage time	$p < 0.001$

and cohesive failures, only group 9 (LT-LT/48 hours) exhibited cohesive failure (Figure 2).

DISCUSSION

The present study examined whether bond strengths between bis-acryl resin and light-cured packable composite resin are adequate to repair provisional restorations formed with bis-acryl and the effect of time elapsed after bonding on the bond strength at the repaired interface of provisional restorations. Light-cured packable composite resins seem to be appropriate for use as the repair material for bis-acryl provisional restorations. The bond strengths of all of the groups showed a tendency to increase with longer storage time, and, in particular, the bonding strength between Luxatemp base and Luxatemp dramatically increased.

Similar to results from previous studies,[9-11] light-cured flowable resin (Z350 flowable) showed higher bond strengths with bis-acryl resin (Luxatemp) compared to bis-acryl resin groups (Luxatemp, Protemp) upon measurement at 10 minutes after bonding. As the repaired provisional restorations usually receive a load immediately after bonding in the clinic, bond strength at 10 minutes after bonding may be the most valid index. It is for this reason that many articles[9,11,14] have recommended the use of light-cured flowable resin to repair provisional restorations made with bis-acryl. Additional advantages of using light-cured flowable resin include ease of application and manipulation, adequate working time, minimal odor, low polymerization shrinkage, and increased marginal accuracy.

For the shear strength tests at 10 minutes and one hour, the bond strengths of light-cured packable composite resins (Z350) were higher than for light-cured flowable resins (Z350 flowable). It can be presumed that the reason for this finding is that light-cured packable composite resin (Z350) has similar structure to light-cured flowable resin (Z350 flowable), and the material strength of these resins is higher than that of light-cured flowable resin (Z350 flowable). Light-cured packable composite resin has the additional virtues of low polymerization shrinkage and the ability to be molded prior to polymerization, compared with light-cured flowable resin. For these reasons, light-cured packable resin can be appropriately used to repair large defects of provisional restorations formed with bis-acryl.

The shear bond strength between bis-acryl resin and light-cured flowable resin was relatively low compared to the findings from previous studies.[9,15] A couple of reasons can be postulated for this discrepancy. First, to diminish differences of surfaces between groups, SiC paper (grit 600) was used in this study; however, the roughness of the SiC paper was relatively fine compared to that of the sandpaper used in previous studies.[15,16] Second, the crosshead speed of the knife-edge shearing rod was 2 mm/min in this study, which was faster than in previous studies.[15-17]

Most of the failure aspects were adhesive failures, except for the bonding between Luxatemp base and Luxatemp after 48 hours. All of the bonding failure aspects between Luxatemp base and Luxatemp after 48 hours were cohesive, and this group was found to have the highest bonding strength. Moreover, groups that showed mixed failure aspects had a relatively high bonding strength. These results suggest that

Table 7: *Distribution of Fracture Aspects on the Debonded Specimen Surface (N=Number)*

	Type of Failure	Luxatemp + Luxatemp	Luxatemp + Protemp	Luxatemp + Z350 (Flowable)	Luxatemp + Z350 (Packable)
10 min	Adhesive	10	10	10	10
	Mixed	—	—	—	—
	Cohesive	—	—	—	—
1 h	Adhesive	10	10	10	10
	Mixed	—	—	—	—
	Cohesive	—	—	—	—
48 h	Adhesive	—	6	7	6
	Mixed	—	4	3	4
	Cohesive	10	0	—	0

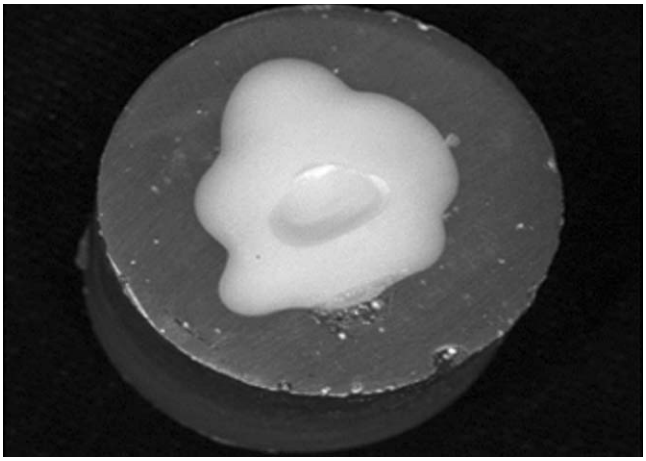

Figure 2. *All specimens from group 9 (LT-LT/48 hours) showed cohesive failure aspects.*

bonding strength at the interface is more important than the mechanical properties of the repair materials.

In general, the chemical similarity of the materials seems to be of great importance in polymer repair.[15] Using a provisional base and repair resins with similar chemical composition appears to provide greater bond strengths compared to using materials of dissimilar composition.[15] In contrast to this apparently common-sense rationale, the bond strength between bis-acryl and bis-acryl was considered to be weak. In this study, the same result was observed upon measurement at 10 minutes after bonding; however, the bond strength between bis-acryl resins rose dramatically with longer storage times. In particular, the bond between Luxatemp base and Luxatemp at 48 hours had the strongest bond strength measured in this study. This result suggests that bis-acryl and bis-acryl composites also have strong bonding but that a relatively long time is required to complete the bonding process. Expanded research about accelerating bonding procedures between bis-acryls may be useful in terms of the clinical use of bis-acryl.

The high bonding strength between Luxatemp and light-cured resin was to be expected because the chemical compositions of bis-acryl and light-cured resin bisphenol A diglycidyl ether dimethacrylate are similar.[18] The reason for the much higher bonding strength between Luxatemp and light-cured resin compared with Luxatemp base and Luxatemp at 10 minutes after bonding may correlate with the differences in polymerization speed: the polymerization of light-cured resin is accelerated with the light-curing machine and reaches complete polymerization in a shorter period of time than does Luxatemp.

To apply the results of this study to the clinical setting would require allowing bis-acryl resin polymerization to process for a relatively long period of time, and further studies to determine the exact time for complete bonding between bis-acryl resins will need to be performed. In addition, the development of methods to accelerate the polymerization of bis-acrylic resins would be a valuable extension of this work.

CONCLUSIONS

1. Light-cured packable composite resin is appropriate for repairing the large defects in provisional restorations formed with bis-acryl resin.
2. With longer storage time, the bond strengths of repaired bis-acryl provisional restorations tend to increase.
3. Strong bonding between bis-acryl base and bis-acryl can be accomplished, but a relatively long period of time is required for complete bonding.

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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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Microleakage of Resin-Modified Glass Ionomer Restorations With Selective Enamel Etching

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Clinical Relevance

In situations where enamel bonding is crucial and saliva contamination is inevitable, selective enamel etching can improve the bond quality of a resin-modified glass ionomer. However, selective enamel etching may lower the quality of the dentin bond.

SUMMARY

Aim: Bonding of resin-modified glass ionomers to enamel is an important quality, especially when saliva contamination is inevitable. This study evaluated if microleakage of a resin-modified glass ionomer improves with selective enamel etching, with or without saliva contamination.

Methods: Class V cavities with the occlusal margin in enamel and the gingival margin on the root were prepared in extracted human permanent teeth and filled with a resin-modified glass ionomer using an acidic primer according to the manufacturer's recommendation or with an additional selective enamel etching step. Preparations were contaminated with saliva before primer application or before restoration placement (n=10). Restored teeth were thermocycled between 5°C and 55°C for 1000 cycles, stained with basic fuchsin, and sectioned. Microleakage distance was measured and analyzed with analysis of variance followed by Duncan *post hoc* test at a significance level of 0.05.

Results: Enamel microleakage was highest when saliva contamination occurred before the placement of resin-modified glass ionomer. Microleakage distances were significantly reduced in the selective etching groups regardless of saliva contamination. However, selective etching of enamel increased microleakage in cementum. The increase in cementum leakage was significantly higher when saliva contamination occurred before restoration placement.

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Conclusion: Selective etching reduces enamel microleakage of a resin-modified glass ionomer even with saliva contamination, but it may increase microleakage at the cementum. The severity of microleakage is affected by the timing of saliva contamination.

INTRODUCTION

The increasing demand for esthetic restorations in all aspects of dentistry continues to revolutionize tooth-colored materials at the clinician's disposal. Resin-based composites are esthetically pleasing with adequate mechanical properties, whereas acid-base glass ionomer cements do not possess comparable strength, esthetic, and preferred setting characteristics but do maintain their special place in clinical applications.[1] Resin-modified glass ionomers have emerged as a hybrid material that combines benefits from both resin-based composites and glass ionomer cements by adding methacrylate functional groups to polyacrylic acids.[2] Recently, a new resin-modified glass ionomer has been introduced with bonded nanofiller technology for improved polish and esthetics (Ketac Nano, 3M ESPE, St Paul, MN, USA).[3]

One crucial factor determining the longevity of tooth-colored restorations is the quality of the bonded interface between the restorative materials and tooth structures. The bonded interface of resin-based composites is easily compromised by contamination from oral fluids.[4] Previous research showed that glass ionomer materials, in particular a conventional glass ionomer, is more tolerant to saliva contamination than composite or the recently introduced resin-modified glass ionomer, which comes with a no-rinse acidic primer.[5] In bonded restorations, by omitting the separate etching and rinsing steps, self-etch adhesives are less technique sensitive than etch-and-rinse systems and thus provide reliable clinical performance in adhesive restorations.[6,7] Self-etch adhesives are also more tolerant to saliva contamination, especially on dentin.[8-10] However, they generally do not etch enamel sufficiently, which results in lower bond strength to enamel than etch-and-rinse adhesives.[11-13] Therefore, a selective etching technique has been proposed to incorporate the advantage of the etch-and-rinse system on enamel with the self-etch system on dentin.[14]

Resin-modified glass ionomers can be a material of choice in compromised patients due to their anticariogenic property. However, when bonding to enamel is important, it would be advantageous if the bonding could be improved by an additional etching step. No studies to date have evaluated the concept of selective enamel etching with and without the presence of saliva contamination on a resin-modified glass ionomer system with its no-rinse acidic primer. The objective of this study was therefore to evaluate if the bond quality of a resin-modified glass ionomer could be improved with selective enamel etching with or without saliva contamination at different stages of the restorative procedure. The assessment of the bonding was carried out with microleakage tests.

METHODS AND MATERIALS

Tooth Preparation and Sample Distribution

Extracted human permanent premolars and molars were collected and kept in normal saline solution (Institutional Review Board exempt category study number 11-01212-XM). After cleaning with pumice slurry, a cylindrical cavity (3-mm diameter, 1.5-mm depth) was prepared at the cementoenamel junction area of the buccal surface using a high-speed handpiece with #245 carbide bur under a copious amount of water coolant. The prepared teeth were randomly divided into six groups, all to be restored with resin-modified glass ionomer Ketac Nano (3M ESPE). In group 1, the prepared teeth were restored following the manufacturer's instructions with the application of an acidic primer (Ketac Nano Primer, 3M ESPE) for 15 seconds, air-dried for 10 seconds, and light cured for 10 seconds before placement of the resin-modified glass ionomer and then light cured for 20 seconds. In groups 2 and 3, the cavities were contaminated with saliva before the primer application and before the glass ionomer placement, respectively. The saliva was applied to the preparation with a saliva-saturated cotton swab (Q-tips) for 5 seconds and dried with compressed air. The saliva was collected on the day of the test and pooled from four individuals. The data for groups 1-3, published previously,[5] were used for comparison. Group 4 had an additional step of selective etching by applying phosphoric acid gel only on the enamel for 20 seconds, followed by rinsing with water and Ketac Nano Primer application as previously described. Groups 5 and 6 had an additional selective enamel etching step whereby saliva contamination happened before the primer application or before the glass ionomer placement, respectively. The restorations were wet polished with an Esthetic Polishing System (EP 200, Brassler USA, Savannah, GA, USA). The experimental groups and steps with saliva contamination are summarized in Figure 1. The sample size was 10 per group.

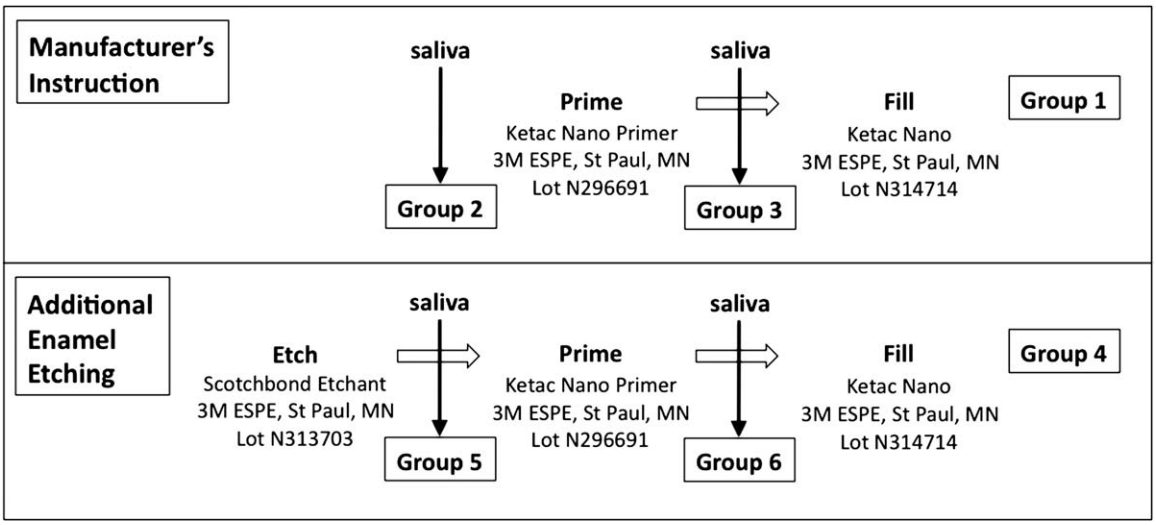

Figure 1. *Diagram summarizing the experimental groups and stages of saliva contamination. The resin-modified glass ionomer was restored according to the manufacturer's instructions in group 1, with saliva contamination before the primer application in group 2 and before the restoration placement in group 3. Group 4 had an additional selective enamel etching step. Groups 5 and 6 also had the selective enamel etching step with saliva contamination before the primer application and before the restoration placement, respectively.*

Microleakage and Statistical Analysis

Root apices were obstructed with utility wax, and the roots were painted with nail polish before the samples were subjected to a thermocycling process, consisting of 1000 cycles alternating between hot water (55°C) and cold water (5°C) with a 30-second immersion time.[14] After thermocycling, the teeth were immersed in 0.5 wt% basic fuchsin solution for 16 hours and sectioned buccolingually through the center of the restoration using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Both sections from each tooth were imaged using a stereomicroscope with a CCD camera (SZX16 and UC30, Olympus, Tokyo, Japan). The microleakage distances at the occlusal (enamel) and gingival (cementum) margins were measured using imaging software (Stream Basic, Olympus Soft Imaging Solution GmbH, Münster, Germany), as shown in Figure 2. Two independent evaluators who were blinded to the group of each tooth performed the measurements. The final microleakage distance was determined as the average of the two evaluators. If a measurement differed more than 10%, the image was reviewed with both evaluators present to determine a consensus distance. For each tooth, the microleakage distances from both sections were averaged into a single number for the enamel or the cementum margin. Microleakage distances were subjected to one-way analysis of variance (ANOVA) followed by the Duncan new multiple range test at a significance level of 0.05. Two-way ANOVA was used to test the effect of the timing of saliva contamination, the effect of selective etching, and their interaction.

RESULTS

Selective etching significantly affected the microleakage of both enamel and cementum regardless of timing of saliva contamination (two-way ANOVA; $p<0.05$). If etching status (etched or not etched) was disregarded, timing of saliva contamination significantly affected the enamel microleakage ($p=0.0001$) but not the cementum microleakage ($p=0.072$). No statistical interaction was shown between the two effects for either enamel ($p=0.4657$) or cementum microleakage ($p=0.4145$).

Microleakage distances at the enamel and cementum margins are shown in Figure 3. The additional step of selective enamel acid etching significantly reduced microleakage distance at the enamel margin but increased microleakage at the cementum margin. Enamel microleakage was highest when saliva contamination occurred before the restoration placement (group 3) but was reduced significantly by the selective etching step (group 6; $p<0.05$). When saliva contamination occurred before the primer application, enamel microleakage was also reduced by selective etching, although the values were not significantly different (group 2 vs group 5).

Microleakage at the cementum margin of the resin-modified glass ionomer restored according to

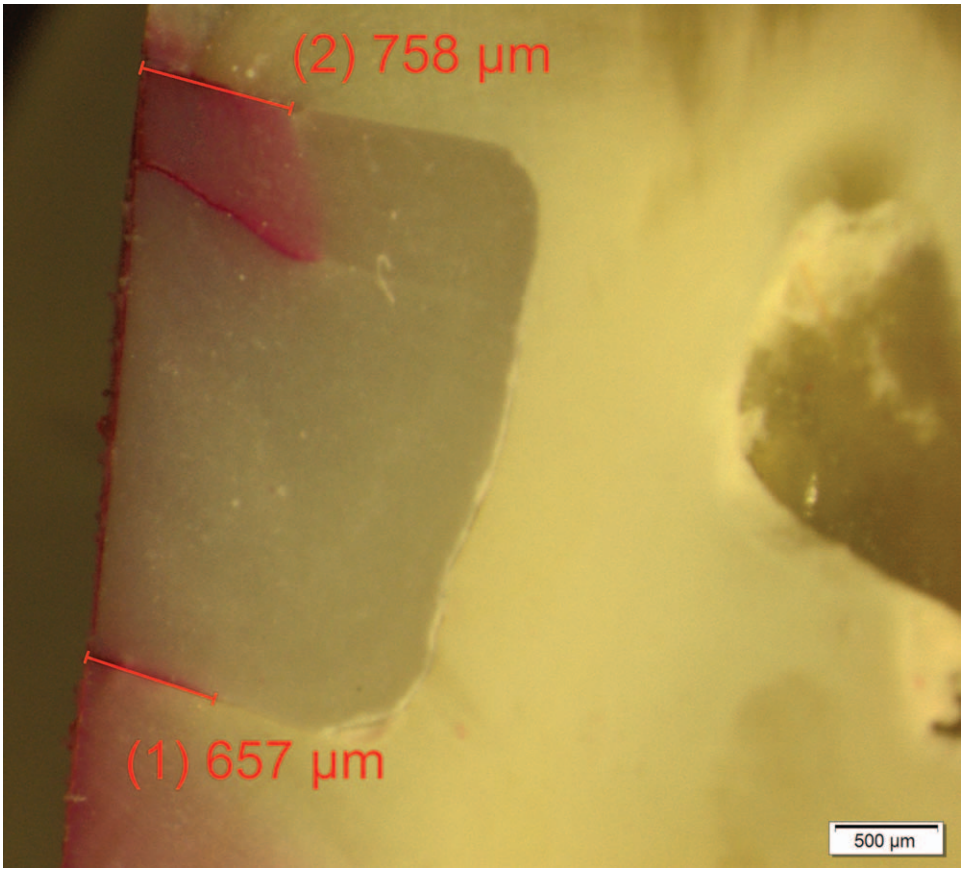

Figure 2. *Using imaging software to measure microleakage distances at the occlusal and gingival margins of the restoration.*

the manufacturer's instructions did not significantly increase with saliva contamination (groups 1-3). The additional selective etching step increased microleakage at the cementum margin (groups 4-6) and became significantly different when saliva contamination occurred before restoration placement (group 6). Group 6 also had the highest microleakage distance.

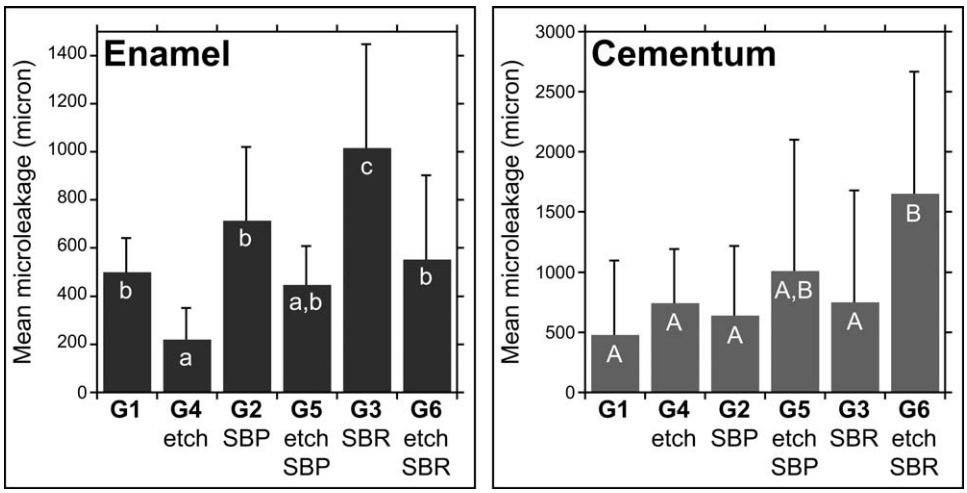

Figure 3. *Microleakage distances (mean and standard deviation) at the enamel and cementum margins. The reference groups (1-3) had no selective enamel etching step.*[5] *Same letters indicate mean values that are not significantly different (analysis of variance followed by Duncan* post hoc *test, significance level = 0.05). SBR, saliva contamination before restoration; SBP, saliva contamination before application of primer.*

DISCUSSION

Enamel Microleakage

Unlike other glass ionomers that use polyacrylic acid to condition the cavity surfaces, the resin-modified glass ionomer in this study used a no-rinse acidic primer for bonding. The results showed that selective etching reduced microleakage of the resin-modified glass ionomer at the enamel margin. Selective enamel etching is an additional step using phosphoric acid to etch only the enamel margin of a cavity, followed by rinsing, prior to the application of an acidic primer. Etching of enamel with phosphoric acid increases the surface area available for bonding and improves the wettability of the enamel surface.[16] The increase in enamel bond quality in this study is mirrored in other studies that tested self-etch adhesive with composites.[12,14,17] Resin-modified glass ionomer and composites thus behaved similarly in terms of qualitative bonding performance to etched enamel, likely because Ketac Nano also uses a resin-based primer that forms a bonding layer on the tooth surface. Since Ketac Nano restorations have shown significant enamel marginal staining in 1 year in a clinical study,[18] the proposed selective enamel etching may help to improve the marginal quality and reduce such staining.

Cementum Microleakage

Although the selective etching step was applied only on the enamel, our results showed that it also affected microleakage at the cementum margin. The cementum leakage increased in all groups after selective enamel etching and was statistically significant higher when saliva contamination occurred before restoration placement (see group 6 in Figure 3). An improved enamel bond for the restoration could have caused a higher failure rate at the cementum margin during polymerization shrinkage or thermocycling because if the enamel bond strength improves, the stress on the cementum margin will increase. Moreover, during rinsing, phosphoric acid may have contacted the dentin surface and depleted hydroxyapatite. A resin-modified glass ionomer forms an ionic bond between its carboxyl groups and calcium in hydroxyapatite and provides micromechanical retention at the interface.[19] Depleted hydroxyapatite from an etched dentin surface was suggested to reduce bonding effectiveness over time,[14] although no significant difference was found in bond strengths of glass ionomer bonded to dentin treated with phosphoric or polyalkenoic acid.[20] Note that Ketac Nano is not applied directly onto the tooth surface. Its primer is comprised of methacrylate-modified polyalkenoic acid (Vitrebond copolymer), 2-hydroxyethyl methacrylate, which is similar to the liquid component of the Ketac Nano restorative material.

The acidic nature of the Ketac Nano primer that was applied to both the enamel and the dentin facilitates adhesion by modifying the smear layer and wetting the tooth surface.[3] If the phosphoric acid used in the selective enamel etching had come into contact with dentin during rinsing, it may also have removed the smear layer and thus affected the bond quality of the cementum margin. Some studies that applied selective enamel etching in combination with a self-etch adhesive reported reduced bond strength on etched dentinal surfaces.[12,17]

Saliva Contamination

There is no consensus in the dental literature about the effect of saliva contamination on the quality of the bond of glass ionomer restorations. Results reported range from no effect of saliva contamination on enamel and dentin bond strength[21,22] to some effect on marginal integrity[23] to substantial bond strength reduction that cannot be recovered with rinsing or etching.[24] Saliva contamination, independent of timing, reportedly did not significantly affect dentin bond strength when a self-etch adhesive (instead of an acid primer) was used with a resin-modified glass ionomer.[25] In the present study, which used the acid primer, we found that saliva contamination increased microleakage distance in both enamel and cementum margins, although the effect was not always statistically significant (Figure 3). Saliva contamination after the application of the acidic primer had higher microleakage than when the contamination occurred before primer application, especially at the enamel margin, where the difference was statistically significant. The current results therefore show that the time of contamination was important for the acidic primer and resin-modified glass ionomer used in this study.

CONCLUSIONS

The results of our study show that a selective enamel etching improved the bond quality of a resin-modified glass ionomer to enamel, also in cases of saliva contamination. On the other hand, microleakage at contaminated cementum margins increased with the selective enamel etching. When enamel bonding becomes crucial, selective etching is an option to be considered when using a resin-modified glass ionomer.

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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 Influence of Elastic Modulus of Inlay Materials on Stress Distribution and Fracture of Premolars

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Clinical Relevance

To produce inlays, it is important to know the type and behavior of material used because the elastic modulus and rigidity of the adhesive restorative material can promote different stress distributions and cusp deflection.

SUMMARY

The purpose of this study was to evaluate the influence the width of the occlusal isthmus and inlay material had on the stress distribution, displacement, and fracture resistance of upper human premolars. For this *in vitro* test, 35 intact upper premolars (UPM) were selected and five were kept intact for the control group (group I). The remaining 30 were divided into two experimental groups (n=15) according to the width of isthmus: conservative (CP) and extensive preparation (EP), one third and more than two thirds of cuspal distance, respectively. Five teeth from each experimental group were left without restoration for negative controls (CPnc and EPnc), and the remaining 10 in each group were subdivided according to the inlay material (resin or ceramic): group CPr, CP + indirect resin; group CPc, CP + ceramic; group EPr, EP + indirect resin; and group EPc, EP + ceramic. The cemented inlays were loaded in a universal testing machine at a crosshead speed of 0.5 mm/min until fracture. The fractured specimens were analyzed with stereomicroscopy, and the values of the fracture resistance evaluated by analysis of variance and Tukey test. For the finite element analyses, an average

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UPM for each group was modeled in Rhinoceros CAD software and imported to Ansys 13.0. An average of 320,000 tetrahedral elements and 540,000 nodes for the seven models were performed using the same experimental simulation setup for each. The models were constrained on the base, and a displacement of 0.02 mm was applied to keep a linear behavior for the analysis. A von Mises stress and total displacement fields were used for the coherence test and the maximum principal stress fields were used for mechanical behavior comparisons. Group I (161.73 ± 22.94) showed a significantly higher mean value than the other experimental groups (EPc: 103.55 ± 15.84; CPc: 94.38 ± 12.35; CPr: 90.31 ± 6.10; EPr: 65.42 ± 10.15; CPnc: 65.46 ± 5.37; EPnc: 58.08 ± 9.62). The stress distribution was different in all of the groups. EPnc showed a higher concentration of tensile stress on the cervical region of the proximal box. CPc and EPc provided a lower tensile stress and a smaller cuspal displacement. Within the limits of this study, the configuration of the inlay preparation is a significant factor in the fracture resistance of premolars: the smaller the amount of remaining tooth, the lower the fracture resistance. In addition, the teeth restored with ceramic materials showed a higher fracture resistance than those restored with composite resin.

INTRODUCTION

With the advent of adhesive dentistry and new and better materials, it is important that professionals be made fully aware of the importance of preserving the natural structures such as enamel, dentin, and pulp vitality. But, it is also important that the preparation be adequate to fulfill the esthetic and functional requirements of the restoration.

The preparation design for an indirect restoration must strike a balance between preserving the tooth structure and obtaining sufficient retention and resistance. Thus, some fundamental aspects are needed to determine the design of a preparation, such as the tooth's structural condition, its functional aspect, esthetic aspect, tooth inclination, retention, occlusion, reconstruction, and the patient's satisfaction.

The resistance to fracture is directly related to the amount of remaining tooth structure. The removal of marginal ridges, the increase in the width of the isthmus, and the increase in the depth gingival of the preparation are the main reasons for the decrease in resistance.[1]

The masticatory loads in the posterior region are much higher than in the anterior region. Among the posterior teeth, the maxillary premolars suffer the most from vertical fractures, leading to the loss of the dental element.[2] This is due to its complex anatomy, location in an area of high masticatory load, and the presence of a very pronounced groove in the buccal root of the first bifurcated premolars that lead to stress concentration, all three of which may cause vertical fractures.

In addition to concerns with maintaining vitality, esthetics is a very important element in meeting the patient's expectations. Indirect restorations have been made in both composite resins and ceramics and show different optical and mechanical properties.

The analysis of the compatibility between the mechanical properties of restorative materials and the tooth structure may assist in the selection of the material that is best for a specific clinical situation. It also has the preparation characteristics that pose the lowest risk of fractures of the restorations.

The restorative material is considered to be a factor that affects the biomechanics (the stress distribution and deflection of the cusp) during occlusal loading. Ceramic materials are fragile and stiff and tend to increase the rigidity of the tooth's structure, thus decreasing the cusp deflection, as was said previously. On the other hand, composite resins show mechanical properties nearer to those of enamel and dentin, but tend to distribute stresses by deformation.[3]

The aim of the current study was to evaluate how the size of the occlusal isthmus and the restorative material can influence stress and displacement distribution and their impact on the fracture resistance of human maxillary premolars. This was done through laboratory testing of fracture resistance and by analyzing the stress distribution and cusp deflection by a method of mathematical three-dimensional finite element analysis.

METHODS AND MATERIALS

Fracture Resistance Test

The brand name, manufacturer, and basic description of the materials used in this study are listed in Table 1.

In accordance with approval from the research protocol by the local ethics committee (052/2010-PH/CEP), 35 fresh and caries-free maxillary premolars

Table 1: *Brand Name, Manufacturer, and Basic Description of the Materials Used*

Material	Manufacturer	Characteristic	Lot
IPS e.max Press	Ivoclar Vivadent	Ceramic lithium disilicate	N30995
Variolink II	Ivoclar Vivadent	Resin cement	M44477
			L46354
Monobond Plus	Ivoclar Vivadent	Silanizing agent	M15219
Signum Ceramis	Heraeus	Composite resin indirect	010131
Adper Single Bond 2	3M ESPE	Adhesive system	N190472ER
Impregum-F	3M ESPE	Impression material polyether-based	421709
			4205\40

were selected, having been extracted for orthodontic treatment. The dimensions were checked by a digital x-ray image by the software Image Tool (UTHSCSA for Windows version 3.0, San Antonio, TX, USA). The exclusion criterion was the crown's occlusal surface area and the angle formed by the two cusps having a value above or below 10% of the average.

Each root was immersed in melted wax up to the 2-mm demarcation line apical to the cementoenamel junction, analyzed by a caliper accurate for simulation of 0.3 mm of periodontal ligament, and then placed in a self-cured polyurethane resin up to the demarcation line in a polyvinyl chloride cylinder 20 mm in height and 18 mm in diameter. After curing, the wax was carefully removed from the root using hot water so that the wax could be replaced with polyether (Impregum F, 3M ESPE, St Paul, MN, USA) to simulate the elastic modulus of the periodontal ligament. The root was then stored in distilled water in a refrigerator at 5°C.

The teeth were then randomly separated into three groups: group I, intact teeth (n=5); group CP, conservative preparation (n=15); and Group EP, extensive preparation (n=15). Groups CP and EP were subdivided into three subgroups; group CPnc – conservative preparation without restoration (negative control); group CPr, conservative inlay + indirect **r**esin; group CPc, conservative preparation + **c**eramic; group EPnc, extensive preparation without restoration (negative control); group EPr, extensive preparation + indirect **r**esin; and group EPc, extensive preparation + **c**eramic.

All preparations were made with a diamond bur No. 3131 (KG Sorensen, Cotia, SP, Brazil) and placed perpendicularly to the long axis of the tooth, with a taper of 6° in the surrounding walls of the cavities. The internal angles were rounded, as is characteristic of the tips used.

The occlusal and proximal boxes of groups CPnc, CPr, and CPc were standardized to a width of 2 mm and a depth of 2 mm using a diamond bur (2 mm) as illustrated in Figure 1.

For the extensive preparations (groups EPnc, EPr, and EPc), two lines mesiodistally generated the occlusal contact point between a steel ball 10 mm in diameter. The intact premolar was first marked at 0.5 mm, the distance considered to be the limit of the width of the isthmus. The occlusal and proximal depths were made the same way as the CP groups (Figure 1).

All teeth were impressed with silicone (Express, 3M ESPE) and sent to the laboratory to manufacture two types of indirect restorations: composite resin (Signum Ceramis, Heraeus, Frankfurt, Germany) and lithium disilicate ceramic (IPS e.max press, Ivoclar Vivadent, Schaan, Liechtenstein). All teeth were etched with 37% phosphoric acid for 15 seconds, washed with a water jet for 15 seconds, and the excess water was removed with absorbent paper. Then, the two layers of adhesive (Single Bond 2, 3M ESPE) were applied and cured following the manufacturer's instructions (Figure 2).

The inlays were ultrasonically cleaned (Ultrasound E15H Elma, South Orange, NJ, USA) with distilled water for 15 seconds then air-dried for 30

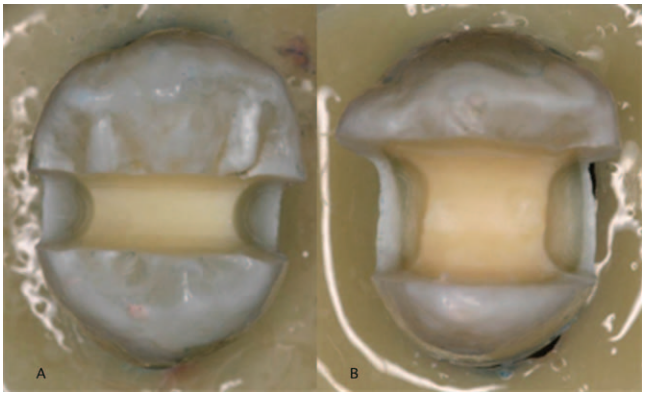

Figure 1. *(A): Conservative Preparation. (B): Extensive preparation.*

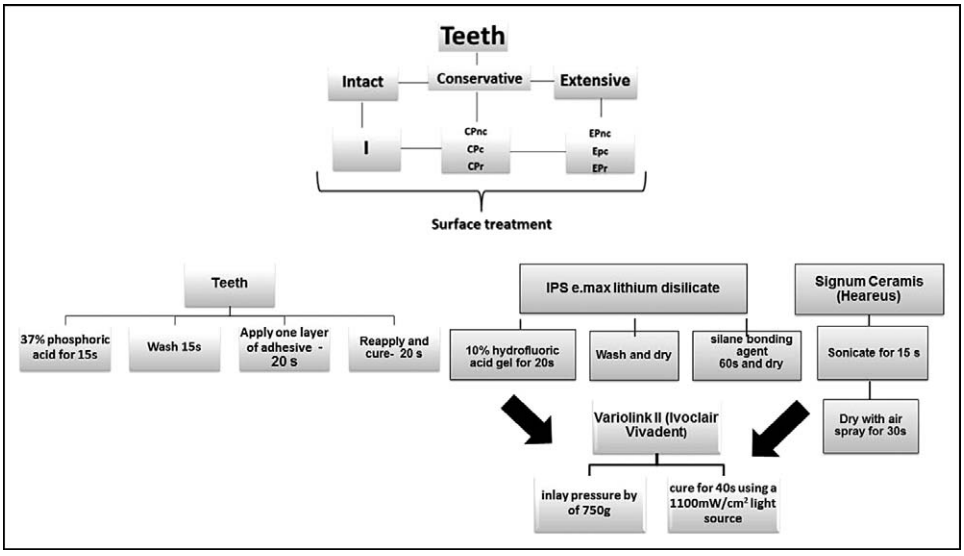

Figure 2. *Scheme of methodology*.

seconds. The ceramic surface was etched with 10% hydrofluoric acid gel for 20 seconds, rinsed thoroughly, and dried with paper towels. A silane-bonding agent (Monobond Plus, Ivoclar Vivadent) was applied for 60 seconds (Figure 2).

The inlays were coated with resin cement (Variolink II, Ivoclar Vivadent), settled in the cavity under 750 g pressure, and the excess removed. The inlays were cured for 40 seconds using a 1100 mW/cm^2 light source (Poly Wireless KAVO, Brazil Ind Com Ltda, Joinville, SC, Brazil), allowed to set for 10 minutes, and then stored in distilled water at 37°C for 24 hours (Figure 2).

The specimens were loaded on the enamel surface until fracture using a sphere 10 mm in diameter in a universal testing machine (EMIC, DL200MF-EMIC Test Equipment and Systems Ltd, Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/min. The data were analyzed by Levene, two-way analysis of variance (ANOVA), and Tukey test with a significance level of 5%. The fragments were analyzed under a stereomicroscope at 20× magnification. The types of fractures are listed in Figure 3.

Finite Element Analysis

The complete tooth structure and polyurethane resin were modeled in the Rhinoceros 4.0 (McNeel North America, Seattle, WA, USA) software, within the BioCad (CTI Campinas, SP, Brazil) protocol,[4] and the *.stp* file was imported into Ansys 13.0 (ANSYS Inc, Houston, TX, USA) for the finite element analysis (FEA). All materials were considered isotropic, linear, homogeneous and their moduli included in Table 2. The contact regions between the structures were considered perfectly bonded, and the mesh, with a quadratic tetrahedral element, was controlled by a sizing method with 0.4 mm resulting in a total of 348,296 nodes and 231,444 elements. The base of the polyurethane was considered fixed in the three axes, and the sphere was displaced by 0.02 mm parallel to the principal axis to keep the analysis in the elastic field.

Table 2: *Mechanical Properties of Materials Used on Finite Element Analysis*

Material	Longitudinal Elastic Modulus, MPa	Poisson's ratio	Reference
Stainless steel	200,000	0.3	Ansys Library 13.0
Dentin	17,600	0.31	Reinhardt and others[27]
Enamel	48,000	0.3	Holmes and others[28]
Polyurethane Axson F16	3,600	0.3	Owner characterization
Ligament	68.9	0.45	Holmes and others[28]
IPS E.max Press	91,000	0.24	Albakry and others[29]
Signum Ceramis	4,854	0.3	Manufacturer data

		Type of failure					
		I	II	III	IV	V	VI
							
Caracteristics Buccal or lingual		enamel	enamel+dentin at middle third	enamel+dentin at cervical line	Restoration+tooth at occlusal third	Restoration+tooth up to cervical line	At adhesive interface
Percentage of total fracture [%]		5.12	35.89	28.20	7.79	15.38	7.69
Experimental groups	**I**	2857	42.85	28.57	0	0	0
	CPnc	0	33.30	66.60	0	0	0
	EPnc	0	42.86	57.1	0	0	0
	CPr	0	80.00	0	20.00	0	0
	CPc	0	25.00	25.00	0	50.00	0
	EPr	0	0	0	60.00	20.00	20.00
	EPc	0	20.00	0	0	40.00	40.00

Figure 3. *Mapping of Fractures and Values of Total Percentage of Fracture in Each Group*

RESULTS

The descriptive statistics (mean, in kgf and standard deviation) for each of the seven groups, the statistical analysis that evaluated the effects of the variables studied on the fracture resistance, and the results are presented in Tables 3, 4, and 5, respectively. Group I (161.73 ± 22.94) had the highest average, followed by groups EPc (103.55 ± 15.84), CPc (94.38 ± 12:35), CPr (90.31 ± 6.10), EPr (65.42 ± 10.15), CPnc (65.46 ± 5.37), and EPnc (58.08 ± 9.62). Groups CPr and CPc and CPnc and EPnc showed no statistical difference between them.

The types of failure are given in percentages and are presented in Figure 3. In groups I and CPr, the most prevalent type of fracture was type II. For Groups CP and EP, the most prevalent type of fracture was type III. The fracture types IV, V, and VI were present in groups EPr, CPc, and EPc, respectively.

Under analysis by FEA, group I had a greater concentration of tensile stress in the central sulcus and also a larger difference in the concentration gradients in the lingual cusp, especially in the middle and occlusal third.

Groups CPnc and EPnc show that as the remaining tooth structure decreases, there is a greater concentration of tensile stress in the wall of the pulp preparation and a greater difference of the concentration gradient as compared with the intact tooth, especially in the lingual cusp (Figure 4).

The distribution of stress showed that the conservative teeth that were restored with ceramics (CPc) had a higher concentration of tensile stress in the sulcus occlusal of the restoration and a higher concentration of compressive stress on the walls of the gingival preparation (Figure 5). For the teeth with conservative preparation and restored with composite resin (CPr), there was a higher distribution of tensile stress in the cusps and a reduced difference in the concentration gradient (Figure 6).

Table 3: *Means and standard deviations of fracture resistance for groups without restoration*[a]

Group	Mean ± SD, kgf	Homogeneous Groups	ANOVA
I	161.73±22.94	A	SS: 46091.7
CPnc	65.46±5.37	B	MS: 23045.9
EPnc	58.08±9.62	B	$p<0.0000$

[a] *Different letters indicate statistically different values (ANOVA/Tukey tests). Abbreviations: SS, Sum of squares; MS, Mean square.*

Table 4: *Means and standard deviations of fracture resistance for groups with conservative preparation*[a]

Group	Mean ± SD, kgf	Homogeneous Groups	ANOVA
I	161.73±22.94	A	SS: 34591.2
CPnc	65.46±5.37	C	MS: 11530.4
CPr	90.31±6.10	B	$p<0.0000$
CPc	94.38±12.35	B	

[a] *Different letters indicate statistically different values (ANOVA/Tukey tests). Abbreviations: SS, Sum of squares; MS, Mean square.*

Table 5: *Means and standard deviations of fracture resistance for groups with extensive preparation*[a]

Group	Mean ± SD, kgf	Homogeneous Groups	ANOVA
I	161.73±22.94	A	SS: 44998.4
EPnc	58.08±9.62	C	MS: 14999.5
EPc	103.55±15.84	B	$p<0.0000$
EPr	65.42±10.15	C	

[a] *Different letters indicate statistically different values (ANOVA/Tukey tests). Abbreviations: SS, Sum of squares; MS, Mean square.*

By restoring the contour of the marginal ridges with restorations, a reduction of the stress concentration can be observed on their dental structures when compared with the prepared teeth without the restoration. The teeth that were restored with ceramics showed a higher concentration of tensile stress in the restoration, unlike what happens with the teeth restored with resin, where stress restoration was transmitted to the dental structure (Figures 5 and 6).

Upon restoring the tooth, there was a decrease in the deflection of the cusps. The groups restored with indirect composite resin (groups CPr and EPr) showed a greater deflection than those restored with ceramics (groups CPc and EPc).

DISCUSSION

The purpose of this study was to investigate the possibility of performing an inlay at the maximum width allowed of the occlusal isthmus, before changing the preparation to an onlay due to the need to protect the cusp. The average occlusal force in the literature is 21.7 kgf (about 212 N).[5] In the region comprising the premolar and molar occlusal areas, these forces vary between 161 N and 351 N, respectively.

In this study, intact teeth showed a mean fracture resistance of 161.73 kgf (1584 N). This is superior to that found by Takahashi and others[6] in 2001, which was 736 N, but lower than that found by Soares and others[7] in 2006, which was 320.5 kgf (3140 N). In all three of these results, the tooth structure is strong enough to weather natural chewing forces since the results exceed the average forces of occlusion. However, this is not what happens in a real situation.

The cusp deflection is a good parameter for analysis when studying fracture resistance. The type of cement used and its interface quality can impact the values of displacement; the higher the adhesive strength, the lower the cusp deflection. Thus, the restorations cemented with conventional resin cement using the total etching technique have

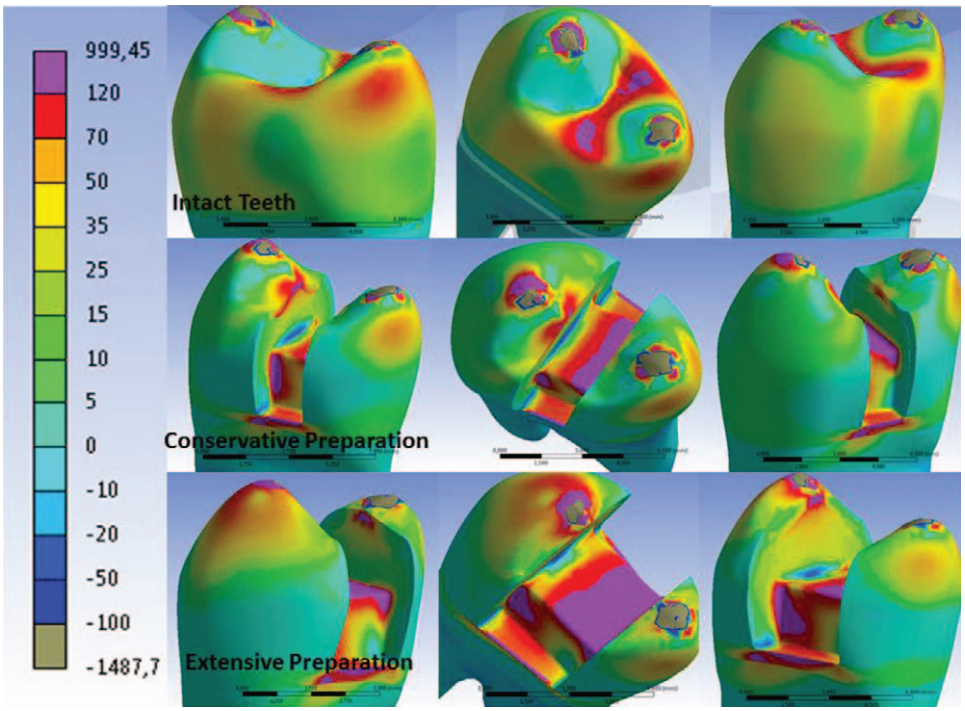

Figure 4. *Maximum principal stress distribution in groups I, CPnc, and EPnc.*

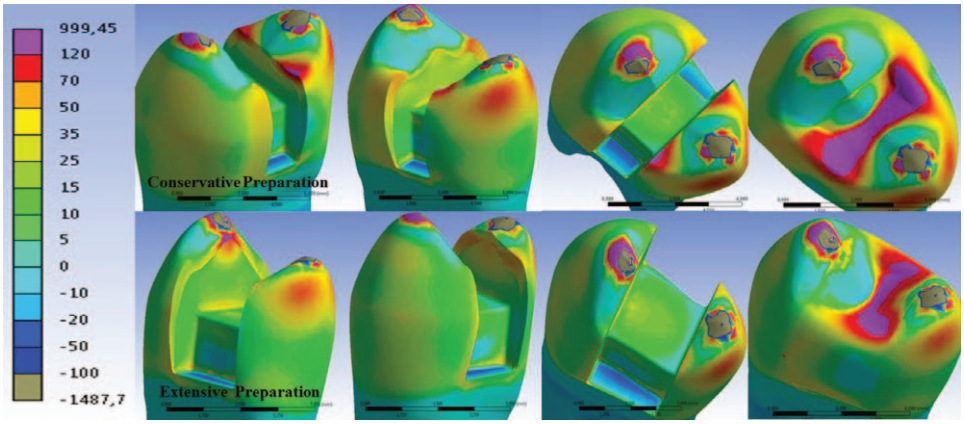

Figure 5. *Maximum principal stress distribution in groups CPc and EPc.*

a better union of the adhesive interface and show a greater resistance to cusp deflection.[8] However, restorations cemented with self-adhesive resin cement have a lower stability with a pronounced cusp deflection and, therefore, are less resistant to fracture.[8]

Analyzing the results, when compared with the intact control (group I), the conservative (CPnc), and extensive (EPnc) preparations showed a 50% reduction of tooth structure and, consequently, a reduction of structural strength and an increase in the risk of fracture.[9]

Though numerically, group CPnc had a greater fracture resistance than group EPnc, there was no statistical difference for a significance level of 5%, and the coefficients of variation were around 18%. We can therefore infer that by removing the marginal ridge, a marked decrease in fracture resistance occurs, regardless of the amount of tissue removed. Soares and others[7] found that inlays with a conservative preparation, even with a higher average of resistance to fracture, also did not differ from inlays with an extensive preparation. This is because the reduction of fracture resistance is due to the removal of marginal ridges rather than the uniting and supporting of the buccal and palatal cusp, as well as to the increase in the isthmus and the depth of preparation in the occlusogingival direction.

The absence of important structures, such as the marginal ridges, exerts an influence on the fracture resistance of teeth. The location of a lost tooth structure is often as relevant as the amount lost, and thus there should be maximum preservation of the prime areas of the teeth, such as the edges and marginal ridges.[10]

The findings that CPnc and EPnc did not show a statistical difference, though they were lower than group I (Table 3), are in agreement with those of Larson and others,[11] who showed that the extent of a cavity that involves the proximal boxes does not cause a significant reduction in the tooth's resistance, since only a small portion of dentin is removed. In the present study, Figure 5 shows that the stress distribution for both cavities studied was

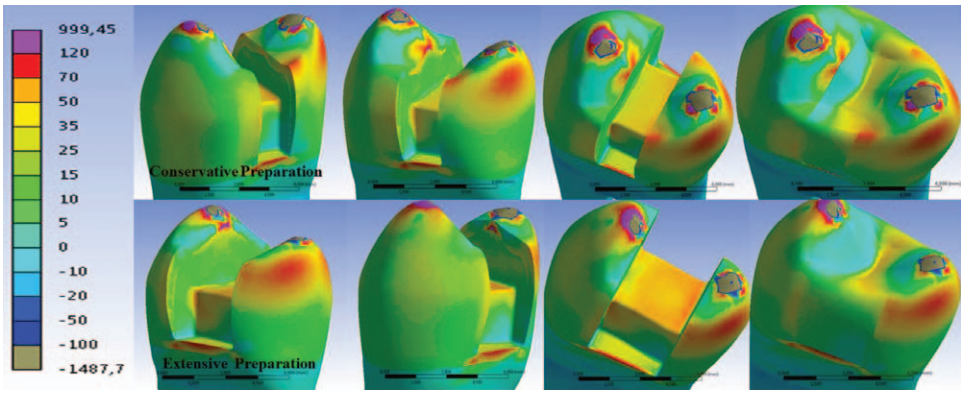

Figure 6. *Maximum principal stress distribution in groups CPr and EPr.*

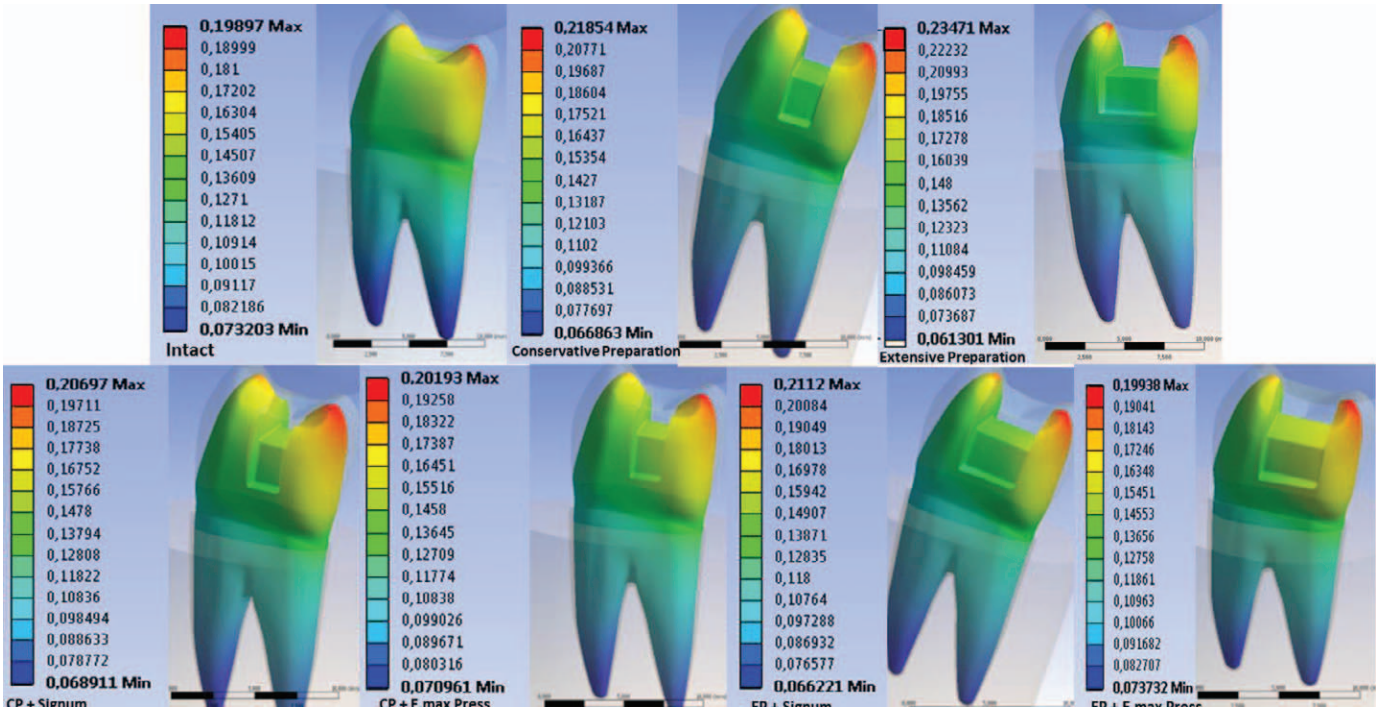

Figure 7. *Total displacement in all groups.*

similar. Figure 4 shows the teeth that are missing a portion of their structures, such as the marginal ridges. There was also a large increase in tensile stress concentration, crossing the occlusal isthmus in the intact portion of the tooth. In groups CP and EP, this stress increases in the region of the pulp wall.

However, the numerical value showed the larger the isthmus the lower the resistance to fracture, despite the nonstatistical difference. The progressive elimination of the dental tissue, such as severe loss of the marginal ridge and dentin remaining above the pulp chamber, is associated with an increased bending of the cusps.[12] According to studies by Mondelli and others,[1] Reeh and others,[13] and Hansen and others,[14] the cusps should be preserved whenever possible to maintain the resistance of the tooth, agreeing with the trend observed in FEA (Figure 7).

Through the analysis of the total displacement, consistency and connectivity of the meshes, von Mises stress fields of the models, and the compared results from the literature, it was shown that it is feasible to use these models in the analysis of stress distribution by means of the FEA. This analysis is a simulation, thus the results are approximate to what occurs *in vitro*. However, the use of FEA is necessary for understanding the failure types and the probable place of the fracture origin.

Group I presented a predominance of fractures in the lingual cusp and occlusal and middle thirds, with cohesive failures in the enamel (types I and II). This probably occurred because the applied load exceeded the proportional limit of the enamel (Figure 3).

As the remaining tooth structure decreases, there is a greater concentration of tensile stress in the pulpal wall of the preparation as compared with the intact tooth, especially in the lingual cusp. This corroborates the findings by Lin and others,[15] who found a higher stress concentration along the lingual-pulpal line angle and, when compared with the intact tooth, found that maximum principal stress was higher as the depth of the pulp wall increased. That is, the greater the depth of preparation, the greater the risk of fracture in restored teeth.

Blaser and others[16] observed that the reduction in tooth resistance is more significant when there is an increase in the depth of preparation, approaching the pulp, compared with the weakening of the occlusal isthmus, when there is an increase in the extent of preparation mesiodistally.

The greater predominance of fractures in groups CPnc and EPnc occurred in the middle third of the occlusal-lingual cusp and in the center of the pulp

wall . This trend can be explained by the presentation of the stress gradient, as shown in Figures 1 and 2, where the path of the fracture is perpendicular to the contour of maximum principal stress.

The groups that have been restored (CPr, EPr, CPc, and EPc) showed fracture resistance values greater than the groups without restoration. According to St-Georges and others,[9] the restored teeth have a 4%-15% increase in fracture resistance compared to the unrestored teeth.

Intact teeth will rarely fracture under masticatory stresses. However, fractures can occur in teeth that have cavity preparations and restorations, either with composite resin or ceramic, because the cavities will weaken the remaining tooth structure. By restoring the tooth, directly or indirectly, the tooth's fracture resistance can be partially restored.[17-19]

In this study, the increase in fracture resistance in the preparation of conservative management was approximately 38% for both groups CPr and CPc; in the extensive preparation there was an increase of 12% for the EPr and 94% for the EPc compared with CPnc and EPnc, showing the influence of the rigidity of the material in this property. The capacity of the adhesive bond interface influences the displacement of the cusp since the rebuilding of the dental structure, the marginal ridge, and the enamel bridge restores the occlusal anatomy. This reduces the deflection of the remaining cusps, regardless of the material used, which reduces the chance of a fracture.

After restoration, the groups with conservative preparation showed similar fracture resistance values for both materials, but the mechanical behavior, which was analyzed by the finite element method, was different. This is because when the conservative preparations were restored with ceramic, there was a higher concentration of stress compared with those restored with composite resin, showing a dissipation of stress in the tooth's restoration. In the groups with extensive preparation, the values of fracture resistance, such as the mechanical behavior between the groups restored with composite resin (EPr) and ceramics (EPc), were different.

For groups CPc and CPr, the type of restorative material had no influence on the fracture resistance. The data showed no statistically significant difference for the two materials used. The elastic modulus of the restorative material also did not affect the results of the fracture resistance. The effect of the adhesion and the quantity of the material removed from the teeth during the preparation did not change the displacement of the cusp. The higher rigidity of ceramics compared to resin and the pertinent smaller deflection of the cusps, with which an adequate bonding to the interface is associated, causes the whole to become more resistant and reduces the probability of a fracture. This is because the stress values are related to the modulus of elasticity of the restorative material.[20,21] This finding corroborates the study by Scherrer and de Rijk[22] which showed that the higher the elastic modulus of the material, the higher the stiffness and then the greater the value of fracture resistance.

For many authors, while adhesively cemented ceramic inlays that extend 1/3 up to 1/2 of the intercuspal distance have similar resistance as sound tooth, this does not occur when the tooth is restored with composite resin.[19,23-26]

Since the elastic modulus of the composite resin is less than the elastic modulus of teeth (enamel and dentin), it will concentrate and transmit to the tooth a tensile stress. In 2011, Desai and Das[19] also showed that in materials exhibiting low elastic modulus, a higher concentration of stress is transferred to the tooth structure. Because of this, there will be a greater deflection of the cusps and, consequently, a higher probability of fracture in the tooth compared to the ceramic restoration, which explains the predominance of the type II fracture (tooth only). In the group restored with ceramics (CPc), where there was a larger tensile stress in the restoration, the material will probably fracture before the tooth structure, which explains the greater number of type V fractures (tooth-restoration) (Figure 3).

The groups with extensive preparation restored with ceramic (EPc) showed a higher value of fracture resistance than those restored with composite resin (EPr). This probably occurred due to the difference in structural rigidity of the system caused by the different modulus of the two restorative materials. The large quantity of ceramic seems to have replaced much of the dentin. As the ceramics have a higher elastic modulus, the flexibility of the structure decreases, causing the offset tooth to be smaller, and thus more resistant than the teeth with conservative preparation (CPc).

Despite the limitations of the FEA models (related to the cement layer, simplification of experimental setup and assumption of a linear behavior), valid results were achieved that could explain the experimental fracture behaviors.The combination of mechanical testing and fracture resistance analysis by finite element methods has shown promise in

assessing the influence of the restorative material and the configuration of the preparation in the failure of indirect restorations.

Thus, this experiment raises the awareness for the need of further studies to formalize a method of occlusal loading that can take place in a more real and ideal simulation (contacts A, B, and C), as well as in endodontic treatment. These analyses take into account the thickness and mechanical properties of the cement, as well as the inclination of the walls of the preparation and the rounding of the line angle.

CONCLUSIONS

According to the methodology employed in this study and within its limitations, we concluded the following: 1) the volume of the cavity preparation is a significant factor influencing stress distribution and resistance to fracture, and 2) ceramic restorative material tends to concentrate more stress inside the inlay and results in lower cusp deflection than the resin; resin tends to transfer more stress to the tooth structure and promote lower fracture resistance than the ceramic.

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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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Effect of Water Storage on the Flexural Strength of Four Self-etching Adhesive Resin Cements and on the Dentin-titanium Shear Bond Strength Mediated by Them

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Clinical Relevance

This study identified that Rely X and G-Cem afforded a significantly higher dentin-titanium adherence compared with Maxcem and SmartCem2.

SUMMARY

Aim: The aim of this study was to evaluate the effect of water storage on the flexural strength (σ_f) of four self-etching adhesive resin cements (SEARC) and on the dentin-titanium shear bond strength (SBS) mediated by them.

Materials and Methods: The selected SEARC were Rely X Unicem, G-Cem, Maxcem, and SmartCem2. For each material, 50 bars (2×2×25 mm) were made and stored in water at 37°C for 1 hour, 1 day, 7 days, 30 days, and 60 days before σ_f was determined via a three-point bend test. Titanium cylinders were bonded to freshly exposed human dentin surfaces using the selected cements. Fifty samples were obtained for each SEARC and were stored in water at 37°C for 1 hour, 1 day, 7 days, 30 days, and 60 days before SBS was determined. The results were statistically analyzed using two-way analysis of variance followed by Scheffé multiple means comparisons (α=0.05). Pearson's correlation coefficient between σ_f and SBS was determined.

Results: Significantly different σ_f and SBS values were obtained for the four cements. With regards to the effect of water storage, the σ_f of all materials increased during the first 7

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days, was not significantly different between materials by 30 days, and then remained relatively constant or decreased for SmartCem2; SBS was not affected by water storage, with the exception of Maxcem, where a significant drop in SBS was detected after 1 day and no deterioration thereafter. No correlation was found between σ_f and SBS.

Conclusions: Under the experimental conditions of this study, 60 days of water storage negatively affected the σ_f of SmartCem2 but did not negatively affect the SEARC-mediated dentin-titanium SBS (Maxcem showed a significant drop in SBS after 1 day but no deterioration thereafter). The dentin-titanium adherence afforded by Rely X and G-Cem was significantly higher than that of Maxcem and SmartCem2.

INTRODUCTION

Over the past 50 years, the practice of adhesive dentistry has changed dramatically. Even if some aspects of cemented prosthodontic restorations have been superseded by dental implantology, adhesive dentistry remains the core of the therapeutic arsenal of restorative dentistry. The principles of adhesion to enamel and dentin, set by the pioneering work of Buonocore,[1] have evolved over the years. Starting from the original total-etch multistep systems, we have witnessed the introduction of total-etch two-step systems, self-etch two-step systems, and finally, self-etch one-step systems, all in an effort to shorten and simplify the application procedure.[2-4] In the case of total-etch two-step systems, the problem of determining the appropriate dentin moisture required for successful bonding renders them technique sensitive.[5] Concerns related to the continuous etching of the substratum by self-etch based systems have been raised,[6,7] along with concerns related to their ability to adequately precondition enamel. Moreover, incompatibility between self-etch based systems and chemically cured resin composites has been identified and the mechanism has been elucidated.[8-11] In spite of these concerns, the attractive aspect of a shorter and simpler application procedure has led to a relatively widespread use of self-etch based systems.[12-14] It should be emphasized, however, that total-etch multistep systems afford the most reliable performance and are considered the gold standard.[2,15]

The evolution of adhesive systems was paralleled by the evolution of luting agents.[16,17] Major landmarks in this evolution were the introduction of zinc polycarboxylate cement[18] (the first cement able to chemically bond to hard tooth tissues), glass ionomer cements,[19] resin modified glass ionomer cements,[20] resin cements, adhesive resin cements, and finally, self-etch adhesive resin cements (SEARCs),[4,17] which have evolved along the principles of self-etch bonding systems. The latter seem to have all the required properties of an ideal luting agent: a resin component to reduce solubility and brittleness, adequate esthetic properties, the ability to precondition and chemically bond to hard tooth tissues, and a shorter and simplified application procedure.[21,22] However, as in the case of self-etch based adhesive systems, the adherence of SEARCs degrades over time.[23,24]

The aim of this study was to assess the effect of water storage on the flexural strength (σ_f) of four commercially available SEARCs and on the dentin-metal shear bond strength (SBS) afforded by them. To simulate a clinically relevant dentin-SEARC-metal interfacial structure, we chose to lute Grade 3 commercially pure titanium (Ti)[25] rods to human dentin. The biocompatibility and corrosion resistance of Ti explain its increased use in dentistry. Several recent publications have investigated bonding to Ti.[26-29] The use of Ti for crown/bridge applications involves attachment to dentin, which was the rationale for selecting Ti for this study. The two null hypotheses tested were 1) there is no difference between the four selected SEARCs with regards to their σ_f and the SBS of dentin-Ti adhesive interfaces mediated by them, and 2) water storage does not affect the σ_f of the four selected SEARCs or the SBS of dentin-Ti adhesive interfaces mediated by them.

METHODS AND MATERIALS

The end surfaces of grade 3 commercially pure Ti rods (3 mm diameter and 20 mm height) were polished perpendicularly to the long axis on 800-grit silicon carbide (SiC) discs under water irrigation, followed by sandblasting (0.2 MPa pressure) with 50 μm alumina. Before luting, the Ti rods were sonicated in acetone for 3 minutes.

The four SEARCs selected for this study, along with pertaining information and group codes, are listed in Table 1. We used 200 caries-free human third molars (with bioethical approval from Université Paris-Descartes in conformity with Law Nr. 2004-800, August 6, 2004) within three months after extraction. The teeth were stored in an aqueous 1% chloramine-T solution at 4°C until used. After the occlusal enamel was flattened, the samples were

Table 1: *Details Regarding the Self-etching Adhesive Resin Cements Used*

Product/Manufacturer/ Lot/Expiry Date/pH	Group Code	Composition According to the Manufacturer
RelyX Unicem 3M ESPE Dental, Seefeld, Germany Lot 414519/2012-03 pH 2	RXU	Powder: Alkaline (basic) fillers, silanated fillers, initiator components, pigments Liquid: Methacrylate monomers containing phosphoric acid groups; methacrylate monomers, initiator components, stabilizers. Filler: 72 wt%; average size <9.5 μm
Maxcem Elite Kerr, Orange, CA, USA Lot: 3645144/2012-09 pH 2.3	MXC	Bis-GMA, UDMA, TEGDMA, GPDM, barium glass filler, fluoro-alumino-silicate glass filler, nano-ytterbium fluoride, fumed silica Filler: 67 wt%; average size 3.6 μm
SmartCem2 Dentsply, Milford, DE, USA Lot: 1004221/2012-04 pH 2.2	SMC2	UDMA, di-and tri-methacrylate resins, phosphoric acid modified acrylate resin, fluoro-alumino-silicate glass, organic peroxide initiator, CQ, phosphene oxide photoinitiator, accelerators, butylated hydroxy toluene, ultraviolet stabilizer; titanium dioxide, iron oxide, hydrophobic amorphous silicon dioxide Filler: 69 wt%; size 0.1-7 μm
G-CEM GC Tokyo, Japan Lot: 1012221/2012-12 pH 2	GC	4-META, UDMA, dimethacrylate, alumino-silicate glass, pigment, dimethacrylate, distilled water, phosphoric ester monomer, initiator, and stabilizer. Filler: 71.4 wt%; average size 4 μm

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; CQ, camphorquinone; GDMA, glycidyl dimethacrylate; MDP, methacryloxydecaethyl phosphate; 4-META, 4-methacryloxyethyl trimelythic anhydride; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

embedded in self-curing acrylic resin (Plexil A6, Escil, Chassieu, France) in a cylindrical mold. The embedded samples were ground perpendicular to the long axis of the tooth, using 800 grit SiC paper under running tap water, to expose an approximately 3×3 mm area of coronal dentin. The opposite side of the resin cylinder was ground parallel to it (checked with a bubble level). Prepared specimens were stored in distilled water at 37°C until used. They were randomly allocated to the four experimental groups, one for each SEARC. The bonding protocol followed manufacturers' instructions.

After the respective SEARCs were applied to dentin, the resin cylinder containing the tooth was fixed in a special device (Figure 1a) to allow control of cement thickness and perpendicular alignment of the Ti rods. The Ti rod was brought into contact with the cement layer and placed under a 70 g load (Figure 1b). The excess cement was delicately removed immediately after assembly, before curing. In the dual-cure mode, the light source was a Demetron LC Curing Light (Kerr Corporation, Orange, CA, USA), activated for 40 seconds at four diametrically opposed locations around the sample, at the level of the adhesive interface, for a total of 160 seconds of curing time. Each bonded sample (Figure 1c) was maintained under load for 10 minutes in a dry place at room temperature, as per manufacturers' instructions. The specimens were then stored in tap water at 37°C for 1 hour, 1 day, 7

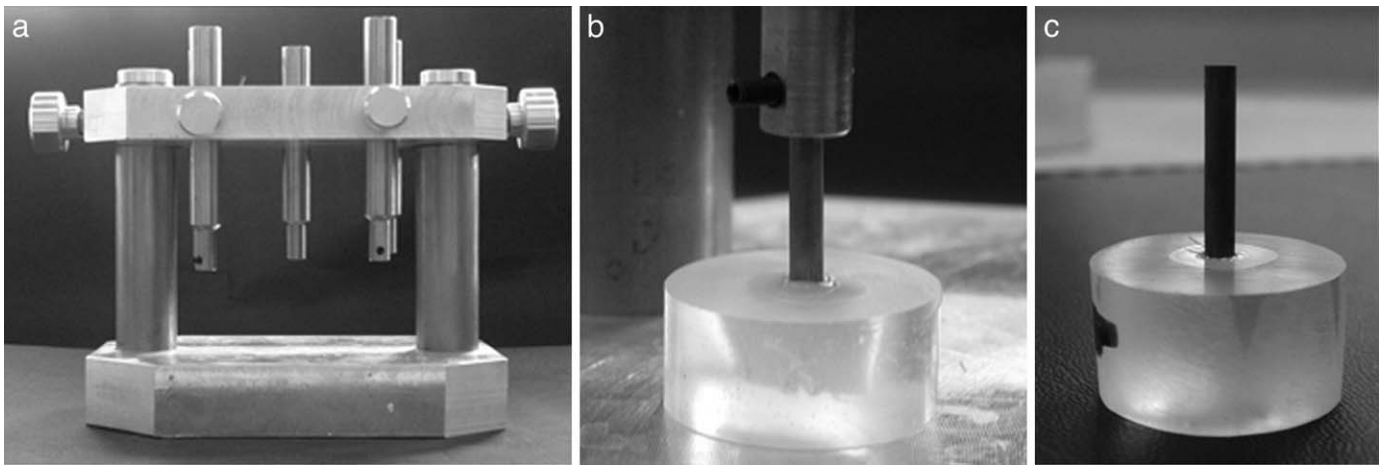

Figure 1. *(a) Device for specimen fabrication. (b) Ti rod and cement, held in contact with dentin surface. (c) Final specimen, with Ti rod cemented onto dentin surface.*

Table 2: *Results of Shear Bond Strength (SBS) Tests (Mean ± SD) and Statistical Analysis**

Group	SBS (in MPa; n=10)				
	Time				
	1 h	1 d	7 d	30 d	60 d
RXU	16.4 ± 4.3^{aA}	12.2 ± 5.2^{aA}	17.2 ± 2.7^{aA}	16.8 ± 8.2^{aA}	18.6 ± 4.6^{aA}
MXC	9.0 ± 2.7^{aB}	4.9 ± 3.2^{abB}	3.7 ± 3.5^{bB}	3.5 ± 3.0^{bB}	4.6 ± 3.1^{bC}
SMC2	5.6 ± 2.0^{aB}	5.7 ± 3.2^{aB}	4.7 ± 3.0^{aB}	3.2 ± 2.0^{aB}	2.1 ± 2.1^{aC}
GC	6.9 ± 3.2^{aB}	7.6 ± 5.0^{aAB}	13.0 ± 3.6^{aA}	11.1 ± 6.0^{aA}	10.1 ± 5.8^{aB}

Abbreviations: GC, G-CEM; MXC, Maxcem; RXU, RelyX Unicem; SMC2, SmartCem2.
** Small letter superscripts refer to within-group analysis; capital letter superscripts refer to within-time analysis. Identical superscripts identify results that are not significantly different.*

days, 30 days, and 60 days before SBS determination (n=10/cement /time period). The specimens were tested using a universal testing machine (LRX, JJ Lloyd Instruments, West Sussex, UK) at a crosshead speed of 0.5 mm/min. Fractured interfaces were viewed and photographed under an optical microscope (40×) to assess the locus and mode of failure.

Using Teflon molds, 50 bars (2×2×25 mm) were made from each SEARC and stored in water at 37 °C for 1 hour, 1 day, 7 days, 30 days, and 60 days (n=10/material/time) before testing in three-point bending mode for σ_f determination. The specimens were tested using a three-point bending device (with a 20 mm span) and the universal testing machine operated at a cross head speed of 0.1 mm/min. The load at fracture, F, was used to calculate σ_f using the formula:

$$\sigma_f = \frac{3FL}{2hc^2}$$

where L is the specimen span, h the specimen width, and c the specimen height.

The results of SBS and σ_f were statistically analyzed (PASW Statistics 18) using two-way ANOVA followed, if warranted, by Scheffé multiple means comparisons (α=0.05). Pearson's correlation coefficient was determined to assess correlation between SBS and σ_f.

RESULTS

The results of SBS and σ_f, along with the results of the statistical analysis, are summarized in Tables 2 and 3, respectively. The results showed that within each group SBS remained unaffected by water storage, with the exception of MXC, where a significant drop in SBS was detected after 1 day but no deterioration was detected thereafter. Comparing the SBS of the different groups at the same time interval showed that the RXU had significantly higher results, with the exception of the 7-day and 30-day results, which were not different from those of GC. The results obtained at 7 days, 30 days, and 60 days for MXC and SMC2 were significantly lower than the others but not different between themselves.

The characterization of the mode of failure of the tested adhesive interfaces indicated that all failures were adhesive at the dentin surface; therefore, no further analysis was warranted.

The results showed that within each material group σ_f increased up to 7 days and thereafter remained either relatively constant over the 60 days (RXU, MXC, GC) or decreased (SMC2) (see Table 3

Table 3: *Results of Flexural Strength (σ_f) Tests (Mean ± SD) and Statistical Analysis**

Group	σ_f (in MPa; n=10)				
	Time				
	1 h	1 d	7 d	30 d	60 d
RXU	67.6 ± 12.5^{cA}	75.9 ± 13.3^{bcB}	91.8 ± 10.4^{abB}	101.4 ± 6.6^{aA}	100.4 ± 14.3^{aBC}
MXC	72.9 ± 5.8^{cA}	97.32 ± 8.8^{bA}	114.2 ± 15.7^{abA}	105.9 ± 9.2^{abA}	116.0 ± 18.1^{aB}
SMC2	65.5 ± 6.4^{dA}	72.2 ± 12.4^{cdB}	92.0 ± 11.6^{abB}	101.4 ± 13.9^{aA}	84.5 ± 9.5^{bcC}
GC	43.3 ± 9.9^{cB}	109.7 ± 12.8^{bA}	126.4 ± 6.5^{abA}	118.1 ± 22.5^{bA}	141.7 ± 9.2^{aA}

Abbreviations: GC, G-CEM; MXC, Maxcem; RXU, RelyX Unicem; SMC2, SmartCem2.
** Small letter superscripts refer to within-group analysis; capital letter superscripts refer to within-time analysis. Identical superscripts identify results that are not significantly different.*

rows, small letter superscripts). Comparing the σ_f of the different groups at the same time interval (see Table 3 columns, capital letter superscripts) showed that even though there were some significant differences between the groups at 1 hour, 1 day, and 7 days, there were no differences between the materials investigated by 30 days. At 60 days, however, the results split again into three homogeneous groups with GC > RXU = MXC > SMC2. Overall, with regards to σ_f, after full maturation (7 days), only SMC2 was affected by long-term water storage (60 days).

Based on the results obtained, both null hypotheses were rejected.

DISCUSSION

The aim of this study was to assess the effect of water storage, between 1 hour and 60 days, on both the SBS afforded by the four SEARCs and their σ_f. The intent was to monitor the effect of water storage on a relevant mechanical property of the luting agent (σ_f) and investigate if it correlates with the adherence. Although significant differences were identified between the SBS obtained with the four SEARCs, the results of the study showed that water storage affected only one material (SMC2), and the effect was evident only between the results obtained at 1 hour and all the others. The conditions of this *in vitro* study are, however, different from those found *in vivo*, where adhesive interfaces are challenged not only by the presence of water at 37°C, but also by the presence of enzymes and exposure to cyclic loading.[30] Another possible explanation for the identified long-term stability of the SEARCs investigated is the absence of hydroxyl-ethyl methacrylate from their composition, a component that could render a material susceptible to water degradation.[31]

The acidity (pH) of the two SEARCs that showed better long-term SBS, that is, RXU and GC, have a lower pH (<2) than the other two materials, MXC and SMC2 (pH > 2.2). This difference in pH may have been responsible for an increase in the rugosity/surface area of dentin, leading to higher SBS. Moreover, the presence of urethanes and/or triethylene glycol dimethacrylate in MXC and SMC2 could render these materials more susceptible to water sorption and degradation.[30] The large variability of the results did not permit the identification of a significant effect of water storage; however, the 1-hour values for MXC and SMC2 were twice those recorded after 60 days.

The analysis of the fractured adhesive interfaces revealed that all of them failed adhesively at the dentin surface. Our results differ from those reported by Pisani-Proenca, [2] who observed a 30% mixed failure in the case of RXU and MXC. The difference between the results may be attributable to the different dentin surface preparations: we used 800 grit SiC while they used 600 grit SiC. A rougher surface may encourage a deeper penetration of the luting agent and provide a larger surface area for bonding, which, in turn, could lead to the mixed failures reported. The use of 800 grit SiC in this study parallels the study by De Munck and others,[22] who reported a majority of adhesive failures as well. They also showed that a pH <2 is not necessarily sufficient to adequately demineralize dentin and lead to the formation of a well-defined hybrid layer.[22] Moreover, the same authors speculated that the high viscosity of the materials does not allow for close wetting of and penetration into dentin. It has been shown that the application of force could counteract these problems.[33] Use of phosphoric acid[22,32] or polyacrylic acid[34] preconditioning before the application of SEARC has also been proposed, but this step seems to defeat the advantages afforded by the use of a SEARC. It has been reported that the use of polyacrylic acid doubled the performance of RXU, a fact explained by the possible interaction between the polyacrylic acid infiltrated into dentin and the fluoro-alumino silicate glass filler present in RXU.[34,35]

The results obtained for σ_f showed an increase from 1 hour to 7 days for all the materials, paralleling the completion of their setting reaction and equilibration with water. It is interesting to note that although there was no statistically significant difference between the four SEARCs at 39 days, at 60 days one SEARC (SMC2) exhibited a significant decrease in σ_f. Most studies report mechanical properties within 30 days but it may be advisable to monitor these properties for longer periods to better predict their long-term performance. The SEARCs tested contained groups capable of forming hydrogen bonds, which may be responsible for the relatively high σ_f determined.[36] The results of this study did not show any correlation between the results of SBS and those of σ_f, which is probably due to the fact that all the failures were adhesive at the dentin SEARC interface. It is possible that correlations would be found in systems where the mechanical properties of the adhesive are actually challenged by the presence of a strong adherence.

CONCLUSIONS

This study identified significant differences in dentin-Ti SBS afforded by the four SEARCs tested along with significant differences in their σ_f values. The adherence afforded by Rely X Unicem and G-Cem was significantly higher than that of Maxcem and SmartCem2. Under the experimental conditions of this study, 60 days of water storage significantly affected the σ_f of SmartCem2 but did not affect the SBS of SEARC mediated dentin-Ti adhesive interfaces (Maxcem showed a significant drop after 1 day but remained constant thereafter). The advantages offered by SEARCs are appealing and their clinical usage is increasing. However, the selection and use of SEARCs, as with that of any novel materials, should be approached with caution and grounded on evidence-based information.

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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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Acid Demineralization Susceptibility of Dental Enamel Submitted to Different Bleaching Techniques and Fluoridation Regimens

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Clinical Relevance

The in-office bleaching technique must be performed in conjunction with a daily fluoride regimen to minimize the damage produced by bleaching dental enamel.

SUMMARY

The aim of the current study was to assess the acid demineralization susceptibility of bleached dental enamel submitted to different fluoride regimens. One hundred bovine enamel blocks (6×6×3 mm) were randomly divided into 10 groups (n=10). Groups 1 and 2 received no bleaching. Groups 3 to 6 were submitted to an at-home bleaching technique using 6% hydrogen peroxide (HP; G3 and G4) or 10% carbamide peroxide (CP; G5 and G6). Groups 7 to 10 were submitted to an in-office bleaching technique using 35% HP (G7 and G8) or 35% CP (G9 and G10). During bleaching, a daily fluoridation regimen of 0.05% sodium fluoride (NaF) solution was performed on groups 3, 5, 7, and 9, while weekly fluoridation with a 2% NaF gel was performed on groups 4, 6, 8, and 10. The samples in groups 2 to 10 were pH cycled for 14 consecutive days. The samples from all groups were then assessed by cross-sectional Knoop microhardness at different depths from the outer enamel surface. The average Knoop hardness numbers (KHNs) were compared using one-way analysis of variance and Tukey tests (α=0.05). The comparison between groups 1 and 2 showed that the demineralization method was effective. The comparison among groups 2 to 6 showed the same susceptibility to acid demineralization, regardless of the fluoridation method used. However, the samples from groups 8 and 10 showed more susceptibility to acid demineralization when compared with group 2 ($p<0.05$). Groups 7 and 9 provided

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similar results to group 2, but the results of those groups were different when compared with groups 8 and 10. The use of 6% HP and 10% CP associated with daily or weekly fluoridation regimens did not increase the susceptibility of enamel to acid demineralization. However, the use of 35% HP and 35% CP must be associated with a daily fluoridation regimen, otherwise the in-office bleaching makes the bleached enamel more susceptible to acid demineralization.

INTRODUCTION

Tooth discoloration is one of the main reasons leading patients to seek dental treatment. Therefore, tooth bleaching has become very popular, and sometimes it is done indiscriminately. One of the factors of success for bleaching treatment depends on the dentist, who must know the properties of these materials, the techniques of application, and the possible side effects.

The effectiveness of tooth bleaching when using carbamide peroxide (CP) or hydrogen peroxide (HP) on discolored vital teeth has been previously reported.[1] The application of CP and HP gels at a concentration of 10%-15% and 6%-10%, respectively, for four to eight hours per day and over a period of three to six weeks are still the most popular at-home bleaching techniques and are suggested as efficient and simple procedures for tooth whitening.[2,3] In-office bleaching was introduced to shorten the treatment time. In this technique, the teeth can be bleached in two or three sessions, as the in-office bleaching uses the same bleaching agents as the at-home treatment, but in higher concentrations.[3]

Both the at-home and in-office techniques of tooth bleaching can lead to tooth sensitivity, which may occur during or after treatment.[4] Several approaches to reduce tooth sensitivity have been used, such as a reduction in application time and frequency of application, temporary interruption of whitening, and the use of an active ingredient such as potassium nitrate.[5] The use of fluoride compounds, such as gels, solutions, or dentifrices, has also been used to prevent tooth sensitivity, to enhance the remineralization of the bleached enamel, and to increase the abrasion resistance of the softened enamel.[6] It is known that fluoride uptake is higher in demineralized enamel when compared to sound tissue.[7] Furthermore, bleaching might render enamel porous, which might allow better diffusion and penetration of the fluoride.[8] Nevertheless, the most effective fluoridation regimen and the best fluoride compound for preventing enamel demineralization during bleaching have not been indicated in previous research.

Previous studies[9,10] have shown that bleaching agents can promote alterations in the enamel surface. Scanning electronic microscopic analysis has shown that morphological alterations of the superficial enamel surface, such as erosion, decalcification, and porosities, might occur following exposure to CP or HP at different concentrations.[11] Additionally, the application of bleaching gels can also affect the calcium and phosphate content and, consequently, may decrease the surface and subsurface microhardness of the enamel.[10]

Despite the protective and remineralizing potential of human saliva, mineral loss is sometimes evident under *in situ* conditions.[12] Saliva increases the microhardness of bleached enamel by providing phosphate and calcium ions, but sometimes remineralization is not complete.[13] As a result, fluoride might contribute to the repair of the microstructural defects of bleached enamel.

It has been observed that bleaching gel might alter the structure of dental enamel, but the literature is unclear about the correct fluoridation method to use to prevent excessive demineralization of dental enamel. The purpose of this present study was to assess the acid demineralization susceptibility of bleached dental enamel submitted to different fluoridation regimens. The null hypothesis was that the susceptibility of dental enamel to acid demineralization is not altered when exposed to the at-home and the in-office bleaching techniques in conjunction with different fluoridation regimens.

METHODS AND MATERIALS

Preparation of Specimens

One hundred freshly extracted bovine incisors were stored in a 0.1% thymol solution (pH 7.0) for at least one month, and the maximum storage time was two months. The crowns were separated from the roots using a water-cooled diamond disc (Isomet; 10.2 cm×0.3 mm, arbour size one-half inch, series 15HC diamond; Buehler Ltd, Lake Bluff, IL, USA) mounted in a sectioning machine (Minitom, Struers Inc, Westlake, OH, USA). The crowns were sectioned to obtain 100 enamel blocks (6×6×3 mm). The labial surfaces were ground flat and polished with water-cooled sandpapers (#600, 800, 1200, and 2400 grit, Saint-Gobain Abrasivos Ltda, Sao Paulo, SP, Brazil) to standardize the substrate. Prior to the experiment the specimens were stored in distilled water.

Table 1: *Description of the Experimental Groups According to the Bleaching Technique and the Fluoridation Regimen*

	Bleaching Technique	Bleaching Agent	Fluoride Concentration	Fluoridation Regimen	pH Cycling
1	None	None	None	None	No
2	None	None	None	None	Yes
3	At home	Hydrogen peroxide 6%	NaF 0.05%	Daily	Yes
4	At home	Hydrogen peroxide 6%	NaF 2%	Once a week	Yes
5	At home	Carbamide peroxide 10%	NaF 0.05%	Daily	Yes
6	At home	Carbamide peroxide 10%	NaF 2%	Once a week	Yes
7	In office	Hydrogen peroxide 35%	NaF 0.05%	Daily	Yes
8	In office	Hydrogen peroxide 35%	NaF 2%	Once a week	Yes
9	In office	Carbamide peroxide 35%	NaF 0.05%	Daily	Yes
10	In office	Carbamide peroxide 35%	NaF 2%	Once a week	Yes

Abbreviation: NaF, sodium fluoride.

Bleaching and Fluoridation Regimen

The enamel samples were randomly divided into 10 experimental groups (n=10). In each sample, a 16-mm^2 area was delimited on the buccal surface. Two layers of varnish sealer (Colorama Maybelline Ltda, São Paulo, Brazil) were applied around this demarcated area.

The samples from group 1 were kept in artificial saliva (20 mM $NaHCO^3$, 3 mM $NaH_2PO^4 \bullet H_2O$, and 1 mM $CaCl_2 \bullet 2H_2O$; pH=7.0),[14] and no experimental procedures were performed on those samples. The samples from group 2 were submitted to pH cycling (see below). The treatments performed on the other samples are described in Table 1. The at-home and in-office bleaching techniques were simulated in each respective group.

The at-home bleaching technique was performed by applying 6% HP gel (groups 3 and 4; Mix Day, Dentalville do Brasil Ltda, Joinville-SC, Brazil; pH=7.0) or 10% CP gel (groups 5 and 6; Whiteness Perfect 10%, FGM Produtos Odontológicos, Joinville, SC, Brazil; pH=6.5) on the exposed enamel surface, according to the manufacturers' instructions, for six hours per day in a humid atmosphere at 37°C. This process was performed daily over a period of 21 days.

After removal of the at-home bleaching gel, the samples from groups 3 and 5 were washed in distilled water and immersed in a colorless, neutral 0.05% sodium fluoride (NaF) solution (Fluor Sol Clear, Dentsply Indústria e Comércio Ltda, Petrópolis, RJ, Brazil) for five minutes. The samples were kept in artificial saliva at 37°C when not in use.

The samples from groups 4 and 6 were covered once a week with a 2% NaF (pH=6.5) gel (Nuprogel, Dentsply) for a period of four minutes. The excess gel was removed and the samples were kept in artificial saliva at 37°C when not in use.

The in-office bleaching technique was performed by applying 35% HP gel (groups 7 and 8; Mix One Supreme, Dentalville; pH=6.4) or 37% CP gel (groups 9 and 10; Whiteness Super, FGM; pH=6.5) on the exposed enamel surface, according to the manufacturers' instructions. Three in-office bleaching sessions were performed on each group on days 0, 7, and 14. At each session, the bleaching gel was applied on the enamel surface for a period of 20 minutes and then rinsed. The gel was applied on the enamel surface two more times.

The fluoridation regimen started at the end of each session. The samples from groups 7 and 9 had the same fluoridation regimen as described for groups 3 and 5. The fluoridation regimen for groups 4, 6, 8, and 10 was the same. After the third bleaching session (day 14), the specimens were stored in artificial saliva for an additional seven days.

The pH of the bleaching agents was verified using a pH meter (Digimed DM-20-Digicrom Analítica Ltda, São Paulo, Brazil) fitted with an electrode (DME-Digimed CV8).

pH Cycling

To perform the acid challenge, samples were submitted to a pH-cycling procedure, modified from a previously described protocol.[15] Groups 2 to 9 were submitted to the acid challenge. The samples were first coated with an impermeable coating, except for the bleached enamel surface. The demineralization solution (pH=5.0) consisted of 2.0 mmol/L calcium (Ca) and 2.0 mmol/L phosphate in a buffering solution of 0.075 mol/L acetate. The remineralization solution (pH=7.0) consisted of 1.5 mmol/L Ca, 0.9 mmol/L phosphate, and 150 mmol/L of potassium chloride. Each specimen was cycled in 5.0 mL of both solutions for six hours in the demineralizing solution

and for 18 hours in the remineralizing solution. This procedure was carried out for 14 days at 37°C. At the end of each five consecutive days of cycling, the samples were immersed in remineralizing solution for two days.

Microhardness Test

At the end of the pH cycling, the samples from all 10 groups were individually embedded in self-curing acrylic resin, with the bleached surface exposed. The samples were longitudinally sectioned to provide the ability to perform the cross-section microhardness analysis. The exposed surfaces were polished with 600- and 1200-grit silicon carbide paper (Saint-Gobain Abrasivos Ltda) in a polishing machine (MetaServ 250, Buehler Ltd) under water cooling followed by 0.3-μm alumina paste applied over a felt disc using the same polishing machine. Next, the specimens were ultrasonically cleaned. Each sample was assessed using a microhardness examination of the enamel, starting at 20 μm from the outer enamel surface, with indentations at 20-μm intervals between 20 μm and 120 μm, and a last indentation was performed at 200 μm from the outer surface. Three Knoop microhardness measurements were performed at each location, with a distance between measurements of 500 μm, to prevent marks from overlapping each other. A static load of 25*g*/10 seconds was applied.

Statistical Analysis

The data were submitted to the D'Agostino normality test. One-way analysis of variance (ANOVA) and the Tukey test, when necessary, were used to analyze the differences in Knoop hardness numbers (KHNs) of all groups. The level of statistical significance was set at 0.05. All analyses were performed using BioEstat 5.3 (Instituto Mamirauá, Tefe, AM, Brazil).

RESULTS

The mean KHN found in groups 1 (no treatment) and 2 (unbleached and pH cycled) are presented in Figure 1. The KHN values were compared at each depth. The graph indicates that demineralization occurred at depths of up to 120 μm, with a greater decrease in hardness for the superficial layers of the enamel. No differences were observed between these two groups at 200 μm.

The results obtained for the at-home bleaching groups were compared to those obtained in group 2 and are presented in Table 2. The KHNs were compared at each depth. The ANOVA showed that no statistically significant differences were found ($p>0.05$).

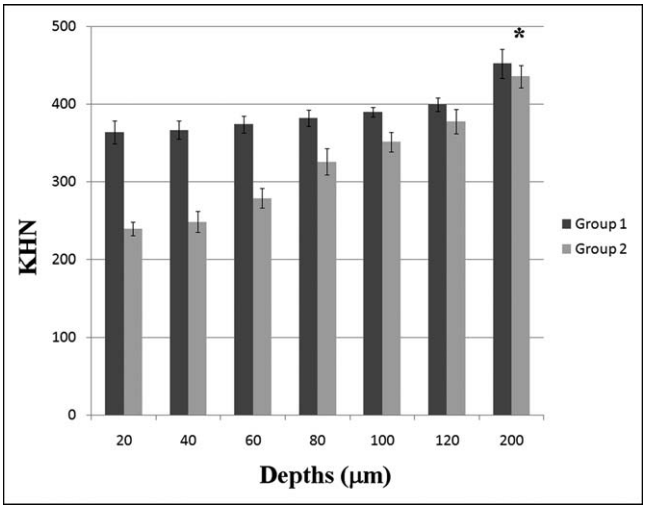

Figure 1. *Comparison among the mean KHNs obtained at each depth for groups 1 and 2. The * symbol indicates no statistical differences (*p*>0.05).*

Table 3 shows the KHNs found for the in-office bleaching groups, as well as the data obtained for group 2 ($p=0.0001$). Groups 2, 7 (35% HP and daily fluoridation with 0.05% NaF), and 9 (35% CP and daily fluoridation with 0.05% NaF) showed similar KHNs at a depth of 20 μm; however, these groups were statistically different from groups 8 (35% HP and weekly fluoridation with 2% NaF) and 10 (35% CP and weekly fluoridation with 2% NaF). The same situation occurred at depths of 40, 60, and 80 μm. The experimental groups showed no statistically significant differences at the deeper layers (100, 120, and 200 μm).

DISCUSSION

The null hypothesis of the present investigation was partially rejected, since the groups treated with 35% CP and 35% HP and that received weekly fluoridation were more susceptible to acid demineralization.

The currently available bleaching agents are primarily composed of CP or HP.[3] HP has low molecular mass, which facilitates its rapid diffusion into enamel prisms and interprismatic spaces.[16] The bleaching agent is capable of being retained in the enamel and exerting a prolonged effect in structures that do not necessarily need to be bleached. This also applies to CP, which dissociates into urea and HP when it comes in contact with dental structures. Thus, it is possible to believe that bleaching causes alterations in dental hard tissues, including erosion,

Table 2: *Mean Knoop Hardness Numbers (KHNs; ±Standard Deviation) Found at Different Depths for Unbleached and At-home–Bleached Samples. No Statistically Significant Differences Were Found (p>0.05)*

Depth, μm	Group 2	Group 3	Group 4	Group 5	Group 6
20	239 (8.8)	249 (13.6)	266 (18.6)	263 (15.4)	259 (21.0)
40	248 (13.7)	256 (9.4)	275 (23.5)	270 (19.9)	269 (22.2)
60	279 (12.3)	282 (13.0)	291 (17.3)	295 (13.9)	286 (20.6)
80	326 (17.3)	323 (22.8)	336 (29.0)	325 (27.4)	321 (21.2)
100	351 (12.9)	352 (25.0)	371 (26.6)	361 (23.2)	353 (26.9)
120	377 (15.4)	380 (19.1)	388 (17.2)	385 (24.9)	386 (21.5)
200	435 (14.4)	432 (21.8)	445 (15.6)	450 (21.0)	439 (24.4)

porosity, and an increase in enamel surface roughness.[11] Although these alterations are not clinically or macroscopically visible, previous studies[10,17,18] have found microstructural changes of enamel induced by bleaching agents, particularly when peroxides are used in high concentrations. In fact, the present study showed that enamel treated with high-concentration bleaching agents was more susceptible to acid demineralization, even when a 2% NaF weekly fluoridation regimen was used.

Some bleaching agents can lead to erosive effects on enamel, so that tooth brushing during bleaching can increase the roughness of the enamel surface.[19] The chemical analysis of enamel after the application of CP and HP in concentrations between 10% and 30% revealed a reduction in Ca levels as well as in the average Ca:phosphorus value of bleached dental enamel.[10,20] The reduction in Ca is attributed to the dissolution of this element by bleaching agents. In addition to reducing the surface hardness, the loss of minerals increases the enamel permeability to acids produced by cariogenic bacteria, leading to the formation of deeper carious lesions.[21]

Similarly, these changes allow the penetration of bleaching gel into the deeper layers of the dental hard tissue, which may promote effects in dentin and pulp.[22] Clinically, the presence of these changes may be suggested by the occurrence of tooth sensitivity during bleaching. Therefore, the use of fluoride compounds throughout bleaching treatment has been proposed to avoid the occurrence of side effects.

The acid challenge used in the current study was based on previous studies[15] and was found to be effective in demineralizing deeper enamel layers. According to Figure 1, layers up to 120 μm distant from the enamel outer surface were affected by the pH cycling. The bleaching treatments and fluoridation regimens used in the present study, as described before, are usually applied in dental offices. The data obtained in the present study suggest that in-office bleaching reduces the mineral content of the dental enamel to an extent that a weekly fluoride application could not restore the initial enamel mineralization. According to the current results obtained for the in-office bleaching groups that were submitted to a daily fluoridation regimen, fluoridation must be present throughout bleaching treatment, at low and constant concentrations, to reduce mineral loss and stimulate remineralization and is not indicated only after each bleaching session.

The use of fluoride compounds is effective in increasing the hardness of enamel samples and preventing mineral loss during at-home bleaching.[23] Fluoride incorporation into the demineralized tooth surface creates a calcium fluoride layer that increases the natural enamel hardness.[24] As described

Table 3: *Mean Knoop Hardness Numbers (KHNs; ±Standard Deviation) Found at Different Depths for Unbleached and In-office–Bleached Samples. Similar Capital Letters in a Row Indicate No Statistically Significant Differences (p=<0.0001)*

Depth, μm	Group 2	Group 7	Group 8	Group 9	Group 10
20	239 (8.8) A	260 (17.2) A	203 (11.3) B	259 (24.0) A	208 (6.3) B
40	248 (13.7) C	268 (24.6) C	217 (8.23) D	265 (22.9) C	219 (10.3) D
60	279 (12.3) E	289 (17.5) E	249 (7.16) F	288 (19.3) E	246 (12.2) F
80	326 (17.3) G	323 (26.57) G	292 (10.19) H	326 (29.6) G	287 (16.4) H
100	351 (12.9) I	357 (28.6) I	342 (7.16) I	368 (22.9) I	335 (37.4) I
120	377 (15.4) J	387 (14.49) J	367 (7.94) J	399 (21.1) J	379 (26.4) J
200	435 (14.4) K	438 (21.13) K	431 (15.8) K	445 (15.3) K	432 (13.14) K

before, bleaching makes the enamel surface porous and rough; it is commonly accepted that fluoride uptake into demineralized enamel is greater when compared to that into sound enamel, since the porous and permeable structure of the demineralized tissue allows deeper diffusion and penetration of the fluoride, and the porosity increases the number of retention sites for the fluoride.[25] Small amounts of fluoride around the tooth effectively inhibit demineralization more than incorporated fluoride and provide a much greater caries-protective potential than does a large proportion of fluoride in the enamel.[25]

In the present study, the excess fluoride gel was rinsed after four minutes of being in contact with the hard dental tissue and the sample was immersed in artificial saliva. Thus, the only contact between the samples and the 2% NaF was on the day of bleaching. It is accepted that topical fluoride promotes remineralization and inhibits demineralization of dental hard tissues.[25] In fact, a weekly application of fluoride was found to be sufficient for the samples submitted to the at-home bleaching treatment, most likely because the bleaching agents used are less concentrated and less aggressive on tooth enamel. Nevertheless, this same fluoridation regimen was not effective for samples bleached with high-concentration bleaching agents, which promote more severe changes in dental tissue.[26] In addition, the results obtained in the current study suggest that fluoridation does not leave the bleached enamel more acid resistant when compared to sound enamel, and, therefore, fluoridation does not reinforce the tooth structure during treatment; however, fluoridation most likely helps to return the level of mineralization of bleached enamel to its initial state.

Some bleaching gels incorporate Ca^{2+} and F^{-} ions in their formulations.[27] These minerals were included because they were thought to be a possible alternative to overcome the adverse effects of bleaching gels on surface enamel, since the ions could diffuse into the enamel structure along with the CP and HP. The deposition of those ions in the enamel may act as a physical barrier, minimizing the contact of the acid to enamel, or providing additional mineral to be dissolved during the acid challenge before the underlying enamel is attacked. This effect is not completely confirmed, but recent research[27] has shown promising results for those bleaching agents.

According to some studies,[28,29] the potential for demineralization depends on the pH of the bleaching agents. In the present study, the pH levels of the bleaching gels were found to be between 6.4 and 7.0. As these bleaching agents are near the neutral value, their pH was thought to have no influence in the present results. Nevertheless, as previous studies have determined that bleaching agents are also able to demineralize tooth enamel, it is possible that the enamel demineralization represents a combination of the action of the bleaching agent concentration and the low pH of the gel.[26,29]

However, there are past studies[30,31] that have shown that bleaching agents do not change the enamel surface and, consequently, do not alter the susceptibility of enamel to acid demineralization. The divergence among the various studies is natural, because there are several variables involved in each study, such as the pH of bleaching gels, the HP gel concentration, the contact period between the tooth and the bleaching gel, and the treatment time, among other factors. Nevertheless, common sense dictates that additional fluoridation is essential during bleaching treatment.

The present study used bovine incisors. While there are some differences between bovine and human enamel (for example, the latter has a keyhole arrangement of the prisms, while interrow sheets or lamellar sheets tend to occur in the ungulates), they are easily obtained; their composition is more homogeneous than that of human teeth, resulting in a more standardized experimental setup; their weight percentage Ca content is equivalent to that of human enamel and shows a similar, gradual decrease from the surface to the dentin-enamel junction; and their matrix proteins are composed of amino acids that resemble those of human enamel.[32]

It should be noted that the present study was performed *in vitro*. It is not known whether these findings would be the same if the study was conducted *in vivo* or *in situ,* using exactly the same products and the same methodology. However, it is known that saliva plays an important role in enamel remineralization.[33] Additionally, tooth brushing with fluoride dentifrices is essential in increasing the acid resistance of enamel and minimizing the effects of bleaching gels.[25] These factors could affect the results found for groups 8 and 10, but clinical studies are needed to confirm or reject the findings obtained here.

CONCLUSIONS

Within the limitations of this *in vitro* study, it may be concluded that the use of low-concentration bleaching agents (6% HP and 10% CP) with the at-

home bleaching technique, associated with a daily or weekly fluoridation regimen, does not increase the susceptibility of enamel to acid demineralization. However, the use of high-concentration bleaching agents (35% HP and 35% CP) may increase the susceptibility of dental enamel to demineralization. To minimize this side effect, it is necessary to use a daily fluoridation regimen, since weekly fluoridation is not capable of inhibiting that adverse effect.

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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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