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Clinical Technique/Case Report

Amelogenesis Imperfecta: Case Study

C Leevailoj • S Lawanrattanakul • K Mahatumarat

Clinical Relevance

This article describes the diagnosis and treatment of a patient with amelogenesis imperfecta from mixed dentition to permanent dentition. This study aims to provide a treatment plan that can be used as a source for other amelogenesis imperfecta cases.

SUMMARY

Amelogenesis imperfecta (AI) refers to a group of rare genetic disorders that involve tooth development and that are passed down through families as a dominant trait. This condition is characterized by abnormal enamel formation caused by gene mutations that alter the quality and/or quantity of enamel. This dental problem can impact both primary and permanent dentition, varies among affected individuals, and results in esthetic and functional problems. This condition caused the patient in the current case report to have a lack of confidence when speaking.

The treatment for amelogenesis imperfecta depends on the severity of the problem and age of the patient. It is crucial to plan a proper remedy, which requires collaboration among dental specialties to execute comprehensive dental treatment in order to provide a long-term solution with adequate esthetics.

The current clinical study presents a patient affected by AI that was diagnosed when the patient was a child. The interdisciplinary treatment continued throughout his childhood and into adult life. The initial treatment consisted of resin composite veneers and stainless-steel crowns to restore the defective tooth structure. The malocclusion of the patient was corrected using a fixed orthodontic appliance that was placed when he had an entire permanent dentition. The treatment plan was eventually intended to include all ceramic crowns and veneers.

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INTRODUCTION

Amelogenesis imperfecta (AI) is defined as a group of hereditary developmental defects of the dental enamel affecting both primary and permanent dentition.[1] AI may be inherited in an X- linked manner or by autosomal dominant, autosomal recessive, or sporadic inheritance patterns.[2,3] The most common type of AI occurs as a result of the autosomal dominant form of transmittance.[4] This anomaly exists independent of any related systemic diseases.[4-6]

There are various classification systems proposed for the different types of AI. Generally, AI is classified into three types—hypoplastic, hypominer-

alization, and hypomaturation[4,7-10]—on the basis of clinical and radiographic findings. The hypoplastic type is defined as a deficiency in the deposition of organic matrix. Therefore, the enamel is reduced in quantity but is mineralized properly, has normal radiopacity, and can be observed as thin, malformed, lacking contact but appearing hard and shiny. The hypomineralization type of AI presents with a deficiency in the mineralization of the formed matrix. The enamel has normal thickness but has a low degree of mineralization. Thus, there is no contrast in the radiopacity between the enamel and the dentin. The enamel is soft, friable, and easy to remove from the dentin. In hypomaturation, there are abnormalities in the final stage of the mineralization process because the enamel crystals remain immature and there is no contrast of radiopacity between the enamel and dentin. The hypomaturation type differs from the hypocalcification type in that the enamel is harder and presents with a mottled opaque white to yellow-brown or red- brown color. However, the enamel is softer than normal and tends to chip from the underlying dentin.

The estimated prevalence of AI is reported to vary between approximately 1:16,000 and 1:700, depending on the population studied and the diagnostic criteria used.[11] Extrinsic disorders, chronological disorders, and localized disorders of tooth formation and metabolic disturbance affecting enamel formation should be considered in the differential diagnosis for AI.[3]

The primary clinical problems of AI include poor dental esthetics, dental sensitivity, and loss of vertical dimension.[12] AI also has been associated with impacted teeth and anomalies in tooth eruption, congenitally missing teeth, pulp calcification, root and coronal resorption, hypercementosis, root malformation, and taurodontism.[13-17] Moreover, the AI patient has an increased probability for having dental caries and plaque accumulation. The common malocclusion frequently associated with AI is anterior open bite. It has been suggested that craniofacial skeletal and enamel formation may share a common ectomesenchymal origin. The incidence of anterior open bite in AI patients varies from 24% to 60%.[13] Thus, restorations are important not only because of esthetic and functional concerns but also because there may be a positive psychological impact for the patient.

Preventive treatment for an AI patient is impossible because AI is genetic. However, there are many options proposed for the treatment of AI-affected teeth, including simple microabrasion,[18] composite veneers,[19] porcelain laminate veneers, onlays,[10,20-22] gold or stainless-steel crowns,[20,23-25] metal ceramic crowns,[10,20,21,23,26] and all-ceramic crowns.[10,20] The treatment planning for patients with AI is related to many factors, including the age of the patient, the type and severity of the disorder, intraoral conditions, and the socioeconomic status of the patient.[21] This clinical report describes the sequence of treatment by interdisciplinary approach to manage a case of AI from childhood to adulthood.

Case Report

A healthy Thai boy, 10 years of age, presented to the Faculty of Dentistry, Chulalongkorn University, with his parents. His major concerns were the appearance of his teeth and sensitivity to cold. His parents mentioned that his mother and grandfather also suffered from the same problem. A specialist in the Department of Oral Medicine examined the patient's disorder with clinical and radiographic examinations and diagnosed the patient with AI (hypoplastic type).

A comprehensive treatment plan was formed at the outset and was divided into three sessions. During preadolescence, restorations were performed to restore the defective tooth structure. Oral hygiene was also reinforced to reduce the risk of caries. In the early permanent dentition, the subject's malocclusion was treated orthodontically using fixed appliances. As the patient's growth ceased, permanent restorations were performed to improve both esthetics and function. The following provides the details of the comprehensive treatment plan.

At the first session, the patient was referred to the pediatric and operative departments for initial restorations to protect the remaining tooth structure against further wear and sensitivity by using resin composite veneers for the anterior and premolar teeth and stainless-steel crowns for the second deciduous molars and first permanent molars. Furthermore, occlusal sealants were performed on the premolar and molar teeth. Oral hygiene instructions were provided. The patient presented to the clinic every six months for an oral hygiene recall and application of 1.23% acidulated phosphate fluoride.

After getting an entire permanent dentition, the subject was referred to an orthodontist at the age of 14 to treat his underlying malocclusion. An intraoral examination revealed an anterior open bite. A posterior open bite and posterior cross bite were found at the right premolar and molar region, respectively. The upper dental midline was centered,

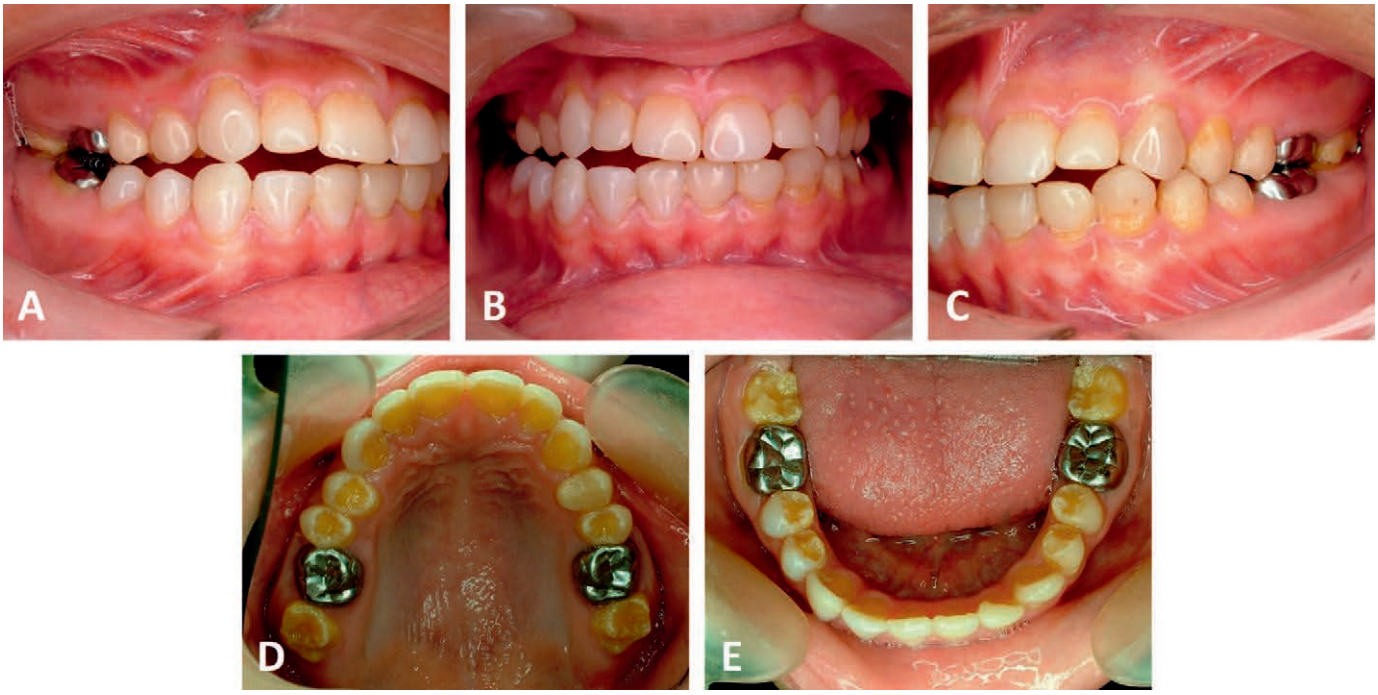

Figure 1. *Intraoral views before orthodontic treatment. (A) Right-side view. (B) Frontal view. (C) Left-side view. (D) Occlusal view of the maxilla. (E) Occlusal view of the mandible.*

but the lower dental midline was shifted to the right by approximately 2 mm. The molar relationship was class I. The canine relationship was class II on the right side and class I on the left side (Figure 1). Space analysis indicated a 2-mm space deficiency in the upper arch. The extraoral examination revealed a convex profile with lower lip retrusion (Figure 2). Cephalometric analysis indicated a class I skeletal relationship.

The treatment objectives were to obtain normal occlusion, normal tooth intercuspation, and normal overbite and overjet and to improve the soft tissue profile. The patient was treated using fixed orthodontic appliances. The posterior cross bite was corrected with maxillary expansion using a Quadhelix appliance (Figure 3), combined with buccal crown torque of the maxillary posterior teeth and lingual crown torque of the mandibular posterior teeth. The open bite was corrected using intermaxillary elastics (Figure 4). The total orthodontic treatment time was six years and eight months.

After completion of orthodontic treatment, the patient was recommended to wear retainers for six months to maintain the occlusion. Subsequently, the patient's overall dental condition and expectations were evaluated. At this point in the treatment, the patient wished to have good dental esthetics and function. At 22 years of age, a clinical and radiographic reevaluation revealed that the interpupillary line and commissural line were parallel. The facial midline was centered. The patient's profile was slightly convex (Figure 5). The lower dental midline was shifted to the right by 2 mm when compared to the upper dental midline. Both overjet and overbite were 1 mm. The canine relationship was class II on the right and class I on the left. The occlusal scheme presented as canine protected occlusion. A clinical exam revealed that some composite veneers were defective. The lower-left second molar presented with an MO amalgam filling. In general, the enamel was hypoplastic and partially broken down. Exposed dentin could be observed (Figure 6). The overall root length and shape for the dentition was normal. The pulp chambers of all teeth were normal (Figure 7). The subject presented plaque- induced gingivitis. No active carious lesions were found.

The proportions of the upper anterior teeth were not symmetrical (Figure 8A). Therefore, the proper width of the teeth was calculated by using the repeated ratio or continuous proportion—the repeated proportion of the central-incisor-to-lateral-incisor width and lateral- incisor-to-canine width (as seen in a frontal view). The normal value is about 0.66 to

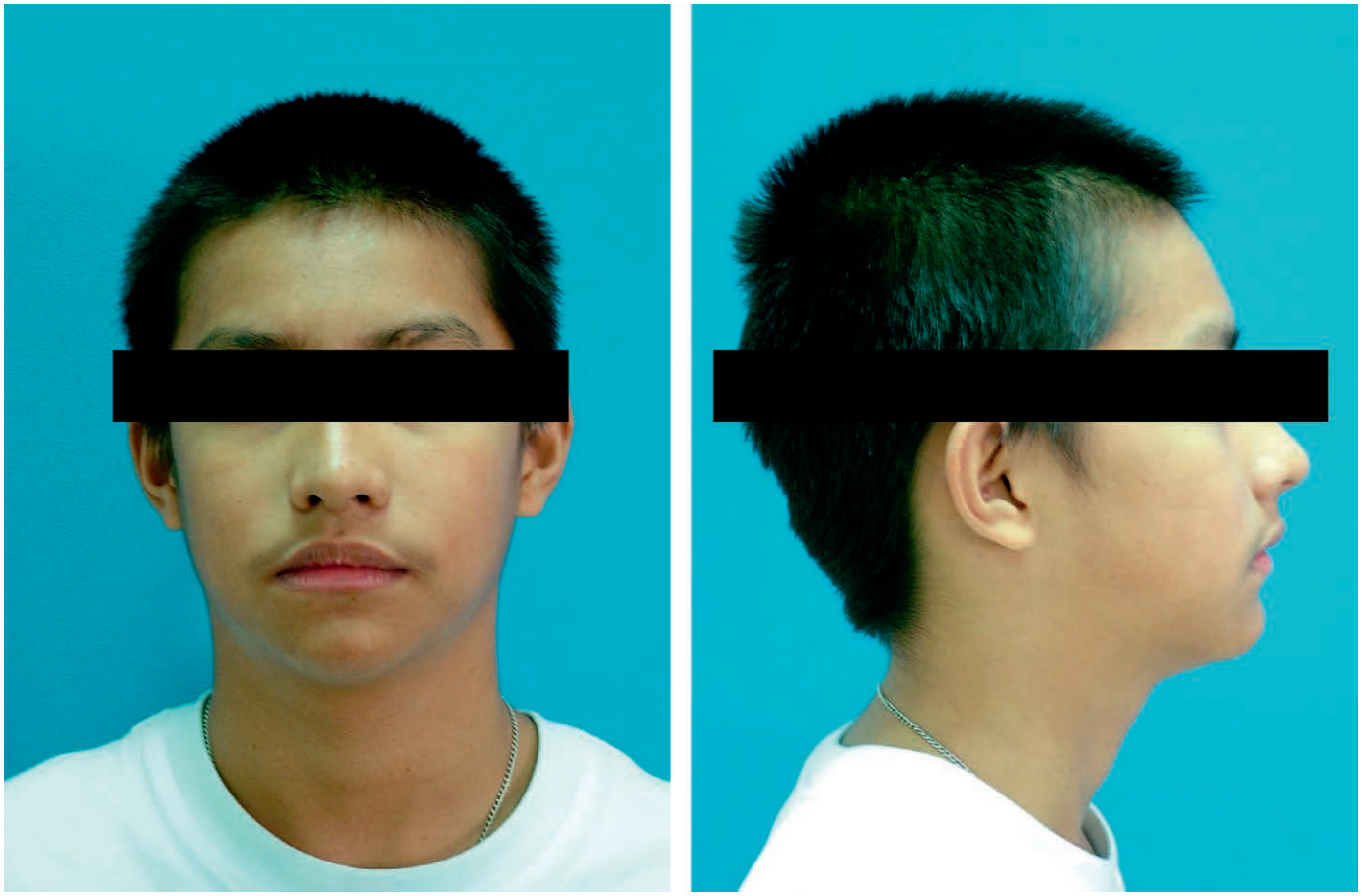

Figure 2. *Extraoral views before orthodontic treatment. (A) Frontal view. (B) Lateral view.*

0.78 (Figure 8B).[27,28] Because of asymmetry of the gingival level, crown lengthening was proposed. However, the patient denied any periodontal surgery.

Due to the patient's concern, full-mouth rehabilitation was planned. The treatment was initiated on the posterior teeth by performing all ceramic crowns on the first permanent molars to provide good

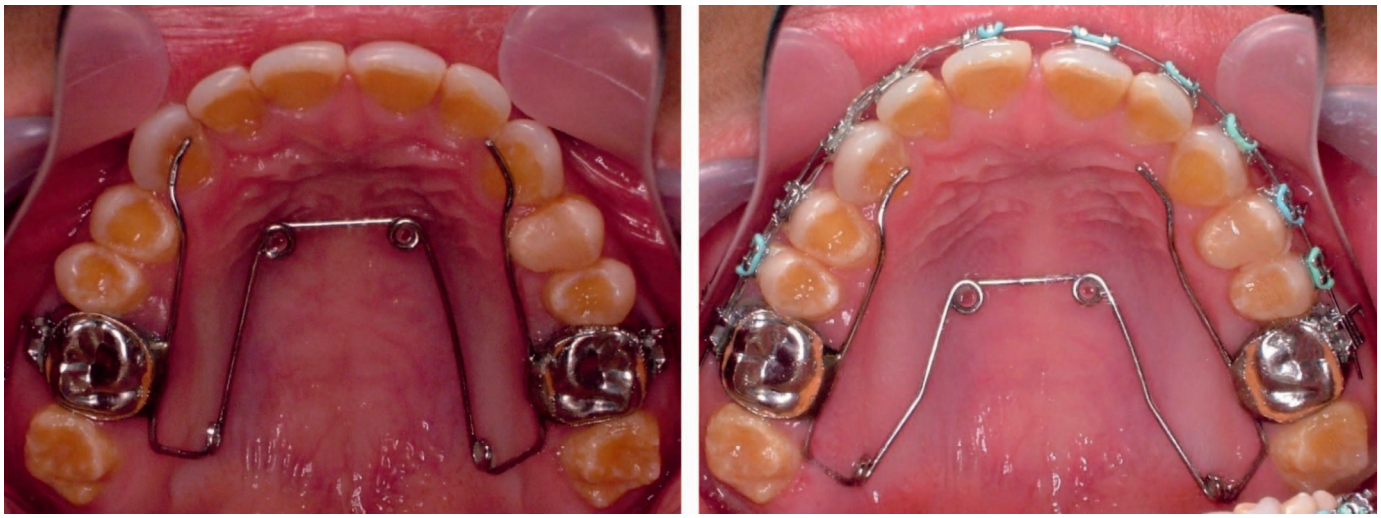

Figure 3. *Quadhelix appliance. (A) 2 months after insertion. (B) 9 months after insertion.*

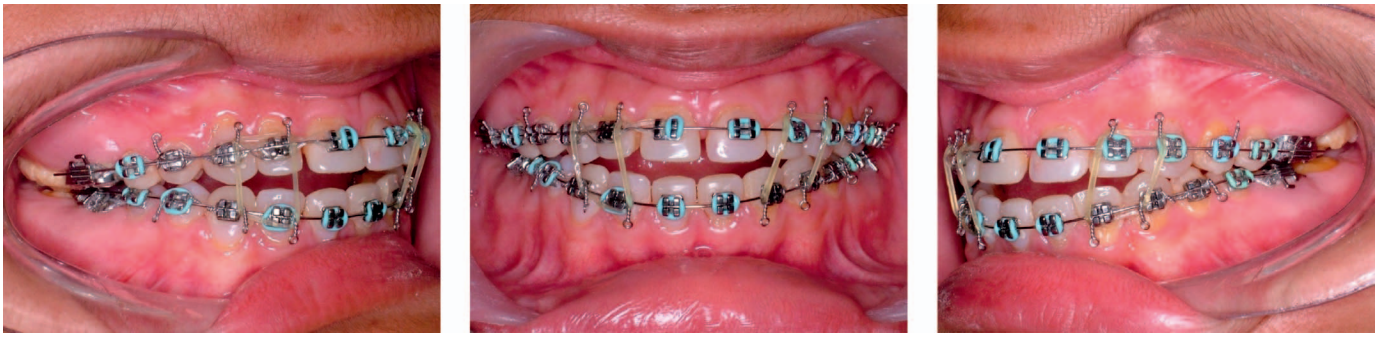

Figure 4. *Intermaxillary elastics. (A) Right-side view. (B) Frontal view. (C) Left-side view.*

posterior support before restoration of the other teeth. Stainless-steel crowns were removed from teeth #3, #14, #19, and #30, and all ceramic crown preparations were performed. For the upper first molars, a 3/4 zirconia core design (Lava) was chosen. For the lower first molars, an all-zirconia crown (Lava) was selected. Shade selection of all crowns was B2 Vita Classic. All crowns were temporarily fixed with Temp bond NE until the patient felt comfortable and reported no occlusal problems. A self-adhesive resin cement (Rely X Unicem, 3M ESPE, St Paul, MN, USA) was used for permanent fixation.

The anterior and premolar area was restored with ceramic and resin composite veneers. The teeth (#5-#12 and #22-#27) were prepared for ceramic veneers using a chamfer margin and incisal bevel preparation (Figure 9). According to minimally invasive treatment guidelines, tooth preparation was performed using a labial silicone index made from a diagnostic wax-up as a guide. The final impression

Figure 5. *Extraoral views before final restorations. (A) Frontal view. (B) Lateral view.*

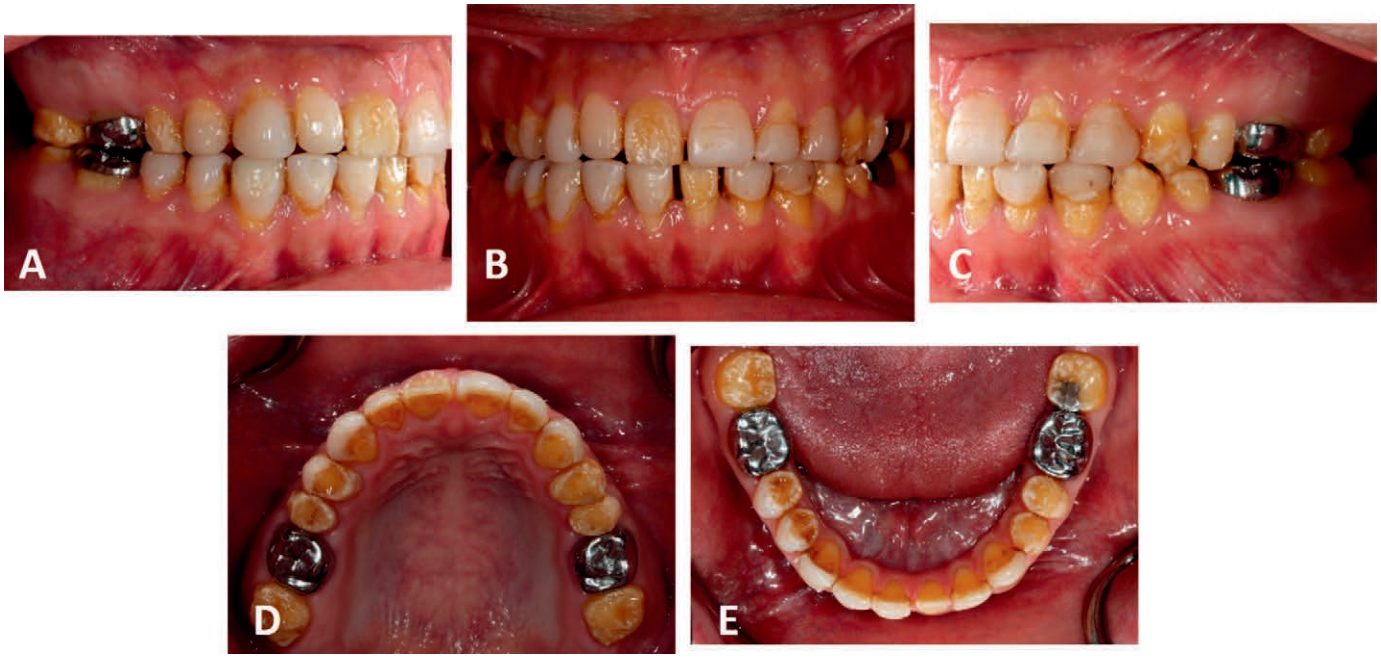

Figure 6. *Intraoral views before oral rehabilitation. (A) Right-side view. (B) Frontal view. (C) Left-side view. (D) Occlusal view of the maxilla. (E) Occlusal view of the mandible.*

was taken using polyvinyl siloxane (Flexitime, Heraeus Kulzer, South Bend, IN, USA). Bite registration and facebow transfer were performed. A palatal silicone index was used to fabricate temporary veneers using resin composite (Premise, Kerr Corp, Orange, CA, USA). Final working casts were mounted on a semiadjustable articulator (Artex CR, AmannGirrbach, Voraralberg, Austria) with a customized anterior guide table. Ceramic veneers were made from pressed ceramic (IPS Empress Esthetic, Ivoclar/ Vivadent, Liechtenstein) with a layering of veneering ceramic to mimic the natural enamel (Figure 10).

Shade selection was B1 Vita Classic. After the ceramic veneers were tried in, the appearance of #9-#11 was more yellowish than the other teeth. This appearance was caused by the underlying dentin color of those particular teeth. Therefore, white

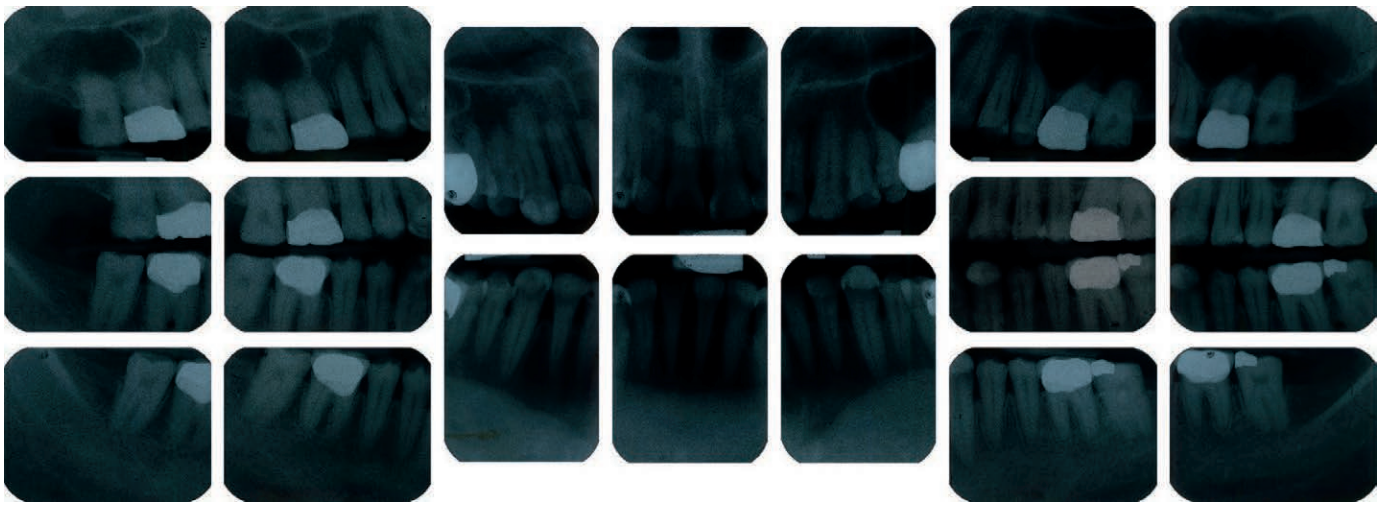

Figure 7. *Full-mouth periapical radiograph.*

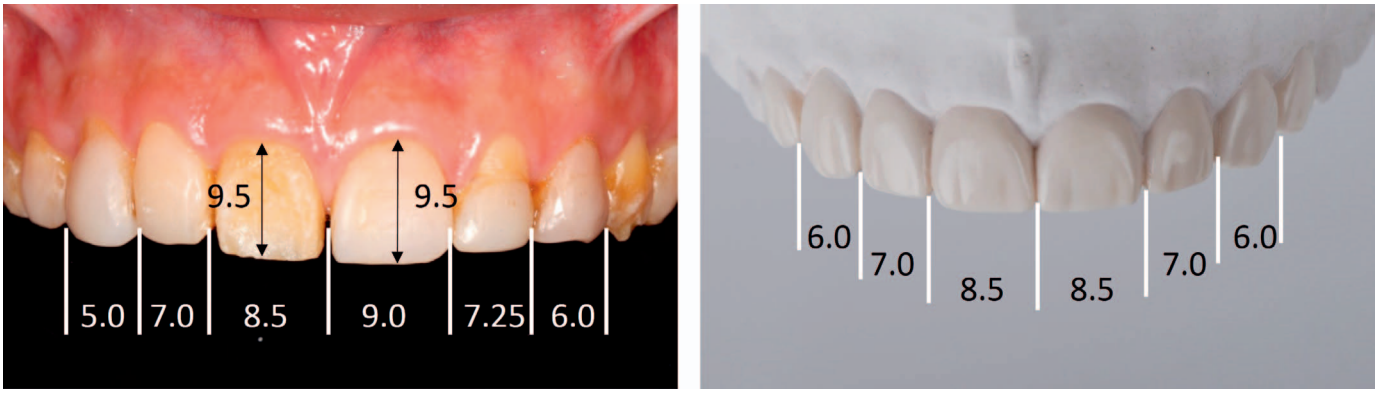

Figure 8. *(A) An asymmetry of the width of upper anterior teeth. (B) A diagnostic wax-up of upper anterior teeth using the repeated ratio for calculating the proper width.*

opaque, white, and white shades of the light-curing resin cement (NX3, Kerr) were chosen, respectively.

The clear shade of the light-curing resin cement was chosen for the remaining teeth (Figure 11). Every procedure was carried out following the manufacturer's instructions. The patient was instructed on the care of his restorations and was provided with an adequate oral home care regimen.

Direct resin composite veneers were performed on the upper second premolars and all lower premolars (#4, #13, #20, #21, #28, and #29). All of the teeth were etched with 37.5% phosphoric acid (Kerr Gel Etchant, Kerr). OptiBond FL (Kerr) bonding agent was then applied. Resin composite (Premise: opaque B1 and body B2, Kerr) was chosen as the restorative material.

Any exposed dentin (and the MO amalgam on #18) was restored with resin composite (Premise: body B3, Kerr). After all treatment had been carried out, the patient was satisfied with the outcome for both esthetics and function (Figure 12). Upper and lower Hawley retainers were made to maintain the tooth alignment (Figure 13). Furthermore, upper and lower trays for GC Tooth Mousse were made to help prevent dental caries. The patient was instructed to wear these trays at least three minutes after brushing, following the manufacturer's recommendations (Figure 14). The patient was recalled at postoperative intervals of one week, one month, three months, six months, and one year.

DISCUSSION

The patient in the current case report had been treated since he was 10 years old. The skeletal growth had not completed yet at that age; therefore, the proper transitional treatment consisted of resin

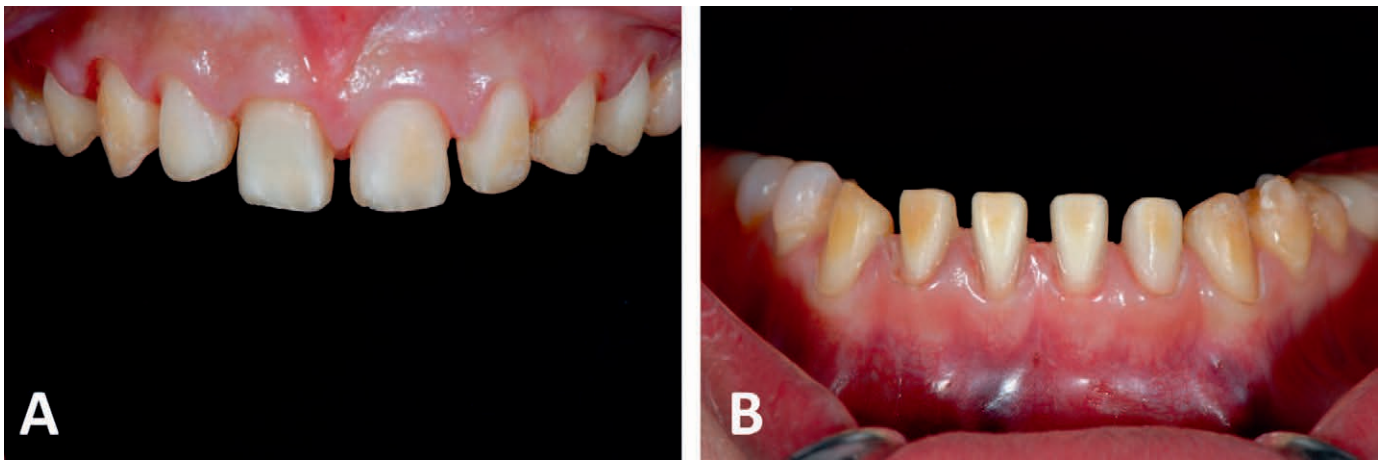

Figure 9. *Preparation for ceramic veneers. (A) 5-12. (B) 22-27.*

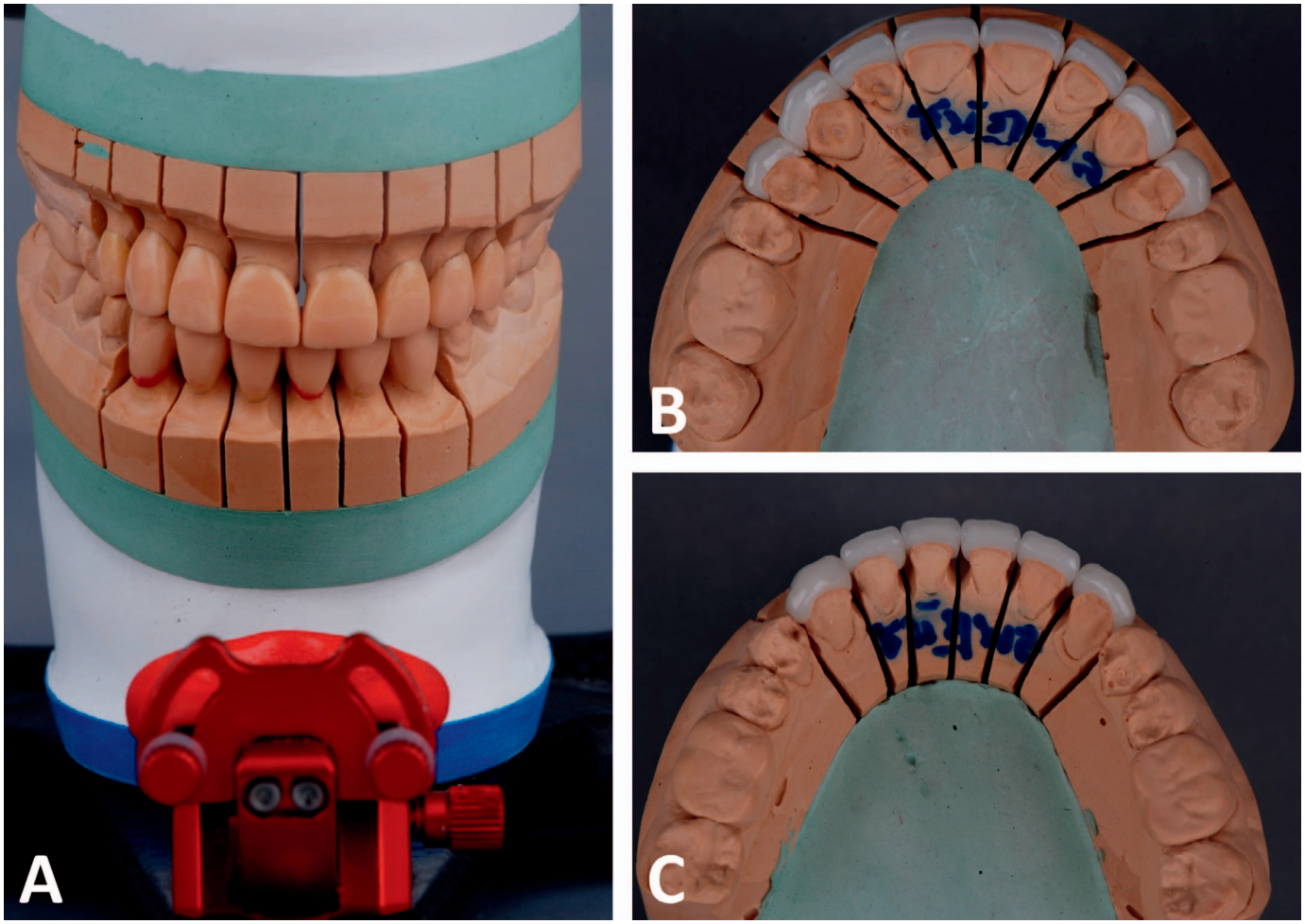

Figure 10. *(A) Waxing for ceramic veneers. (B) Ceramic veneers 5-12. (C) Ceramic veneers 22-27.*

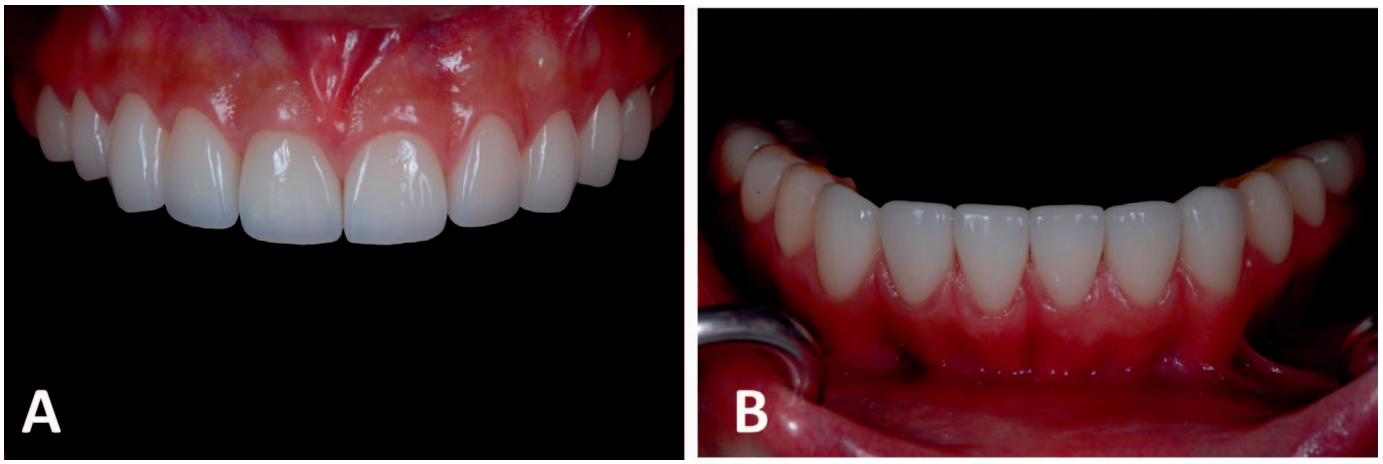

Figure 11. *Ceramic veneers after cementation. (A) 5-12. (B) 22-27.*

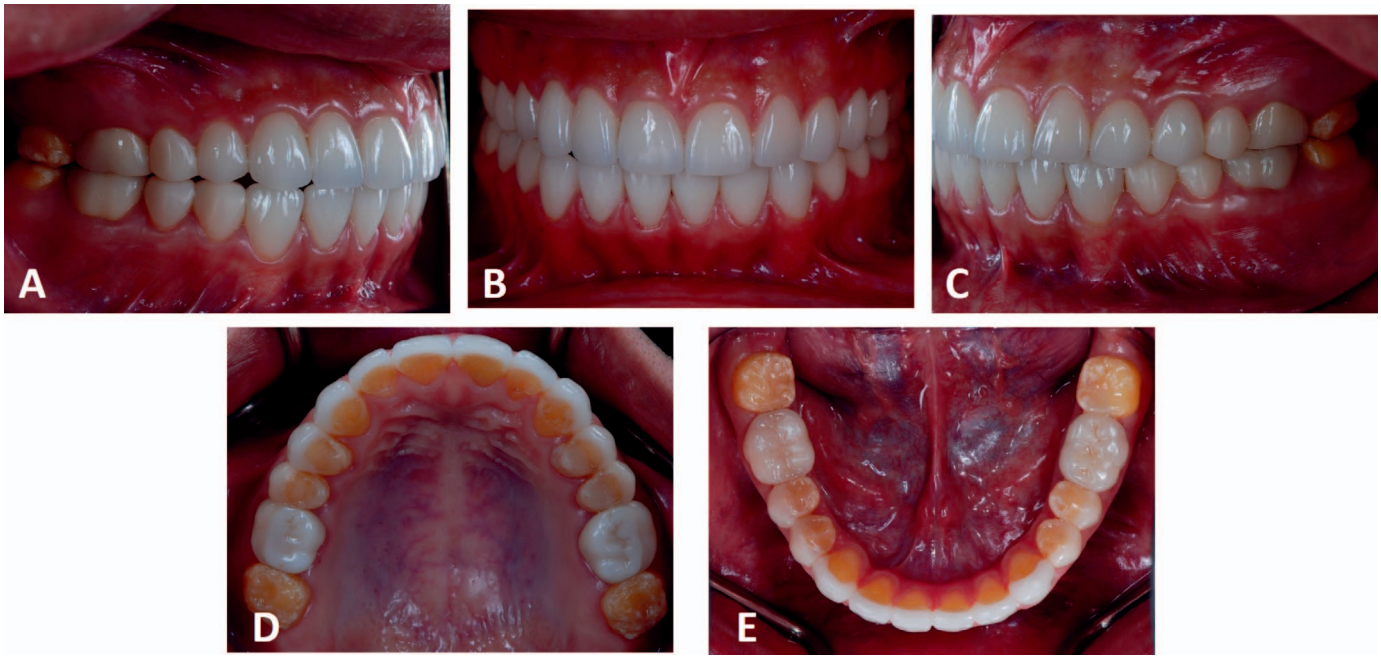

Figure 12. *Final restorations six months after insertion (ceramic veneers at 5-12 and 22-27; resin composite veneers at 4, 13, 20, 21, 28, and 29; and all ceramic crowns at 3, 14, 19, and 30). (A) Right-side view. (B) Frontal view. (C) Left-side view. (D) Occlusal view of the maxilla. (E) Occlusal view of the mandible.*

composite veneers and stainless-steel crowns. This treatment relieved tooth sensitivity and improved esthetics. Additionally, the resin composite veneer is considered the best way to preserve tooth structure since it requires a minimal preparation.[12,29,30] However, there has been some evidence that for AI patients, especially those with poorly mineralized and friable enamel, the outcome of bonding between a resin composite and enamel is not as efficient as it is with normal enamel.[31] Because of tremendous advances in the field of esthetic dentistry, especially in bonding to dentin, it is currently possible to restore function and esthetics to an acceptable level for areas with poor bonding substrates, as found in patients with AI.[32,33]

With regard to the final restorations, the stainless-steel crowns were converted to all-ceramic crowns. It has been found that there was no significant difference in the five-year success rates between single unit zirconium oxide–based ceramic crowns and single-unit porcelain-fused-to-metal crowns.[34] Although all ceramic crowns seem to deliver a better biocompatible material and esthetics, they are prone to fracture, particularly for the veneering layer. Therefore, 3/4 zirconia cores and all zirconia crowns were selected for use with this patient. Additionally, zirconia cores require only 0.5 mm of occlusal reduction, reducing the amount of enamel preparation. For the incisors and first premolars, ceramic veneers and resin composite veneers were chosen due to the minimal loss of labial enamel and no loss of lingual enamel. Because the patient had a limited budget, ceramic veneers were used only in the anterior zone. Crown restoration, which is a relatively invasive technique, is not justified in this case because the lingual surface of the teeth was intact. Moreover, veneer restorations have been proven to achieve a success rate of 80%-100% for patient satisfaction.[35,36]

The case study of Vailati and Belser[37] suggested another idea for the treatment of generalized initial dental erosion. Those authors provided minimally invasive treatment using a thin occlusal ceramic onlay that required only 0.5-0.6 mm of enamel reduction and a palatal veneer for maxillary anterior teeth (IPS e. max Press, Impulse 01, Ivoclar Vivadent). Not only was the patient in that study satisfied with his outcome, but the process used in that study requires minimal preparation. However, that method demands much technical expertise for both the clinician and the dental technician.

Because of an irregularity of the enamel surface for patients affected with AI and the subsequent higher risk of developing dental caries, customized

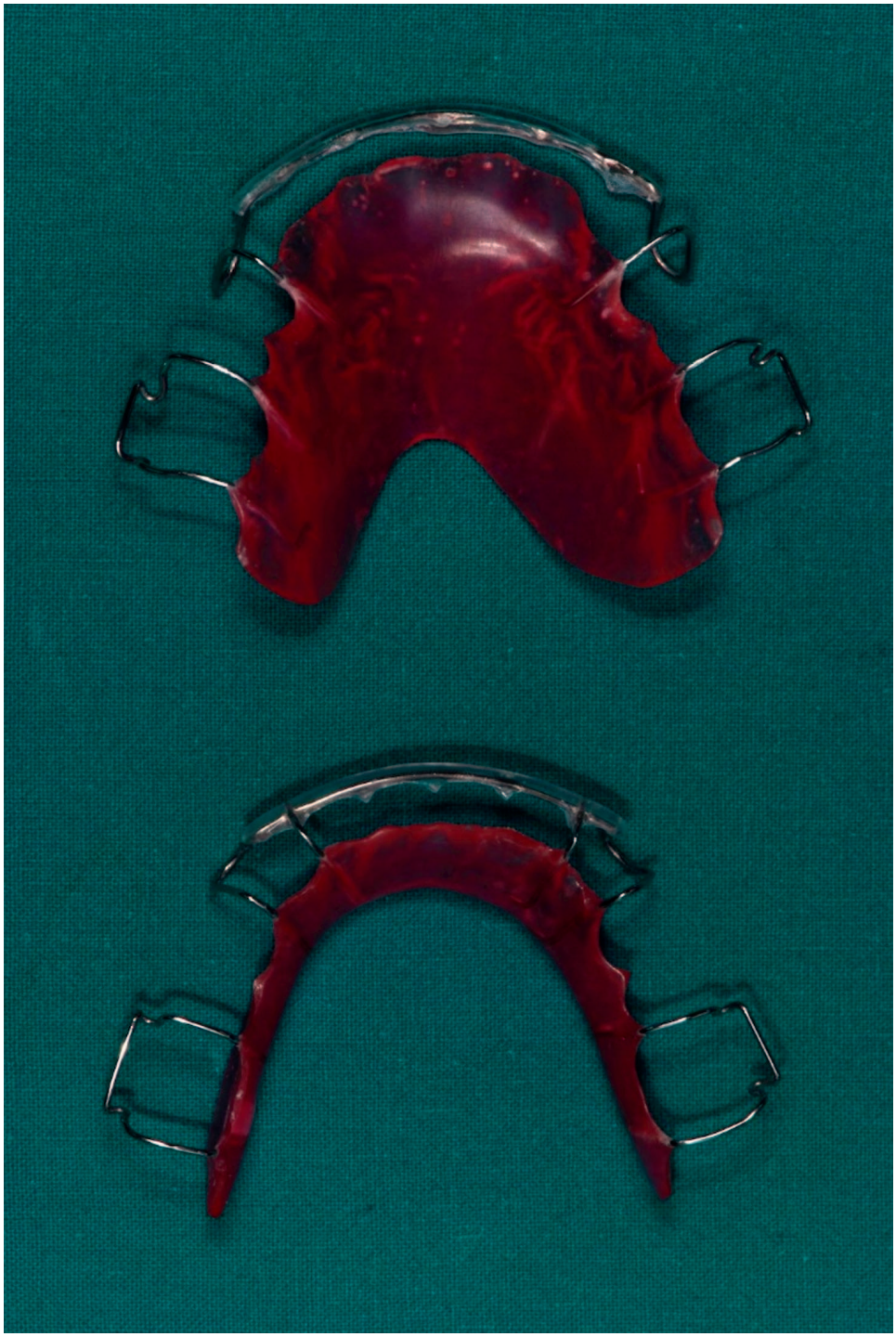

Figure 13. *Hawley retainers*.

trays (both upper and lower trays) and GC Tooth Mousse were presented to the patient. The GC Tooth Mousse was selected because it is a water-based cream containing RECALDENT (CPP-ACP: casein phosphopeptide–amorphous calcium phosphate) and its component, CPP-ACP, has been proven to promote the remineralization process.[38,39]

CONCLUSION

As AI disorders noticeably affect the primary dentition, it is crucial to obtain a proficient diagnosis and effective treatment plan. An interdisciplinary approach should be used to treat the patient starting at the first stage, as not only is the tooth structure preserved but the esthetics and functional approach

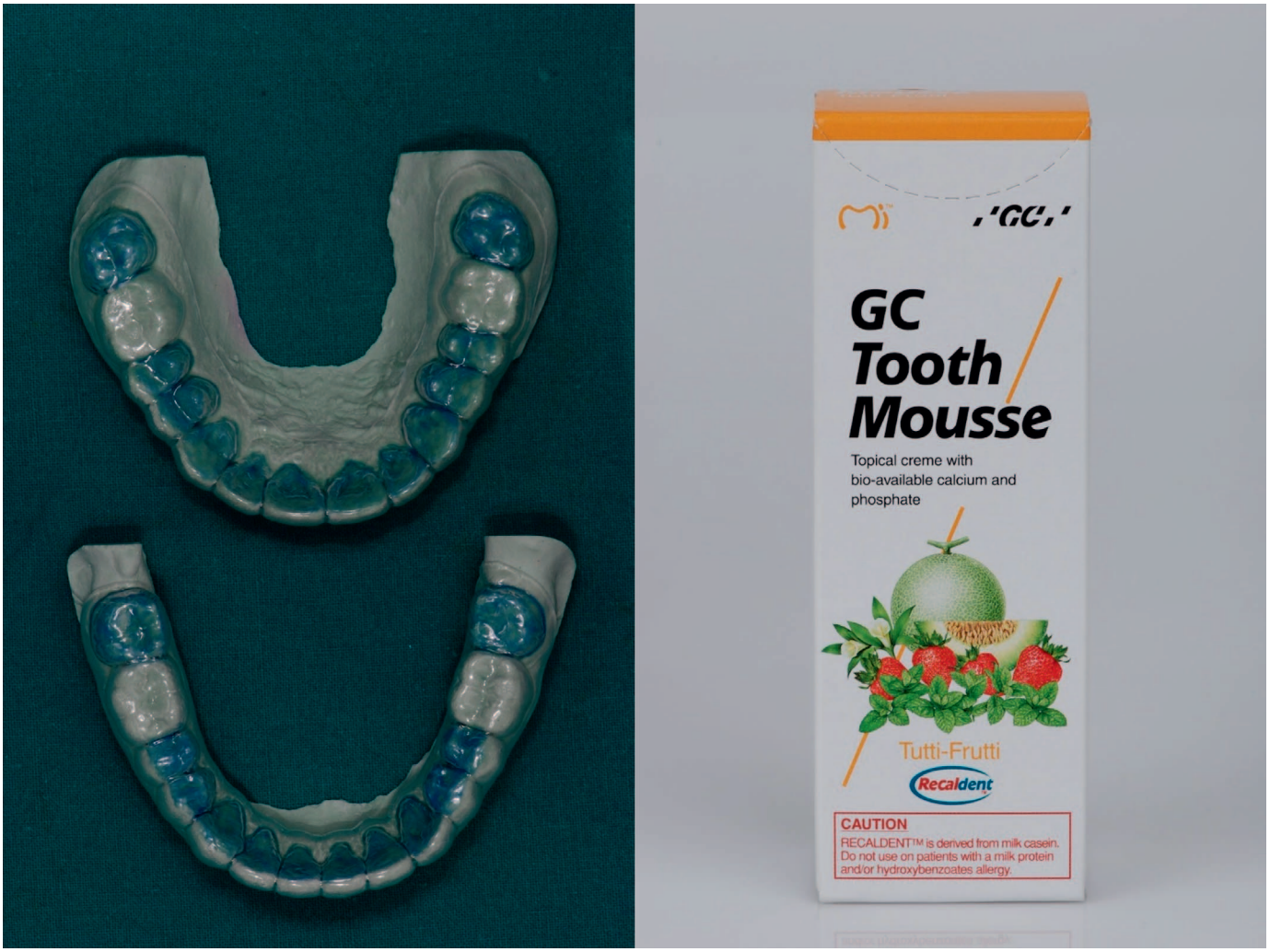

Figure 14. *Upper and lower trays with GC Tooth Mousse.*

are feasible as well. This case study demonstrates a thorough treatment plan that could be applied to other cases as guidance for an AI patient from the period of mixed dentition.

Conflict of Interest

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

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Clinical Research

Randomized Clinical Trial to Evaluate MTA Indirect Pulp Capping in Deep Caries Lesions After 24-Months

U Koc Vural • A Kiremitci • S Gokalp

Clinical Relevance

The application of MTA and calcium hydroxide showed similar clinical performance over a 24-month period.

SUMMARY

Objective: This clinical study aimed to assess the efficacies of mineral trioxide aggregate (MTA) and calcium hydroxide [$Ca(OH)_2$] in the treatment of deep carious lesions by the direct complete caries removal technique.

Methods and Materials: A total of 100 permanent molar/premolar teeth were capped with either $Ca(OH)_2$ (n=49) or MTA (n=51) and restored with composite resin in 73 patients. Periapical radiographs were acquired prior to the treatment as well as at six, 12, and 24 months posttreatment. Two calibrated examiners performed the clinical and radiographic assessment of the periapical pathology and pulpal symptoms. Intergroup comparisons of the observed values were performed using the Fisher exact test. Significance was predetermined at $\alpha = 0.05$.

Results: The recall rates were 100% at six and 12 months posttreatment and 98.6% at 24 months posttreatment. Four teeth capped with $Ca(OH)_2$ (two each at six and 12 months posttreatment) and two capped with MTA (one each at 12 and 24 months posttreatment) received endodontic emergency treatment because of symptoms of irreversible pulpitis, which were clinically and/or radiographically established. There were no significant differences in pulp vitality between the two pulp-capping agents at six, 12, or 24 months posttreatment ($p=0.238$, $p=0.606$, and $p=0.427$, respectively).

Conclusions: Both pulp-capping materials were found to be clinically acceptable at 24 months posttreatment.

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INTRODUCTION

Infected deep carious lesions can be clinically managed either by preservation of the tooth tissue or by root canal therapy. Preservation of the dental

pulp vitality is important in the treatment of deep carious lesions. The success of the pulp-capping procedure relies primarily on the type of capping material, patient's age, periodontal condition, stage of root formation, size and nature (traumatic/mechanical/carious) of the exposure, and status of microbial contamination of the site.[1]

Conservative approaches for the treatment of deep carious lesions still show favorable prognosis for pulpal healing. Alternative approaches for the vital treatment of deep carious lesions include complete caries removal, stepwise excavation, indirect pulp capping, and partial caries removal. Complete caries removal, which involves the removal of all infected and affected carious dentin, is preferred by a majority of the professionals for the treatment of deep carious lesions.[2,3] In this procedure, the pulp is covered with a protective base at areas where the remaining dentin thickness is $\leq$ 0.5 mm. Although stepwise excavation has gained popularity in recent times, the results of a previous histological study indicate that infected dentin should be completely removed to arrest the carious process.[4]

A wide array of materials have been used in the treatment of deep carious lesions for maintaining pulp vitality, including calcium hydroxide [$Ca(OH)_2$], which is regarded as the gold standard for pulp capping.[1] Following pulp exposure, pulpal tissue is usually in disarray, and subsequent capping with $Ca(OH)_2$ can cause coagulation necrosis, in spite of which stimulation of the vital pulp can still elicit a response.[5] However, several disadvantages of $Ca(OH)_2$ have been reported, including high solubility in oral fluids[6] and the formation of tunnel defects in dentin bridges, leading to the failure of capping over time.[1] New materials, including mineral trioxide aggregate (MTA), have been tested as alternatives to $Ca(OH)_2$. The use of MTA has increased following its approval by the US Food and Drug Administration. Although it was first introduced as a root canal–filling material in 1993, MTA has shown promising results in many areas, particularly in direct pulp capping.[1] This material is essentially composed of Portland cement and bismuth oxide in a 4:1 proportion and is capable of releasing calcium ions into the environment.[7] The application of MTA has been evaluated in several experiments; it has been found to exhibit good sealing ability, biocompatibility, and resistance to bacterial penetration; in addition, it is insoluble, unlike $Ca(OH)_2$, which resorbs over time.[8,9] Witherspoon[10] reported MTA as being an optimum material for application in vital pulp therapy. These advantages have led investigators to evaluate the possibility of its use as an alternative to $Ca(OH)_2$ in pulp capping.

Although comparative studies on the treatment of deep carious lesions have been performed previously, only a few studies have compared the efficacies of MTA and $Ca(OH)_2$ in the treatment of deep carious lesions by the complete caries removal technique. The present clinical study aimed to compare the efficacies of MTA and $Ca(OH)_2$ (control group) as indirect pulp treatment (indirect pulp-capping) materials using the complete caries removal technique in patients with deep carious lesions. The null hypothesis tested was that no significant differences would be observed between the pulps capped with MTA and $Ca(OH)_2$ at six, 12, and 24 months posttreatment.

METHODS AND MATERIALS

In this single-blinded, randomized, controlled clinical trial, MTA was evaluated as the test pulp-capping material and $Ca(OH)_2$ as the control. This study was not operator blinded because of the different application procedures of the two materials.

This clinical study was approved by the local ethics committee with the reference number HEK 11/106. Informed written consent was obtained from all participants after they were clearly instructed regarding the reporting of their pain history and the influence of this report on the treatment they would receive. This study was conducted in full accordance with the World Medical Association Declaration of Helsinki.

The study population was comprised of patients with deep carious lesions who were admitted to the outpatient clinic at our department. A single operator, trained in the standardization of the procedures, performed all of the restorative treatments. Clinical assessment was performed by electric pulp testing (Parkell, Farmingdale, NY, USA), thermal testing using an air-water syringe, palpation, and percussion as well as the evaluation of the presence of signs of inflammation. Periapical radiographs were acquired prior to the treatment as well as at baseline (one week) and six, 12, and 24 months posttreatment; they were assessed to exclude any signs of irreversible pulpitis. All the teeth were clinically and radiographically examined to ensure the absence of widening of the periodontal ligament or periapical lesions.

The inclusion criteria were the presence of deep carious lesions involving 75% or more of the dentin

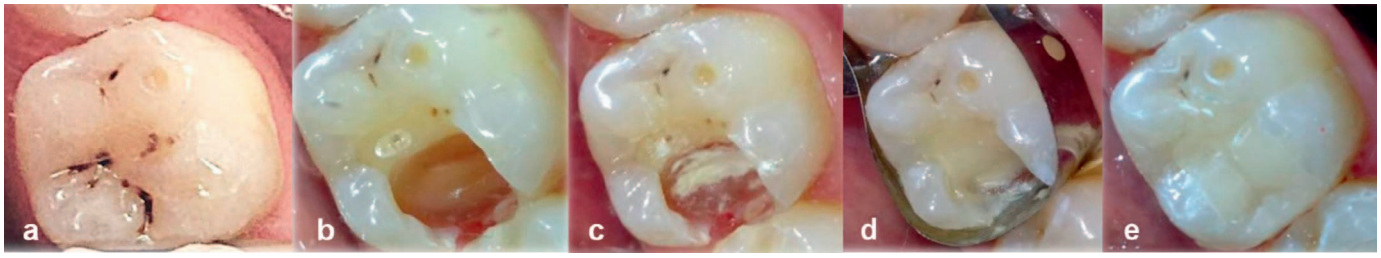

Figure 1. *Restoration procedure of an indirect pulp capping. (a): Preoperative stage. (b): Cavity preparation. (c): mineral trioxide aggregate application. (d): Glass ionomer cement (GIC) base. (e): Final restoration.*

without pulp exposure, restorable permanent posterior teeth without a history of spontaneous and severe pain, abscesses, and a sinus tract or other clinical signs of irreversible pulpitis. A maximum of two teeth per patient were included in this study. A simple randomization method was used for selection—the first tooth selected was assigned to be treated with MTA and the second with $Ca(OH)_2$.

Isolation was performed using a saliva ejector and cotton rolls. Cavities were prepared using sterile diamond burs at high speed under water cooling. After gaining suitable access through the cavitated enamel, superficial, soft, and infected dentin was excavated using sharp hand instruments, particularly wide spoon excavators. The remaining caries were removed using a single-use steel bur, applied at a low speed to avoid pulp exposure (Figure 1a,b). Patients were asked whether they experienced severe sensitivity or pain during the preparation, and, if required, a local anesthetic was administered. Teeth that developed pulp exposure were excluded from the study.

The teeth were then divided into two experimental groups. Indirect pulp capping was performed using $Ca(OH)_2$ (Dycal, Dentsply/Caulk, Dentsply International Inc, Milford, DE, USA) in group I and MTA (Dentsply Tulsa Dental, Johnson City, TN, USA) in group II. In cases where two teeth were restored in the same patient, one of the teeth was capped with MTA, while the other was capped with $Ca(OH)_2$.

All of the materials (Table 1) were applied according to the manufacturers' instructions.

While MTA powder was mixed with its respective solution for application, $Ca(OH)_2$ paste was mixed in a 1:1 ratio. The capping materials were placed with the passive application on the deepest part of the cavity (Figure 1c). Only light pressure was applied

Table 1: *Description of the Materials*

Material	Composition	Manufacturer
Dycal	Base paste:	Dentsply/Caulk, Dentsply International Inc (Milford, DE, USA)
	1,3-Butylene glycol disalicylate, zinc oxide, calcium phosphate, calcium tungstate, iron oxide pigments	
	Catalyst paste: Calcium hydroxide, N-ethyl-o/p-toluene sulfonamide, zinc oxide, titanium dioxide, zinc stearate, iron oxide pigments (dentin shade only)	
ProRoot MTA	Oxides: lime(CaO), silica (SiO_2), aluminum oxide (Al_2O_3), ferric oxide (Fe_2O_3), bismuth trioxide	Dentsply Tulsa Dental (Johnson City, TN, USA)
	Tricalcium aluminate, tetracalcium aluminoferrite, tricalcium aluminate	
Riva Light Cure	Compartment 1: polyacrylic acid, tartaric acid, 2-hydroxyethyl methacrylate, dimethacrylate cross-linker, acidic monomer	Southern Dental Industries (Bayswater, Australia)
	Compartment 2: fluoroaluminosilicate glass powder	
Prime and Bond NT	Di- and trimethacrylate resins, PENTA (dipentaerythritol penta acrylate monophosphate), nanofillers—amorphous silicon dioxide, photoinitiators, stabilizers, cetylamine hydrofluoride, acetone	Dentsply DeTrey (Konstanz, Germany)
Gradia Direct Posterior	Methacrylate monomers, silica, fluoro-alumino-silicate glass, prepolymerized filler, pigments, catalysts	GC (Tokyo, Japan)

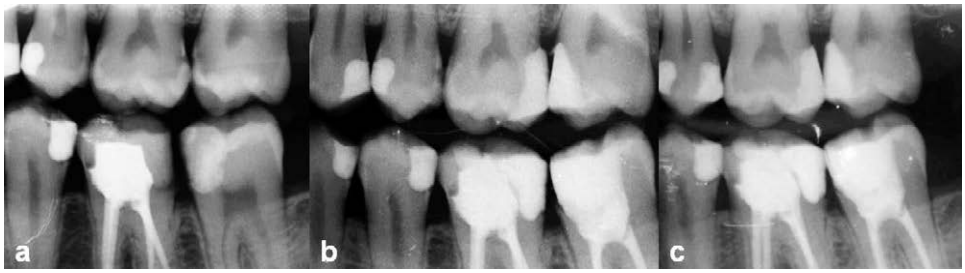

Figure 2. *X-rays of maxillary left first and second molar; #14 indirectly capped with mineral trioxide aggregate, #15 indirectly capped with $Ca(OH)_2$. (a): Preoperative stage. (b): 12 months. (c): 24 months.*

on the MTA mixture using a wet cotton pellet to ensure adaptation of the material onto the dentin. When necessary, excess material was carefully removed using hand instruments.

Following the application of the pulp-capping materials, a light-cured glass ionomer cement base (Riva Light Cure LC, Southern Dental Industries, Bayswater, Australia) was applied and light cured for 20 seconds using a light-emitting diode (LED) device (Radii Plus, Southern Dental Industries; 1500 mW/cm^2) (Figure 1d). The teeth were than etched using 37% phosphoric acid (Condac 37, FGM, Setubal, Portugal) for 30 seconds, and the one-bottle adhesive system Prime and Bond NT (Dentsply DeTrey, Konstanz, Germany) was used. A microfilled hybrid composite resin material (Gradia Direct Posterior, GC, Tokyo, Japan) was then incrementally inserted into the cavities and light cured for 20 seconds using the same LED device, thus completing the restoration process (Figure 1e). The teeth were then polished using diamond-composite finishing burs, discs, and rubber cones. Phosphor plate images were acquired using a dental X-ray unit (Soredex, Tuusula, Finland; 70 kVp, 7 mA) using a paralleling technique, with Rinn film holders to achieve standardization.

The patients were informed of the recall appointments at baseline (one week) and six, 12, and 24 months posttreatment (Figure 2). During the observation periods, two calibrated and experienced dentists assessed all subjective symptoms, including pain or tooth sensitivity to various stimuli. The treatment was recorded as being clinically successful when pulp vitality was observed along with a normal response to thermal, electrical, and tactile tests without signs of spontaneous pain, and it was considered to be radiographically successful in the absence of radiolucency and periodontal ligament space widening.

The data were statistically analyzed using the chi-square, frequency, and Fisher exact tests. Significance was predetermined at $\alpha = 0.05$. Power analysis of noninferiority tests of two independent proportions was performed by using NCSS 2007/PASS program. The margin (Δ), the maximum acceptable extent of clinical noninferiority, was defined as 6%. Power of this study was calculated as 82%.

RESULTS

A total of 100 permanent molar/premolar teeth of 73 patients were capped randomly with either $Ca(OH)_2$ (n=49) or MTA (n=51) and restored using composite resin. The patients included 47 women (64.4%) and 26 men (35.6%), with a mean age of 21 years (20.93±3.48). The recall rates were 100% at six and 12 months posttreatment and 98.6% at 24 months posttreatment. Molars accounted for 62% of the total number of treated teeth. The distribution of teeth evaluated in the present study is shown in Table 2.

At 24 months posttreatment, one of the patients who was treated with $Ca(OH)_2$ failed to attend the recall; this patient, however, confirmed the absence of discomfort in the treated tooth via telephone. Four teeth indirectly capped with $Ca(OH)_2$ (two each at six and 12 months posttreatment) and two indirectly capped with MTA (one each at 12 and 24 months posttreatment) received endodontic emergency treatment because of symptoms of irreversible pulpitis, which were clinically and/or radiographically established (Table 3). The success rates of pulp vitality were 91.7% for the $Ca(OH)_2$-treated teeth and

Table 2: *Distribution of Tooth Type by Location*

Dental Arch	Tooth Type	n	%
Calcium hydroxide (n=49)			
Upper (n=29)	Premolar	16	16.0
	Molar	13	13.0
Lower (n=20)	Premolar	7	7.0
	Molar	13	13.0
Mineral trioxide aggregate (n=51)			
Upper (n=23)	Premolar	10	10.0
	Molar	13	13.0
Lower (n=28)	Premolar	5	5.0
	Molar	23	23.0
Total		100	100

Table 3: *Clinical Assessment of Tooth Vitality Distribution*

Material	Condition of Pulp Tissue			
	Normal (Vital)		Irreversible Pulpitis	
	n	%	n	%
Calcium hydroxide (n=49)				
Baseline	49	100.0	—	—
Six months	47	95.9	2	4.15
12 months	45	91.8	2	4.15
24 months	44	91.7	—	—
Total			4	8.3
Mineral trioxide aggregate (n=51)				
Baseline	51	100.0	—	—
Six months	51	100.0	—	—
12 months	50	98.0	1	1.95
24 months	49	96.01	1	1.95
Total			2	3.9

96.01% for the MTA-treated teeth; the results of the Fisher exact test revealed no statistically significant differences in pulp vitality between the two capping materials at 24 months posttreatment (p=0.427; Table 3).

At 24 months posttreatment, the frequency of observed symptoms was found to have reduced in both groups. Correlations between the type of pulp-capping material and the frequency of symptoms observed at six, 12, and 24 months posttreatment (p=0.588, p=0.087, and p=0.202, respectively) were found to be insignificant. The symptoms most frequently observed were sensitivity to cold/heat; the distribution of the symptoms according to the type of pulp-capping material is summarized in Table 4.

At 24 months posttreatment, four of the six teeth that were subsequently subjected to root canal treatment were found to be symptomatic at the preoperative time; however, this relationship was not found to be significant (p=0.690).

In the present study, restoration failure due to the loss of materials was not observed in either of the groups at six, 12, and 24 months posttreatment.

DISCUSSION

In this clinical study, we compared the efficacies of MTA and $Ca(OH)_2$ as pulp-capping materials in the treatment of teeth with deep dentin carious lesions using the complete caries removal technique. The results of the present study revealed the high efficacy of complete caries removal using a one-visit approach, along with the high success rate of indirect pulp capping, which was found to be as high as 94% regardless of the material used. This high success rate may be attributed to accurate diagnosis, complete removal of the carious tissue (thus halting the progression of the carious process), and well-sealed restoration preventing microleakage. The remnant cariogenic bacteria in the cavities would have died on the elimination of their source of nutrition because of the tight restoration, resulting in the arrest of the carious lesions.

In most countries, caries in permanent/deciduous teeth make up the most prevalent chronic disease among both children and adults,[11,12] and, therefore, caries removal is a frequently performed operation in dental clinics. However, there are no standard guidelines describing the criteria for the excavation technique, depth, or instruments to be used for caries removal. Different treatment options for deep carious lesions have been reported in the literature. Stepwise caries excavation with a one- or two-visit approach, which is the most discussed technique in the literature, is gaining popularity of late. In the one-visit approach, the caries are partially removed, and permanent restoration is performed with tight sealing of the cavity. In the two-visit approach, an intermediate restoration is initially performed, and the cavity is reopened in the second visit,[13] making the restoration process more complicated, time consuming, and costly. Complete caries removal, which involves the removal of all of the decayed and

Table 4: *Clinical Assessment of the Distribution of Symptoms with Calcium Hydroxide and Mineral Trioxide Aggregate (MTA) at the Pretreatment, Baseline, and Six-, 12-, and 24-Month Posttreatment Examinations*

	MTA				Calcium Hydroxide			
	Cold	Heat	Bite	Percussion	Cold	Heat	Bite	Percussion
Pretreatment	24	16	7	16	17	6	4	10
Baseline	7	1	—	1	6	2	2	2
Six months	5	—	—	2	2	—	1	2
12 months	3	—	—	1	7	1	1	1
24 months	2	—	—	—	3	1	—	—

infected dentin in one visit, has been reported to be the preferred method of treatment.[2,13-16] This method offers the advantage of completing permanent restoration in one visit because of the lack of need for reopening the cavity for caries removal in a subsequent step in addition to there being no doubts regarding the progression of the remaining carious lesion into the pulp. Browning[17] compared the stepwise and complete caries removal techniques and reported a 90% success rate of treatment as well as no significant differences in the outcomes between the two methods. Rohan and others [13] and Franzon and others[18] reported greater treatment success with complete caries removal in comparison to the stepwise approach.

Calcium hydroxide plays an important role in the treatment of deep carious lesions by controlling the growth of microflora and stimulating the pulp.[17] Although MTA exhibits an antibacterial effect on some of the facultative bacteria and no effect on the strictly anaerobic bacteria,[19] it continuously exhibits high pH levels (12.5) for eight weeks. Although this limited antibacterial effect of MTA is less than that demonstrated by $Ca(OH)_2$ paste, the ability of the former to resist the potential penetration of microorganisms appears to be high.[10]

In the present study, the number of observed symptoms was higher in the $Ca(OH)_2$ group than in the MTA group at 12 months posttreatment. Browning and Swift[17] reported that, at one year posttreatment, preoperative pain was significantly less likely to be classified as indicating success. However, in the present study, the frequency of the symptoms reported was reduced in both groups at 24 months posttreatment, and there was no significant correlation between the intensity of the symptoms and treatment outcome; four of the six teeth that were subsequently subjected to root canal treatment were symptomatic at the preoperative time. This result is concurrent with that reported in the literature.[17]

In clinical studies involving pulp therapy, histopathological guides have been used as the gold standard.[1] However, the application of a standard histological reference for the comparison of the two materials evaluated in the present study was not possible because of its design.[20] In the present study, the lesions exhibited a decrease in symptoms such as sensitivity to cold/heat or percussion at the 24-month follow-up. These clinical symptoms are typically observed over time following the removal of caries lesions with sclerotic or reparative dentin formation.[9,13,21,22]

In the present study, no differences were observed in the outcomes between the two indirect pulp-capping materials, and both materials exhibited very similar success rates. Therefore, the null hypothesis was accepted. However, the potential clinical relevance of the difference of 4.32% in the success rates of MTA and $Ca(OH)_2$ can be discussed. Results of clinical and histological studies conducted by exposing the pulp tissue have highlighted the better performance of MTA in comparison with that of $Ca(OH)_2$ in both animal and human teeth, with the former exhibiting faster pulpal healing, early formation of hard and tight tissue barriers within three weeks, and less inflammation, hyperemia, and necrosis as well as thicker dentinal bridges and more frequent odontoblastic layer formation.[1,9,23-25] Additionally, MTA has been reported to demonstrate an improved ability to maintain pulp tissue integrity and produce negligible pulpal necrosis[1,26,27] in the treatment of deep dentin caries.

Since bacteria and/or their toxic products are capable of passing through tubules to induce inflammatory responses in the dental pulp,[28,29] remaining dentin thickness and tubular permeability are crucial factors in the induction of pulpal inflammatory response.[30] To the best of our knowledge, there is no device for the measurement of the remaining dentin thickness in clinical conditions.

Calcium hydroxide, which is the gold standard among indirect pulp-capping materials, is still popular because of its easy handling, paste form, and lower cost compared to MTA. However, Chisini and others[31] reported that, because of their updated knowledge of contemporary literature, MTA was mostly used by professionals working in the university environment, which indicates that MTA might gain more popularity in the future.

CONCLUSIONS

Indirect pulp capping accompanying complete caries removal exhibited a satisfactory success rate in the treatment of deep carious lesions. Both $Ca(OH)_2$ and MTA were found to be clinically effective at 24 months posttreatment. Further clinical research is required to confirm the long-term results of the current study.

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Regulatory Statement

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

guidelines and policies of Hacettepe University. The approval code for this study is HEK 11/106.

Conflict of Interest

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

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Thirty-Six-Month Clinical Comparison of Bulk Fill and Nanofill Composite Restorations

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Clinical Relevance

Bulk fill restorative resin might be a good alternative to conventional resin composites in terms of marginal adaptation and discoloration under clinical conditions.

SUMMARY

Objectives: The aim of this study was to evaluate the clinical performance of a nanofill and a bulk fill resin composite in class II restorations.

Methods and Materials: In accordance with a split-mouth design, 50 patients received at least one pair of restorations, restored with a nanofill resin composite (Filtek Ultimate [FU]) and with a bulk fill resin composite (Tetric EvoCeram Bulk Fill [TB]). Each restorative resin was used with its respective adhesive system according to the manufacturers' instructions. A total of 104 class II restorations were placed by two operators. The restorations were blindly evaluated by two examiners at baseline and at six, 12, 18, 24, and 36 months using modified US Public Health Service Ryge criteria. The comparison of the two restorative materials for each category was performed with the chi-square test (α=0.05). The baseline scores were compared with those at the recall visits using the Cochran Q-test.

Results: At six, 12, 18, and 24 months, the recall rate was 100%, 98%, 94%, and 82%, respectively, with a retention rate of 100%. At 36 months, 81 restorations were evaluated in 39 patients with a recall rate of 78%. For marginal adaptation, four restorations from the TB group and 10 from the FU group rated as Bravo. Two restorations from the TB and eight restorations from the FU group showed marginal discoloration. There were statistically significant differences between the two restorative resins in terms of marginal adaptation and marginal discoloration ($p<0.05$). No differences were observed between the restorative resins in terms of retention ($p>0.05$). One restored tooth from the FU group was crowned. The retention rates for the TB and the FU groups were 100%. In the FU group, two

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restorations showed slightly rough surfaces, and two showed a slight mismatch in color. None of the restorations showed postoperative sensitivity, secondary caries, or loss of anatomic form.

Conclusions: The tested bulk fill restorative resin demonstrated better clinical performance in terms of marginal discoloration and marginal adaptation.

INTRODUCTION

The use of resin composites has become a routine procedure for the restoration of posterior teeth.[1,2] Several factors have contributed to their acceptance, including esthetics, improved mechanical properties to match amalgam's performance, and mercury concerns.[2,3] Additionally, their preparation design is more conservative. Although resin composites are being continually developed in order to improve their mechanical and physical properties, polymerization shrinkage still remains a main challenge.[4,5] Polymerization shrinkage stress that is concentrated at the adhesive interface can lead to marginal breakdown, gap formation, marginal leakage, and even cuspal deflection, secondary caries, and restoration loss.[6,7]

The incremental placement technique is the common procedure adopted in clinical practice to avoid depth-of-cure limitations and to overcome polymerization shrinkage stress.[8-10] However, there are various disadvantages of the incremental placement technique, such as voids that can be entrapped between these layers, contamination risk between layers, difficulty in the placement of layers in small cavities that are difficult to access, and increased chair time.[11]

One of the major changes in resin-based composite technology is the introduction of bulk fill resin composites. This category has been launched to simplify and shorten application time, making the clinical procedure more user friendly.[12] Bulk fill resin composites can be placed up to 4-mm in thickness with proper polymerization and low polymerization shrinkage.[13-16] Various strategies are implemented by different manufacturers to accomplish increased depth of cure and lower shrinkage. The addition of stress-relieving monomers, more reactive photoinitiators, and prepolymerized particles result in lower polymerization shrinkage.[17,18] Moreover, the increased translucency of these resins allows greater light transmission and adequate depth of cure.[19] For resin composites, not only adequate polymerization but also proper marginal adaptation are important to ensure optimum clinical behavior. In most of the studies, the performance of bulk fill restorative resins was found to be similar to incrementally placed conventional resins in terms of marginal integrity.[20-23] Although the mechanical and physical properties of bulk fill restorative resins have been evaluated under *in vitro* conditions,[24-29] the ultimate test for a dental restorative material is its clinical durability and effectiveness. This test is needed to validate results from the *in vitro* studies. Therefore, the aim of this clinical study was to compare the clinical performance of a bulk fill resin composite with a conventional resin composite for three years. The null hypothesis tested was that there would be no difference between bulk fill and conventional resin composites.

METHODS AND MATERIALS

Approval for the clinical trial was obtained from the Human Ethics Committee of the Hacettepe University School of Dentistry, and all patients signed a written informed consent form. The inclusion criteria for patients were as follows: good oral hygiene, minimal periodontal disease, absence of deleterious habits (eg, mouth breathing, nail biting, bruxism, and tooth clenching or grinding), and the presence of at least two similar-sized approximal primary caries lesions in premolar and molar teeth. Teeth with secondary caries or in need of replacement of existing restorations were not included in the study. Subjects were excluded from participating in the study if they had a history of adverse reaction to the test materials, were pregnant or lactating, had poor oral hygiene, or had systemic disease and severe periodontal disease and were potentially unable to attend recall visits.

A total of 50 patients—24 males and 26 females—ages 24 to 55 years participated in the study. Bitewing radiographs of the teeth to be restored were taken preoperatively. The teeth to be restored had a normal class I occlusal relationship with natural dentition and had adjacent teeth in contact. The minimal sample size of each group was found to be 40 restorations according to power analysis.

The teeth to be restored were first cleaned with a nonfluoridated prophylaxis paste on a rubber cup and then rinsed with water. Class II slot preparations were completed with a diamond straight (flat end) and round burs (Diatech, Heerbrugg, Switzerland) at high speed with water coolant. The dimension of the preparation was determined by

Table 1: *Composition of the Materials Used in the Study*

Materials	Composition	Mode of Application
Filtek Ultimate (3M ESPE, St Paul, MN, USA) (N214468)	Bis-GMA, UDMA, TEGDMA, bis-EMA, PEGDMA, silica filler, zirconia filler	Insert incrementally in 2-mm increments. Light cure for 40 s.
Adper Single Bond 2 (3M ESPE) (9XN)	Bis-GMA, HEMA, copolymer of acrylic/ itaconic acids, diurethane dimethacrylate, glyceroldimethacrylate, water and ethanol	Apply two consecutive coats of Adper Single Bond adhesive to etched enamel and dentin. Dry gently for 2 to 5 s. Light cure for 10 s.
Tetric EvoCeram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein) (P84129)	Bis-GMA, UDMA, bis-EMA, barium alumina silicate glass filler, ytterbium fluoride, spherical mixed oxide	Insert up to 4 mm thick. Light cure for 20 s.
Excite F (Ivoclar Vivadent) (R06148)	HEMA, methacrylate, phosphonic acid acrylate, highly dispersed silicone dioxide, initiators, stabilizers and potassium fluoride	Apply adhesive to etched enamel and dentin and agitate the adhesive for at least 10 s. Dry gently for 2 to 5 s. Light cure for 10 s.

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

the extent of the caries. The caries removal was completed using a round steel bur in a slow-speed hand piece. The tissue removal was terminated when the dentin was hard on probing. Isolation was accomplished using a rubber dam.

Two different restorative resins were placed in each patient, resulting in a total of 104 restorations. Half of the preparations were restored using the bulk fill resin composite Tetric EvoCeram Bulk Fill (TB, Ivoclar Vivadent, Schaan, Liechtenstein) (n=52), and half of them were restored with Filtek Ultimate (FU, 3M ESPE, St Paul, MN, USA) (n=52) with their respective etch-and-rinse adhesives according to the manufacturers' instructions. The materials used in the study are shown in Table 1.

An Ivory #1 retainer and matrix band with wooden wedges were utilized for placement of the resin composite. The restorative material Filtek Ultimate was applied using an incremental filling technique beginning at the gingival wall. The increments, not exceeding 2-mm in thickness, were added to complete the proper anatomical form. Each increment was polymerized for 20 seconds with an LED light-curing unit (440-465nm) (Starlight s Mectron s.p.a., Carasco, Italy) with an irradiance of >1400 mW/cm^2. The output of the curing unit was checked after each patient with a curing radiometer (Demetron, Danbury, CT, USA). The restorative material Tetric EvoCeram Bulk Fill was placed in bulk up to 4-mm thickness and cured for 20 seconds with the same curing unit. The randomization of restorative material was done using a table of random numbers. Occlusal adjustments were made using articulating paper. Finishing was completed with finishing diamond burs (Diatech), and polishing was accomplished with aluminum oxide disks of decreasing abrasiveness (Sof-Lex, 3M ESPE) and rubber points (Kerr Corp, Orange, CA, USA). The clinical procedure of tooth preparation and placement of restorations was performed by the same operator.

Two experienced double-blinded dentists not involved with the placement of the restorations performed the evaluation. The dentists were calibrated to a predetermined level of inter- and intra-examiner agreement of at least a Kappa value of 95% for each criteria. The restorations were evaluated at baseline, at 6, 12, 18, 24, and 36 months using modified US Public Health Service Ryge criteria[30] and scored as Alpha, Bravo, or Charlie. Alpha corresponds to excellent, Bravo to clinically acceptable, and Charlie to clinically unacceptable results. Postoperative sensitivity was assessed by blowing a stream of compressed air for three seconds at a distance of 2 to 3 cm from the restoration under isolation from the adjacent teeth with gauze and by moving the probe over the restored tooth surface. Subjects were also questioned regarding sensitivity to cold/hot or stimuli. Color photographs at 1:1 magnification were taken at baseline and each recall. In case of disagreement, a consensus was reached based on assessment of the photographs. Bitewing radiographs were also taken at the end of a three-year recall.

The statistical analyses were carried out with the IBM SPSS version 22.0 software package (SPSS, Chicago, IL, USA). The restoration groups for each category were compared using the Pearson chi-square test, and the Cochran Q-test was used to compare the changes across different time points within each restorative material (α=0.05).

Table 2: *Distribution of Materials According to Tooth Type and Arch*

	Maxillary Arch		Mandibular Arch		Total
	Premolar	Molar	Premolar	Molar	
Tetric EvoCeram Bulk Fill	21	13	6	12	52
Filtek Ultimate	21	15	6	10	52
Total	42	28	12	22	104
	70		34		

RESULTS

A total of 104 restorations were placed in 50 patients. The distribution of the restorations is displayed in Table 2. Fifty restorations (48%) were placed in molars, whereas 54 (52%) were placed in premolars. Table 3 presents the results of the clinical evaluation of the restorations.

Retention and Recall Rates

At six, 12, 18, and 24 months, the recall rates were 100%, 98%, 94%, and 82%, respectively, with a retention rate of 100%. One restoration from the FU group received a crown at the 24-month recall. At the end of 36 months, 81 restorations were evaluated in 39 patients with a recall rate of 78% and a retention rate of 100%.

Marginal Adaptation

All restorations from Tetric Bulkfill EvoCeram group showed perfect adaptation until the 24-month evaluation. At 24 months, one restoration from the TB group and eight from the FU group were rated as Bravo.

At 36 months, there was a crevice along the margin of four restorations from the TB group and 10 restorations from the FU group that were detectable with an explorer. In terms of marginal adaptation, significantly better adaptation was observed in the TB group at the 24-month (p=0.015) and 36-month recalls (p=0.048).

Marginal Discoloration

None of the restorations showed marginal discoloration until the 18-month evaluation. At 18 months, two restorations from the FU group showed slight discoloration. At 24 months, while none of the restorations showed discoloration from the TB group, seven from the FU group showed discoloration. At the end of 36 months, two restorations from the TB and eight from the FU group rated as Bravo. Significant differences were observed between the two groups at the 24-month (p=0.005) and 36-month recalls (p=0.048).

Color Match

From the FU group, two restorations at 12 and 18 months, one restoration at 24 months and two restorations at 36 months showed a slight mismatch in color within the normal range of the adjacent tooth structure.

Surface Texture

At 24 and 36 months, only the surface of two restorations from the FU group was rougher than the surrounding enamel.

Table 3: *Results of the Clinical Evaluation of the Restorations*

Evaluation Criteria	Score	Six-Month n (%)		12-Month n (%)		18-Month n (%)		24-Month n (%)		36-Month n (%)	
		TB (n=52)	FU (n=52)	TB (n=51)	FU (n=51)	TB (n=49)	FU (n=49)	TB (n=43)	FU (n=42)	TB (n=41)	FU (n=40)
Marginal adaptation	Alpha	52 (100)	52 (100)	51 (100)	51 (100)	49 (100)	48 (98)	42 (97.7)	34 (80.9)	37 (90.3)	30 (75)
	Bravo	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	1 (2)	1 (2.3)	8 (19.1)	4 (9.7)	10 (25)
Marginal discoloration	Alpha	52 (100)	52 (100)	51 (100)	51 (100)	49 (100)	47 (95.9)	43 (100)	35 (83.3)	39 (95)	32 (80)
	Bravo	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	2 (4.1)	0 (0)	7 (1.7)	2 (5)	8 (20)
Color match	Alpha	52 (100)	52 (100)	51 (100)	49 (96)	49 (100)	47 (96)	43 (100)	41 (97.6)	41 (100)	38 (95)
	Bravo	0 (0)	0 (0)	0 (0)	2 (4)	0 (0)	2 (4)	0 (0)	1 (2.4)	0 (0)	2 (5)
Surface texture	Alpha	52 (100)	52 (100)	51 (100)	51 (100)	49 (100)	49 (100)	43 (100)	40 (95)	41 (100)	38 (95)
	Bravo	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	2 (5)	0 (0)	2 (5)

Abbreviations: TB, Tetric EvoCeram Bulk Fill; FU, Filtek Ultimate.

Postoperative Sensitivity

One patient from the TB group reported postoperative sensitivity that disappeared at the 12-month recall. No differences were detected for the rest of the evaluated criteria ($p>0.05$).

For the TB group, significant differences were observed between baseline and 36 months only for marginal adaptation ($p=0.010$). For the FU group, differences were found between baseline and 24 months and baseline and 36 months for marginal discoloration ($p<0.05$) and adaptation ($p<0.05$).

DISCUSSION

To the extent of the authors' knowledge, there are only limited studies that compare the clinical effectiveness of bulk fill resin composites. Two of them were conducted by van Dijken and Pallesen.[31,32] In one of their studies, five-year clinical durability of the flowable bulk fill resin composite SDR capped with CeramX Mono was compared with CeramX Mono that had been placed incrementally. No difference was observed between restorations with and without SDR in any of the evaluated criteria. None of the restorations showed postoperative sensitivity. Tooth and restoration fracture was also observed.[31] In their other clinical study, they also found that the bulk fill flowable SDR showed highly acceptable clinical results comparable to the conventional 2-mm incremental technique in the three-year follow-up. None of the restorations showed marginal discoloration. Although not statistically different, poor marginal adaptation was seen in incrementally placed restorations.[32]

The results of our study do not correlate with these previous studies mentioned above. This might be related to the bulk fill resin used in our study. In the mentioned studies, bulk fill flowable resin composites were compared. However, in our study, bulk fill resin without the need of capping was investigated, and significant differences were found between bulk fill and conventional resin composite that was placed incrementally in terms of marginal discoloration and marginal adaptation at the end of 36 months. The tested resin composites performed differently in these criteria over the 36-month evaluation period, leading to rejection of the tested hypothesis. In another short-term clinical study, bulk fill restorations demonstrated comparable performance to conventional posterior composite resin.[33] However, these results were obtained at the end of 12 months. Even in our study, there were no differences between tested restoratives at any evaluated criteria until 18 months of recall. The important issue is to evaluate the performances and survival of restoratives for a long time period to reach better conclusions. The results might vary according to the recall time. As seen in our clinical study, clinical outcomes had changed after long-term recalls.

The integrity of the adhesive bond at the tooth/resin interface plays an important role in the clinical success and survival of restorations. The reason for better marginal adaptation observed in bulk fill resin composite might be related to its lower polymerization stress. Moreover, the manufacturer states that TB contains a shrinkage stress reliever, which is a special filler functionalized with silane to minimize polymerization shrinkage (scientific documentation).[34] It is known that stresses are affected by the composition and filler content of resin composite, its elastic modulus.[4,35]

TB contains a mixture of bisphenol-A diglycidyl dimethacrylate, urethane dimethacrylate, and ethoxylated bisphenol A dimethacrylate, all of which are high-molecular-weight monomers with high viscosity and low polymerization shrinkage. On the other hand, FU beside these monomers has a diluent monomer, triethylene glycol dimethacrylate that reduces its viscosity. Because of its smaller molecules, it might have a negative effect on polymerization shrinkage.[36] Due to its low molecular weight, it increases water sorption.[37] The use of prepolymerized filler particles as in TB also contributes to a lower elastic modulus.[38] In a recent study comparing other bulk fill resins, the elastic modulus of TB was found to be moderate.[39] A direct relationship between marginal integrity and polymerization contraction/stress has been reported in some *in vitro* studies.[22,40,41] Although better performance of bulk fill resin composite was found in terms of marginal adaptation in the present study, comparable results with traditional incrementally placed resin composites were reported in most of the *in vitro* studies.[21,42,43] In a recent *in vitro* study,[21] Tetric EvoCeram Bulk Fill demonstrated similar gap formation to conventional resin composite. Al-Harbi and others[23] also analyzed the cervical marginal integrity of class II preparations restored with bulk fill vs incrementally placed resin composites. They found similar marginal integrity compared to conventional incremental fill composites. Concurring with these results, Fronza and others[22] also found that TB was not different in percentage of internal gap formation from incrementally placed resin composite. However, in clinical conditions, restorations are subjected to temperature changes and, more important, masticatory stresses. These factors cause a strain accumula-

tion that leads to chemical and mechanical degradation.[42] Therefore, it might not be accurate to directly compare our findings with the results obtained from *in vitro* studies.

The perfect seal between restoration and tooth has great importance in preventing microleakage and its clinical consequences, such as marginal discoloration, recurrent caries, and pain. There are only few *in vitro* studies evaluating the microleakage of bulk fill resin composites. In one of them, flowable bulk fill resin showed less leakage in comparison with nanohybrid resin composite at dentinal margins. However, they showed similar microleakage values at enamel margins.[43] In another study, no difference was observed in cervical microleakage when an incrementally filled conventional resin composite was compared with a bulk fill flowable resin composite base in class II preparations.[44] However in both studies, flowable bulk fill composites were used. In the present study, bulk fill restorations led to significantly better clinical outcomes regarding marginal discoloration. This can be explained by the lower elastic modulus of TB (10 GPa) than FU (12 GPa). Taking into account that the same six restorations rated as Bravo for both marginal discoloration and adaptation, the higher marginal discoloration observed in the FU group may have been related to the poor marginal adaptation. The contraction stress might have caused a dislodgment of resin composite from the tooth margin, thereby inducing gap formation. These defects along the margin might have facilitated susceptibility to staining.

Although not statistically different, two restorations from the FU group showed slightly rougher surfaces. This might be related to void entrapment during the increment filling technique. Surface texture is important since rougher surfaces would cause surface staining and increase plaque retention and bacterial adhesion. The intrinsic filler particle size of resin composites also determines the smoothness of restoration.

It might have been expected that subjects with bulk fill restorative might have suffered from postoperative sensitivity more than incrementally placed restoratives. However, at the end of the study, none of the patients complained of sensitivity. Adequate depth of cure of the bulk fill resin used in the present study might have contributed to this result. The bulk fill resin TetricEvoCeram Bulk used in the present study contains a new photoinitiator system, Ivocerin, a dibenzoyl germanium compound that has a higher photocuring activity than camphorquinone, as it absorbs visible light over a wider range of wavelengths from 370 to 460 nm.[17,34]

In the present study, two tested restorative resins were used with their respective adhesive systems. The authors decided to compare two different restorative resins as they are optimized by their manufacturer. Marginal adaptation and discoloration might be affected by the adhesive type. However, it might not be accurate to think that the adhesives were the sole responsible factor for the observed difference between the two restorative materials in terms of adaptation and discoloration since both of the adhesive systems used were etch-and-rinse adhesives. On the other hand, their pH values were quite different (Adper Single Bond 2 = 4.1; Excite F = 2.5). The lower acidity of Excite F might contribute to a deeper etching pattern and improved marginal adaptation.

Bravo-scored restorations are considered to be clinically acceptable. A slight lack of marginal adaptation or superficial discoloration does not require further treatment and might be considered negligible. However, degradation between restoration and the tooth interface might continue and cause restoration loss over time. Therefore, clinical research studies with long-term follow-up are needed to confirm the efficacy of bulk fill resin composites.

Moreover, further investigations should be conducted in patients with deleterious/parafunctional habits as well as in other type of cavities that are larger than a slot preparation to support these findings.

CONCLUSIONS

Within the limitations of this study, the 36-month evaluation of a bulk fill restorative resin compared to a nanofill showed better clinical performance with reference to marginal discoloration and marginal adaptation.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Department of Restorative Dentistry, Hacettepe University School of Dentistry. The approval code for this study is 04(KA-15004).

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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Preemptive Use of Naproxen on Tooth Sensitivity Caused by In-Office Bleaching: A Triple-Blind, Crossover, Randomized Clinical Trial

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Clinical Relevance

The administration of single-dose naproxen to prevent tooth sensitivity caused by in-office bleaching had limited effectiveness.

SUMMARY

Objectives: A triple-blind, randomized, crossover clinical trial evaluated prior use of non-steroidal anti-inflammatory naproxen on sensitivity reported by patients undergoing in-office tooth bleaching.

Methods and Materials: Fifty patients were subjected to two sessions of in-office tooth bleaching with 35% hydrogen peroxide in a single application of 40 minutes for two sessions, with an interval of seven days between applications. One hour prior to the procedure, each patient randomly received a single dose of naproxen (500 mg) or placebo. The patient's sensitivity level was evaluated during and immediately after the bleaching using two scales (verbal and visual analog); the verbal scale only was repeated after 24 hours. The effectiveness of the bleaching procedures was evaluated with the Bleachedguide scale. Relative risk to sensitivity was calculated and adjusted by session, while comparison of overall risk was performed by the McNemar test. Data on the sensitivity level for both scales and shade were subjected to the Friedman, Wilcoxon, and Mann-Whitney tests (α=0.05).

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Results: The use of naproxen only decreased the absolute risk and intensity of tooth sensitivity reported immediately after the second session. On the other hand, no measurable effect was observed during or 24 hours after either session. The sequence of drug administration did not affect the bleaching effectiveness.

Conclusions: Preemptive use of naproxen only reduced tooth sensitivity reported by patients immediately after the second session of bleaching.

INTRODUCTION

Tooth bleaching is a noninvasive procedure used with great success to treat teeth with color alteration that compromises esthetics.[1-4] Among the techniques used for bleaching vital teeth, the technique performed in-office is usually indicated for certain clinical conditions, including for patients presenting with a contraindication for using the at-home technique (such as those with gastrointestinal disorders) or those who prefer not to use bleaching trays.[5-7] The in-office techniques are performed using high concentration hydrogen peroxide (H_2O_2) for a shortened exposure time compared to the time needed to perform the technique at home. The H_2O_2 radicals are responsible for whitening the tooth; however, these radicals are also able to diffuse through the dentin and reach the dental pulp, resulting in inflammatory reactions of this tissue.[8-10]

The presence of these components generates oxidative reactions that release inflammatory chemical mediators and induce cellular apoptosis.[11,12] Clinically, these reactions result in tooth sensitivity during and after the bleaching process, which is the main adverse effect associated with this procedure. Clinical trials have reported elevated absolute risk of developing tooth sensitivity during and after in-office bleaching, as more than 90% of patients subjected to the procedure report sensitivity.[2-4] Sensitivity reported by patients is transient and disappears a few hours after the procedure; however, the presence of tooth sensitivity can impair the continuity of treatment and compromise its results.[13,14]

Considering the high rate of tooth sensitivity reported by patients, several approaches have been evaluated that are aimed at reducing the sensitivity caused by dental bleaching, including the use of desensitizing agents[15-18] or antioxidants[19] prior to the bleaching procedure. However, these protocols increase the number of operatory steps, and clinicians prefer to limit the use of time-consuming protocols. The preemptive use of anti-inflammatories has also been attempted, but the drugs that have been evaluated were unable to prevent or reduce postbleaching sensitivity.[20-23] The limited action of these anti-inflammatories on bradykinin and substance P, which are important mediators of pain caused by tooth bleaching,[24,25] can be related to the absence of favorable outcomes in previous trials.[20-23] A common anti-inflammatory drug acting on these mediators is naproxen, which can help control tooth sensitivity.[26] Naproxen sodium is a propionic acid derivative that inhibits the cyclooxygenase pathway, thus preventing the release of inflammatory mediators, such as prostaglandins and bradykinin.[27] Further, naproxen is an effective treatment for postoperative pain when administered in a single dose,[26-28] while this drugs reaches its peak of plasma concentration two to four hours after the oral administration.[29]

The effectiveness of naproxen on bradykinin and its pharmacokinetics motivated this study to evaluate whether this drug may be effective in the prevention of tooth sensitivity caused by in-office bleaching. Thus, this randomized controlled clinical trial aimed to evaluate the effectiveness of preemptive prescription naproxen versus placebo on the absolute risk of postoperative tooth sensitivity (primary outcome) associated with in-office bleaching. The hypothesis was that administrating a single dose of naproxen prior to an in-office bleaching procedure would reduce the absolute risk of postoperative tooth sensitivity. The intensity of tooth sensitivity and color evaluation, both secondary outcomes, were also evaluated.

METHODS AND MATERIALS

This clinical trial was approved by the scientific review committee and by the committee for the protection of human subjects of the local university (CAAE: 37578714.4.0000.5546). The protocol of study was registered at https://clinicaltrials.gov under number NCT02463552 and followed the CONSORT statements.

Study Design

This study was a randomized, triple-blind, placebo-controlled clinical trial with a crossover design. The patients included were submitted to two in-office bleaching sessions, receiving a placebo (control) or naproxen prior to the bleaching procedure, while different treatments were allocated for each session.

A delay of one week between the session ("washout") was established. The study was conducted at the school of dentistry of the local university from November 2014 to July 2015.

Inclusion and Exclusion Criteria

Patients included in the study were at least 18 years old and in good general and oral health. Participants were recruited by advertisements attached to boards located on university buildings. Patients with caries, restoration, severe discoloration (eg, stains caused by tetracycline), enamel hypoplasia, gingival recession, dentin exposure, cracks visible on buccal enamel, pulpitis, or endodontics on any of the six upper anterior teeth were excluded. Participants who had undergone a previous bleaching procedure, presented prior tooth sensitivity, had a known allergy to any component of the medication used in the study, were under continuous use of anti-inflammatory or analgesic drugs, smoked, had parafunctional habits, were using oral removable or fixed orthodontic appliances, or were pregnant or breast-feeding were excluded. The tooth shade of eligible participants was evaluated using the VITA Bleachedguide 3D-MASTER (Vita-Zahnfabrik, Bad Sackingen, Germany) scale, and only the participants presenting all six upper anterior teeth matched to the shade 2.5 M2 or the darker tab of this scale were included. Following color evaluation, a slight airstream was applied over the buccal surfaces of the upper maxillary teeth; patients reporting any tooth sensitivity at this time were excluded.

Sample Size Calculation

The sample size calculation was based on primary outcome data (absolute risk measured immediately after the procedure) from a previous study using a similar bleaching protocol.[20] The calculation was performed for a superiority trial with a binary outcome, considering a power test of 80%, a significance level of 5%, and a decrease of 30% for the experimental treatment compared to the control in a crossover design. Thus, 48 patients were required, and 50 patients were included in the randomization to address the possibility of dropout during the follow-up.

Random Sequence Generation and Allocation Concealment

A randomized list was computer generated by a person not involved in the intervention or evaluation. The participants were defined as a block in the randomization process, and the sequence of treatment (placebo or naproxen) was randomly set for each block by using computer-generated tables. The sequence was inserted into sealed envelopes numbered from 1 to 50 that were opened by the operator only at the moment of the intervention. The patients were numbered according to the sequence of enrollment. Neither the participant nor the operator knew the group allocation determining blinding to the protocol.

Baseline Measurements

Two evaluators were previously calibrated to assess tooth shade using the Bleachedguide scale. The agreement evaluation was performed by analyzing the shade of the upper teeth of six patients not included in the study two times, with a three-day interval between. The kappa coefficient was used to calculate for intraevaluator (evaluator 1=0.80; evaluator 2=1.00) and interevaluator (0.70) agreement. Thus, the calibrated evaluators evaluated the shade of the anterior upper teeth prior to the bleaching procedure based on a match of color between the scale tabs and the middle third of the tooth crown. The shade tabs selected were converted to scores ranging from 1 (whiter shade—0 M1) to 15 (darker shade—5 M3). Considering a possible effect of dental anxiety of sensitivity reported by patients, the Corah Dental Anxiety Scale was used to determine each patient's level of anxiety related to the procedure.[30] Each answer to the survey instrument was scored on a scale from 1 to 5 (four questions), and the sum of the scores was used to determine the level of anxiety: low—lower than 12, moderate—between 12 and 14, and high—higher than 14.

Intervention

Identical capsules containing either 500 mg of naproxen or placebo (inert content) were manufactured by the local university's pharmacy department. The capsules were manufactured by individuals not involved in the intervention or evaluation and were placed into two bottles identified by letters according to the treatment. Neither the researchers responsible for the intervention and evaluation nor the patients knew the content of each capsule. One hour prior to the in-office bleaching procedure, the patient received a single dose of a capsule containing naproxen or placebo. Dental prophylaxis was performed with pumice and water using a rubber cup, and a light-polymerized resin dam (Top Dam, FGM, Joinville, Brazil) was applied over the gingival tissue corresponding to the teeth to

be bleached. A 35% hydrogen-peroxide–based whitening agent (Whiteness HP Blue, FGM) was used according to the manufacturer's recommendation but without the prior application of desensitizing topical to not affect the evaluations of tooth sensitivity. The whitening agent was applied over the buccal surfaces of the teeth and remained in position for 40 minutes, while any bubbles from oxygen release were released with the aid of a disposable microapplicator. After the peroxide removal, the enamel was polished with felt disks. A second session of bleaching was performed after one week, during which time the patient received the opposite treatment (naproxen or placebo) from the one received prior to the first session.

Evaluations

The tooth sensitivity reported by patients was recorded using both a visual analog scale (VAS) and a verbal rating scale (VRS). For the VAS, the patient set his or her sensitivity level by pointing with a pen to the colored 10-cm scale, which ranged from green (no pain) to red (extreme pain). The distance between the marking and the green border of the scale was recorded. Tooth sensitivity was also scored according to VRS, where 0 = none, 1 = mild, 2 = moderate, 3 = considerable, and 4 = severe. Tooth sensitivity was evaluated during the bleaching procedure and immediately after bleaching agent removal. Tooth sensitivity was recorded again 24 hours after the procedure using only the VRS. The VRS defined the presence (score different from 0) or absence of tooth sensitivity in all time assessments. This binary outcome (main outcome) was used to define the risk to tooth sensitivity. A new shade evaluation of the six anterior maxillary teeth was performed one week after each bleaching session. All evaluations were performed by two evaluators blinded to the allocation assignment.

Statistical Analysis

The analysis followed the intention-to-treat protocol and involved all the participants, who were randomly assigned. The statistician was blinded to the study groups. Data from each patient's profile were analyzed regarding distribution of age, gender, and level of anxiety for each sequence of treatment. Age data were analyzed by the Mann-Whitney rank sum test due to the absence of normal distribution (Kolmogorov-Smirnov, $p<0.05$). The proportions of each gender and level of anxiety were analyzed by the Fisher exact and chi-square tests, respectively.

Based on the presence of any tooth sensitivity (scores different from 0 at VRS), the absolute risk, the odds ratio, and the relative risk, as well as confidence intervals (95%), were calculated for each treatment for each evaluation/session of bleaching. For each session, differences in the presence/absence ratios were analyzed by the Fisher exact test. For the overall risk related to each treatment, the odds ratio was adjusted to the independent variable "session of bleaching" using the Mantel-Haenszel statistic. The homogeneity of the odds ratio was analyzed by the Breslow-Day and Tarone tests. Then the estimated odds ratio was converted to relative risk, and the overall presence/absence ratios were analyzed by the McNemar test, considering the study design (crossover).

For both scales of tooth sensitivity evaluation, the data from the scores observed in each evaluation/session of bleaching were subjected to a Mann-Whitney rank sum test. The average scores for each treatment were compared using a Wilcoxon signed rank test.

For color evaluation, comparisons were performed among the sequences of treatment. Changes in the number of shade guide units (ΔSGU) were calculated from baseline to express the color alteration. Data were submitted to two-way repeated measures analysis of variance. All statistical analyses were performed considering a significance level of 95%.

RESULTS

The participants' flow diagram in the different phases of the study is shown in Figure 1. One hundred fifty-two patients were excluded during the enrollment due to presence of prior tooth sensitivity, restorations, teeth previously bleached, or whiter than 2M2 in order to not affect the outcomes measurements. None of the patients discontinued the intervention or presented adverse effects during the intervention. Demographic data from patients included in the study are displayed in Table 1. The age of the included patients presented a median of 23 years and no significant difference was observed between the patients allocated for each sequence of treatment. Both sequences of allocation presented the same distribution as related to gender with a predominance of females (64.0%). For both sequences of treatment, most patients included in the study demonstrated low anxiety (82.0%) to tooth bleaching.

The results of risk to tooth sensitivity are presented in Table 2. The treatment received by

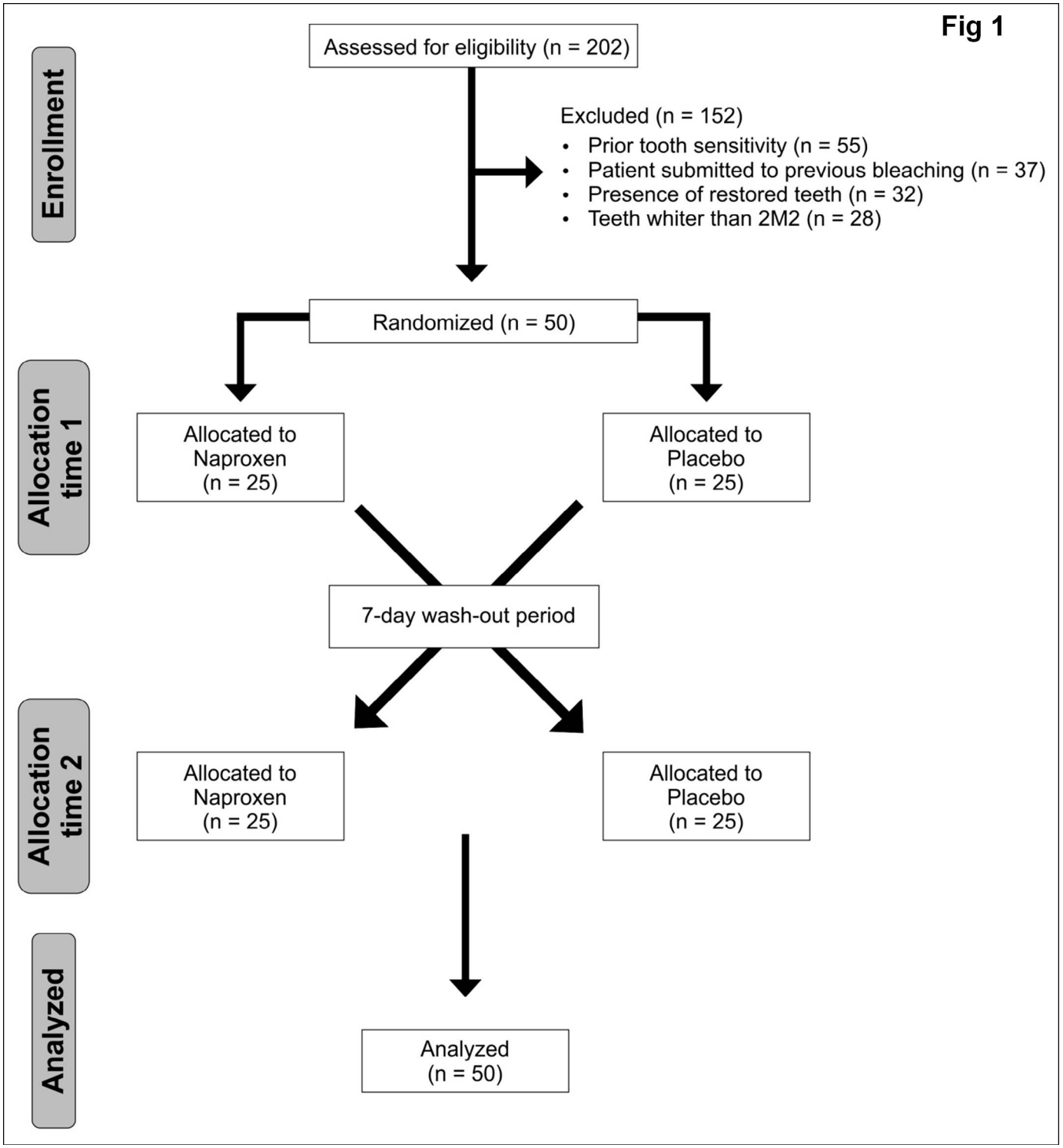

Figure 1. *Flow diagram of the clinical trial.*

the patient did not affect the risk of tooth sensitivity caused by bleaching for the evaluations performed during the procedure or after 24 hours. On the other hand, the use of naproxen reduced the risk of tooth sensitivity in 57% of patients when measured immediately after the second session of bleaching. The same effect was not observed in the first session of tooth bleaching.

Table 1: *Baseline Demographic Characteristics of the Study Population*

Age (y)	Median (First/Third Quartiles)	
Total	23 (21/26)	
Placebo/naproxen	23 (21/26)	$p = 0.883$[a]
Naproxen/placebo	23 (22/26)	
Gender		
Total	Number (%)	
Male	18 (36.0%)	
Female	32 (64.0%)	
Placebo/naproxen		
Male	9 (36.0%)	
Female	16 (64.0%)	$p = 1.000$[b]
Naproxen/placebo		
Male	9 (36.0%)	
Female	16 (64.0%)	
Level of anxiety	Proportion (%)	
Total		
Low anxiety	41 (82.0%)	
Moderate anxiety	6 (12.0%)	
High anxiety	3 (6.0%)	
Placebo/naproxen		
Low anxiety	21 (84.0%)	
Moderate anxiety	3 (12.0%)	
High anxiety	1 (4.0%)	$p = 0.836$[c]
Naproxen/placebo		
Low anxiety	20 (80.0%)	
Moderate anxiety	3 (12.0%)	
High anxiety	2 (8.0%)	

[a] *Mann-Whitney rank sum test.*
[b] *Fisher exact test.*
[c] *Chi-square test.*

Regarding the level of sensitivity, results from the VRS and the VAS are presented in Tables 3 and 4, respectively. At evaluations performed during the tooth bleaching and after 24 hours (only for the VRS), treatment did not affect the level of sensitivity. However, a lower level of tooth sensitivity was observed in patients who received naproxen in the evaluation performed immediately after the second session regardless of the scale used. During the second session, only the VAS demonstrated reduced tooth sensitivity for the patients who received naproxen. On the other hand, no effect of preemptive use of naproxen was observed after the first session.

The results of color evaluation are presented in Table 5. Two-way repeated measures analysis of variance showed a significant effect only for the moment of evaluation ($p<0.001$), whereas the sequence of allocation ($p=0.590$) and the interaction ($p=0.057$) were not significant. Regardless of the sequence of allocation, higher values of ΔSGU were observed after the second session of tooth bleaching.

DISCUSSION

Despite the cytoplasmic extensions of the odontoblasts, dentinal fluid, and enzymes from pulp tissue to hinder peroxide penetration through the pulp chamber, it is common for patients submitted to in-office bleaching to report postoperative tooth sensitivity.[2-4,8,13] This sensitivity is related to inflammatory processes induced by the presence of peroxide and its products in pulp tissue, resulting in a reduction of cell proliferation, metabolism, viability, pulp-reparative capacity, and pain.[11] Inflammatory processes are extremely complex, and several mediators of pain and inflammation are released by host cells in response to specific stimuli. Thus, oxidative stress caused by peroxide penetration into the pulp chamber increases the level of mediators of inflammation such as prostaglandins, bradykinin, and substance P.[24,25,31] Among these mediators, the presence of bradykinin was seen in the inflammatory process in pulp tissue, and this nonapeptide plays a pivotal role in the production of pain.[32]

In the present study, nonsteroidal anti-inflammatory naproxen was preemptively administered to reduce the risk of postoperative tooth sensitivity caused by in-office bleaching. Interestingly, preemptive naproxen administration was only effective in reducing tooth sensitivity (both risk and level) reported at the second bleaching session, mainly the sensitivity measured immediately after the last session. Thus, the hypothesis of study was rejected. One important observation was that only VAS showed a reduction of tooth sensitivity with naproxen when the evaluation was carried out during the second bleaching procedure. Contrary to the VRS, which uses discrete data (score), continuous data are obtained with the VAS, allowing higher sensitivity to measure slight differences.

In addition to peroxide concentration, the number of exposures of pulp tissue to a bleaching agent is closely related to the inflammatory response.[33-35] Therefore, an increased level of inflammatory mediators can be expected after the second session compared to that observed after the first session. It was demonstrated that the action of naproxen over bradykinin depends on the presence of prostaglandins;[27] thus, it was speculated that a higher level of pulp inflammation after the second session increased the effectiveness of naproxen to reduce tooth sensitivity. However, even in the second session, using

Table 2: *Results for Risk to Tooth Sensitivity Observed for Each Treatment*

Session	Moment of Evaluation	During		Immediately After		24 h After	
	Treatment	Naproxen	Placebo	Naproxen	Placebo	Naproxen	Placebo
First session	Presence of sensitivity (yes/no)	14/11	10/15	10/15	9/16	4/21	1/24
	Absolute risk (95% CI)	0.56 (0.37-0.73)	0.40 (0.23-0.59)	0.40 (0.23-0.59)	0.36 (0.20-0.56)	0.16 (0.06-0.35)	0.04 (0.01-0.20)
	Odds ratio (95% CI)	1.91 (0.62-5.88)		1.19 (0.38-3.72)		4.58 (0.47-44.17)	
	Relative risk (95% CI)	1.27 (0.73-2.23)		1.11 (0.55-2.26)		4.00 (0.48-33.33)	
	p-value[a]	0.396		1.000		0.349	
Second session	Presence of sensitivity (yes/no)	9/16	14/11	6/19	14/11	1/24	4/21
	Absolute risk (95% CI)	0.36 (0.20-0.56)	0.56 (0.37-0.73)	0.24 (0.12-0.43)	0.56 (0.37-0.73)	0.04 (0.01-0.20)	0.16 (0.06-0.35)
	Odds ratio (95% CI)	0.44 (0.14-1.38)		0.25 (0.07-0.83)		0.22 (0.02-2.11)	
	Relative risk (95% CI)	0.64 (0.34-1.20)		0.43 (0.20-0.93)		0.25 (0.03-2.08)	
	p-value[a]	0.256		0.042		0.349	
Average	Odds ratio (95% CI)[b]	0.93 (0.43-2.01)		0.56 (0.25-1.25)		1.00 (0.28-3.60)	
	Relative risk[c]	0.96		0.70		1.00	
	p-value[d]	0.789		0.264		0.617	

Abbreviation: CI, confidence interval.
[a] *Fisher exact test.*
[b] *Mantel-Haenszel common odds ratio estimate.*
[c] *Based on odds ratio, estimated.*
[d] *McNemar test.*

preemptive naproxen did not affect the risk of tooth sensitivity reported by patients during the bleaching procedure and after 24 hours, while a reduced effect on sensitivity was observed during the procedure. These outcomes are related to the pharmacokinetics of naproxen after oral administration. Oral naproxen reaches its peak of plasma concentration after two to four hours.[29] Therefore, considering the methodology used in the present study, the plasma concentration of naproxen was reached during or a few minutes after the bleaching procedure. The study design with preemptive treatment administered one hour prior to the bleaching procedure was defined based on clinical protocols to prevent postoperative pain using a single dose of drug.[36,37] However, the findings of the present study suggest that this time delay might not be enough to allow more effective action of some anti-inflammatory drugs on the prevention of trans-operatory pain.

Regarding the absence of difference between the placebo and naproxen treatments after 24 hours, it is important to emphasize that the risk to tooth sensitivity was lower than expected (absolute risk ranging from 5% to 20%) regardless of the treatment. Furthermore, the risk to sensitivity reported during and immediately after the bleaching session was lower than the average observed in prior studies evaluating in-office bleaching procedures (0.63).[38] The same was observed for the level of sensitivity reported in the present study, while prior studies reported an average of 2.8 cm on the VAS.[38] The reduced sensitivity was probably due to the bleaching agent being more alkaline than those used in prior studies[2-4,20] and to the presence of calcium in the agent.[13] Furthermore, the elimination half-life (t½) of naproxen is achieved approximately 18 hours after its administration,[29] while slight effects of a single-dose use can be expected after 24 hours. This

Table 3: *Results (Medians and First and Third Quartiles) for Level of Tooth Sensitivity Observed for Each Treatment Using the Verbal Rating Scale*

Moment of Evaluation	First Session			Second Session			Average		
	Naproxen	Placebo	*p*-Value[a]	Naproxen	Placebo	*p*-Value[a]	Naproxen	Placebo	*p*-Value[b]
During	1.00 (0.00/2.00)	0.00 (0.00/1.00)	0.225	0.00 (0.00/1.00)	1.00 (0.00/1.00)	0.087	0.00 (0.00/1.00)	0.00 (0.00/1.00)	0.833
Immediately after	0.00 (0.00/0.00)	0.00 (0.00/1.00)	0.098	0.00 (0.00/0.00)	1.00 (0.00/1.00)	0.038	0.00 (0.00/1.00)	0.00 (0.00/1.00)	0.261
24 h after	0.00 (0.00/0.00)	0.00 (0.00/0.00)	0.162	0.00 (0.00/0.00)	0.00 (0.00/0.00)	0.162	0.00 (0.00/0.00)	0.00 (0.00/0.00)	1.000

[a] *Mann-Whitney rank sum test.*
[b] *Wilcoxon signed rank test.*

Table 4: *Results (Means [Standard Deviations]) for Level of Tooth Sensitivity Observed for Each Treatment Using the Visual Analog Scale*

Moment of Evaluation	First Session			Second Session			Average		
	Naproxen	Placebo	*p*-Value[a]	Naproxen	Placebo	*p*-Value[a]	Naproxen	Placebo	*p*-Value[b]
During	2.51 (2.09)	1.66 (2.05)	0.108	0.99 (1.01)	1.88 (1.85)	0.018	1.75 (2.11)	1.77 (1.94)	0.587
Immediately after	2.09 (2.19)	2.00 (2.25)	0.406	1.03 (1.54)	2.24 (2.31)	0.004	1.56 (1.95)	2.12 (2.26)	0.039

[a] *Mann-Whitney rank sum test.*
[b] *Wilcoxon signed rank test.*

t½ of naproxen also demonstrated that the one-week washout period used in this study, which is nine times higher than the t½, was enough to avoid any residual effect of the naproxen administered in the first session.

In addition to evaluating tooth sensitivity, color alteration reached with the bleaching procedures was also evaluated. Clinical trials evaluating bleaching procedures commonly expect at least 30 days for color to stabilize prior to final color measurement.[39] A shorter time was used in the present study because the main aim was to evaluate tooth sensitivity reported by patients instead of the effectiveness of the bleaching technique. Thus, the statistical analysis performed for color evaluation did not aim to compare the effect of treatment (naproxen or placebo) on bleaching effectiveness. Instead, the color evaluation aimed to determine whether bleaching procedures performed in patients allocated in both sequences of treatments resulted in tooth bleaching, demonstrating the action of hydrogen peroxide which is also responsible for tooth sensitivity. In fact, the bleaching procedure carried out in the present study resulted in a similar bleaching effect observed as prior studies using the same tool for color evaluation.[4,38,40,41] A recent meta-analysis analyzing data from clinical trials demonstrated an average of 5.3 (±2.8) ΔSGU when in-office tooth bleaching was carried out, which is slightly superior to the color alteration observed in the present study.[38]

Table 5: *Means (Standard Deviations) of ΔSGU[a]*

Sequence of Treatment	After First Session	After Second Session	Pooled Average
Placebo/naproxen	3.9 (1.0)	4.4 (0.9)	4.3 (1.3) a
Naproxen/placebo	3.7 (1.1)	4.9 (1.2)	4.1 (1.0) a
Pooled average	3.8 (1.0) A	4.7 (1.1) B	

Abbreviation: ΔSGU, change in the number of shade guide units.
[a] *For pooled average, different letters indicate significant statistical differences (α=0.05).*

An approximate 57% reduction of tooth sensitivity was observed only after the second session, when a reduction in the intensity level of sensitivity was also reported. This reduction on risk was slightly superior to that observed using a desensitizing gel containing nitrate potassium/sodium fluoride in previous studies (30% to 46%).[16,42] However, overall, administrating a single dose of naproxen prior to a bleaching procedure did not affect risk and level of tooth sensitivity. The crossover design of the present study aimed to reduce bias related to population (age, gender, and so on) in relation to tooth sensitivity. Despite this experimental design eliminating the difference between evaluated treatments, the population studied presented a predominance of young (median age of 23 years) and female patients (64%). Pain threshold differences between genders have been demonstrated, so the outcomes found in the present study could be different if more men were evaluated.[43] Furthermore, outcomes from young patients cannot generalize to a general population of older adults.[38,44] Further studies evaluating different populations or other anti-inflammatories, doses, or administration protocols are required to clarify the evidence about the preemptive administration of anti-inflammatories on tooth sensitivity caused by in-office bleaching.

CONCLUSION

The preemptive administration of naproxen in a single dose one hour prior to an in-office bleaching procedure has limited effect on the risk and level of tooth sensitivity reported by patients and a reduction in sensitivity was only observed immediately after a second session. Moreover, no alteration on bleaching effectiveness was observed by administration of naproxen.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the committee for the protection of human subjects of the Federal University of Sergipe. The approval code for this study is CAAE: 37578714.4.0000.5546.

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

Battery Charge Affects the Stability of Light Intensity from Light-emitting Diode Light-curing Units

A Tongtaksin • C Leevailoj

Clinical Relevance

Partial discharge of the battery may affect the stability of light intensity. Therefore, the light intensity of light-emitting diode (LED) light-curing units with an initially high intensity should be checked regularly.

SUMMARY

This study investigated the influence of battery charge levels on the stability of light-emitting diode (LED) curing-light intensity by measuring the intensity from fully charged through fully discharged batteries. The microhardness of resin composites polymerized by the light-curing units at various battery charge levels was measured. The light intensities of seven fully charged battery LED light-curing units—1) LY-A180, 2) Bluephase, 3) Woodpecker, 4) Demi Plus, 5) Saab II, 6) Elipar S10, and 7) MiniLED—were measured with a radiometer (Kerr) after every 10 uses (20 seconds per use) until the battery was discharged. Ten 2-mm-thick cylindrical specimens of A3 shade nanofilled resin composite (PREMISE, Kerr) were prepared per LED light-curing unit group. Each specimen was irradiated by the fully charged light-curing unit for 20 seconds. The LED light-curing units were then used until the battery charge fell to 50%. Specimens were prepared again as described above. This was repeated again when the light-curing units' battery charge fell to 25% and when the light intensity had decreased to 400 mW/cm^2. The top/bottom surface Knoop hardness ratios of the specimens were determined. The microhardness data were analyzed by one-way analysis of variance with Tukey test at a significance level of 0.05. The Pearson correlation coefficient was used to determine significant correlations between surface hardness and light intensity. We found that the light intensities of the Bluephase, Demi Plus, and Elipar S10 units were stable. The intensity of the MiniLED unit decreased slightly; however, it remained above 400 mW/cm^2. In contrast, the intensities of the LY-A180, Woodpecker, and Saab II units decreased below 400 mW/cm^2. There was also a significant decrease in the surface microhardnesses of the resin composite specimens treated with MiniLED, LY-A180, Woodpecker, and Saab II. In

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conclusion, the light intensity of several LED light-curing units decreased as the battery was discharged, with a coincident reduction in the units' ability to polymerize resin composite. Therefore, the intensity of an LED light-curing unit should be evaluated during the life of its battery charge to ensure that sufficient light intensity is being generated.

INTRODUCTION

Dental patients have become increasingly interested in highly esthetic treatment outcomes. Therefore, the use of light-cured resin composites has grown to meet this demand. A resin composite effects a cure as a result of the chemical reaction between its dimethacrylate resin monomer units, which produces a rigid cross-linked polymer network.[1] The degree of conversion of a resin composite is defined as the percentage of reacted C=C bonds of the monomer molecules. Resin composite requires an optimum degree of conversion to generate good physical and mechanical properties.[2]

Chemical bonding among layers of resin composite relies on copolymerization between new resin monomers and residual unreacted C=C bonds of the monomer molecules.[3] However, an insufficient degree of conversion can result in reductions in mechanical properties, wear resistance, and color stability as well as increased water sorption and secondary caries.[4-7] Ferracane and others[7] showed that a dental composite's resistance to abrasive wear could be improved by increasing its degree of conversion. The factors influencing the degree of conversion include resin composite composition,[8] fillers,[9] type of photoinitiator,[10] temperature,[11] type of light-curing unit, curing time, light guide tip positioning, light wavelength, and light intensity.[12]

By 2007, the use of light-emitting diode (LED) light-curing units had increased because of their many advantages, such as their high light intensity, ease of use, reasonable price, and long life span. Following technological improvements, there are currently many brands of LED light-curing units available. Although each LED light-curing unit brand has different specifications, such as size, weight, and price, the most important factors are the properties of its emitted light. A light-curing unit should produce a light intensity of more than 400 mW/cm^2.[13] Several surveys[14-16] found that not all light-curing units in dental practices produced a sufficient intensity. Indeed, approximately 27% of the light units examined had a light output of only 200 mW/cm^2 or less. It is important that the light intensity remains high and stable to ensure adequate resin composite polymerization. In addition, numerous factors can interfere with efficient functioning of the light-curing unit, such as contamination of the light guide, damage to the fiber optic bundle,[17] reduced light output after repeated sterilizations,[18] and dwindling battery power. However, there are no studies concerning the effect of battery power on the stability of the light intensity of an LED light-curing unit throughout the life of the battery charge.

Table 1: *LED Light-curing Units*

Unit	Manufacturer
LY-A180	Anyang Zongyan Dental Material Co, Ltd, Anyang City, China
Bluephase	Ivoclar Vivadent, Schaan, Liechtenstein
Woodpecker	Guilin Woodpecker Medical Instrument, Guilin, Guangxi, China
Demi plus	Kerr, Orange, CA, USA
Saab II	Foshan Keyuan Medical Equipment Co, Ltd, Foshan, China
Elipar S10	3M ESPE, St Paul, MN, USA
MiniLED	Acteon, Mettmann, Germany

The aim of the present study was to evaluate the stability of the light intensity from different brands of LED light-curing units by measuring the intensity from the point at which the battery was fully charged until the point at which the battery was fully discharged. Furthermore, resin microhardness was used to evaluate the performance of the light intensity during the life of the battery charge.

METHODS AND MATERIALS

Light Intensity Measurement

Seven new different, fully charged LED light-curing units were used in the present study (Table 1). The light intensity was measured 0 mm from the tip of the light guide by means of an LED radiometer (Kerr, Orange, CA, USA). The light guide was positioned over the radiometer sensor, and the light intensity was recorded in mW/cm^2. To represent the clinical situation, the LED light-curing units were used repeatedly for 20 seconds until the battery was fully discharged. The light intensity measurements were obtained after every 10 uses. When the light-curing unit batteries were fully discharged they were fully recharged, and this procedure was repeated for five charging cycles.

Resin Microhardness Measurement

An A3 shade nanofilled resin composite (PREMISE, Kerr) was used in the present study (Table 2). The

Table 2: *Resin-based Composite Material Used in the Study*

Composite	Matrix	Photo Initiator	Particle Size, μm (Mean)	Filler Type
Premise (Kerr, Orange, CA, USA)	Bis-EMA/TEGDMA	CQ/amine	PPF, 30-50	Prepolymerized filler, barium glass, silica filler
			Silica, 0.02	
			Barium, 0.4	

Abbreviations: Bis-EMA, Ethoxylated bisphenol A dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; CQ, camphorquinone; PPF, Prepolymerized filler.

resin composite was placed in a cylindrical stainless-steel mold (4 mm in diameter and 2 mm deep). The resin was covered with a transparent celluloid strip and a glass microscope slide to remove the excess material. Each specimen was irradiated by means of a fully charged light-curing unit for 20 seconds and then stored in the dark for 15 minutes. Ten specimens were prepared for each light-curing unit. The LED light-curing units were then used until the battery charge fell to 50%. Specimens were prepared again as described above. This was repeated again when the light-curing units' battery charge fell to 25% and when the light intensity had decreased to 400 mW/cm^2.

The microhardness measurements were made by means of a universal testing machine (FM-700e TYPE D, FUTURE-TECH, Kawasaki-City, Japan) on the top and bottom surfaces of the resin samples. Three measurements were made per surface, 1 mm from each other for each specimen under a 100 g load for 10 seconds.

Knoop hardness $= F/A = F/Cd^2$ where F = applied load (kgf); A = the unrecovered projected area of indentation (mm^2); d = measured length of long diagonal (mm); and $C = 0.07028$.

The mean Knoop hardness and hardness ratio (hardness ratio = Knoop hardness of the bottom surface/Knoop hardness of the top surface) of the specimens were calculated. The hardness data were analyzed by one-way analysis of variance/Tukey post hoc test at a significance level of 0.05. The Pearson correlation coefficient was used to determine significant correlations between surface hardness and light intensity.

RESULTS

We analyzed the light intensities of the various LED units throughout the life of their respective battery charge (Figure 1; Table 3). The light intensity of the Saab II unit gradually decreased from the start of the charge to approximately 0 mW/cm^2 at the end of the charge. The Woodpecker and LY-A180 units demonstrated light intensities that were stable throughout most of the charge; however, at the end of the charge, their intensities decreased rapidly to nearly 0 mW/cm^2. In contrast, the light intensity of the MiniLED unit at the end of the charge cycle was only slightly lower than at the start. The Bluephase, Demi Plus, and Elipar S10 units produced a stable light intensity throughout the life of their respective batteries.

To identify the effects of changes in light intensity on resin polymerization, we determined the Knoop hardness and hardness ratios of the irradiated samples (Table 4). We observed that the hardness values were coincident with the light intensity produced by the units. In the groups in which the light intensity decreased, the samples' hardness also decreased. When the battery charge decreased to 50%, the top and bottom surface hardnesses of the Saab II group samples were significantly lower than those polymerized with a fully charged battery

Table 3: *Light Intensity of the LED Light-curing Units and the Number of Irradiations*

	Light Intensity (Fully Charged), mW/cm^2	Light Intensity (Discharged Battery), mW/cm^2	No. of Irradiations per Charge (20 seconds/use)
LY-A180	850	200-250	145-165
Bluephase	900	900	80-90
Woodpecker	950	50	595-608
Demi Plus	1000-1200	1000-1200	380-406
Saab II	1200	0	360-390
Elipar S10	1250	1250	240-254
MiniLED	2000	1900-2000	90-104

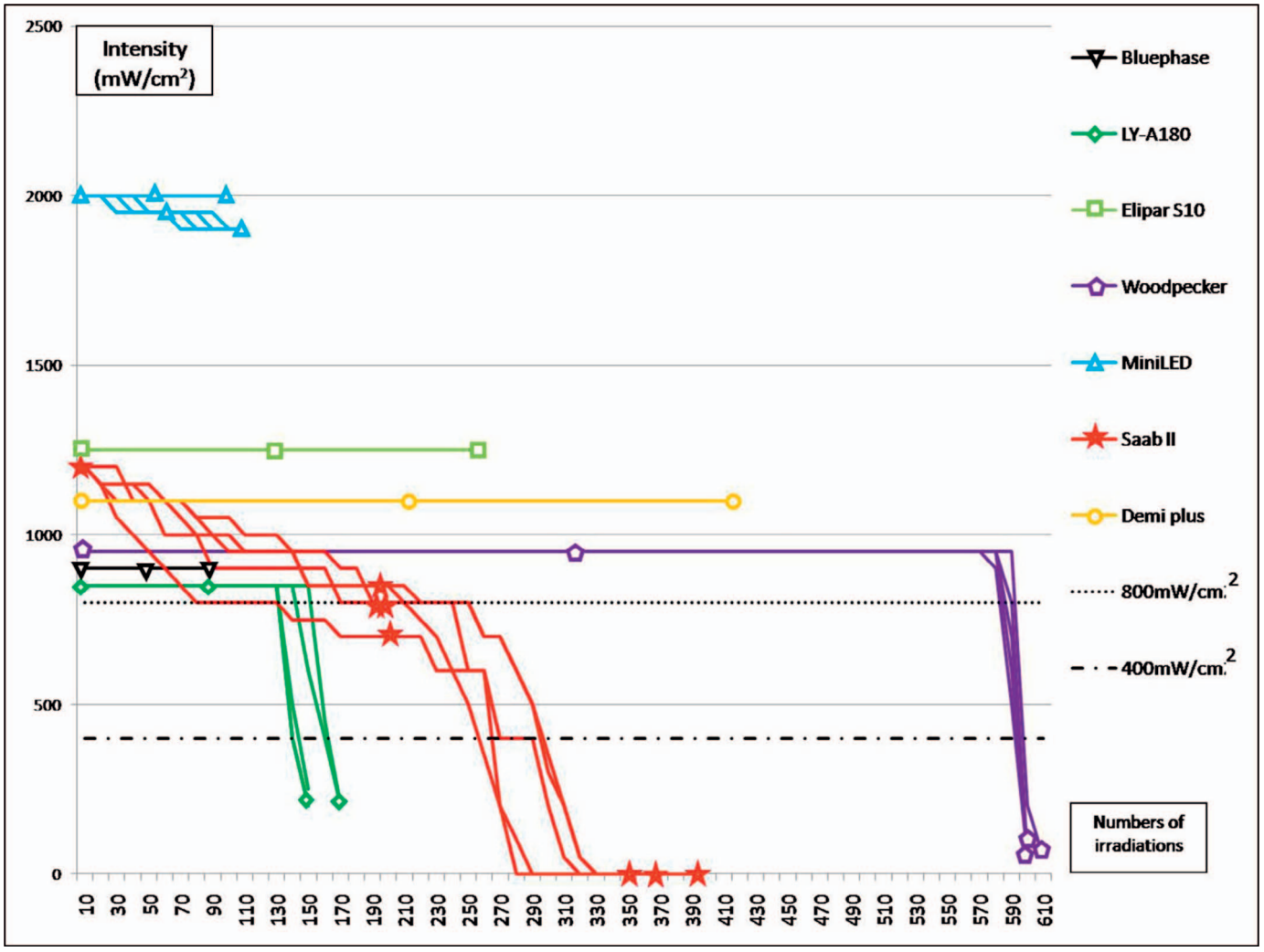

Figure 1. *Light intensity of the LED light-curing units during use.*

($p<0.05$). When 25% of the battery charge remained, the hardnesses of the Woodpecker and the top surface hardnesses of MiniLED group samples were significantly lower than when the batteries were fully charged ($p<0.05$). When the light intensity in the LY-A180, Woodpecker, and Saab II groups became lower than 400 mW/cm^2, the hardnesses of their samples were significantly lower compared with those at 100% charge ($p<0.05$).

The light-curing unit groups all demonstrated hardness ratios greater than 80% when their batteries were fully charged and when they were 50% discharged. However, when only 25% of the battery charge remained, the hardness ratios of the Saab II samples were lower than 80%. Furthermore, when the light intensities of the LY-A180, Woodpecker, and Saab II units decreased to 400 mW/cm^2, the hardness ratios of their samples were lower than 80%.

Results of the Pearson correlation between surface hardness and light intensity revealed that there was a significant direct correlation between top surface hardness and light intensity ($r=0.607$, p[two-tailed]≤ 0.01), and there was a significant direct correlation between bottom surface hardness and light intensity ($r=0.727$, p[two-tailed]≤ 0.01) (Figure 2).

DISCUSSION

The present study examined the light intensity from seven brands of LED light-curing units over the life of the respective battery charge in each unit. According to the manufacturers, the light intensity of these units ranged from 850 to 2000 mW/cm^2. However, we found that during the battery life of some brands the light intensity decreased. This indicates that the battery power level affected the

Table 4: *The Knoop Hardnesses and Hardness Ratios of the Resin Composites during Usage Simulation*

	Battery	100%		50%		25%		400 mW/cm²	
		Hardness	Ratio	Hardness	Ratio	Hardness	Ratio	Hardness	Ratio
LY-A180	Top	45.91[a]	90.59%	46.20[a]	90.97%	45.53[a]	91.15%	40.51[b]	59.76%*
	Bottom	41.59[a]		42.03[a]		41.51[a]		24.21[b]	
Bluephase[R]	Top	44.23[a]	88.04%	43.71[a]	87.08%	43.56[a]	87.75%	-	-
	Bottom	38.94[a]		38.06[a]		38.22[a]		-	
Woodpecker[R]	Top	44.27[a]	92.86%	45.80[a]	89.18%	44.1[b]	87.85%	39.87[c]	71.51%*
	Bottom	41.11[a]		40.84[a]		38.74[b]		28.52[c]	
Demi[TM] plus	Top	45.81[a]	92.68%	46[a]	91.68%	45.67[a]	91.39%	-	-
	Bottom	42.46[a]		42.18[a]		41.74[a]		-	
Saab II	Top	42.61[a]	93.65%	40.06[b]	93.07%	39.36[b]	77.41%*	39.36[b]	77.41%*
	Bottom	39.90[a]		37.29[b]		30.47[c]		30.47[c]	
Elipar[TM] S10	Top	45.06[a]	91.78%	45.43[a]	90.88%	44.28[a]	92.15%	-	-
	Bottom	41.35[a]		41.29[a]		40.80[a]		-	
MiniLED[R]	Top	46.26[a]	93.92%	46.40[a]	92.91%	45.28[b]	94.63%	-	-
	Bottom	43.45[a]		43.17[a]		42.85[a]		-	

(*) indicates a hardness ratio lower than 80%.
Different letters in a row indicate statistically significant differences in hardness ($p<0.05$).

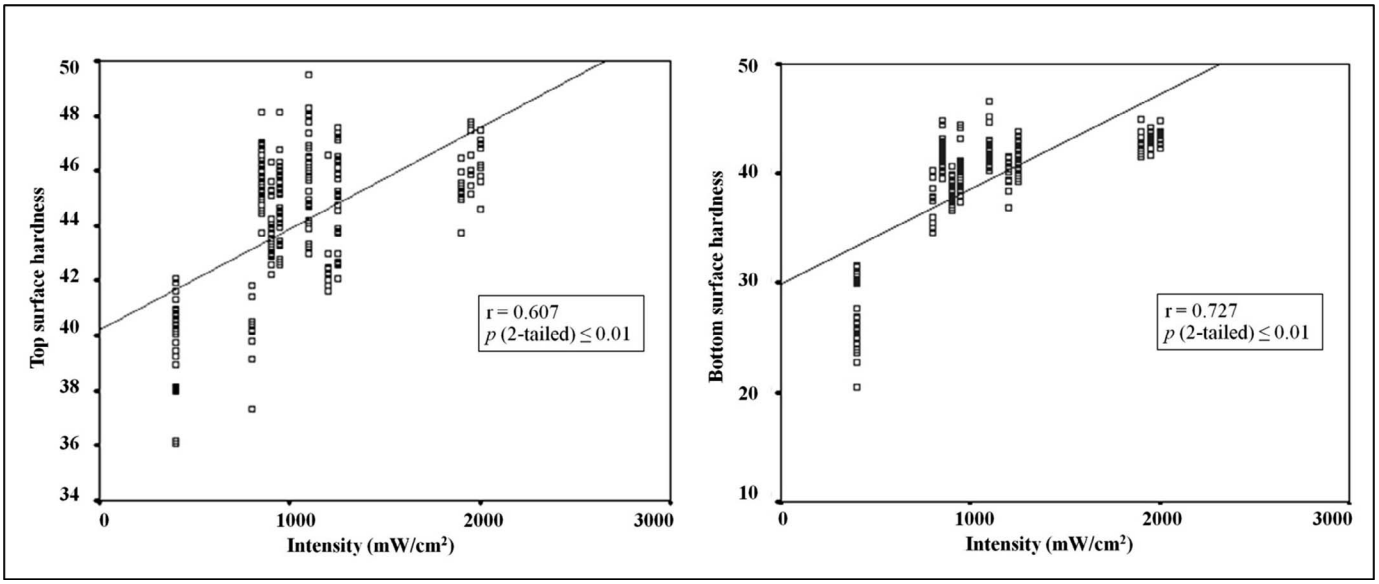

Figure 2. *Pearson correlation between surface hardness and light intensity.*

light intensity of several of the LED light-curing units we evaluated.

It has been reported[13] that the minimum light intensity required for adequate cure of 2 mm of resin composite is approximately 400 mW/cm^2, with an exposure time of 40 seconds. In addition, this study recommended that a light-curing unit with a light intensity lower than 233 mW/cm^2 not be used because this intensity cannot optimally polymerize any depth of resin composite. There are many possible combinations of light energy density (intensity×time) that can be used to achieve an effective resin cure. Most new LED light-curing units produce a light intensity that is greater than 800 mW/cm^2; thus, the irradiation time may be reduced to 20 seconds for a 2-mm-deep resin composite restoration.[19-21] The light intensities of the Saab II, LY-A180, and Woodpecker units all decreased below both 800 and 400 mW/cm^2 as their batteries became discharged. The number of times (20 seconds each) the Saab II unit could be used from the point at which the battery was fully charged until it was fully discharged was approximately 360-390 times/charge. The light intensity was initially 1200 mW/cm^2 and gradually decreased. When the Saab II unit was used 260 times (approximately 70% of the number of irradiations/charge), the light intensity was below 800 mW/cm^2. The Saab II unit was able to be used 110 more times (30% of the number of irradiations/charge) with a decreasingly low intensity that neared 0 mW/cm^2 when the battery was fully discharged, which can affect the degree of polymerization of resin composite.[22] The LY-A180 and Woodpecker units exhibited a stable light intensity of 850 mW/cm^2 and 900 mW/cm^2, respectively, throughout most of their charge. However, at the end of the charge, the light intensity dropped rapidly below 800 mW/cm^2. These units could be used 25 and 20 more times (15% and 3% of the charge, respectively) before their batteries were fully discharged. The MiniLED unit initially produced a very high light intensity of 2000 mW/cm^2 that gradually decreased until the battery was discharged. However, at that point the intensity was at 1900 mW/cm^2, which is higher than the 800 mW/cm^2 considered optimal. We observed that battery power influenced the light intensity output for all of the light-curing units evaluated. We speculate that in some light-curing units on the market, the battery may produce a varied line voltage, which affects the stability of the light intensity output.[23] However, the present study simulated light-curing unit use for only five charging cycles, which is a short portion of the LED lifetime. Further research is required to investigate the light intensity from an LED light-curing unit throughout its lifetime.

Hardness testing is a reliable and commonly used method to evaluate how well a resin has been cured. The Knoop microhardness test is one of the best methods for determining the hardness of resin composites, and a good correlation has been reported[24,25] between the degree of conversion of a resin composite and its Knoop microhardness value. In our study, we found that the hardness values were coincident with the light intensity. The top and bottom surface hardnesses of the Saab II samples at 50% and 25% of the battery charge and at 400 mW/cm^2 were significantly lower than those at 100% charge, and the lowest hardness was found when the light intensity was 400 mW/cm^2. This can be explained by the fact that the light intensity of the Saab II unit gradually decreased throughout its battery charge. In contrast, only the top surface hardnesses of the MiniLED samples decreased at 25% of its battery charge, reflecting its light intensity pattern, which decreased from 2000 mW/cm^2 to 1900 mW/cm^2. Furthermore, the lowest top and bottom surface hardnesses of the LY-A180 and Woodpecker unit samples were observed when their light intensity was 400 mW/cm^2. This is likely because the light intensities of these units were stable at 100%, 50%, and 25% of the battery charge, until at the end of the cycle the light intensities dramatically decreased below 400 mW/cm^2. Based on our results, when the light intensity was higher than 400 mW/cm^2, the Knoop hardness values were still high. In clinical practice, we suggest that the light intensities of LED light-curing units should be more than 400 mW/cm^2 to optimize the degree of conversion and increase the mechanical properties of resin composites.

The degree of polymerization of a resin composite should be the same throughout its depth, and its hardness ratio should be 1 or approximately 1. As the curing light passes through the bulk of the resin composite, its intensity is greatly reduced as a result of light-scattering, thus decreasing curing effectiveness.[26] Light-scattering likely accounted for the differences in hardness between the top and bottom surfaces of our samples. The hardness ratio should be equal to or greater than 80% for visible-light-cured composites.[27] When the light intensities of the LY-A180, Woodpecker, and Saab II units (25% of the battery charge and 400 mW/cm^2 were coincident only for the Saab II unit) were at 400 mW/cm^2, the hardness ratios of their samples were less than 80%. This indicates that an effective cure depth of 2 mm of

the resin composite could not be achieved with 20 seconds of a 400 mW/cm^2 light intensity.

In the present study, the distance from the light guide tip to the resin composite surface was 0 mm, and the direction of the light guide was perpendicular to the surface. However, in clinical practice, there can be hard-to-reach restoration areas requiring polymerization, and it is difficult to position the light guide close to and perpendicular to these surfaces. Thus, the amount of light energy delivered to a restoration under ideal laboratory conditions may be greater than that which can be achieved clinically.[28]

Clinically, it is important to select stable light intensity light-curing units and to monitor light intensity by means of radiometry or manufacturers' light intensity indicators during the LED lifetime. In this study, some LED light-curing units underperformed when the battery charge decreased to 25%. This can be avoided if the dental staff replaces the light-curing units on the charging stand regularly or keeps the battery fully charged.

CONCLUSIONS

The present study demonstrated that the light intensities of some LED light-curing units currently on the market are stable throughout their battery charge, regardless of the remaining battery power. However, there are some LED light-curing units whose light intensity stability is dependent on battery power, which can affect the degree of conversion of a resin composite. Evaluating the light intensity of a LED light-curing unit regularly during its use is recommended, especially as the unit ages.

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 Light-Curing Exposure Time, Shade, and Thickness on the Depth of Cure of Bulk Fill Composites

A Rodriguez • P Yaman • J Dennison • D Garcia

Clinical Relevance

Bulk fill composites can achieve higher depth of cure than conventional composite, but they do not always achieve a clinically adequate depth of cure. Time exposure and shade influence the depth of cure, especially at 4-mm thickness.

SUMMARY

Purpose: To investigate the effect of different light exposure times, shades, and thicknesses on the depth of cure (DOC) of bulk fill composites.

Methods and Materials: Two bulk fill composites, Tetric EvoCeram Bulk Fill (TBF) and Sonic Fill (SF), and a conventional composite, Filtek Supreme Ultra (FSU), were evaluated. Samples (n=10) were made using two different shades (light and dark), thicknesses (2 and 4 mm) and exposure times (20 and 40 seconds). A Tukon 2100B-testing machine was used to obtain three Knoop hardness numbers (KHNs) measured at the top and bottom of each sample, and DOC was calculated as the bottom/top ratio. Statistical analysis was done using a Student *t*-test for comparisons between groups with a Bonferroni correction of $p < 0.004$.

Results: Top hardness values ranged from 79.79 to 85.07 for FSU, 69.49 to 91.65 for SF, and 51.01 to 57.82 for TBF. Bottom KHNs ranged from 23.54 to 73.25 for FSU, 45.74 to 77.12 for SF, and 36.95 to 52.51 for TBF. TBF had the lowest overall KHNs. Light-curing exposure time, shade, and material thickness influenced the DOC in most groups, especially at 4-mm depths. A higher bottom/top ratio was achieved when a 40-second cure was compared to a 20-second cure, when light shades were compared to dark shades, and when 2-mm increments were compared to 4-mm increments.

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INTRODUCTION

Resin-based composites (RBCs) were introduced as restorative materials that would substitute for lost

tooth structures. Since the 1960s, the main components of RBCs have been modified to achieve improved mechanical, biological, and esthetic properties.[1] The filler particles enhance the mechanical properties of RBCs, such as modulus of elasticity, flexural strength, and hardness of the material; alter the thermal expansion behavior; and reduce their dimensional change.[2-5] They also reduce the polymer content and consequently the polymerization shrinkage. There is also improvement in wear resistance, translucency, opalescence, radiopacity, water absorption, intrinsic surface roughness; and polishability as well as enhanced esthetics and handling properties.[3,6] The filler sizes and shapes have been modified through time, and now most RBCs are hybrids, containing micro- and nanofiller particles and filler clusters.[1,3,7,8] According to Lohbauer and others,[9] RBCs should have a filler content of 60wt% to 87wt% to achieve acceptable characteristics.

The most common monomers used in RBCs are the bisphenolglycidyl methacrylate created by Bowen in 1956[10] and urethane dimethacrylate by Foster and Walker in 1974.[11] These are usually diluted with low-viscosity monomers, such as triethylene glycol dimethacrylate and ethylene glycol dimethacrylate.[2,3,12,13] The reaction in which monomers get activated and join together to form polymers is called polymerization.[12,14,15] For light-cured RBCs to polymerize, they require a photoinitiator that has an absorption spectrum that coincides with the output of the light-curing unit (LCU) used.[3,4,16-20]

Depth of cure (DOC) of composites is generally studied to verify that the light-curing technique used for each specific material is appropriate to adequately cure the RBC so that it will have good mechanical properties and optimal conversion of unreacted monomers. In this way, microleakage, which can lead to secondary caries and toxicity of unreacted materials, is minimized. The DOC of composite resins may be affected by the composite photoinitiator, filler type, matrix, shade and translucency, intensity, and spectral output of the curing unit and possibly the technique used when placing the composite.[18]

The DOC of RBCs can be evaluated *in vitro* following standard laboratory tests.[21] Microhardness tests have been shown to be reliable to determine the DOC of RBCs.[22,23] Knoop hardness numbers (KHNs) and Vickers hardness tests have shown strong correlation between them as well as with tests used to determine degree of conversion,[22,23,25,26] whereas the International Organization for Standardization's scrapping method overestimates the DOC of composites.[21-23,26,27]

The bottom-to-top ratio (B/T) of KHNs is a good estimate of the completeness of polymerization at a specific depth from the light source. An RBC is considered adequately cured for clinical use when the microhardness measurements of the bottom are 80% or higher than the measurements at the top of the same sample.[24,26-29,36]

In 1994, Rueggerberg and others[30] determined that for 2-mm- and 3-mm-thick samples, duration, intensity of the light exposure, and their interaction contributed to the degree of conversion of RBCs. They recommended using 60 seconds of light cure with an intensity of at least 400 mW/cm^2 for each 2-mm layer of composite. More recently, in 2013, Ilie and others[31] suggested that when using an LED curing light, a minimum of 20 seconds should be used for a 2-mm layer of composite. Other studies have shown that the DOC of RBCs is material specific[25,26,29,32-37] and that, in general, increasing the light cure exposure time increased the DOC.[30,31,36,38]

The shade of the composite can also affect the DOC. Authors have shown that lighter shades with greater translucency achieve higher DOC than darker shades.[24,32,35]

Recently introduced RBCs can be cured in 4-mm increments (bulk fill) using a short exposure time, according to the manufacturers. Czasch and others[34] showed that bulk fill placement of these composites had a lower degree of conversion only at low polymerization times. The physical properties of several bulk fill composites have been studied for creep deformation,[39] elastic modulus, nanohardness,[40] cuspal deflection, microleakage,[41,42] shrinkage, surface gloss, light sensitivity, and surface wear.[43]

The purpose of this study was to evaluate the effect of different curing light exposure times, composite shades, and material thicknesses on the DOC of bulk fill composites. The null hypothesis was that for each composite material, there was no difference in DOC when light exposure time, composite shade, or sample thickness was varied.

METHODS AND MATERIALS

The materials used in this study are presented in Table 1. Cylindrical composite samples were made using a standardized split brass mold 10 mm in diameter and either 2 or 4 mm thick. The molds were cleaned with 2 × 2 gauze soaked in alcohol and then

Table 1: *Filler Content, Filler Composition, Monomer Composition, Depth of Cure (DOC), and Time Exposure Recommended by the Manufacturers and Shades of RBCs Used in This Study*

Filler Content (%Vol)	Filler Composition	Monomer Composition	DOC	Time Exposure	Shades
Filtek Supreme Ultra (3M ESPE, St Paul, MN, USA)					
63.3%	Silica, zirconia, and aggregated zirconia/silica cluster fillers	Bis-GMA, UDMA, TEGDMA, Bis-EMA, PEGDMA	2 mm	20 s	A1B, A3B
Sonic Fill (Kerr, MNF, Orange, CA, USA)					
69%	Barium, aluminum, boron, silicate	Ethoxylated bisphenol-A-dimethacrylate, bisphenolA-bis (2-hydroxy-3-mehacryloxypropyl) ether	5 mm	20 s	A1, A3
Tetric EvoCeram Bulk Fill (Ivoclar/Vivadent Amherst, NY, USA)					
61%	Barium glass filler, ytterbium trifluoride, mixed oxide	Bis-GMA, Bis-EMA, UDMA	4 mm	10 s	IVA, AVB

Abbreviations: Bis-EMA, bisphenolglycidyl ethyl-methacrylate; Bis-GMA, bisphenolglycidyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

lubricated with mold-releasing agent (Al-Cote, Dentsply, Woodbridge, ON, Canada). The composites were inserted into the brass molds, and excess was removed with a metal spatula. The material was covered with a clear Mylar matrix, and a 1-mm-thick glass slide was pressed and stabilized with clips. The assembled samples were light cured with the Bluephase LED curing light (Ivoclar/Vivadent Amherst, NY, USA) using a low-power setting (583 mW/cm^2) as measured with a radiometer (Cure Rite Radiometer, Efos Inc, Mississauga, ON, Canada) during the study.

Ten samples for each of the selected shades (A1 and A3 for Filtek Supreme Ultra and SonicFill and IVA and IVB for Tetric EvoCeram) were light cured with each of the different time exposures (20 and 40 seconds). Once light cured, the glass slide was removed, and the samples were pushed out of the mold with finger pressure. A total of 240 samples were fabricated.

The samples were finished with a 600-grit silicon carbide paper (Mager Scientific, Dexter, MI, USA) on a rotary grinder-polisher (Micro Star 2000, Inc, Concord, ON, Canada) at a rotation speed of 102 rpm for one minute and then polished for an additional three minutes with moderate finger pressure with 800-grit silicon carbide paper (Mager Scientific). All samples were rinsed with water, dried, and stored in a manila envelope in a dark drawer at room temperature for 24 hours.

A Tukon 2100B-testing machine (Wilson Instrument, Norwood, MA, USA) was calibrated for microhardness testing. The Knoop diamond indenting tool was used to apply a 200-g load on the stabilized samples with a dwell time of 15 seconds. Three measurements were recorded for both the top and the bottom surfaces of each sample. The indentations were measured using 20× magnification by positioning the cursors on both ends of the diagonal and measuring the length. The Tukon 2100B-testing machine gives the (KHNs), which are calculated using the formula

$$HK = L/I^2C_P,$$

where L represents the load applied (kg), I is the length of the long diagonal indentation (mm), and C_p is a constant to the projected area of the indentation (0.07028). The average of the three KHN readings on each surface were calculated and entered as a single data point to calculate the B/T ratio of each sample. Statistical analysis was done using a *t*-test for multiple comparisons between groups with a Bonferroni correction of $p < 0.004$.

RESULTS

Two light-curing exposure times, 20 and 40 seconds, and two shades were evaluated at 2- and 4-mm thicknesses to determine the effect on the DOC of one control and two bulk fill composites. Shade A1 for Filtek Supreme Ultra (FSU) and Sonic Fill (SF) and IVA for Tetric EvoCeram Bulk Fill (TBF) will be referred to as light, or "L," and shade A3 for FSU and SF and IVB for TBF as dark, or "D." The averaged top and bottom hardness for the three materials at the different times and shades are shown in Table 2. TBF had the lowest overall hardness numbers compared to the other materials, but this is related primarily to composition.

Table 2: *Bottom and Top Means and Standard Deviations (SD) of Knoop Hardness Numbers of Different Resin-Based Composites*

	2 mm L		2 mm D		4 mm L		4 mm D	
	20 s Mean (SD)	40 s Mean (SD)	20 s Mean (SD)	40 s Mean (SD)	20 s Mean (SD)	40 s Mean (SD)	20 s Mean (SD)	40 s Mean (SD)
Filtek Supreme Ultra								
Top	82.00 (0.72)	79.79 (0.67)	83.23 (2.18)	81.04 (4.62)	85.07 (4.27)	83.09 (1.71)	84.23 (2.65)	84.03 (1.49)
Bottom	73.25 (0.73)	72.56 (0.92)	58.55 (4.72)	70.94 (4.45)	39.99 (5.68)	55.58 (4.36)	23.54 (2.94)	46.00 (4.39)
SonicFill								
Top	82.77 (3.26)	91.65 (2.85)	69.49 (3.59)	75.94 (1.48)	72.57 (2.86)	81.17 (1.28)	74.28 (1.74)	74.53 (2.02)
Bottom	74.49 (2.18)	77.12 (2.61)	62.32 (1.67)	70.81 (2.05)	59.18 (4.70)	71.77 (2.84)	45.74 (3.83)	60.18 (2.19)
Tetric EvoCeram Bulk Fill								
Top	55.95 (0.88)	57.79 (0.57)	57.82 (0.73)	53.72 (0.69)	51.01 (1.76)	54.85 (1.97)	56.14 (1.29)	52.66 (1.90)
Bottom	47.99 (1.41)	52.51 (0.38)	50.40 (1.04)	48.94 (1.05)	36.95 (2.34)	45.64 (2.63)	36.95 (2.93)	39.16 (1.73)

Exposure time comparisons are shown in Table 3. For 2-mm-thick samples, the exposure time did not have a significant influence on the DOC except for light shade SF and dark shade FSU. At 4-mm thickness, all materials had a statistically significant higher DOC when cured for 40 seconds compared to 20 seconds.

The different shades of composites (Table 4) did not have a significant influence in 2-mm-thick samples except for FSU at 20-second and SF at 40-second exposure times. When 4-mm-thick samples were evaluated, the light shade for all materials had a statistically significant higher DOC compared to the dark shade.

In general, 2-mm-thick samples achieve a higher DOC than 4-mm-thick samples (Table 5). At 2-mm thickness, only the dark shade FSU samples did not achieve an adequate DOC when cured for 20 seconds (.70). For light shades at 4 mm, only SF (.82) had an average DOC higher than 80% cure after 20 seconds of exposure, but both SF (.88) and TBF (.83) had 80% cure after 40 seconds. For a darker shade at 4 mm, only SF (.81) had 80% DOC after 40 seconds of exposure.

DISCUSSION

This study measured top and bottom KHNs to calculate the DOC for two bulk fill composites and a conventional composite by evaluating different light-curing exposure times, shades, and thicknesses.

All top KHNs were higher than their corresponding bottom hardness for all samples (Table 2). As mentioned in previous studies, the hardness of the RBC materials decreases when the thickness of the material increases.[22,26,35,37] In the present study, top and bottom hardness values were material specific (Table 2), coinciding with results of other studies.[25,26,29,32-37] FSU had the smallest range of top KHN (5.27) followed by TBF (6.80). SF had a wider range of top KHN (22.15) and also had the highest top surface value of all groups (91.65). In general, TBF had the lowest top KHN (51.01). The wide range of top KHNs of SF might be related to its surface characteristics, which looked grainy and dark under the microscope, making it difficult to read the Knoop

Table 3: *Depth-of-Cure B/T Ratios of Groups Comparing Exposure Times; Mean (Standard Deviation) of Filtek Supreme Ultra (FSU), SonicFill (SF), and Tetric EvoCeram Bulk Fill (TBF)*

	2mm L		p value	2mm D		p value	4mm L		p value	4mm D		p value
	20s	40s		20s	40s		20s	40s		20s	40s	
FSU	0.89 (0.01)	0.91 (0.01)	1	0.7 (0.07)	0.88 (0.08)	<0.001	0.47 (0.08)	0.67 (0.06)	<0.001	0.28 (0.04)	0.55 (0.06)	<0.001
SF	0.90 (0.03)	0.84 (0.04)	0.003	0.90 (0.04)	0.93 (0.03)	0.128	0.82 (0.06)	0.88 (0.04)	0.003	0.62 (0.05)	0.81 (0.04)	<0.001
TBF	0.86 (0.02)	0.91 (0.01)	0.045	0.87 (0.02)	0.91 (0.02)	0.611	0.73 (0.05)	0.83 (0.05)	<0.001	0.66 (0.05)	0.74 (0.04)	<0.001

Table 4: *Depth-of-Cure B/T Ratios of Groups Comparing Shades; Mean (Standard Deviation) of Filtek Supreme Ultra (FSU), SonicFill (SF), and Tetric EvoCeram Bulk Fill (TBF)*

	2mm 20s		p value	2mm 40s		p value	4mm 20s		p value	4mm 40s		p value
	L	D		L	D		L	D		L	D	
FSU	0.89 (0.01)	0.7 (0.07)	<0.001	0.91 (0.01)	0.88 (0.08)	0.261	0.47 (0.08)	0.28 (0.04)	<0.001	0.67 (0.06)	0.55 (0.06)	<0.001
SF	0.90 (0.03)	0.90 (0.04)	0.61	0.84 (0.04)	0.93 (0.03)	<0.001	0.82 (0.06)	0.62 (0.05)	<0.001	0.88 (0.04)	0.81 (0.04)	<0.001
TBF	0.86 (0.02)	0.87 (0.02)	0.13	0.91 (0.01)	0.91 (0.02)	1	0.73 (0.05)	0.66 (0.05)	0.001	0.83 (0.05)	0.74 (0.04)	0.003

hardness indentations. The surface appearance might be related to the ultrasonic vibration by the hand piece used to apply the composite and the higher filler content. The different hardness between materials might be related to their filler content by volume SF (69%), FSU (63.3%), and TBF (61%) as well their components (Table 1). None of the materials had KHNs comparable to the hardness of enamel as verified by Cardoso and others[44] (253±26 to 478±39 KHN).

It is possible that the sizes, radiopacity, translucency, and pigments of these filler particles influenced the light transmission through the material, affecting the DOC.[31] The combination of monomers used in the three materials was also different, which, according to some studies, can influence the material properties.[3,13,16-19]

The photoinitiators used are not specified by the manufacturers, and the DOC and other properties of a material depend on the photoinitiator's emission spectrum coinciding with the light output of the LCU.[3,4,16-20] A slight mismatch in the wavelength of the light source and the sensitivity of the photoinitiator could limit the ability to successfully maintain free radicals, which are responsible for the polymerization process.[14,15]

Exposure Time

Some authors have found that when increasing the thickness of the samples, the exposure time should also increase to achieve a higher DOC.[22,30,33,36-38] In general, the materials studied showed that the exposure time had a greater effect on the DOC of 4-mm samples compared to 2-mm samples (Table 3).

FSU was used as the control in this study. The manufacturers recommend light curing "body" (B) shades in 2-mm increments for 20 seconds. In the present study, 2-mm increments of A1B shade reached an adequate DOC when light cured for 20 seconds (0.89). This outcome was comparable to the results of the study done by Ilie and others,[1] who suggested that a minimum of 20 seconds of light-curing exposure time should be applied to a 2-mm increment of material light cured with an LED LCU. According to the present study, 2-mm samples of A3B shade required a higher exposure time (40 seconds) to obtain a B/T ratio higher than 0.80 (0.88).

Table 5: *Depth-of-Cure B/T Ratios of Groups Comparing Thickness; Mean (Standard Deviation) of Filtek Supreme Ultra (FSU), SonicFill (SF), and Tetric EvoCeram Bulk Fill (TBF)*

	L 20s		p value	D 20s		p value	L 40s		p value	D 40s		p value
	2mm	4mm		2mm	4mm		2mm	4mm		2mm	4mm	
FSU	0.89 (0.01)	0.47 (0.08)	<0.001	0.7 (0.07)	0.28 (0.04)	<0.001	0.91 (0.01)	0.67 (0.06)	<0.001	0.88 (0.08)	0.55 (0.06)	<0.001
SF	0.90 (0.03)	0.82 (0.06)	0.001	0.90 (0.04)	0.62 (0.05)	<0.001	0.84 (0.04)	0.88 (0.04)	0.012	0.93 (0.03)	0.81 (0.04)	<0.001
TBF	0.86 (0.02)	0.73 (0.05)	<0.001	0.87 (0.02)	0.66 (0.05)	<0.001	0.91 (0.01)	0.83 (0.05)	<0.001	0.91 (0.02)	0.74 (0.04)	<0.001

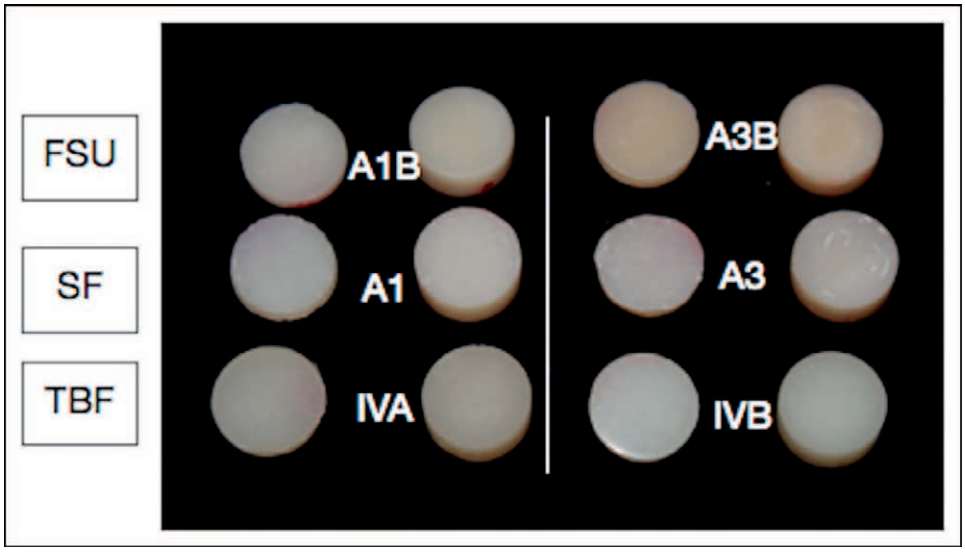

Figure 1. *Samples of light and dark shades of the composites illustrating variation in shade as related to thickness of the samples; 2 mm (left) and 4mm (right).*

SF samples of 2-mm-thickness shade A1 had a higher DOC when cured for 20 seconds (0.90) compared to 40 seconds (0.84). The statistical difference between these groups was significant (p=0.003, which is very close to the Bonferroni correction value of p<0.004). If the sample size would have been larger, there is a possibility of not finding any difference between light curing for 20 or 40 seconds for 2-mm-thick light shade samples. In 4-mm-thick samples, increasing the light exposure time increased the DOC of samples evaluated (0.82 to 0.88), as has been stated in other studies.[22,30,33,36-38]

The effect of time exposure on DOC of TBF was similar to FSU. Two-millimeter samples of either IVA or IVB shade showed an adequate DOC when light cured for 20 seconds. When thickness was increased to 4 mm, samples exposed to a 40-second light cure showed significantly higher DOC than samples cured for 20 seconds, but only the light shade had a value above 0.80 (0.83).

Shade

The effect of shade has shown that lighter shades reach a higher DOC than darker shades (Table 4). Koupis and others[35] compared shades A2 and A4 light cured for 40 seconds, finding higher DOC on A2 samples. Moore and others[24] compared shades B1, A3, and D3 and also found higher DOC for the lightest shade (B1). In the present study, FSU had a higher DOC when A1B shade was compared to A3B, except in the 2-mm samples light cured for 40 seconds, where there was no statistically significant difference. When the shade of the A3B FSU composite is compared to other dark shade samples, it can be noticed that FSU has a higher chroma, possibly containing more pigments or less translucency, which leads to a lower DOC at higher depths compared to other materials (Figure 1).

The comparisons of shade for SF showed that there was no influence of shade on 2-mm-thick samples light cured for 20 seconds, whereas 2-mm-thick samples light cured for 40 seconds showed a significantly higher DOC for A3 shade (0.93) compared to A1 (0.84). A similar result was found by Lazarchik and others,[32] who established that at certain thicknesses, 2-mm-thick samples of shade A4 had a tendency to be harder than the A1 shade. There is a possibility that the darker shade had a higher concentration of photoinitiators that would be activated at decreased depths (2 mm), or it is more translucent to compensate for the pigmentation.[32] As seen in Figure 1, the A3 shade of SF was different from the A3B shade of FSU. Light shade samples of 4-mm depths light cured for either 20 or 40 seconds had a higher DOC than the dark shade groups; this has also been shown in previous studies.[24,32,35]

At 2-mm depths, the shades of TBF did not influence the DOC. For 4-mm-deep groups, IVA shade reached significantly higher DOC than IVB.

Thickness

All the materials had significantly lower DOC for 4-mm samples compared to 2-mm samples, except for SF shade A1 cured for 40 seconds, which had no difference in DOC between thicknesses (Table 5). Other studies have also established that DOC decreases when material thickness increases.[22,26,35,37]

FSU did not reach an adequate DOC for 2-mm shade A3 samples cured for 20 seconds (0.47) or for any 4-mm-thick sample. One of the reasons for this can be attributed to the composition of the FSU composite, which has microfilled clusters that can increase the light dispersion, as mentioned by DeWald and others.[23] Koupis and others[35] also stated that the filler composition of RBCs influences their translucency, hardness values, and DOC.

According to the manufacturer, SF should reach a 5-mm DOC of more than 86% when light cured for 20 seconds with a light output of 550 mW/cm^2. The manufacturer does not mention the mechanism and/or components that this material uses to achieve a higher DOC compared to conventional composites. All SF 2-mm samples had a higher DOC than the 4-mm samples, except for the light shade cured for 40 seconds, where the DOC of 2- and 4-mm thickness was statistically similar. Most likely, the translucency of the light shade composite and its photoinitiator and monomer compositions permit the light to activate the polymerization process throughout a 4-mm increment of material when light curing for a 40-seconds time period.

For SF, all the groups evaluated had a mean B/T ratio higher than 0.80, except 4-mm samples of A3 shade cured for 20 seconds (0.62). From the groups that achieved a mean DOC of 0.80, the 2-mm A1 shade cured for 40 seconds, 4-mm A1 shade cured for 20 seconds, and 4-mm A3 shade cured for 40 seconds had around 25% of the samples below a B/T ratio of 0.80. This shows that this material does not always achieve an adequate DOC even if it is light cured for 40 seconds. With the results of this study, it is difficult to predict a DOC of 5 mm when using SF as recommended by the manufacturer.

TBF is advertised to achieve a DOC of 4 mm when light cured with an LED LCU $\geq$1000 mW/cm^2 for 10 seconds. In the present study, a light output much lower than the recommended one was used (583 mW/cm^2) for a longer period of time (20 or 40 seconds). The manufacturers claim that this material has an additional photoinitiator (polymerization "booster") that ensures a long working time. The exact properties of this photoinitiator that may influence the properties of the composite are unknown. All 2-mm-thick samples of TBF reached an adequate DOC above a B/T ratio of 0.80. From the 4-mm-thick samples, only the IVA shade cured for 40 seconds achieved an acceptable DOC (0.83), although 25% of these samples were below the 0.80 B/T ratio.

CONCLUSIONS

- At greater thickness (4 mm), a longer exposure time increases the DOC for all materials tested.
- Shade had a greater influence at increased depths, showing higher DOC for light shades compared to dark shades.
- Bulk fill composites achieved the highest DOC. At 4-mm thickness, all samples of only SF A1 shade samples achieved a DOC higher than 0.80. SF A3 shade and TBF IVA shade samples achieved an average DOC above 0.80, but about 25% of the samples did not reach that threshold.

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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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Short- and Long-Term Bond Strength Between Resin Cement and Glass-Ceramic Using a Silane-Containing Universal Adhesive

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Clinical Relevance

Use of various "silane solutions" provides different bond strength values and patterns of failure according to subjection to short- or long-term water storage. Use of an adhesive layer following a separate silane application may improve long-term clinical ceramic/resin cement bonding performance.

SUMMARY

This study aimed to evaluate the effect of various silane-containing solutions on bonding between resin cement and glass ceramic after 24 hours and after six months of water storage. Glass-ceramic plaques (IPS e.max CAD) were sandblasted with aluminum oxide, etched with 10% hydrofluoric acid (HF), and divided into five "silane treatment" groups: RelyX Ceramic Primer (RCP), RelyX Ceramic Primer and Single Bond Plus (RCP+SB), Scotchbond Universal (SBU), Clearfil Ceramic Primer (CP), and no solution (HF-only control). Each group was divided into two "storage time" subgroups: 24 hours or six months in 37°C water. Eighteen resin cement cylinders (RelyX Ultimate) were bonded to each treatment group substrate (n=18) and then subjected to microshear testing. Failure mode was analyzed using scanning electron microscopy. Debond data were analyzed using a two-way analysis of variance and the Tukey *post hoc* test (α=0.05) as well as Weibull distributions. The factors "silane treatment," "storage time," ($p<0.0001$), and their interaction were statistically significant ($p<0.0010$). Group means (MPa±SD), RCP (24 hours: 27.2±3.1; six months: 18.0±4.9), and HF-only control (24 hours: 21.1±3.4; six months: 15.7±5.8) showed a reduced bond strength after six months of water storage, while RCP + SB (24 hours: 23.4±4.4; six months: 22.2±5.4), SBU (24 hours: 18.8±3.0; six months: 17.2±3.6), and CP (24 hours: 21.7±4.3; six months: 17.4±4.8) remained

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constant. Weibull analysis revealed that more reliable bond strengths were obtained after six months for SBU and RCP + SB. Evaluation by scanning electron microscopy revealed that all groups demonstrated hydrolytic degradation at six months of storage, but RCP + SB and SBU indicated less. Use of a separate application of silane and adhesive system improved short and long-term ceramic/resin cement bond strength.

INTRODUCTION

In recent years, all-ceramic restorations have been highly utilized in dentistry because of their biocompatibility, high mechanical properties, and optimal esthetics.[1] However, in order to bond inorganic restorative materials to organic tooth tissues, surface preparation and application of coupling agents[2] to both dental tissues[3] and ceramic material are essential.[4] Also, the presence of an intermediate material, such as resin cement, can improve the overall mechanical behavior of the restoration.[5,6] All of these factors need to be considered to ensure adequate, long-lasting bonding with optimal clinical performance.[7]

Two main strategies are employed to enhance resin cement/glass-ceramic bonding: mechanical and chemical. Silanes provide chemical adhesion to silica-containing ceramic substrates[5] and are often coupled with the use of acid etchants on ceramic surfaces (hydrofluoric acid [HF]) in order to dissolve part of the glassy phase, facilitating mechanical interlocking of the resin cement.[4,5,8,9] This combination of both mechanical and chemical strategies is currently the most accepted procedure for enhancing resin cement/glass-ceramic bonding.[2,4,5]

Silane coupling agents have been applied as adhesion promoters in dentistry since 1977.[10] These silane primer molecules contain two different, reactive functional groups: one end reacting with methacrylates and the other reactive toward silica in glassy structures. This latter group can exist as a nonfunctional silane (containing alkoxy [-OR] groups) that, after hydrolysis, reacts with inorganic substrates[2] in order to achieve not only a linear but also a three dimensional, cross-linked network between ceramic and resin.[11-13] The alkoxy groups of silane-coupling agents must be activated by a hydrolysis process (SiOR→SiOH) to be able to bond to silica in glass ceramic through OH groups, following a condensation step in which water is released.[2]

Other methods can also be used to improve ceramic-resin bonding, such as airborne-particle abrasion with 50-μm aluminum oxide[5,14] and employment of primers containing silane mixed with phosphate acid monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP). These phosphate acid monomers are used because of their ability to chelate bond with metallic cations present in some ceramics (mainly the polycrystalline ceramics) as well as in hydroxyapatite while having the ability to covalently bond with methacrylate groups of resin cements.[14,15] Thus, when silane and a phosphate acid monomer are combined in a ceramic priming agent, a "universal ceramic primer" is created because it has the potential to chemically bond to a high number of restorative and tooth substrates.

Thus, these phosphate acid monomers are also found in some enamel-dentin adhesives (self-etch or multimode) to act as coupling agents between composite resin and calcium present in hydroxyapatite of dental tissues.[16] Silane is incorporated in those adhesives, providing a class of materials called "universal bonding solutions," and are to be used with enamel, dentin, ceramic, and metal substrates. Because silane and MDP are incorporated into those adhesive compositions, these products may also be indicated to enhance bonding between resin cements and glass and nonglass ceramics. Therefore, these universal systems have a great potential for providing adhesion to a wide variety of substrates and for simplification of many dental procedures through use of only one product for many clinical purposes.[17]

Manufacturers recommend this broad range of "silane primers" to be employed indistinctly (whether silane is mixed with other components or not) when performing glass-ceramic pretreatment. To date, there is no evidence comparing the effect of these different kinds of silane primers (particularly the silane-containing, multimode adhesives with conventional silane-only primers) on glass-ceramic/resin cement bonding after short- and long-term storage.

Thus, the purpose of this *in vitro* study was to evaluate the effect of a silane-containing, universal adhesive on the microshear bond strength between resin cement and glass ceramic after 24 hours and after six months of water storage. The null hypotheses tested were 1) that there are no significant differences in ceramic/resin cement bond strengths provided by the different silane treatments employed and 2) that there are no significant differences in

ceramic-resin cement bond strengths after 24 hours and after six months of water aging.

METHODS AND MATERIALS

Specimen Preparation and Group Division

One hundred and eighty sintered plaques of lithium disilicate glass ceramic (IPS e-max lithium disilicate CAD/CAM, A2 color, Ivoclar, Vivadent, Schaan, Liechtenstein), measuring 3 ± 0.1 mm in length, 3 ± 0.1 mm in width, and 2 ± 0.1 mm in thickness, were prepared. Specimens were sandblasted with aluminum oxide (50 μm; Bio-Art, São Carlos, Brazil) at 2 bar pressure for five seconds on only one surface at a working distance of 5 mm in order to standardize ceramic surface roughness after the milling process. Specimens were ultrasonically cleaned for five minutes in distilled water and air-dried. Surfaces were then etched with HF (10% hydrofluoric acid gel, Porcelain Conditioner Dentsply, Petropolis, Brazil) for 20 seconds.[18] A previously perforated, double-faced adhesive tape (having circular perforations measuring the same as the resin cement cylinders to be built) was placed on each ceramic surface in order to delineate the ceramic/resin cement bonding area (one cylinder per ceramic plaque).

A variety of manufacturer-recommended silane treatment protocols for the ceramic surfaces were formed among five groups: 1) RelyX Ceramic Primer (3M ESPE, St Paul, MN, USA) consisted of a single silane primer (RCP) and was applied actively onto the ceramic surface for 60 seconds, followed by thorough drying using oil-free air until complete solvent evaporation (20 seconds); 2) RelyX Ceramic Primer was also applied actively for 60 seconds and thoroughly dried, and then an adhesive system (Adper Single Bond Plus, 3M ESPE) was applied in one coat for 15 seconds, air-dried for five seconds, and left unpolymerized in order to be light cured together with the resin cement (RCP+SB); 3) Scotchbond Universal (SBU), a "silane-containing," universal adhesive system (3M ESPE), was actively applied for 20 seconds, air-dried for five seconds, and also left unpolymerized; 4) Clearfil Ceramic Primer (CP), containing both silane and MDP (Kuraray Noritake Inc, Okayama, Japan), was applied to the ceramic surface in one coat and air-dried until solvent evaporation (20 seconds); and 5) a control, HF-only treatment group (HF-control), having no silane treatment, using only the previously HF-etched ceramic surfaces. Materials used are described in Table 1.

Thirty-six resin cement cylinders (RelyX Ultimate, 3M ESPE) (1±0.1 mm in diameter and 1±0.1 mm in height) were built up on treated ceramic surfaces for each group (one specimen was bonded to only a single ceramic plaque) with the aid of a silicon mold containing a single circled compartment per plaque. Resin cement was injected onto the mold using the manufacturers' auto-mixing tip, without separating the tip from the dispensed mass, until filling the compartment completely. A half-millimeter-thick glass slide was then placed over the top of the mold, and each cylinder was exposed for 40 seconds (Optilight Max, Gnatus, Brazil; light output: 600 mW/cm^2). The ceramic-plaque/resin-cement assemblies were removed from the molds after five minutes, water rinsed, and dried. All groups were randomly divided into two subgroups (n=18) for testing the effect of water storage duration at 37°C: 1) 24 hours and 2) six months.

Microshear Bond Strength Test and Statistical Analysis

After each storage time, specimens were dried and attached to a holding device using cyanoacrylate cement (420 Super Bonder Instant Adhesive, Loctite, Henkel Corp, Westlake, OH, USA) and placed on the platen of a universal testing machine (Instron 4411, Instron Corp, Canton, MA, USA) to perform microshear bond testing (μSBS), following methodology developed by Shimada and others.[19] An uplifting shear load was applied to the extreme base of each resin cement cylinder at a crosshead speed of 0.5 mm/min through the aid of a thin wire (0.20-mm diameter) placed strictly parallel to and in contact with the adhesion area on the substrate. Using the cross-sectional area of each specimen, the resulting bond strength was calculated and expressed in MPa, after which a group mean was computed (n=18). All data were statistically analyzed (Minitab version 17.2.1, Minitab Inc, State College, PA, USA). A two-way analysis of variance (silane treatment vs storage time) was performed followed by the Tukey pairwise *post hoc* test at a preset alpha of 0.05 to evaluate bond strength values. Pretesting bond failures were not considered in the statistical analysis. In addition, Weibull distribution plots were also generated. Failure mode prevalence was statistically analyzed using the chi-square test (α=0.05) to assess the effect of silane treatment on failure mode within each storage time.

Failure Pattern Analysis

Visual analysis of the fractured surfaces was performed using scanning electron microscopy. Specimens were mounted on aluminum stubs,

Table 1: *Materials Used and Their Compositions*

Material	Type of Material	Manufacturer/Lot Number	Composition[a]	Application Steps
IPS e.max CAD	Lithium disilicate glass ceramic, A2	Ivoclar, Vivadent, Schaan, Liechtenstein/N76665	SiO_2 , Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, Al_2O_3, MgO, coloring oxides	
RelyX Ceramic Primer (RCP)	Ceramic primer (silane)	3M ESPE St Paul, MN, USA/N406850	MPS, ethanol, water	Apply actively for 60 s, then thoroughly air-dry
Adper Single Bond Plus (SB)	Total-etch adhesive system	3M ESPE/N334650BR	Bis-GMA, HEMA, dimethacrylates, ethanol,water, photoinitiators, a methacrylate functional copolymer of polyacrylic and polyitaconic acids and silica nanofiller	Apply actively for 15 s in one layer and air-dry for 5 s and do not polymerize separately
Scotchbond Universal (SBU)	Multimode adhesive system	3M ESPE/504115	MDP, dimethacrylate resins, HEMA, Vitrebond TM Copolymer, filler, ethanol, water, initiators, silane	Apply actively for 20 s and air-dry for 5 s and do not polymerize separately
Clearfil Ceramic Primer (CP)	Ceramic primer	Kuraray Noritake Dental Inc, Okayama, Japan/ 00023D	MPS, MDP, ethanol	Apply on the entire surface, then thoroughly air-dry
RelyX Ultimate	Resin cement, A2	3M ESPE/505370	Base paste: methacrylate monomers, radiopaque silanated fillers, initiator, stabilizer, rheological additives Catalyst paste: methacrylate monomers, radiopaque alkaline (basic) fillers, initiator, stabilizer, pigments, rheological additives, fluorescence dye, dark cure activator for Scotchbond Universal	Apply the resin cement with an automixing tip (provided by manufacturer) without separating it from the dispensed mass until filling the compartment

Abbreviations: Bis-GMA, bisphenol A-diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MPS, methacryloxypropyltrimethoxysilane (pre-hydrolyzed silane).
[a] *Product composition according to materials safety data sheets provided by the manufacturers.*

sputter coated with gold/palladium (SCD 050, Balzers, Schaan, Liechtenstein), and then examined using scanning electron microscopy (SEM) (JSM 5600 LV, JEOL, Tokyo, Japan) operating at 15 kV and a working distance of 15 mm. Images of representative areas of the fractured surfaces were obtained at different magnifications for fracture pattern morphology evaluation. Fracture classification was assigned using the following criteria: AD-CC, adhesive failure between ceramic and resin cement; AD-AC, adhesive fracture between the adhesive system layer (when employed) and the resin cement; C-AS, cohesive failure in adhesive system (when employed); C-CE, cohesive failure within the ceramic; C-RC, cohesive failure in resin cement; and MIX, a mixture of different kinds of fractures within the same specimen.

RESULTS

The statistical report indicated data were normally distributed (Anderson-Darling test, $p=0.251$), and homoscedasticity was proven (Bartlett test, $p=0.080$), both at a preset alpha of 0.05, indicating appropriate use of parametric methods for data analysis. The two-way analysis of variance revealed that the factors "Storage Time" and "Silane Treatment" both significantly influenced μSBS values ($p<0.0001$). Likewise, the interaction between the two factors was also significant ($p=0.0010$). Descriptive statistics for the μSBS results and statistical grouping of data sets for each experimental group are provided in Table 2. Regarding groups stored for 24 hours, the conventional silane (RCP) group presented higher bond strength values than all other experimental groups, except for the group where a

Table 2: *Microshear Bond Strength Values (MPa, mean± SD) Among the Test Groups Stored for Either 24 Hours or Six Months in 37°C Water*

Ceramic Silane Treatment[a]	Storage Time (37°C Water)	N[b]	Number of Pretest failures	Micsoshear Bond Strength (MPa)[c]	Decrease in Bond Strength With Storage Time (MPa [%])	Statistical Grouping Within Storage Time[d]
RCP	24 h	18	0	27.2 (±3.1)	9.2 (34%)	A
	6 mo	18	1	18.0 (±4.9)		ab
RCP + SB	24 h	18	0	23.4 (±4.4)*	1.2 (5%)	AB
	6 mo	18	0	22.2 (±5.4)*		a
SBU	24 h	18	0	18.8 (±3.0)*	1.6 (9%)	B
	6 mo	18	2	17.2 (±3.6)*		b
CP	24 h	18	0	21.7 (±4.3)*	4.3 (20%)	B
	6 mo	18	0	17.4 (±4.8)*		b
HF-control	24 h	18	0	21.1 (±3.4)	5.4 (26%)	B
	6 mo	18	1	15.7 (±5.8)		b

[a] *RCP, RelyX Ceramic Primer; RCP + SB, RelyX Ceramic Primer and Adper Single Bond 2; SBU, Single Bond Universal; CP, Clearfil Ceramic Primer; HF/control, no silane, 10% hydrofluoric acid only.*
[b] *Sample size.*
[c] *Debond strength values with an asterisk between 24 hours and six months for each treatment are not significantly different.*
[d] *For statistical grouping, groups identified by similar uppercase letters (24-hour values) or lowercase letters (six-months values) are not significantly different ($p \leq 0.05$).*

conventional silane followed by an adhesive system layer was applied (RCP+SB), which also showed no statistical difference with the other groups (Table 2). Moreover, after six months of water storage, the RCP + SB and RCP groups obtained the highest results (respectively), but RCP was not statistically different from the other groups (CP, SBU, and HF-control) (Table 2). Only the RCP and the HF-control groups demonstrated a significant decrease in bond strength values between 24 hours and six months of water storage, while groups RCP + SB and SBU presented more stable and uniform data distributions for this comparison (Table 2).

Weibull distributions are graphically presented in Figure 1, and associated parameters are summarized in Table 3. The analysis showed, in general, that after 24 hours of aging, all groups were more reliable (higher Weibull modulus) and presented higher characteristic bond strengths (higher scale value) than after six months of storage (Table 3). Accordingly, the Weibull distributions after 24 hours of storage appear more vertical (higher slope, m), and bond strengths appeared more toward the higher end of strength values (on the right side of Figure 1) than were plots of similar bonding conditions but stored for only six months. The highest modulus and characteristic strength values were recorded for RCP/24 hours (10.8 and 28.5, respectively), and the lowest were seen using the HF-control/six months (3.1 and 17.6, respectively) (Table 3). Groups CP and SBU presented the least variation among Weibull moduli, and groups RCP + SB and SBU demonstrated the least variation in characteristic strength between the two aging conditions (Table 3; Figure 1). Conversely, groups RCP and HF-control demonstrated the most change in Weibull parameters between the two storage durations (Table 3; Figure 1).

The chi-square test revealed that failure mode is affected by silane treatment for both storage conditions ($p<0.0001$). The failure analysis results (Figure 2) correlated with the bond-strength data, and all fractures occurred within the adhesion zone: no ceramic fractures were noted. Groups that were not treated with an adhesive system (RCP, CP, and HF-control) presented mainly cohesive failures within the resin cement (C-RC) after 24 hours of water storage (Figures 2, 3a, 6a, and 7a). Moreover, 24-hour groups that were pretreated with an adhesive system (RCP+SB and SBU) presented mainly adhesive failures between the adhesive layer and the resin cement layer (AD-AC), showing an entire adhesive system layer adhering to the ceramic substrate. However, both layers morphology showed some differences between them (RCP+SB and SBU). The adhesive system layer from the RCP + SB group was seen to be uniform with the presence of some filler particles that were remnants from the resin cement layer (Figure 4a). Conversely, the SBU adhesive layer presented a very irregular pattern, showing some degree of discontinuity in the adhesive layer, with areas that apparently were not in contact with the resin cement and some that were before failure occurred (Figure 5a). After six months of storage,

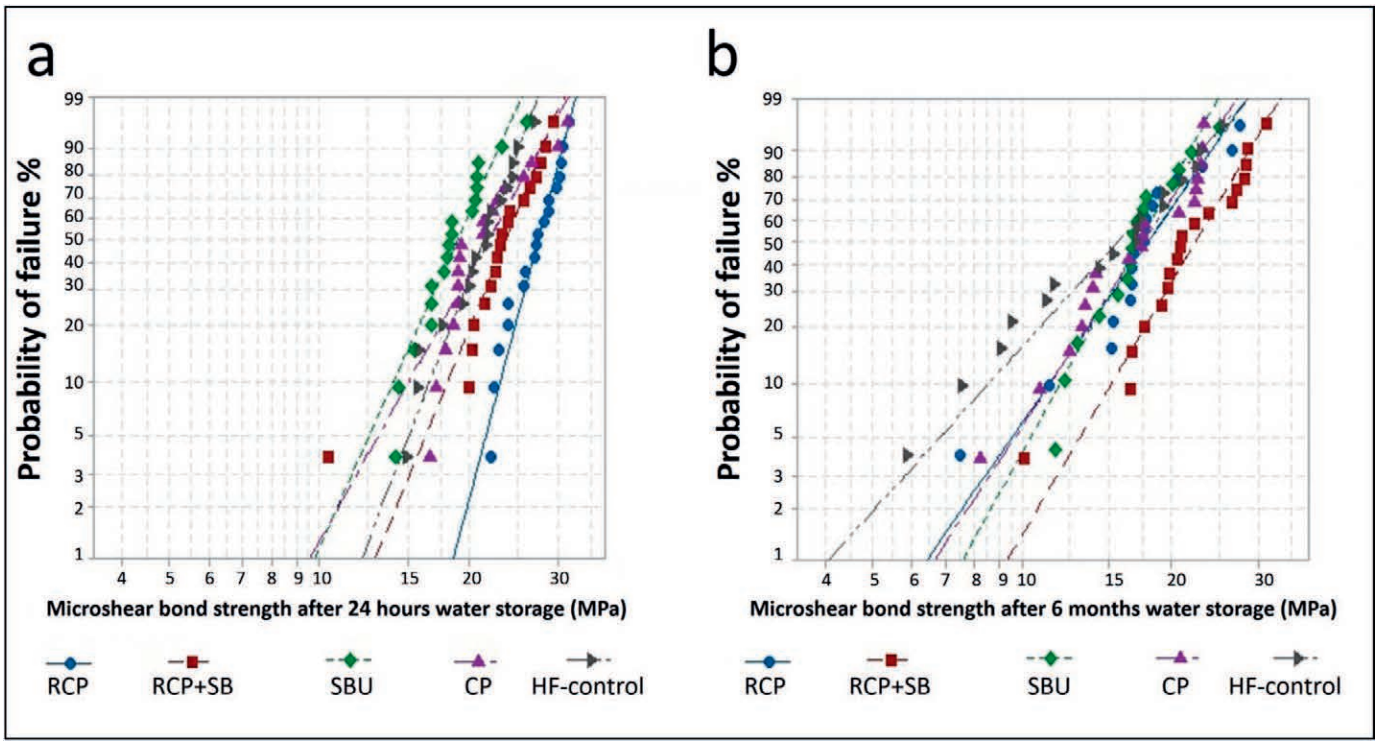

Figure 1. *Weibull distributions of microshear bond strength data related to water storage time at 37°C: (a) 24 hours and (b) six months. RCP, RelyX Ceramic Primer; RCP + SB, RelyX Ceramic Primer and Adper Single Bond 2; SBU, Single Bond Universal; CP, Clearfil Ceramic Primer; HF/control, no silane, 10% hydrofluoric acid treatment only. Lines drawn represent the Weibull curve shape.*

Table 3: *Weibull Modulus (*m*) and Weibull Characteristic Strength (σ_0) Values Among the Test Groups Stored for Either 24 Hours or Six Months in 37°C Water*

Ceramic Silane Treatment[a]	Storage Time (37°C Water)	N[b]	Number of Pretest Failures	Weibull Modulus (*m*)[c]	Weibull Modulus (95% Confidence Interval)[d]	Change in Weibull Modulus (and % With Storage)	Characteristic Strength (σ_0) (MPa)[e]	Characteristic Strength (95% Confidence Interval)[f]	Change in Characteristic Strength With Storage (MPa [%])
RCP	24 h	18	0	10.8	7.4-15.6	6.7 (61.8)	28.5	27.2-29.8	8.7 (30.7)
	6 mo	18	1	4.1	2.9-5.9		19.7	17.5-22.3	
RCP + SB	24 h	18	0	7.0	4.9-10.2*	2.2 (31.4)	24.9	23.3-26.7*	0.7 (2.7)
	6 mo	18	0	4.8	3.3-7.0*		24.3	22.0-26.9*	
SBU	24 h	18	0	6.4	4.6-8.9*	1.25 (19.5)	20.1	18.6-21.7*	1.5 (7,4)
	6 mo	18	2	5.1	3.6-7.4*		18.6	16.8-20.6*	
CP	24 h	18	0	5.1	3.7-7.1*	0.75 (14.7)	23.5	21.4-25.9*	4.3 (18.3)
	6 mo	18	0	4.3	3.0-6.4*		19.2	17.2-21.5*	
HF-control	24 h	18	0	7.5	5.2-10.8	4.37 (58.1)	22.5	21.1-24.0	4.8 (21.6)
	6 mo	18	1	3.1	2.1-4.6		17.6	15.0-20.7	

[a] *RCP, RelyX Ceramic Primer; RCP + SB, RelyX Ceramic Primer and Adper Single Bond 2; SBU, Single Bond Universal; CP, Clearfil Ceramic Primer; HF/control, no silane, 10% hydrofluoric acid only.*
[b] *Sample size.*
[c] *Determines how vertical the data distribution line is and infers how reliable the bond strength is. The higher the Weibull shape number (modulus,* m*), the more vertical the graph and the more reliable the treatment.*
[d] *Confidence intervals with an asterisk for each treatment are not significantly different, as they overlap.*
[e] *Determines the characteristic bond strength at a 63.2% failure probability. The higher the characteristic strength, the higher the bonding effectiveness.*
[f] *Confidence intervals with an asterisk for each treatment are not significantly different, as they overlap.*

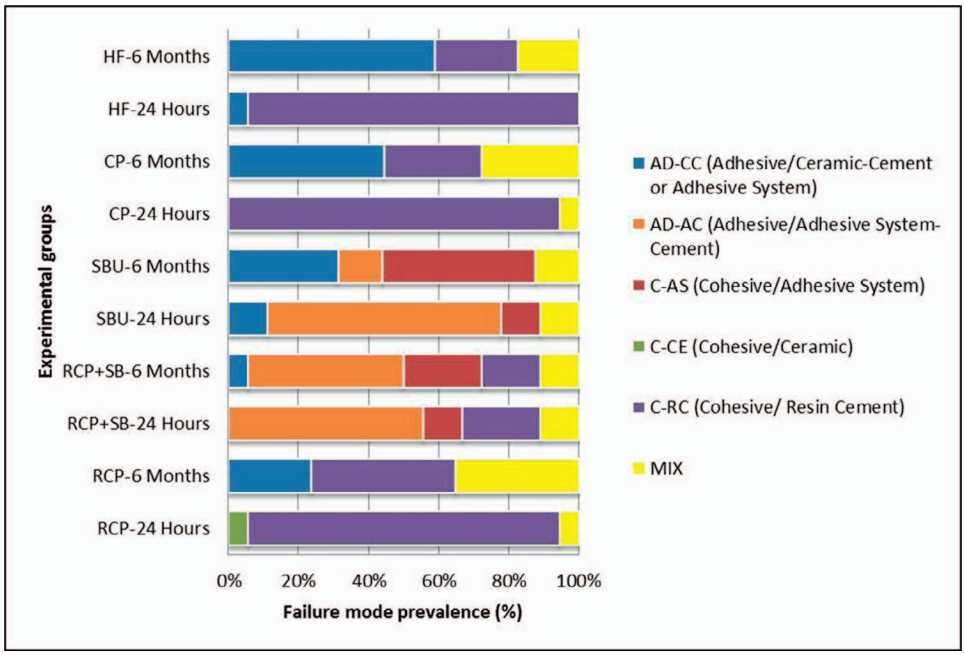

Figure 2. *Characterization of bond failure analysis from SEM images. RCP, RelyX Ceramic Primer; RCP + SB, RelyX Ceramic Primer and Adper Single Bond 2; SBU, Single Bond Universal; CP, Clearfil Ceramic Primer; HF/control, no silane, 10% hydrofluoric acid only.*

increased prevalences of adhesive failures between the resin cement or adhesive system and ceramic (AD-CC) and mixed failure patterns (MIX) were detected in the CP and HF-control groups, with more frequency of AD-CC failures for both (Figures 2, 6b, and 7b). In the case of RCP, a higher prevalence of AD-CC and MIX failure patterns was observed after six months of water storage, but a slightly higher prevalence of the C-RC type of failure was noted as a more irregular pattern than was observed after 24

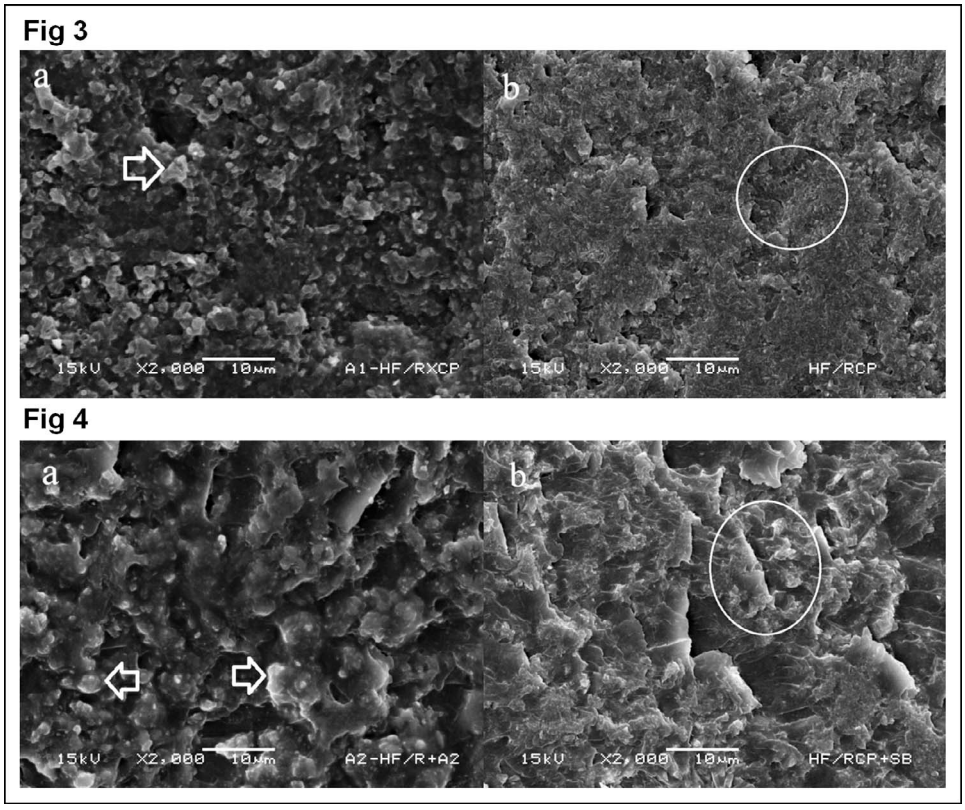

Figure 3. *Representative SEM micrographs of the more prevalent failure patterns observed in group RCP: (a) after 24 hours of water storage, where a C-RC failure can be seen along with the presence of resin cement filler particles (arrow), and (b) after six months of water storage, also showing a C-RC failure presenting an irregular/worn pattern within the surface (circle) in addition to showing fewer filler particles, as shown in Figure 3a.*

Figure 4. *Representative SEM micrographs of the more prevalent failure patterns observed in group RCP + SB: (a) after 24 hours of water storage, where an AD-AC failure is seen showing a regular adhesive layer with a few resin cement filler particles embedded within (arrow), and (b) after six months of water storage, also showing an AD-AC failure presenting an irregular/worn pattern within the surface (circle) but this time showing almost no filler particles embedded on the adhesive layer.*

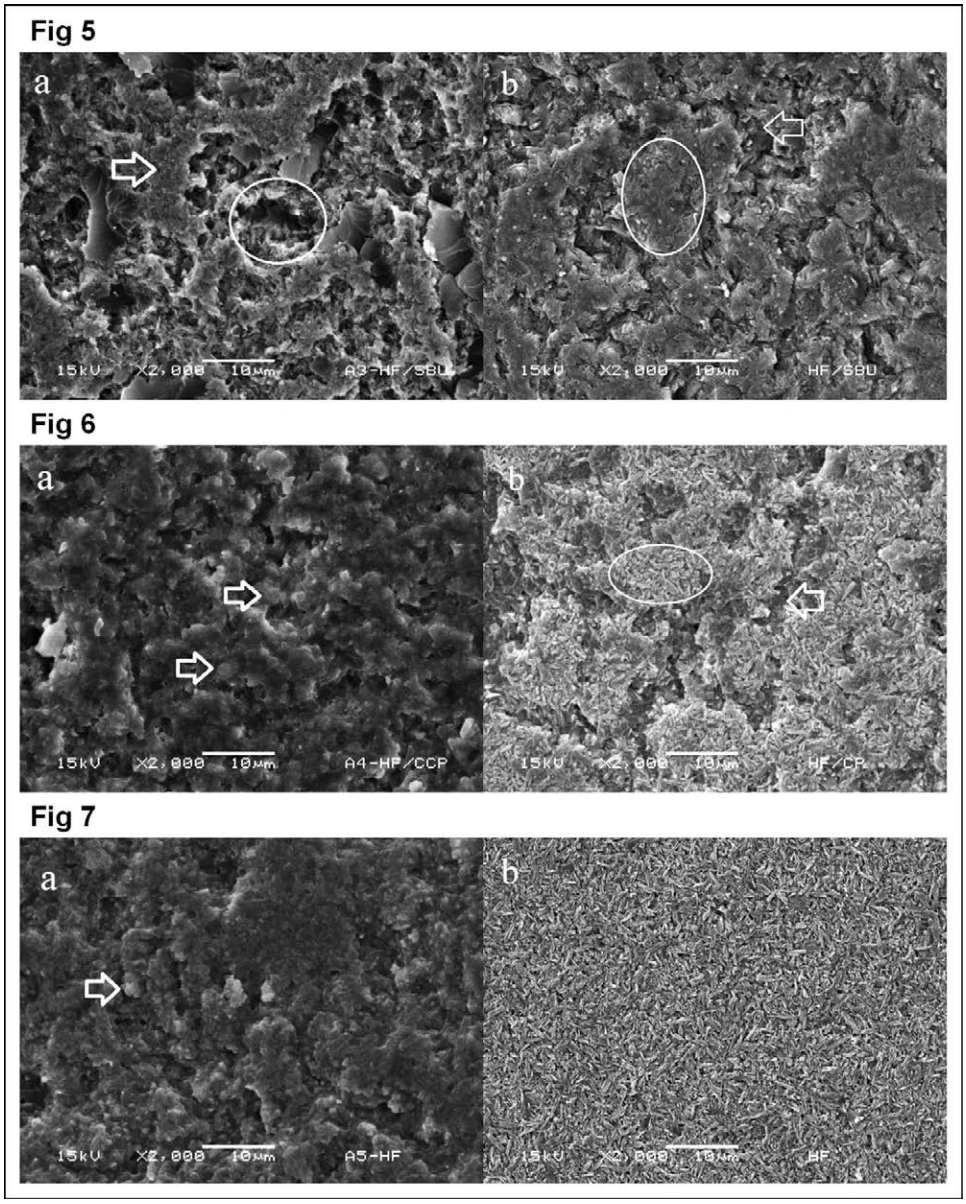

Figure 5. *Representative SEM micrographs of the more prevalent failure patterns observed in group SBU: (a) after 24 hours of water storage, where an AD-AC failure is seen showing a discontinuous and irregular adhesive layer formed by some areas of apparent deficient contact with the resin cement (circle) and some other areas that seem to have contacted the resin cement at the moment when failure occurred (arrow), and (b) after six months of water storage, showing a C-AS failure but very close to the ceramic surface, where a mixture of regions is noted, some with a thin layer of adhesive covering the ceramic surface (circle) and other areas with ceramic crystals exposed (arrow).*

Figure 6. *Representative SEM micrographs of the more prevalent failure patterns observed in group CP: (a) after 24 hours of water storage, where a C-RC failure is seen showing a continuous resin cement layer with some filler particles embedded (arrows), and (b) after six months of water storage, showing an AD-CC failure, with the ceramic surface and crystals exposed (circle) as well as some depressions caused by the airborne particle abrasion (arrow), producing an irregular pattern.*

Figure 7. *Representative SEM micrographs of the more prevalent failure patterns observed in the HF-only group (control): (a) after 24 hours of water storage, where a C-RC failure is seen, showing a continuous resin cement layer with some filler particles embedded (arrows), and (b) after six months of water storage, showing an AD-CC failure, where a regular layer formed by ceramic (lithium disilicate) crystals is noted. In this case, no depressions from airborne particle abrasion are seen, as in Figure 6b; instead, a completely uniform crystal surface is evident.*

hours of aging (Figures 2 and 3a,b). For the RCP + SB group, failure pattern prevalence was very similar after 24 hours or six months of aging, with more prevalence of other failure types after six months of storage but still demonstrating the AD-AC failure pattern most frequently (Figure 2). Furthermore, the adhesive layer morphology after six months was more irregular, and no resin cement remnant remained embedded in the adhesive layer, different from the pattern noted at 24 hours of storage (Figure 4a,b). More variation among failure patterns were noted for SBU after six months (Figure 2), being predominantly cohesive failure of the adhesive system (C-AS). This pattern was demonstrated by a thin, discontinuous adhesive layer, very close to the ceramic surface, where even some regions of underlying ceramic could be observed (Figure 5b). This observation indicates that, apparently, only a small portion of the adhesive layer remained attached to the ceramic surface for this bonding condition.

DISCUSSION

The results of this *in vitro* study show that the various silane treatments employed significantly influenced the ceramic/resin cement bond strength results, and thus the first null hypothesis was rejected. Because water aging also affected the bonding performance of resin cement to the glass

ceramic, the second null hypothesis was also rejected. Thus, it can be said that variations in chemical formulations of commercial silane primers have a significant effect on their effectiveness as ceramic/resin cement bonding promoters. Because a significant interaction between factors (material and storage time) was detected, the specific effectiveness of bond strength was found to be product and treatment specific. In general, RCP and RCP + SB obtained the best results, while SBU and CP did not improve ceramic/resin cement bonding when compared to the use of only HF (Table 2).

The lower bond strengths obtained by SBU and CP, compared with the conventional silane (RCP) after 24 hours of storage, are in accordance with previous work.[20,21] Kalavacharla and others[20] found that SBU required initial application of a separate silane layer to improve adhesion between resin composite and lithium disilicate ceramic after 24 hours of water storage and thermocycling (10,000 cycles). Conversely, Kim and others[21] concluded that two universal adhesives did improve glass-ceramic/resin cement bonding when compared with a negative control group (only 9.5% HF alone); however, a separate application of silane followed by an adhesive showed better results. Those findings are similar with the data obtained in the present work after 24 hours of storage. One possible explanation is that, as SBU (pH 2.7) and CP (pH 3) are more acidic than the conventional silane (RCP, pH 4.6),[2] the silane contained in both SBU and CP may have been continuously hydrolyzed and reacting during storage and consequently been inactivated to some degree before being used. Additional influences may have affected the performance of SBU and CP, such as the addition of ingredients other than silane (mainly in the universal adhesive), resulting in less silane quantity per area of bonded substrate[22] in contact with the ceramic surface in contrast to the primer containing silane only. Intimate contact between silane and the ceramic surface is fundamental because with one silane coat, three oligomer layers are formed on the surface[23] and only the first layer is capable of forming chemical adhesion to the glass; the outermost layers may be detrimental.[24]

Also, elimination of solvents and other by-products formed during the silane condensation reaction may be hindered through the development of a dense polysiloxane polymer network.[25] A previous study showed that increased time to evaporate solvent in universal adhesives is needed (from the manufacturer-recommended 5 seconds to a longer time of 25 seconds) to improve its performance.[26]

The above assumption appears to be confirmed from evidence presented in the SEM images. In the SBU 24-hour group, some discontinuity areas (probably due to deficient solvent elimination) were observed, pointing to an incomplete, intimate contact with resin cement at the moment of polymerization (Figure 5a). Failure to form these bonds leads to stress concentration areas and a lowering of adhesive/resin cement adhesion, which may be consistent with the greater prevalence of AD-AC failure type obtained for this group (Figure 2). A similar pattern was previously reported for the same materials: RelyX Ultimate with Scotchbond Universal using feldspathic-ceramic[27] and CAD/CAM composite blocks (LAVA Ultimate, 3M ESPE).[28] In those papers, the authors infer that the observed voids are possibly the consequence of water in-flow from wet dentin tubules, probably due to undercured areas in the adhesive system/resin-cement assembly when the materials were not cured separately (adhesive and resin cement).[27,28] In the present work, dentin was not used, and it is more appropriate to consider that water or humidity arose from the silane/ceramic condensation reaction or the incompletely eliminated solvent. Moreover, for the groups RCP, CP, and HF, mostly cohesive failure within the resin cement (C-RC) was noted after 24 hours of storage (Figures 2, 3a, 6a, and 7a). This failure type is associated with higher bond strength values in accordance with previous work.[18,25,29,30]

After six months of water aging, a different scenario was observed. RCP and the HF-control showed a significant drop in bond strength compared to their 24-hour values (Table 2). Conversely, groups RCP + SB (highest mean), SBU, and CP still provided more consistent bond strength values between 24 hours and six months of water storage, with RCP + SB demonstrating the least effect of water storage (only 5%) (Table 2). Consistently, failure pattern prevalence of the RCP + SB group remained almost the same between 24 hours and six months (Figure 2). In the case of SBU, water degradation may have reduced the cohesive strength of the adhesive layer, indicating that the fracture may have occurred very close to the adhesive/ceramic interface (Figure 5). Even though SBU bond strength also decreased with water storage, it could be assumed that the presence of its hydrophobic monomers prevented, to some degree, a fully adhesive failure between ceramic and the adhesive, as happened with the CP and HF-control groups.

The better performance exhibited by RCP + SB and SBU against water aging may be due to the

presence of hydrophobic monomers and MDP, which may better resist hydrolytic degradation because of their chemical nature. On the other hand, groups using a conventional silane (RCP) and HF-only treatment alone (HF-control) show no such protection against hydrolytic degradation: a 34% and a 26% decrease in bond strength with storage time, respectively. Such findings are supported by SEM failure analysis where those groups exhibited increased adhesive failure-type prevalence (AD-CC) and some other signs of hydrolytic effects, such as less resin cement particle presence (Figures 2 and 3) or even exposed lithium disilicate crystals (Figures 2 and 7).

Weibull analysis also showed a decrease in the reliability and characteristic bond strength for the RCP and HF-control groups after six months of water storage; for RCP + SB, SBU, and CP, no such decrease was detected (Figure 1; Table 3). These results indicate that, although all groups exhibited higher Weibull parameters at 24 hours of water storage than after six months, groups RCP + SB and SBU were less affected by water aging (Figure 1; Table 3). This finding may support the theory that a mostly hydrophobic adhesive system, used in conjunction with a silane coupling agent, may better resist hydrolytic degradation of the siloxane network. Weibull analysis is a useful tool in associating bond strength with probability of failure. Previous studies used this type of analysis to evaluate dental materials strength,[31] ceramic/resin cement bond strength,[32,33] and zirconia/resin cement bonding.[34] Using this evaluation method, the present work demonstrated that RCP + SB and SBU provided the most reliable treatments in the long term (Figure 1; Table 3).

In terms of bond strength, RCP + SB performed better than SBU (Tables 2 and 3; Figure 1). This finding could be explained by the fact that when applying the silane and the adhesive separately, the full ceramic surface is first contacted by silane molecules, possibly causing a higher chemical interaction between silane and the ceramic. Consequently, evaporation of solvent and condensation reaction by-products may be more efficient using the separate silane application than when silane is mixed with other components, as also suggested by others.[21] This result suggests that an adhesive layer (preferably hydrophobic), placed subsequent to silane application, may be useful in delaying hydrolytic degradation of the siloxane network formed at the ceramic/resin cement interface in addition to other benefits, such as increasing resin cement wettability,[35-37] serving as a shock-absorbing layer,[38] and increasing bond stability[39] and bond strength between the glass ceramic and resin cement.[40]

The selection of products and treatments used in this investigation demonstrated different results related to water storage duration. Consequently, use of a silane coupling agent, together with a mostly hydrophobic adhesive system (applied separately), may be recommended for glass-ceramic pretreatment. Future investigations should evaluate the ceramic/resin cement bonding stability (mechanical and chemical analysis) produced by silane-containing universal adhesives and ceramic primers for longer time periods and among a more diverse group of ceramic substrates.

CONCLUSIONS

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

1) Placement of conventional silane followed by an adhesive system application improved ceramic/resin cement bond strength after both short- and long-term water aging.
2) Application of separate silane and adhesive components and a silane-containing, universal adhesive demonstrated the least decrease in microshear bond strength after long-term water storage.
3) The conventional silane and the HF–only controls showed reduced bond strength from 24 hours to six months of water storage, while silane, followed by adhesive application, the universal adhesive, and the universal ceramic primer, did not change during that time period.

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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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Influence of Different Curing Modes on Polymerization Behavior and Mechanical Properties of Dual-Cured Provisional Resins

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Clinical Relevance

For dual-cured provisional resins, the light-curing mode may contribute to the enhancement of their mechanical properties and reduction of chair time.

SUMMARY

This study determined the influence of curing mode on polymerization behavior and mechanical properties of dual-cured provisional resins. Three dual-cured bisacryl-based provisional resins were used: Tempsmart (TS; GC Corp), Luxatemp Automix Solar (LX; DMG Chemisch Pharmazeutishe Fabrik GmbH), and Integrity Multi·Cure (IG; Dentsply Caulk). A self-cured bisacryl-based provisional resin, Protemp Plus (PP; 3M ESPE) and a conventional poly(methyl methacrylate) (PMMA) provisional resin, Unifast III (UF; GC Corp) were used as controls. The inorganic filler content and coefficients of linear thermal expansion of the test materials were measured. Six specimens of each material were used to determine the flexural strength, elastic modulus, and resilience. The changes in ultrasound velocity during polymerization were measured. The average inorganic filler contents of the provisional resins, apart from UF, ranged from 24.4 to 39.3 wt%. The highest inorganic filler content was determined for LX, whereas TS showed the lowest value among the tested materials. The average coefficients of thermal expansion of the tested provisional resins ranged from 77.3 to 107.7 ($\times 10^{-6}$/°C). TS and IG showed significantly

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lower thermal expansions than the other tested provisional resins. The mean flexural strengths of the provisional resins ranged from 70.4 to 122.6 MPa, the mean elastic moduli ranged from 1.8 to 3.7 GPa, and the mean resilience of the provisional resins ranged from 1.1 to 2.3 MJ/mm^3, respectively. Dual-cured provisional resins showed significantly higher flexural strengths than the PMMA resin. However, in all cases, the light-curing mode showed significantly higher flexural strengths than the self-curing mode. In the initial polymerization phase, dual-cured resins in the light-curing mode showed a rapid increase in the speed of sound (*V*) during light irradiation, followed by a slower increase. Conversely, the dual-cured resins in the self-curing mode showed a slower initial increase, followed by a rapid increase. Although no significant difference in *V* was observed between 10 and 15 minutes in the light-curing mode of all tested dual-cured resins, a significantly higher *V* value was obtained at 15 minutes than at 10 minutes in the self-curing modes for LX and IG. Regardless of the curing mode, tested dual-cured provisional resins showed superior mechanical properties than the conventional PMMA provisional resin. However, dual-cured provisional resin flexural properties and polymerization behavior were affected by the curing mode. This study indicated that the light-curing mode might be recommended for all dual-cured provisional resins because of the enhancement of their mechanical properties and reduction of chair time.

INTRODUCTION

Provisional restorations are fabricated to maintain tooth position, seal prepared teeth, protect margins, and establish appropriate vertical dimension during the interim period between preparing a tooth and seating a final restoration. In addition to their immediate protective, functional, and stabilizing value, interim restorations are useful for determining the functional occlusion and esthetic parameters, thereby enabling an optimum treatment outcome to be identified before definitive procedures have been completed.[1-4] Although digital dentistry, including computer aided design and manufacturing technology, has been developed, provisional restorations can be essential for responding to patient demands for detail and achieving a realistic trial that is similar to the final restorations. In some cases, provisional restorations are required to function for extended intervals and provide long-term stability while the adjunctive treatment is completed. In particular, for complex treatments, including dental implant therapy, maintaining the long-term durability of provisional restorations in the oral environment is required.[5-7]

In the last decade, bis-acryl–based resins containing inorganic fillers have been introduced and used extensively as provisional materials. This type of provisional resin can maintain long-term stability in the oral environment because of enhanced mechanical properties and wear resistance.[8-10] Bis-acryl–based resins have different curing modes, and several new products use a dual-curing mode. However, dual-cured resin-based materials have been reported to exhibit poor mechanical properties when the polymerization reaction is initiated by chemical curing alone.[11-14] Thus, the curing mode might affect the mechanical properties of the dual-cured provisional resins.

When considering the handling properties of provisional resins, the working and setting times are important in clinical situations. To achieve precise fitting to the abutment tooth during the fabrication of provisional restorations, proper flow, wetting, and the plastic properties of materials before complete polymerization are required. Changes in these properties depend on polymerization kinetics. However, quantitatively determining the polymerization kinetics of resin-based materials as a function of time using destructive testing methods such as flexural, compressive, or hardness measurements is difficult. By contrast, the ultrasonic method enables the noninvasive analysis of materials and monitoring of changes in their properties as a function of time. Ultrasonic devices have been used to detect carious lesions and to measure the setting process of resin cements or core buildup resins.[15-17] Because the speed of sound is sensitive to the viscoelastic properties of materials, the polymerization behavior of a material can be quantified by monitoring ultrasonic propagation.[18]

The purpose of this study was to determine the influence of curing mode on the mechanical properties and polymerization behavior of dual-cured bis-acryl provisional resins using an ultrasonic device. In addition, the mechanical properties and polymerization behavior of dual-cured bis-acryl provisional resins were compared with those of a conventional poly(methyl methacrylate) (PMMA) provisional resin and with those of a self-cured bis-acryl provisional

Table 1: *Materials used in this study*

Code	Provisional resins (shade: lot no.)	Main components	Manufacturer
TS	Tempsmart (A2: 1505171)	Bis-GMA, UDMA, dimethacrylate, silane treated amorphous silica, photo initiator, pigment	GC Corp
LX	Luxatemp Automix Solar (A2: 715884)	Multifunctional methacrylate, glass filler, catalyst, stabilizer, additives	DMG
IG	Integrity Multi Cure (A2: 140808)	Bis-acrylate, multifunctional methacrylate, barium boroaluminosilicate glass filler, catalyst, photoinitiator, stabilizer	Dentsply Caulk
PP	Protemp Plus (A2: Base 498865) (A2: Catalyst 495535)	Dimethacrylate (BISEMA 6), silane treated silica, silane treated amorphous silica, ethanol, 2, 2′-[(1-methylethylidene)-bis(4,1-phenyleneoxy)] bisethyl diacetate, benzyl-phenyl-barbituric acid	3M ESPE
UF	Unifast III (A2: Powder 1103231) (Liquid 1507291)	Powder: ethyl-methyl methacrylate polymer, polymethylmethacrylate, barbituric acid derivative, organic copper compound, pigments	GC Corp
		Liquid: methyl methacrylate, *N*,*N*-dimethyl-*p*-toluidine trimethylolpropane, ethylene glycol dimethacrylate	

Abbreviations: Bis-GMA, bisphenol A glycidyl dimethacrylate; UDMA, urethane dimethacrylate.

resin. The null hypotheses to be tested were the following: 1) dual-cured bis-acryl provisional resins would not differ depending on curing mode with respect to mechanical properties and polymerization behavior, and 2) dual-cured bis-acryl provisional resins would not differ in their features from a PMMA provisional resin or a self-cured bis-acryl provisional resin.

METHODS AND MATERIALS

Materials

Three dual-cured bis-acryl provisional resins were used: 1) Tempsmart (TS; GC Corp, Tokyo, Japan), 2) Luxatemp Automix Solar (LX; DMG Chemisch-Pharmazeutishe Fabrik GmbH, Hamburg, Germany), and 3) Integrity Multi Cure (IG; Denstply Caulk, Milford, DE, USA). A self-cured bis-acyl provisional resin Protemp Plus (PP; 3M ESPE, St Paul, MN, USA) and a conventional PMMA provisional resin UniFast III (UF; GC Corp) were used as control materials (Table 1). A visible-light curing unit (Optilux 501, SDS Kerr, Danbury, CT, USA) was used, and the light irradiance (average, 600 mW/cm^2) of the curing unit was checked using a dental radiometer (model 100, SDS Kerr).

Inorganic Filler Content

Inorganic filler contents of the materials were measured using thermogravimetry/differential thermal analysis (TG/DTA6300 thermogravimeter, Seiko Instruments Inc, Tokyo, Japan). For each provisional resin tested, a mixed paste (50 mg) was heated in the thermogravimeter from 25°C to 800°C at a heating rate of 10°C/min until the organic components were completely incinerated. The weight of the residual resin paste was automatically measured by the built-in highly sensitive and accurate horizontal differential balance, and the inorganic filler content (wt%) was calculated using the compensating blank curve. Three measurements were conducted to obtain an average inorganic filler content (wt%). According to the manufacturer's material safety data sheet, the PMMA resin UF does not contain inorganic filler; therefore, the inorganic filler content measurement for UF was omitted.

Coefficient of Linear Thermal Expansion

The coefficients of linear thermal expansion of the test materials were measured using a thermomechanical analyzer (TMA/SS6300, Seiko Instruments Inc). For each provisional resin tested, a mixed provisional paste was condensed into a cylindrical Teflon (Sanplatec Corp, Osaka, Japan) mold 8.0 mm high and 3.0 mm in diameter. For dual-cured resins in the light-curing mode, light irradiation was carried out from the top and bottom sides of the mold for 40 seconds each, and the mold was then cut with a sharp scalpel and removed from the cured provisional resin. To ensure polymerization, the center of the specimen was irradiated perpendicular to the axis for 40 seconds, and the irradiation was then repeated for the opposite side. These tests were not performed for dual-cured resins in the self-curing mode. Because heat curing during the course of thermal expansion testing may induce the polymerization of the unreacted monomer, an equivalent

degree of conversion may be achieved regardless of curing modes. Therefore, the tests were performed only in the light-curing mode. Before the measurements were conducted, specimens were stored under dark conditions at 25 ± 1°C for 24 hours. Three specimens per provisional resin were prepared and separately tested in a thermomechanical analyzer at a heating rate of 2°C/min from 25°C to 100°C. Thermal expansion measurements were conducted four times per specimen; however, the first measurement outcome was discarded as unreliable. The average coefficient of linear thermal expansion ($\times 10^{-6}$/°C) was measured over a temperature range of 50°C, which is the same as that used in standard thermal cycling experiments, starting at a low temperature of 30°C to avoid any influence of fluctuation in ambient temperature.

Flexural Properties

The flexural properties of provisional resins were tested according to ISO #4049 specifications. Following the manufacturer's instructions, we mixed and compacted each provisional resin into a stainless-steel split mold of dimensions $25 \times 2 \times 2$ mm; the mold was positioned on a glass slide. After the hardened specimen was removed from the mold, all six sides were wet polished with #1200 silicon carbide papers (Fuji Star type DDC, Sankyo Rikagaku Co, Ltd, Saitama, Japan). Dual-cured provisional resins were evaluated with or without light irradiation. For the light-curing mode, resin paste was condensed into the mold, the middle third of the specimen was first irradiated for 30 seconds, and the remaining thirds were irradiated for 30 seconds each. All specimens were stored under dark conditions for 24 hours in distilled water at 37 ± 1°C before measurements. After the storage time, six specimens per test group were subjected to the three-point bending flexural strength test (span length, 20.0 mm) using a universal testing machine (model 5500R, Instron Corp, Canton, MA, USA) at a cross-head speed of 1.0 mm/min until breaking of the specimen. The flexural strength (F) and the modulus of elasticity (E) were determined from the stress–strain curve using computer software (Bluehill v. 2.5, Instron Corp, Canton, MA, USA) linked to the testing instrument. The modulus of resilience (R) was calculated using the following equation: $R = F^2/2E$.[19]

Ultrasonic Measurement

The ultrasonic measurement system used in this study was comprised of a pulser-receiver (5900PR, Panametrics, Waltham, MA, USA), two different types of transducers (M203 and V112, Panametrics), and an oscilloscope (WaveRunner LT584, LeCroy, Tokyo, Japan). One of the transducers (V112) was built into the sample stage. A transparent cylindrical mold (2.0 mm in height and 4.0 mm in diameter) was placed on the stage on top of the built-in transducer (M203). Following the manufacturer's instructions, we mixed the provisional resins and condensed them into the transparent mold at a temperature of 23 ± 1°C and relative humidity of 50 ± 5%. For dual-cured provisional resins in the light-curing mode, light guide-tips were placed on horizontally opposite sides of the mold, sandwiching the sample. The sample was irradiated for 30 seconds simultaneously from both sides, beginning 30 seconds after mixing. In the self-curing mode, this step was omitted.

Ultrasound measurements were begun 30 seconds after mixing for both polymerization modes. The transducers were connected to the pulser-receiver in through-transmission mode, and 16-μJ pulses were applied at a pulse repetition frequency of 500 Hz. The signal was captured using an oscilloscope. No heat generation was observed from the transducers during measurement, and the system was calibrated following standard procedures before each specimen was measured. The pulses were applied continuously from 30 seconds after mixing. Transmission times were recorded every 10 seconds until 5 minutes after mixing and then every 30 seconds until 15 minutes after mixing. The propagation speed of ultrasound (V) was calculated from sample thickness and transmission time. The three dual-cured provisional resins were tested in both the self-curing and light-curing modes, and PP and UF were tested only in the self-curing mode. Five samples were tested in each condition for all of the materials tested.

Scanning Electron Microscopy Observations

The surfaces of the polymerized materials were polished to a high gloss with abrasive discs (Fuji Star type DDC, Sankyo Rikagaku Co, Ltd) followed by a series of diamond pastes to 0.25-μm particle size (DP-Paste, Struers, Ballerup, Denmark). The polished surfaces were then subjected to argon ion beam etching (IIS-200ER, Elionix, Tokyo, Japan) for 40 seconds, with the ion beam directed at the polished surface (accelerating voltage, 1.0 kV; ion current density, 0.4 mA/cm^2). The surfaces were then coated with a thin film of gold in a vacuum coating evaporator (Quick Coater, model SC-701, Sanyu Denchi Inc, Tokyo, Japan). Observations were carried out using a scanning electron microscope

Table 2: *Inorganic filler contents of provisional resins*[a]

Code	Inorganic filler content (wt%)	Tukey group	Coefficient of linear thermal expansion ($\times 10^{-6}$/°C)	Tukey group
TS	24.4 (0.5)	d	77.3 (1.7)	a
LX	39.3 (0.3)	a	99.4 (2.5)	b
IG	37.5 (0.8)	b	80.7 (2.4)	a
PP	30.8 (0.6)	c	105.8 (1.6)	c
UF	—	—	107.7 (0.8)	c

[a] *Standard deviation is noted in parentheses. Same lowercase letters in vertical columns indicate no difference at the 5% significance level.*

(SEM; FE-8000, Elionix) operated at an accelerating voltage of 10 kV.

Statistical Analysis

A one-way analysis of variance (ANOVA) followed by Tukey's honestly significant difference (HSD) test (α=0.05) was used for analysis of inorganic filler content, coefficient of thermal linear expansion, flexural strength, elastic modulus, and resilience of the provisional resin. Ultrasound velocity measurements for all resins and conditions were compared using one-way ANOVA followed by Tukey's HSD test (α=0.05). This comparison was performed for measurements taken at the same time point for each of four times. The statistical analyses were performed using a software system (Sigma Plot ver. 11.0, SPSS Inc, Chicago, IL, USA).

RESULTS

Inorganic Filler Content and Coefficients of Thermal Expansion

The average inorganic filler contents and coefficients of thermal expansion are listed in Table 2. The average inorganic filler contents of the provisional resins, apart from UF, ranged from 24.4 to 39.3 wt%. The highest inorganic filler content was determined for LX, whereas TS showed the lowest value among the tested materials, and all differences were statistically significant. The average coefficients of thermal expansion of the tested provisional resins ranged from 77.3 to 107.7 ($\times 10^{-6}$/°C). TS and IG showed significantly lower values than the other tested provisional resins. The difference between LX, PP, and UF was statistically significant.

Flexural Properties

The results of flexural properties testing are shown in Table 3. The mean flexural strengths of the provisional resins ranged from 70.4 to 122.6 MPa, and the mean elastic moduli ranged from 1.8 to 3.7 GPa. Regardless of curing mode, dual-cured provisional resins showed significantly higher flexural strengths than the conventional PMMA resin (UF). However, for all of the dual-cured resins tested, significantly higher flexural strengths were observed for the light-curing mode than for the self-curing mode. The bis-acryl resins, apart from LX, showed higher elastic moduli than UF. In addition, for dual-cured resins, the light-curing mode showed higher elastic moduli than the self-curing mode, except for TS. The average resilience of the provisional resins ranged from 1.1 to 2.3 MJ/mm^3. All dual-cured tested resins, regardless of curing mode, showed signifi-

Table 3: *Flexural strength, elastic modulus, and resilience of provisional resins*[a]

Code	Flexural strength (MPa)	Tukey group	Elastic modulus (GPa)	Tukey group	Resilience (MJ/mm^3)	Tukey group
Light-curing						
TS	118.1 (4.3)	a,b	3.7 (0.3)	a	1.9 (0.2)	b,c
LX	92.2 (2.8)	e	2.4 (0.2)	c	1.8 (0.2)	c
IG	122.6 (5.1)	a	3.4 (0.3)	a	2.2 (0.3)	a,b
Self-curing						
TS	106.7 (2.6)	c,d	3.4 (0.4)	a	1.8 (0.2)	c
LX	82.3 (3.7)	f	1.8 (0.1)	d	1.9 (0.1)	b,c
IG	113.5 (2.5)	b	2.9 (0.1)	b	2.3 (0.1)	a
PP	109.5 (2.9)	b,c	3.3 (0.3)	a,b	1.8 (0.1)	c
UF	70.4 (1.3)	g	2.2 (0.1)	c,d	1.1 (0.1)	d

[a] *Standard deviation is noted in parentheses. Same lowercase letters in vertical columns indicate no difference at the 5% significance level.*

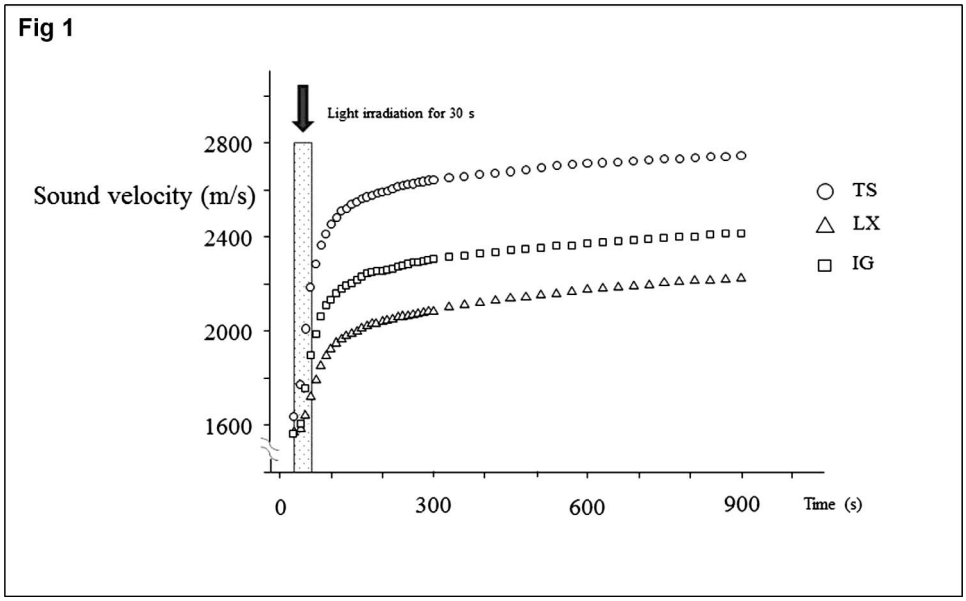

Figure 1. *Changes in* V *values in light-curing mode as a function of time from 30 to 900 seconds after mixing.*

cantly higher resilience than UF. In addition, no significant difference was observed between the light-curing and self-curing modes.

Ultrasonic Measurement

The results for ultrasonic velocity (*V*) in provisional resins are shown in Figures 1 and 2 and in Table 4. Changes in *V* values as a function of time depend on the type of provisional resin and curing mode. In the light-curing mode, a rapid increase in *V* values during light irradiation followed by a slower increase was observed (Figure 1). Although there was no significant difference in the *V* values of TS between five and 10 minutes, LX and IG showed significantly higher *V* values at 10 min than at five minutes. TS showed significantly higher *V* values than the other dual-cured resins at each time point.

In the self-curing mode, a much slower increase in *V* values, followed by a rapid increase was observed. However, TS, IG, and PP (Figure 2) had short initial segments (approximately 30 seconds) that exhibited little change, followed by a sharp increase. By contrast, LX and UF had a long flat segment that lasted for approximately 180 seconds before *V* substantially began to increase. LX and IG showed significantly higher *V* values at 15 minutes than at 10 minutes, whereas TS, PP, and UF exhibited no significant differences between 10 and 15 minutes. Although TS showed significantly higher *V* values than the other dual-cured resins in the self-curing

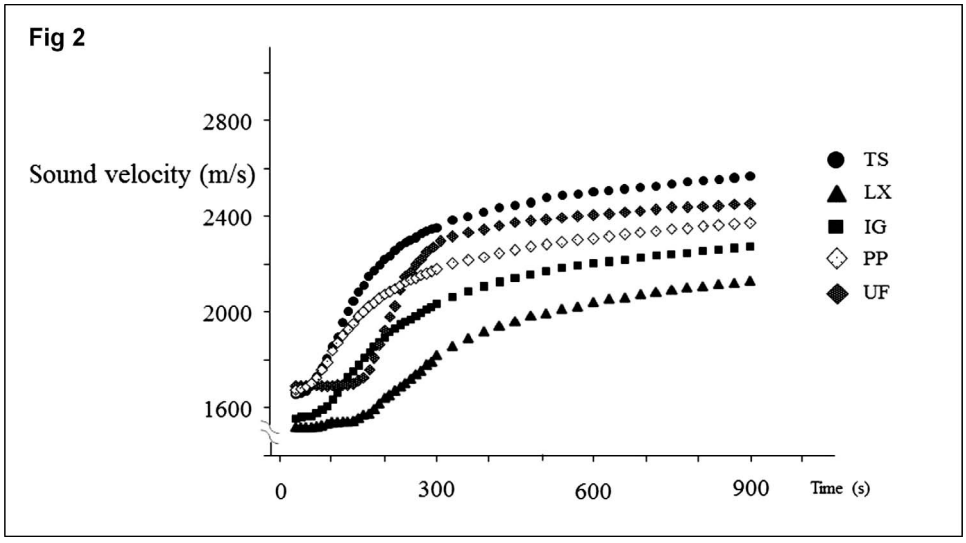

Figure 2. *Changes in* V *values in self-curing mode as a function of time from 30 to 900 seconds after mixing.*

Table 4: *Changes in* V *values in provisional resins as a function of time from 30 seconds to 15 minutes*[a]

Code	30 seconds	One minute	Five minutes	10 minutes	15 minutes
Light-curing					
TS	1661.8 (13.5)^{aA}	2186.9 (39.0)^{bA}	2645.6 (56.0)^{cA}	2714.6 (65.6)^{cdA}	2746.2 (71.2)^{dA}
LX	1570.9 (20.8)^{aB}	1720.4 (57.3)^{bC}	2091.0 (48.9)^{cD}	2176.4 (47.5)^{dD}	2223.8 (50.1)^{dDE}
IG	1559.3 (26.8)^{aB}	1899.8 (22.0)^{bB}	2305.9 (39.7)^{cB}	2371.9 (44.1)^{dC}	2417.1 (51.4)^{dC}
Self-curing					
TS	1656.8 (16.7)^{aA}	1695.7 (37.5)^{aC}	2353.5 (67.3)^{bB}	2501.5 (72.7)^{bcB}	2567.8 (72.5)^{cB}
LX	1538.0 (26.9)^{aB}	1542.5 (28.7)^{aD}	1827.1 (44.5)^{bE}	2046.0 (56.2)^{cE}	2136.8 (58.0)^{dE}
IG	1556.3 (6.5)^{aB}	1566.0 (10.4)^{aD}	2035.8 (41.1)^{bD}	2202.4 (31.3)^{cD}	2274.5 (31.6)^{dD}
PP	1674.1 (36.5)^{aA}	1702.4 (35.6)^{aC}	2180.1 (69.6)^{bC}	2307.0 (82.6)^{cC}	2372.2 (87.2)^{cCD}
UF	1691.8 (91.6)^{aA}	1693.2 (92.7)^{aC}	2294.0 (94.2)^{bBC}	2407.8 (69.3)^{bcBC}	2454.1 (92.1)^{cdBC}

[a] *Standard deviation is noted in parentheses. Same lowercase letters in horizontal rows indicate no difference at the 5% significance level. Same capital letters in vertical columns indicate no difference at the 5% significance level.*

mode at each measuring point, in the case of the light-curing mode, no significant difference was detected with UF at any time point.

SEM Observations

SEM micrographs of the five tested materials after argon-ion etching are presented in Figures 3 through 7. Clear differences in filler size, shape, and distribution were observed. Nano-sized filler particles were observed for TS, and these fillers were more densely packed than those observed for LX and IG (Figures 3 through 5). In the case of LX, 0.02- to 2.5-μm irregular glass filler particles were observed (Figure 4b). In the case of IG, although filler shape and distribution were similar to those of LX, the typical filler size was much smaller than that of LX, approximately 0.02 to 1 μm (Figure 5b). For PP, 0.05- to 0.2-μm spherical filler particles were observed (Fig. 6b). The conventional PMMA resin UF contains 20- to 50-μm spherical polymer particles (Fig. 7b).

DISCUSSION

Marginal adaptation of provisional restoration is important for maintaining a healthy condition of periodontal tissue and for protecting dental pulp from bacterial invasion. One of the desirable mechanical properties of provisional materials is their expansion rate, which is similar to that of tooth substrate. Changes in temperature might induce dimensional changes in the substrates, which could lead to channels forming in the materials.[20] Relatively large differences in the thermal expansion rate between the tooth and provisional restoration may induce gaps in the vicinity of the finishing margin, resulting in penetration of oral fluids and degradation of the temporary cement.

In this study, the average coefficients of thermal expansion of the tested materials ranged from 77.3 to 107.7 ($\times 10^{-6}$/°C), which is approximately seven to 10 times higher than tooth thermal expansion and noticeably higher than the coefficients of thermal expansion of resin composites.[20] A higher inorganic filler content in resin-based materials is expected to reduce thermal expansion because of the low thermal expansion coefficients of the inorganic fillers commonly used in this application. However, despite of its higher inorganic filler content, LX exhibited a significantly higher thermal expansion than the dual-cured resins TS and IG. Furthermore, we found no correlation between inorganic filler content and the thermal expansion. Furuichi and others[21] reported that no correlation exists between the inorganic filler content and coefficient of thermal expansion of resin cements and concluded that the coefficients of thermal expansion were material dependent. Therefore, in bis-acryl resins, other factors such as the type of matrix resin and/or size of filler particles might significantly affect the thermal properties, as in the case of resin cements. Some bis-acryl resins include a plasticizer that reduces the glass-transition temperature (*Tg*) of polymers by weakening the links between the polymer chains and increasing their mobility.[22] However, TS does not contain a plasticizer, which may explain why it exhibited lower thermal expansion despite its lower inorganic filler content.

Fracture-related material properties such as fracture resistance, elasticity, and the marginal degradation of materials under stress have typically been evaluated through the determination of material parameters, such as compressive, tensile, and flexural strengths and fracture toughness.[23,24] From the results of the flexural tests, dual-cured resins

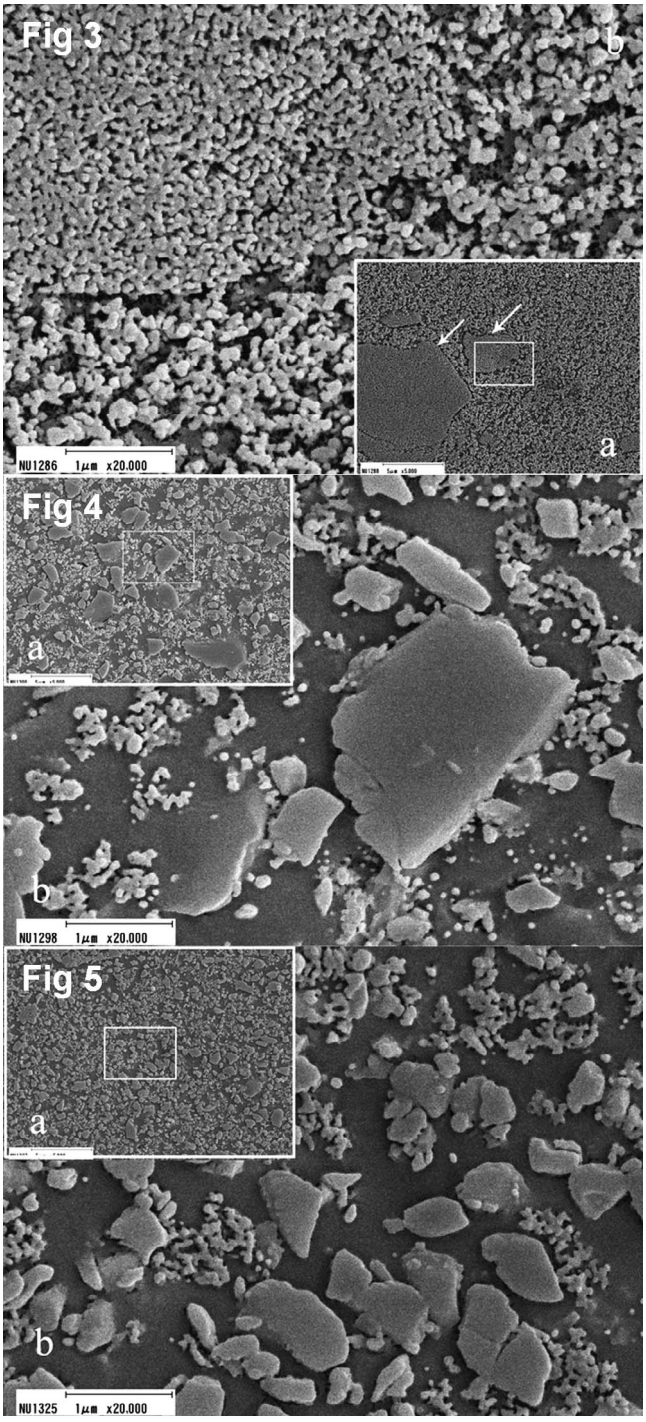

Figure 3. *SEM micrographs of the argon ion-etched surface of TS resin at magnifications of (a) 5000× and (b) 200,000×.*
Figure 4. *SEM micrographs of the argon ion-etched surface of LX resin at magnifications of (a) 5000× and (b) 200,000×.*
Figure 5. *SEM micrographs of the argon ion-etched surface of IG resin at magnifications of (a) 5000× and (b) 200,000×.*

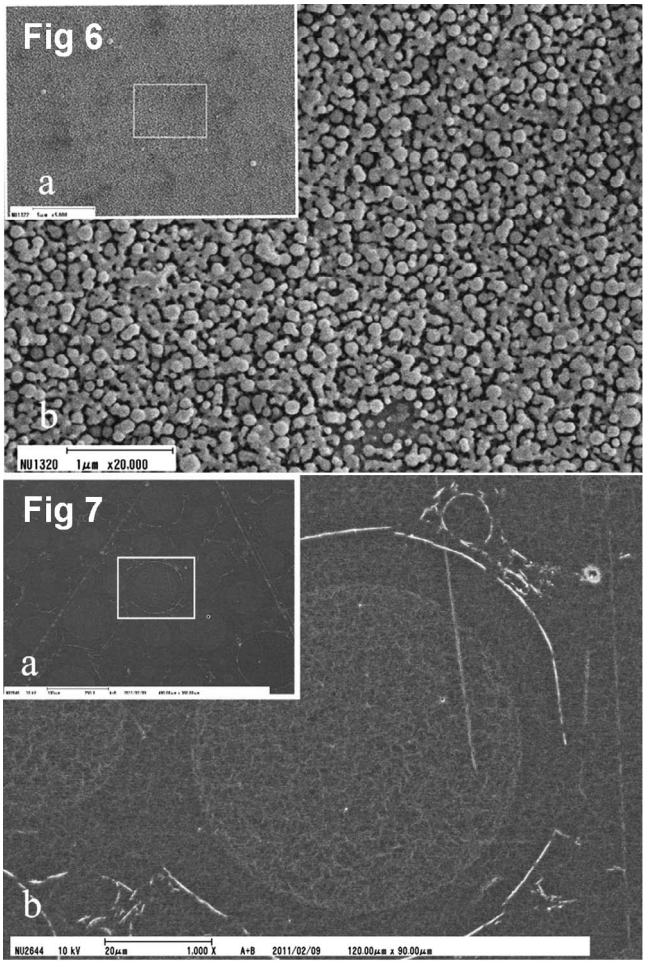

Figure 6. *SEM micrographs of the argon ion-etched surface of PP resin at magnifications of (a) 5000× and (b) 200,000×.*
Figure 7. *SEM micrographs of the argon ion-etched surface of UF resin at magnifications of (a) 100× and (b) 1000×.*

showed significantly higher flexural strength and resilience than the conventional PMMA resin, regardless of curing mode. Although the mechanical properties of UF were inferior to those of dual-cured resins regardless of curing mode, the flexural properties of PP were similar to those of dual-cured resins. Therefore, with respect to the mechanical properties, the null hypothesis that dual-cured bis-acryl resins would not differ from a conventional PMMA resin or a self-cured bis-acryl provisional resin was partially rejected. Bis-acryl resins contain multifunctional monomers that increase strength via cross-linking with other monomers[25] and also include inorganic fillers that can distribute the load stress and inhibit crack propagation.[8] In particular, the presence of filler particles increases the fracture energy and creates a pinning effect in crack propagation.[26] On the other hand, conventional

PMMA resin is composed of monofunctional, low-molecular-weight, linear molecules that demonstrate lower strength and rigidity.[27,28] Moreover, the bis-acryl resins used in this study were in cartridge-based dispensing systems, which might contribute to a more accurately proportioned and consistent mix. By contrast, the PMMA resin was hand mixed, and air entrapment may have occurred and resulted in lower strength values.[10]

Among the dual-cured resins tested, LX exhibited significantly lower flexural strength than the others, regardless of curing mode. SEM observations of the polished surfaces revealed that LX had a wider filler particle size distribution and larger interparticle spacing than the other dual-cured resins. We speculated that the larger interparticle spacing interferes with the pinning effect in crack propagation due to filler particles. In this study, bis-acryl resins, apart from LX, showed significantly higher flexural moduli than the UF resin. In general, resin-based materials exhibit an increase in elastic modulus with increasing inorganic filler load.[29,30] However, to maintain flexibility and plasticity after polymerization, some bis-acryl resins contain a plasticizer. The plasticizer is believed to not be chemically bonded to the plastic network.[22] Although it has relatively low filler load, TS lacks a plasticizer, which is speculated to have contributed to its relatively high elastic modulus.

The results of ultrasonic measurements indicate that the change of *V* as a function of time depends on the type and curing mode of provisional resins. A comparison of light-curing and self-curing modes of dual-cured resins reveals that the curves of *V* values are clearly different (Figures 1 and 2). When light irradiation was conducted for 30 seconds, a rapid increase in the *V* value was observed. Although significant differences were observed in the *V* values between 30 seconds and 1 minute after mixing in light-curing mode, there was no significant difference between 30 seconds and 1 minute in the self-curing mode. When the 15-minute *V* value was defined as 100% for each tested material, *V* values from 77% to 80% were observed at 1 minute in the light-curing mode of dual-cured resins. However, in the self-curing mode of dual-cured resins, *V* values at 1 minute ranged from 66% to 69%. Therefore, the null hypothesis that dual-cured provisional resins would not differ depending on curing mode with respect to mechanical properties and polymerization behavior was rejected.

Although the *V* values of dual-cured resins in the light-curing mode were material dependent, the rising curves exhibited similar shapes. Photoinitiated polymerization has been speculated to occur quickly after an induction period associated with the consumption of polymerization inhibitor and dissolved oxygen by initiator-derived radicals.[31] By contrast, in the case of the self-curing mode, the line graphs showed different trends among the tested resins. In particular, TS, IG, and PP have short initial segments with little change, followed by a sharp rise; by contrast, LX and UF have a long flat segment before *V* begins to rise significantly. This shows that LX has a longer working time than the other dual-cured provisional resins. It can be inferred that changes in *V* values might be attributable to differences in composition, including the composition of the catalyst system. In addition, this result indicates that the working time and setting time of provisional resins in self-curing mode are much more material dependent than in light-curing mode. During the polymerization process, the material properties of the resins change from a viscous liquid state to a glassy solid state. Although ultrasonic velocity cannot directly measure the degree of conversion or the strength of the material, it can monitor the viscoelastic properties of the resins in bulk.[32] In addition, ultrasonic measurements enable visualization and quantification of polymerization behavior over time. This information helps researchers not only to grasp the characteristics of polymerization kinetics but also to predict mechanical properties as a function of time.

The main clinical implication of this study is that dual-cured provisional resins have acceptable properties for clinical use in either mode. The results obtained in this study indicated that the light-curing mode might be recommended for all dual-cured provisional resins because of the enhancement of their mechanical properties and reduction of chair time. In addition, to avoid the negative effects of contact with oral fluid in the initial stage of polymerization, the light-curing mode should be used to induce the polymerization reaction for dual-cured provisional resins when provisional restorations are fabricated in situ.

CONCLUSION

Within the limitations of this *in vitro* study, dual-cured provisional resins exhibited mechanical properties superior to those of the conventional provisional PMMA resin UF, they also showed flexural properties similar to those of the self-cured bis-acryl–based provisional resin PP. A comparison of the light-curing and self-curing modes of dual-cured

resins revealed that the light-curing mode tended to result in higher flexural properties than the self-curing mode. On the basis of the results of ultrasonic measurements, the V value varies as different functions of time, depending on the type of provisional resin and curing mode.

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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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Influence of Emission Spectrum and Irradiance on Light Curing of Resin-Based Composites

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Clinical Relevance

Clinicians should be aware that while broad-spectrum LED curing lights can enhance the properties at the top of a restoration made of resin composites that contain "alternative" photoinitiators, the use of these lights could result in inferior properties at the bottom, since little violet light reaches this area of the restoration.

SUMMARY

Purpose: This study examined the influence of different emission spectra (single-peak and broad-spectrum) light-curing units (LCUs) delivering the same radiant exposures at irradiance values of 1200 or 3600 mW/cm^2 on the polymerization and light transmission of four resin-based composites (RBCs).

Methods and Materials: Two prototype LCUs that used the same light tip, but were either a single-peak blue or a broad-spectrum LED, were used to deliver the same radiant exposures to the top surfaces of the RBCs using either standard (1200 mW/cm^2) or high irradiance (3600 mW/cm^2) settings. The emission spectrum and radiant power from the LCUs were measured with a laboratory-grade integrating sphere coupled to a spectrometer, and the light beam was assessed with a beam profiler camera. Four RBCs (Filtek Supreme Ultra A2, Tetric EvoCeram A2, Tetric EvoCeram T, and TPH Spectra High Viscosity A2) were photoactivated using four different light conditions: single-peak blue/standard irradiance, single-peak blue/high irradiance, broad-spectrum/standard irradiance, and broad-spectrum/high irradiance. The degree of conversion (N=5) and microhardness at the top and bottom of 2.3-mm-diameter by 2.5-mm-thick specimens (N=5) were analyzed with analysis of variance and Tukey tests. The real-time light transmission through the RBCs was also measured.

Results: For all light conditions, the 2.3-mm-diameter specimens received a homogeneous

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irradiance and spectral distribution. Although similar radiant exposures were delivered to the top surfaces of the RBCs, the amount of light energy emitted from the bottom surfaces was different among the four RBCs, and was also greater for the single-peak lights. Very little violet light (wavelengths below 420 nm) reached the bottom of the 2.5-mm-thick specimens.

The degree of conversion and microhardness results varied according to the RBC ($p<0.05$). The RBCs that included alternative photoinitiators had greater microhardness values at the top when cured with broad-spectrum lights, while at the bottom, where little violet light was observed, the results were equal or higher when they were photoactivated with single-peak blue lights. With the exception of the microhardness at the top of TPH, equivalent or higher microhardness and degree-of-conversion values were achieved at the bottom surface when the standard (1200 mW/cm^2) irradiance levels were used compared to when high irradiance levels were used.

Conclusions: Considering the different behaviors of the tested RBCs, the emission spectrum and irradiance level influenced the polymerization of some RBCs. The RBCs that included alternative photoinitiators produced greater values at the top when cured with broad-spectrum lights, while at the bottom, results were equal or higher for the RBCs photoactivated with single-peak blue lights.

INTRODUCTION

Camphorquinone is the most commonly used photoinitiator in resin-based composites (RBCs), but it has a yellow color that may influence the final color of the restoration.[1] This can be problematic if the patient has bleached teeth or otherwise requires a light-colored restoration. Consequently, some resin manufacturers have started to use "alternative" photoinitiators, such as Lucirin TPO and Ivocerin. These photoinitiators do not impart such a yellow color to the final restoration, but they are most reactive to lower wavelengths of light[2] close to 410 nm compared to camphorquinone, which is most sensitive to light at 468 nm, or below 320 nm.

Light-curing units (LCUs) that use light-emitting diodes (LEDs) rather than halogen lights produce a relatively narrow wavelength of light and deliver little below 420 nm. Since they may not be able to adequately activate these alternative photoinitiators, some manufacturers have included in their LCUs several different LED chips that emit light at different wavelengths, thus producing a broader emission spectrum.[3,4] However, it is difficult to achieve a uniform distribution of light at the tip of the LCU, and this inhomogeneity can lead to nonuniform polymerization of dental restorations.[5-8]

Another trend is to try to make clinical procedures faster. Instead of using a 40-second light exposure time, some manufacturers claim that their LCUs deliver such a high irradiance that they can adequately polymerize RBC restorations in as little as one second. However, studies have shown that light-curing RBCs for short exposure times is not ideal.[9-11] This is of concern because inadequate photoactivation of the RBC can negatively affect the properties of the restoration, resulting in a lower degree of double-bond conversion and inferior mechanical properties.[12-14] Also, the degree of conversion of the resin has a direct correlation with biocompatibility of the RBC restoration in the mouth.[15] Moreover, the irradiance of the LCU used in the photoactivation of the RBC may affect the polymerization kinetics, and a rapid polymerization reaction may increase the polymerization contraction stresses and have detrimental effects on the restoration.[16,17]

Since there are many different types of LCU available, dentists should be aware of differences that exist among units and the impact these differences can have on the RBC. They should know when it is appropriate to use each type of LCU.[18,19] To provide the dentist with this information, this study aimed to verify the influence on the polymerization of four different RBCs with different emission spectra from single-peak and broad-spectrum LED units, which delivered either a standard irradiance that is typical of most contemporary LCUs, or a high irradiance typical of a powerful LCU. The null hypotheses were the following:

1) There will be no difference between the degree of double-bond degree of conversion and the Knoop microhardness of RBCs achieved when light cured with the single-peak blue or the broad-spectrum curing lights.
2) There will be no difference between the degree of double-bond conversion and microhardness of RBCs when light cured under the standard irradiance, or the high-irradiance conditions.
3) There will be no correlation between the degree of double-bond conversion and Knoop microhardness

values of RBCs when light cured using the different exposure conditions.

METHODS AND MATERIALS

Four RBCs were used in this study: Filtek Supreme Ultra—shade A2B (3M ESPE, St Paul, MN, USA), Tetric EvoCeram —shade A2 (Ivoclar Vivadent, Amherst, NY, USA), Tetric EvoCeram—shade T (Ivoclar Vivadent), and TPH Spectra High Viscosity—shade A2 (Dentsply, Milford, DE, USA). They were photoactivated using either a custom-designed single-peak blue light or a broad-spectrum light, both made by Ultradent (South Jordan, UT, USA). The optics (lenses) in both units were identical, both had a 9.6-mm tip diameter, and only the LED emitters were different. The electrical current to the LED emitters of both LCUs could be altered by the user, thus allowing the units to deliver various irradiance levels.

Characterization of the LCUs

The emitted radiant power and emission spectrum were measured through a 2.3-mm-diameter aperture into a FOIS-1 integrating sphere (Ocean Optics, Dunedin, FL, USA) coupled to the USB 4000 fiber-optic spectrometer (Ocean Optics). Before the measurements, the system was calibrated using an LS-1-CAL-INT calibration lamp (Ocean Optics). For each light condition, five measurements of the spectral radiant power emitted between 350 and 550 nm were made. Using results from the integrating sphere, the electrical current to the LED chip was adjusted so that the same radiant exposure was delivered in four exposure conditions: 1) single-peak blue light with standard irradiance (SS), 2) single-peak blue light with high irradiance (SH), 3) broad-spectrum multipeak light with standard irradiance (MS), and 4) broad-spectrum multipeak light with high irradiance (MH).

As previously described,[20] to assess the radiant power distribution across the LCU tip, the beam profile analysis was made using a charge-coupled device (CCD) digital camera with a 50-mm-focal-length lens (SP620U, Ophir-Spiricon, Logan, UT, USA) that was fixed at a predetermined distance from the diffusing surface of a translucent ground-glass target (DG2X2-1500, Thor Laboratories, Newton, NJ, USA). A custom-made blue filter (International Light Technologies, Peabody, MA, USA) was used to flatten the spectral response of the CCD camera. The photonic count received by each camera pixel was calibrated with BeamGage version 6.6 software (Ophir-Spiricon). The effect of the ambient light was eliminated before the data collection by using the UltraCal feature in the BeamGage software.

The image received by the camera was collected using the BeamGage software 10 seconds after activating the LCUs using the standard irradiance conditions and three seconds after activating the LCUs under the high-irradiance conditions. Color-coded and calibrated irradiance images were generated using the mean values of emitted radiant power that had been previously collected. The graphics software program Origin Pro version 9.1 (OriginLab, Northampton, MA, USA) was used to scale the two-dimensional images that were then visually compared.

Degree of Conversion

Degree of double-bond conversion measurements were made in gray opaque molds that were 2.3 mm in diameter and 2.5 mm in height. The molds were filled with uncured RBCs and placed directly over the diamond at the center of the Golden Gate Attenuated Total Reflectance (ATR) platform (Specac, Orpington, Kent, UK) attached to a Tensor 27 Mid IR FT-IR spectrometer (Bruker, Billerica, MA, USA). A Mylar strip was placed over the top of the uncured resin and pressed flat with a glass slab. The slab was removed and the specimen was exposed for 15 seconds using the standard irradiance level or five seconds using the high irradiance level. These exposure times delivered the same radiant exposures to the RBCs. The mid IR spectra were collected for five seconds before light curing and were recorded continuously in real time for four minutes at a resolution of eight wavenumbers and collection rate of two scans per second. Five specimens were made with each RBC and each light condition for a total of 80 specimens.

Knoop Microhardness

After the degree-of-conversion analysis, the specimens were removed from the ATR platform. Twenty-four hours after light curing the specimen, three Knoop microhardness indentations were made in the top and the bottom of each specimen, using a load of 15 gf for eight seconds in a microhardness-testing device (HM 123, Mitutoyo, Kawasaki, Kanagawa, Japan).

Light Transmission

The FOIS-1 integrating sphere coupled to the USB 4000 fiber-optic spectrometer was used to measure

Table 1: *Emitted Radiant Power (mW), Calculated Irradiance (mW/cm^2), and Radiant Exposure (J/cm^2) ± Standard Deviations From Each of the Four Light Conditions: Single Standard, Single High, Multi Standard, and Multi High*

Polymerization Mode	**Power** (mW)	**Irradiance** (mW/cm^2)	**Radiant Exposure** (J/cm^2)
Single standard	49.6 ± 0.7	1193.8 ± 17.6	17.9 ± 0.26
Single high	149.9 ± 0.7	3607.9 ± 16.6	18.04 ± 0.08
Multi standard	51.1 ± 0.1	1229.9 ± 3.2	18.4 ± 0.05
Multi high	150.1 ± 1.1	3612.7 ± 26.0	18.1 ± 0.13

the light transmission through the RBC. The RBCs were inserted in a single portion into the same gray opaque molds used for the degree-of-conversion analysis and covered with Mylar strips. Then they were pressed between two glass mixing slabs to produce flat top and bottom surfaces. These molds, now filled with uncured RBC, were placed at the aperture of the integrating sphere and photoactivated at 0-mm distance. SpectraSuite software (Ocean Optics) was used to record, in real time, the spectral radiant power transmitted through each RBC.

After the transmitted spectral radiant power had been measured, the light beam profile through the cured specimen was measured using the beam profiler camera with a 50-mm-focal-length lens (SP620U, Ophir-Spiricon) and a custom-made blue filter (International Light Technologies). The molds filled with the cured RBC were placed at a fixed distance from the camera, and the LCU tip was placed in contact with the molds. Then the LCU was activated, and the light beam transmitted through the cured RBC was examined. Narrow-band-pass interference filters that had a maximum bandwidth centered at 400 or 460 nm and a full-width half-maximum tolerance of 10 nm (Edmund Industrial Optics, Barrington, NJ, USA) were used to collect separate beam profile images of the transmitted violet and blue lights. The images were collected using BeamGage version 6.6 software (Ophir-Spiricon). Only the high-irradiance-level light conditions were beam profiled through the different RBCs.

Statistical Analysis

Analysis of variance, followed by the Tukey *post hoc* multiple comparison test (α=0.05), was applied. To determine if there was a statistically significant difference between the degree of conversion and Knoop microhardness obtained under the different light exposure conditions, tests were applied separately for each RBC since each RBC has a different composition. In addition, the Pearson correlation test was applied to verify if there was a correlation between degree of conversion and microhardness measured at the bottom of the specimens.

RESULTS

The emitted radiant power, calculated irradiance, and radiant exposure values from each of the four light conditions are reported in Table 1. The emission spectrum and the wavelength peaks of the four light conditions are shown in Figure 1. The light beam profiles showed that there was a homogeneous distribution of the light through the 2.3-mm aperture (Figure 2) in all cases.

Means and standard deviations of the Knoop microhardness values at the top of the specimens are reported in Table 2. Photoactivation with the SS light resulted in statistically higher hardness values, while photoactivation with the SH light resulted in lower values. Specimens photoactivated with the broad-spectrum lights presented intermediate hardness values. The degree of double-bond conversion and Knoop microhardness values at the bottom are also reported in Table 2. Filtek Supreme Ultra A2 photoactivated with the SS light (single-peak blue

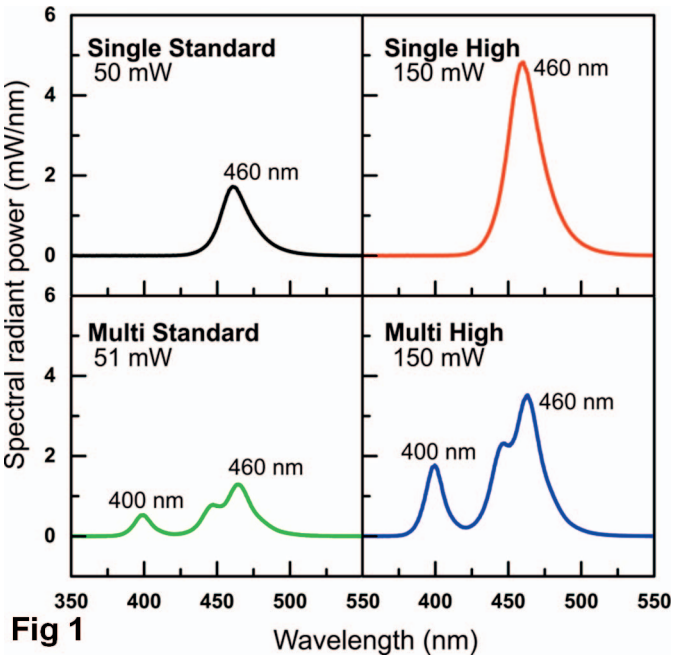

Figure 1. *Emission spectrum, power (mW) and wavelength peaks (nm) from the two lights for each of the four light conditions.*

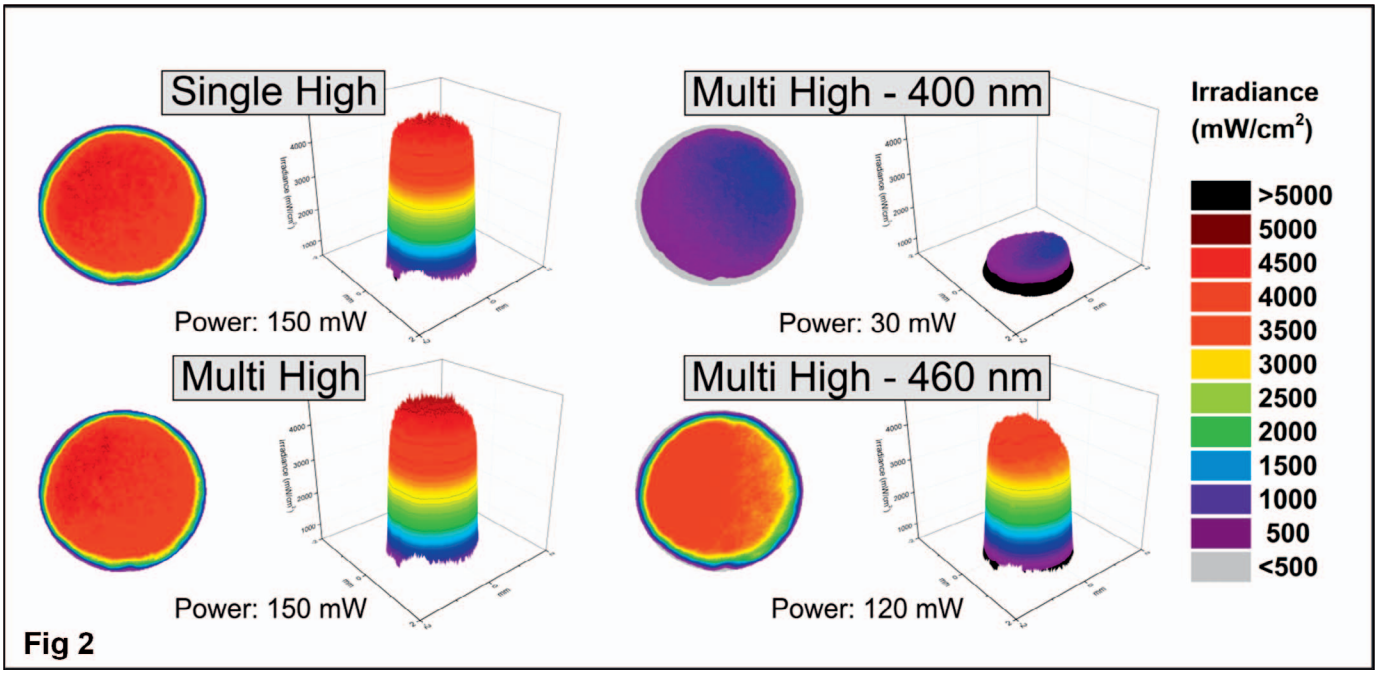

Figure 2. *Two- and three-dimensional light beam profiles of the lights recorded through the 2.3-mm aperture together with the power (mW) and scaled irradiance (mW/cm^2) using the single high (SH) and multi high (MH) light conditions. The images were taken both without a filter and with either a 400-nm or a 460-nm ±5 nm narrow-band-pass filter and scaled using the power values (mW) calculated from the integrated area under the emission spectrum (violet 30 mW, blue 120 mW).*

light with standard irradiance) achieved a significantly higher degree of conversion compared to when the MH light (multipeak light with high irradiance) was used, while, when photoactivated with the SH light (single-peak light with high irradiance) and MS light (multipeak light with standard irradiance), it presented intermediate values. The microhardness at the bottom of the specimen followed the same trends as the degree-of-conversion results.

Specimens of Tetric EvoCeram A2 and Tetric EvoCeram T made at the standard irradiance setting resulted in a higher degree of double-bond conversion values when either the single-peak blue (SS) or the broad-spectrum (MS) light was used. The specimens photoactivated with the SS light showed the highest degree-of-conversion results at the bottom for the Tetric EvoCeram A2, but there was no statistical difference between values obtained with blue light and broad-spectrum light for the Tetric EvoCeram T.

For the TPH Spectra High Viscosity A2, degree-of-conversion and microhardness values were not statistically different for specimens photoactivated with either the single-peak blue or the broad-spectrum lights. However, regarding degree of conversion and microhardness values at the bottom, the use of lower irradiances resulted in higher values, while for the top microhardness, specimens photoactivated with high irradiances presented higher microhardness values. The Pearson correlation test showed a significant linear correlation between the degree-of-conversion and the microhardness values at the bottom for all the RBCs tested (Figure 3).

The real-time light transmission results showed that the emitted radiant power that was measured through the 2.5-mm-thick RBCs was dependent on the choice of RBC and varied from only about 1.5% to 8% of the power received at the top, depending on the RBC and the light condition used (Figure 4). The radiant exposures delivered to the bottom of the specimen are reported in Table 3. Of the emitted power, the violet light emitted from the bottom decreased from about 20% of the total power delivered to the top, to only 2.5% to 8% of the total power that was emitted at the bottom (Figure 5). The light transmission increased with exposure time, and there was more light transmission through the translucent Tetric EvoCeram—shade T. The scaled beam profile images illustrate how little of the violet light reached the bottom of the specimens (Figure 6).

DISCUSSION

Previous studies compared light curing using different designs of single-peak blue and broad-spectrum lights.[21-24] This study allowed better standardization

Table 2: *Means ± Standard Deviations for Degree of Conversion at the Bottom (%) and Knoop Microhardness (KHN) for Each of the Four Light Conditions (ext.)*

	Top Hardness (KHN)				Bottom Hardness (KHN)	
	Filtek Supreme Ultra A2B	Tetric EvoCeram A2	TPH Spectra High Viscosity A2	Tetric EvoCeram T	Filtek Supreme Ultra A2B	Tetric EvoCeram A2
Single standard	85.7 ± 1.3 A	32.0 ± 1.1 B	50.4 ± 2.7 B	36.6 ± 2.7 B	31.6 ± 2.5 A	16.5 ± 0.6 A
Single high	79.3 ± 2.5 C	31.9 ± 1.3 B	63.3 ± 2.5 A	29.8 ± 2.6 C	29.1 ± 2.3 AB	14.7 ± 0.8 B
Multi standard	82.0 ± 1.8 BC	55.1 ± 1.6 A	50.2 ± 2.6 B	49.3 ± 4.2 A	30.2 ± 1.8 AB	15.7 ± 1.1 AB
Multi high	83.0 ± 1.8 AB	55.6 ± 1.7 A	63.0 ± 1.5 A	50.2 ± 4.1 A	26.8 ± 0.5 B	16.3 ± 0.2 B

*Statistical differences between light conditions are described with superscript letters, the same letters within a column indicate no significant difference in the value

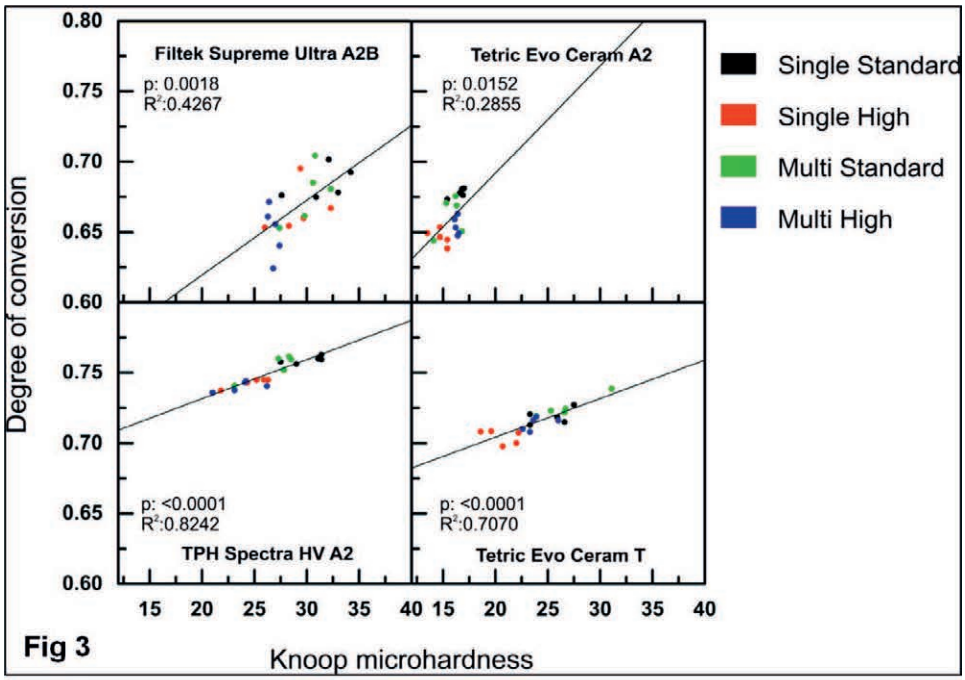

Figure 3. *Scatter graph showing the correlation between Knoop microhardness and degree of conversion for the four resin-based composites for each of the four light conditions (p- and R^2-values indicated for the Pearson correlation test).*

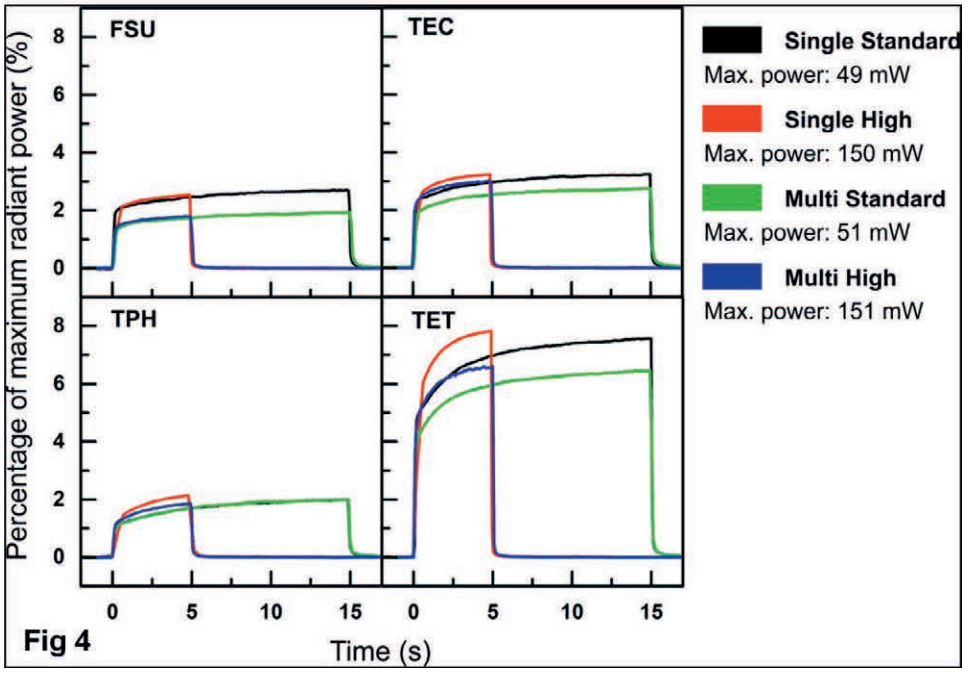

Figure 4. *Percent of maximum power transmitted in real time through the 2.5-mm-thick specimens for each of the four light conditions and for Filtek Supreme Ultra—shade A2B, Tetric EvoCeram—shade A2, Tetric EvoCeram—shade T, and TPH Spectra High Viscosity—shade A2. For TPH, the green line (MS) overlies the black line (SS). Note how the light transmission through the resin-based composites increased with exposure time.*

Table 2: *Extended.*

	Bottom Hardness (KHN)		Degree of Conversion at the Bottom (%)			
	TPH Spectra High Viscosity A2	Tetric EvoCeram T	Filtek Supreme Ultra A2B	Tetric EvoCeram A2	TPH Spectra High Viscosity A2	Tetric EvoCeram T
Single standard	30.1 ± 1.8 A	25.3 ± 1.9 A	68.5 ± 1.2 A	67.8 ± 0.3 A	75.9 ± 0.3 A	71.9 ± 0.6 AB
Single high	24.7 ± 1.8 B	20.6 ± 1.5 B	66.6 ± 1.7 AB	64.7 ± 0.6C	74.3 ± 0.3 B	70.4 ± 0.5 C
Multi standard	27.0 ± 2.2 AB	26.7 ± 2.7 A	67.7 ± 2.0 AB	66.2 ± 1.4 B	75.5 ± 0.9 A	72.5 ± 0.8 A
Multi high	23.7 ± 1.9 B	23.9 ± 1.3 AB	65.1 ± 1.9 B	65.5 ± 0.6 BC	74.0 ± 0.3 B	71.4 ± 0.5 BC

of the light conditions because both lights used the same tip and construction, with changes only in the emission spectra from the LED emitters. The four light exposure conditions delivered similar radiant exposures (Table 1), and the standard and high radiant powers were similar for the single-peak blue and broad-spectrum lights. The shapes of the emission spectra was the same for the blue lights, with peaks at 460 nm, and for the broad-spectrum lights, with peaks at 400 and 460 nm at both settings (Figure 1). For all conditions, the beam profile of the lights showed a homogeneous irradiance distribution through the 2.3-mm aperture. This is relevant since it is known that the distribution of light can be inhomogeneous across the light tip, and this inhomogeneity may affect resin polymerization.[6,18,25] The distributions of the violet and blue light components from the LCUs were also homogeneous, as seen in beam profile images taken through the 400- and the 460-nm narrow-band-pass filters (Figure 2).

The RBCs achieved different microhardness values, as observed in Table 2, and this can also be noted observing the different distributions on the scatter graphs (Figure 3). Tetric EvoCeram A2 achieved the lowest microhardness values, probably due to the different formulation of this product. All the RBCs presented lower microhardness values than expected at the bottom of the specimens. This likely occurred due to the optical characteristics of the opaque molds. The 2.5-mm height is more than the increment thickness recommended by the manufacturer, and the opaque gray molds prevented any light transmission. This should not be considered a limitation of the study because the aim was to measure not the depth of cure of the RBCs but rather the influence of the different exposure conditions on their photoactivation at a clinically relevant thickness (2.5 mm).

The first null hypothesis was rejected since using the broad-spectrum lights increased the top microhardness of two RBCs: Tetric EvoCeram A2 and Tetric EvoCeram T. This result is not unexpected since the manufacturer states that both of these RBCs use the alternative photoinitiators and these initiators are activated by the violet light present in broad-spectrum lights. Previous studies have also reported improved properties when RBCs with these alternative photoinitiators are photoactivated with broad-spectrum lights.[23,26] Consequently, it is recommended that broad-spectrum lights should be used to photoactivate RBCs that have alternative photoinitiators because the top microhardness values would be enhanced. This should reduce the wear and better support occlusal loading.

At the bottom of the specimen, there was no difference between microhardness values when the single-peak blue or the broad-spectrum lights were used, even for the RBCs with alternative photoinitiators in their composition. It might be expected that RBCs that include only camphorquinone in their formulation would achieve a higher microhardness when photoactivated with a single-peak LCU compared to that achieved with a broad-spectrum LED unit delivering the same radiant power because more light energy is delivered in the 460 nm region to the CQ from the single-peak LCU. However, there was no difference between RBCs photoactivated with

Table 3: *Radiant Exposure (J/cm^2) Delivered to the Bottom of the Specimens Made of Each of the Four Resin-Based Composites, Using Each of the Four Light Conditions. (Energy Calculated by Integrating the Area Under the Curve of the Real-Time Power Graph and Dividing It by the Area of the Molds)*

	Filtek Supreme Ultra A2B	Tetric EvoCeram A2	TPH Spectra High Viscosity A2	Tetric EvoCeram T
Single standard	0.42	0.54	0.31	1.24
Single high	0.40	0.52	0.32	1.23
Multi standard	0.34	0.48	0.31	1.11
Multi high	0.31	0.51	0.29	1.11

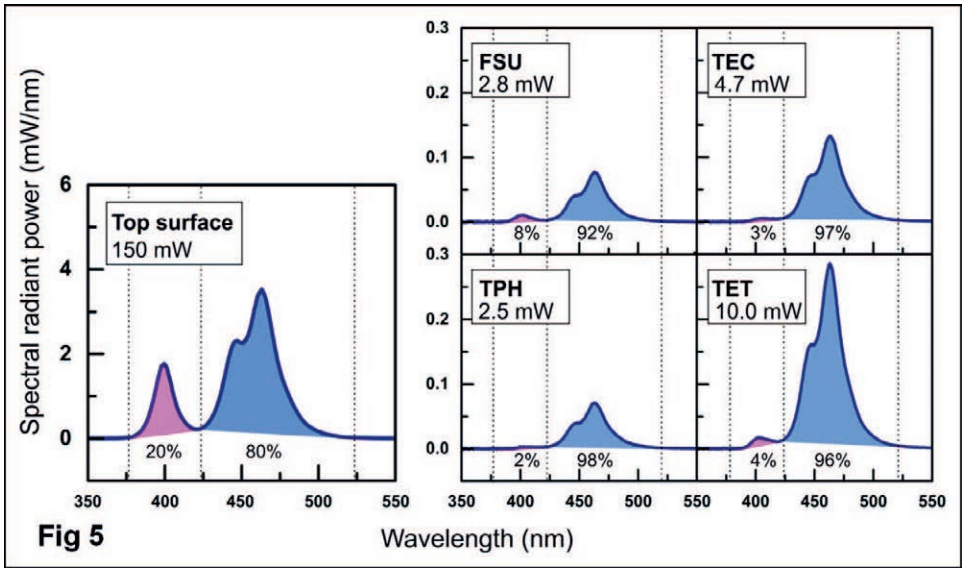

Figure 5. *Radiant power delivered to the top surface and percentage of this radiant power from the violet and blue LEDs that was emitted at the bottom through 2.5 mm of Filtek Supreme Ultra—shade A2B, Tetric EvoCeram—shade A2, Tetric EvoCeram—shade T, and TPH Spectra High Viscosity—shade A2 when using the broad-spectrum high-irradiance light. Note the greater light transmission through the translucent Tetric EvoCeram—shade T and the difference in the transmitted power values compared to what was delivered to the top.*

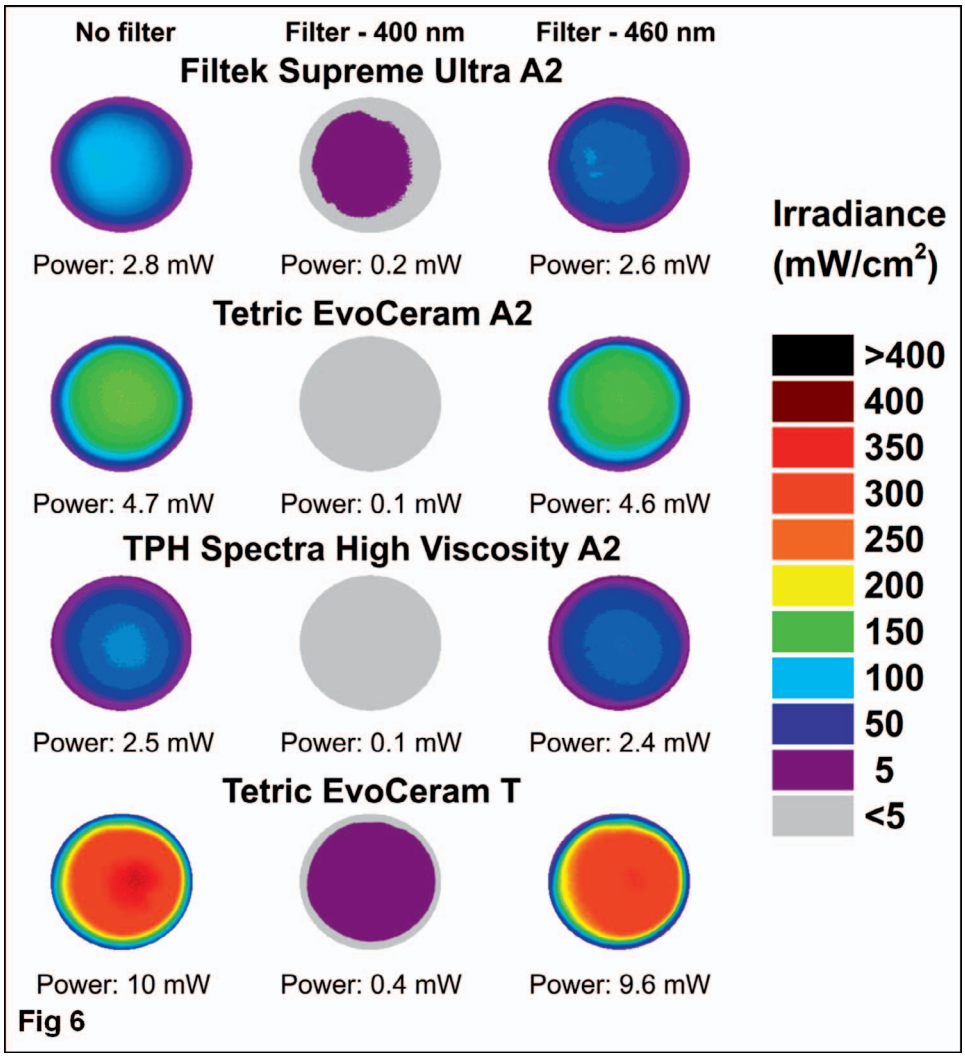

Figure 6. *Scaled two-dimensional irradiance beam profile images viewed through 2.5-mm-thick specimens of the four RBCs. The images were taken without a filter and with either a 400-nm or a 460-nm ±5 nm narrow-band-pass filter. The images were scaled using the power values (mW) calculated from the integrated area under the emission spectrum.*

single-peak or broad-spectrum lights. This probably occurred because more than sufficient radiant exposure was delivered within the blue wavelength range from both lights to photocure the RBCs.

Regarding degree of conversion, except for the Tetric EvoCeram A2 where specimens photoactivated with the SS light achieved a significantly higher degree of double-bond conversion, results were similar to the microhardness outcomes at the bottom, with no difference between specimens photoactivated with single-peak blue and broad-spectrum lights. This likely occurred because all the specimens received more than enough energy at the bottom.

As previously reported, the real-time radiant power transmitted through these four RBCs increased during polymerization [27]; that is, the RBCs became more transparent as they polymerized (Figure 3), but it is still greatly reduced compared to what is delivered to the top surface. Although the curing lights delivered very similar radiant exposures, the radiant power delivered from the broad-spectrum lights is a combination of violet and blue light and thus the light delivered in the blue region is less compared to the single-peak blue lights. Since almost none of the violet light reaches the bottom of the specimen, radiant power delivered in this spectral region is effectively "wasted" at the bottom, but not near the top surface. This helps to explain why the radiant exposure delivered at the bottom of the Filtek Supreme Ultra specimens was 0.3 J/cm^2 when photoactivated with the broad-spectrum lights, while it was higher (0.4 J/cm^2) when the single-peak blue light was used. The radiant exposure delivered at the bottom of the translucent shade of Tetric EvoCeram specimens (1.1 J/cm^2 when photoactivated with the broad-spectrum lights), was also higher (1.2 J/cm^2) when the single-peak blue light was used. The ratio of blue and violet light transmitted appears to be affected by the composition of the material since there was a greater percentage of violet light transmitted through the Filtek Supreme Ultra A2 than through the other RBCs (Figure 5). This is likely due to the exclusive use of nanosized filler particles in Filtek Supreme Ultra that result in less light scattered in the 400 nm range when compared to the other RBCs that use larger sized filler particles. Of note, since twice as much light was transmitted through the Tetric EvoCeram T than through the Tetric EvoCeram A2, even though both were the same brand of RBC, the shade of the material influences the amount of both blue and violet light transmission. The increased amount of energy delivered at the bottom of Tetric EvoCeram T (Table 3 and Figure 4) may explain why there was no significant difference between degree of double-bond conversion when using the single-peak blue and the broad-spectrum lights for this translucent shade, while there was a significant difference between the degree of conversion using the same lights for the A2 shade of the same RBC (Table 2).

The reduced transmission of violet light at depth is a concern if violet light is required to activate some of the photoinitiators used in the RBC, since the biocompatibility of restorations is directly correlated with the degree of conversion of RBCs.[15] If the alternative initiators at the bottom of restorations, close to the pulp or to the gingival tissues, are not used up, there may be increased release of unwanted leachates from the RBC. The concern is that the RBC may appear to be adequately polymerized if sufficient blue light is delivered to activate the camphorquinone photoinitiator, but still may contain the unused alternative photoinitiators at the bottom.

The second null hypothesis was also rejected. Equivalent or higher microhardness and degree-of-conversion values were achieved at the bottom surface when 1200 mW/cm^2 was used compared to 3600 mW/cm^2. Not all RBCs responded in the same manner to the different irradiance levels. This may be related to the different composition among these four products, in that they likely have different polymerization kinetics. High-irradiance photoactivation could potentially cause a lower degree of conversion because there are more radicals being formed at the same time, which in turn leads to more biradical terminations.[10] Slower polymerization rates should produce fewer polymer growth centers, leading to a higher density of linear chains.[28,29] Also, using a high irradiance should produce polymers with higher cross-linked density than when using low irradiance because of the rapid formation of the polymeric chain. These differences would lead to different mechanical properties of the material, such as hardness, but without necessarily altering its degree of conversion.

It is important to note that the previous studies that analyzed polymerization kinetics used very low irradiances,[30] from 3.1 to 50 mW/cm^2, or very high irradiances[31] of up to 7500 mW/cm^2, while in the present study, the 'low' irradiance was close to 1200 mW/cm^2, and the higher irradiance was close to 3600 mW/cm^2 (Table 1). Thus, the low irradiance used in the present study should not be considered low, but instead is a representative standard irradiance value for contemporary curing lights. The high irradiance

levels used in this study are similar to several high-irradiance light curing units.

The third null hypothesis was rejected since the Pearson correlation showed a direct linear correlation between bottom microhardness and degree-of-conversion results (Figure 3). Previous studies have also reported positive correlations between microhardness and degree of conversion.[32,33] Since these two independent tests produced the same outcomes, there is more confidence in the conclusions of this study.

Considering the results of this study, for shallow cavities or thin increments, broad-spectrum lights should be used to photocure RBCs that use alternative photoinitiators in their composition. When restoring deeper cavities with a single increment of composite using a broad-spectrum light, the exposure time should be increased since little violet light penetrates down to 2.5 mm. The aim is to deliver the same radiant exposure to the bottom of the composite as would be delivered using a single peak light.

Regarding the use of high irradiance lights, this study supports previous reports [9-11] that high irradiance levels and short (five-second) exposure times offer no benefit when photoactivating RBC restorations. Lower irradiance and longer exposure times may produce RBCs with better physical properties and are preferable in clinical situations where small errors in the angle or the position of the tip of the LCU would have less of a negative effect on the total amount of energy delivered to the restoration. For example, if one second of exposure was lost during a five-second exposure of a RBC, this would represent a decrease of 20% of the total radiant exposure. If one second of exposure was lost during a 15-second exposure at the lower irradiance values, this would represent only a 7% decrease in the total radiant exposure to the RBC.

CONCLUSIONS

Considering the limitations of this study and the different behaviors of the tested materials, the emission spectra from the LCUs influenced the polymerization of the tested RBCs. The microhardness of the materials that used alternative photoinitiators in their composition was enhanced at the top surface with the use of broad-spectrum lights. However, this effect was lost at the bottom surface, where little violet light was observed. Also, different shades of the same brand allowed different amounts of light to reach the bottom of the RBC.

Even when the same radiant exposure was delivered, the irradiance levels influenced the polymerization of the tested RBCs. Equivalent or higher microhardness and degree-of-conversion values were achieved at the bottom surface when 1200 mW/cm^2 was used compared to 3600 mW/cm^2.

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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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Fluoride Ion Release of Self-Adhesive Resin Cements and Their Potential to Inhibit *In Situ* Enamel and Dentin Demineralization

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Clinical Relevance

Fluoride release and demineralization inhibition in enamel and dentin of self-adhesive resin cements varied within brands and was mostly lower than that of conventional glass ionomer cement.

SUMMARY

Objective: This study evaluated *in situ* the potential of a glass ionomer and self-adhesive resin cements to inhibit enamel and dentin demineralization around indirect restorations exposed to cariogenic challenge. The cumulative fluoride release (CFR) of materials was measured in water and acid. Methods: Seventy blocks cut from human molars received two indirect composite restorations (one in enamel and another in dentin) luted with Ketac Cem EasyMix (GIC, positive control), SeT (SeT), Maxcem Elite (Max), Smart Cem2 (Smart), and RelyX Unicem 2 (Unicem2). Fourteen volunteers wore palatal appliances containing five blocks exposed to a cariogenic challenge (20% sucrose solution, eight times per day, seven days). Knoop microhardness (KH) at two distances from the margins and three depths from the outer surface determined enamel and dentin demineralization. Disc-shape specimens of materials were immersed in daily-replaced deionized water or lactic acid solutions. KH and CFR data were analyzed by analysis of variance, Games-Howell test, and Tukey test (α=0.05). Results: The overall KH ranking was GIC > SeT > Max > Smart = Unicem2 in both enamel and dentin (">" means $p<0.05$). SeT was the only resin cement that resulted in enamel and dentin KH comparable

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to that of GIC at most distances and depths. In water, CFR rank of materials was GIC > SeT = Max > Smart = Unicem2. In acid, the rank was similar, except that Set was significantly superior to Max. Conclusion: SeT inhibited demineralization in enamel and dentin quite comparably to GIC. All resin cements released lower cumulative amounts of fluoride than the glass ionomer cement.

INTRODUCTION

Secondary caries make up the most important cause of failure and replacement of direct[1] and indirect dental restorations.[2] Fluoride-releasing materials supposedly help prevent or reduce caries progression, which could decrease the need for additional restorative treatments. Glass ionomer cement (GIC), resin-modified glass ionomer cement, polyacid-modified composite (compomer), composite, and adhesive systems are fluoride-containing dental restoratives available on the market.[3]

The cariostatic properties of restorative materials are associated with the amount of fluoride released and incorporated into the adjacent tooth structure.[4] Fluoride is intentionally added to materials as an anticaries component, or the fluoride is released as part of the natural setting reaction of the material (eg, glass ionomers).[5] Fluoride release varies according to the source, size, and concentration of fluoride-containing filler particles as well as the composition, solubility, and permeability of the resin matrix in composite materials.[5-8]

Luting procedures of indirect restorations include the use of zinc phosphate, glass ionomer, and resin cements. Fluoride-containing luting cements could be effective in preventing or reducing secondary caries around the edges of indirect restorations. Glass ionomer and zinc phosphate luting cements have been investigated regarding their effect against secondary caries in enamel and root dentin adjacent to indirect restorations.[9,10] Self-adhesive resin cements (SARCs) were developed with claims of shorter application time and less technique sensitivity. SARCs act through a mechanism of infiltration on dental tissues combined with a chemical reaction between phosphate methacrylates and hydroxyapatite crystals.[11,12] Although some SARCs contain fluoride sources in their composition, limited information exists regarding their fluoride release,[13] and no data are available regarding their potential to reduce or prevent demineralization on the enamel and root dentin adjacent to indirect restorations exposed to a cariogenic challenge.

The anticaries mechanism of restoratives containing fluoride was previously evaluated by *in situ* studies.[14-17] This model reproduces the dynamics of caries development on either enamel or dentin using palatal appliances by promoting demineralization of the tooth surface. Different treatment strategies for the prevention or control of carious lesions are tested in a controlled manner and during a short period of time.[18,19] Moreover, the model provides a link between clinical and laboratory conditions.[18]

The objectives of this study were 1) to evaluate the *in situ* effect of fluoride-containing self-adhesive resin cements on the decrease of enamel and root dentin demineralization under cariogenic challenge and 2) to evaluate the cumulative release of fluoride ions from the cements in water and in an acidic solution. The two null hypotheses tested were that there would be no difference among hardness of enamel and root dentin adjacent to the cements and no difference among the cumulative fluoride release of the cements.

METHODS AND MATERIALS

Experimental Design

The factors under study for anticaries effect were luting cement at five levels—one conventional glass ionomer and four self-adhesive resin cements—and position at six levels—each one uniquely determined by one of the two distances from the preparation margin and one of the three depths from the outer enamel and dentin surface.[20] The factors under study for cumulative fluoride release were the luting cement and media solution at two levels: deionized water and lactic acid.

Anticaries Effect

A randomized, double-blind *in situ* design was conducted in one phase of seven days, during which 14 volunteers wore palatal appliances containing five human dental blocks containing indirect restorations luted with five cements in enamel and dentin cavities. Thirty-five sound third human molars that had been extracted and stored in the Pontifícia Universidade Católica do Paraná Tooth Bank were used. The inclusion criteria were sufficient volume of root and absence of caries lesions and fractures or imperfections in enamel and root dentin. Dental surfaces were manually scaled and then cleaned using a rotary bristle brush with a slurry of pumice and water. The cleaned teeth were stored in 0.05% chloramine-T solution at 4°C for a maximum of four months. The cervical third of each crown and root

Table 1: *Materials Used in the Present Study*

Group	Material (Manufacturer)	Composition[a] (Lot Number)
Unicem 2	RelyX Unicem2 (3M ESPE, St Paul, MN, USA)	Base paste: methacrylate monomers containing phosphoric acid groups, silanated glass powder, initiator components, stabilizers, rheological additives. Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives. (1312800922)
Smart	Smart Cem2 (Dentsply, Weybridge, UK)	Base paste: silanated barium-boron-fluoro-alumino-silicate glass, polymerizable dimethacrylate resin, strontium fluoride, hydrophobic amorphous silica. Catalyst paste: barium boron fluoro-alumino-silicate glass, urethane dimethacrylate resin, urethane modified bisphenol-A-diglycidylether dimethacrylate (Bis-GMA), dipentaerythritol pentaacrylate phosphate, hydrophobic amorphous silica. (120419)
Max	Maxcem Elite (Kerr, Orange, CA, USA)	Glycerol dimethacrylate dihydrogen phosphate, multifunctional methacrylate monomers, proprietary self-curing redox activator, camphorquinone, stabilizer. Filler load (67%wt): fluoro-aluminio-silicate glass, fumed silica, barium glass, ytterbium fluoride. (506742)
SeT	SeT PP (SDI, Bayswater, Australia)	Fluoro-alumino-silicate glass (60 to 70%wt), urethane dimethacrylate, camphorquinone, acidic monomer. (S1303061)
GIC	Ketac Cem EasyMix (3M ESPE)	Powder: aluminum-calcium-lanthanum-fluorosilicate glass. Liquid: water, copolymer of acrylic maleic, tartaric and benzoic acid. (524470)

[a] *Data provided by the manufacturer.*

was sectioned longitudinally and transversally into 70 blocks using a diamond disc (D943-100, Kerr Rotary, Orange, CA, USA). Each block had the side opposite to the restoration manually ground to achieve a 2-mm thickness using #1200 SiC sandpaper. The final dimensions (7×4 mm) of the blocks were checked with a digital caliper (CD-15CX, Mitutoyo Corp, Tokyo, Japan). Two cavities (1.5-mm depth×1.5-mm diameter)—one in enamel and the other in dentin—were prepared on each block using a cylindrical diamond self-limiting bur (MADC-015, Kerr Rotary) under constant water cooling. The diamond burs were replaced every 10 preparations (five blocks). The blocks were stored at 37°C and at 100% relative humidity for 24 hours.

The dental blocks were sterilized by autoclaving and randomly divided into five experimental groups (n=14). The cavities received indirect restorations of a resin-based laboratory composite (SR Adoro, Ivoclar Vivadent, Schaan, Liechtenstein). Luting materials consisted of four translucent dual-cured self-adhesive resin cements and one conventional glass ionomer cement (positive control). The materials were manipulated according to the manufacturers' instructions. Details of the materials are presented in Table 1. The resin cements were light cured for 40 seconds with a light-curing device (Elipar Free Light II, 3M ESPE, St Paul, MN, USA) monitored to 1000-mW/cm^2 irradiance every five specimens.

Fourteen adult subjects (six males and eight females; mean age 21 years, range 18 to 25 years) were selected for the *in situ* study. The inclusion criteria were 1) good general and oral health, 2) low caries activity, 3) nonusage of antibiotics two months prior to the study, 4) nonsmoker, and 5) no history of ingestion of drugs that affect salivary flow. The exclusion criteria were 1) any infectious disease, 2) not having used the sucrose solution in accordance with the research recommendations, and 3) poor oral hygiene. All volunteers signed the informed consent form detailing the main aspects of the research and all recommendations and guidelines regarding the procedures performed during the experiment.

Acrylic palatal appliances were constructed for each volunteer with five cavities (8×5×3 mm) for the restored blocks. A polyester mesh was positioned over the blocks to protect their surfaces from mechanical attrition, leaving a 1-mm space for biofilm accumulation.

The volunteers used the appliance for seven days. A previous study[21] showed that 20% sucrose exposure (eight times per day) significantly induced mineral loss of enamel after seven days of biofilm accumulation. Oral hygiene was performed three times a day with a fluoride toothpaste (Colgate Toothpaste Triple Action, 1500 mg F/g, Colgate-Palmolive Company, North York, ON, Canada) and a soft bristle dental brush (Oral-B Advantage Plus Soft Bristle Toothbrush, Procter & Gamble, Cincinnati, OH, USA) given by the main researcher. The appliances were not brushed to avoid disturbing the biofilm. The volunteers were advised to remove the appliance only during meals and oral hygiene. The cariogenic challenge was carried out by drop-

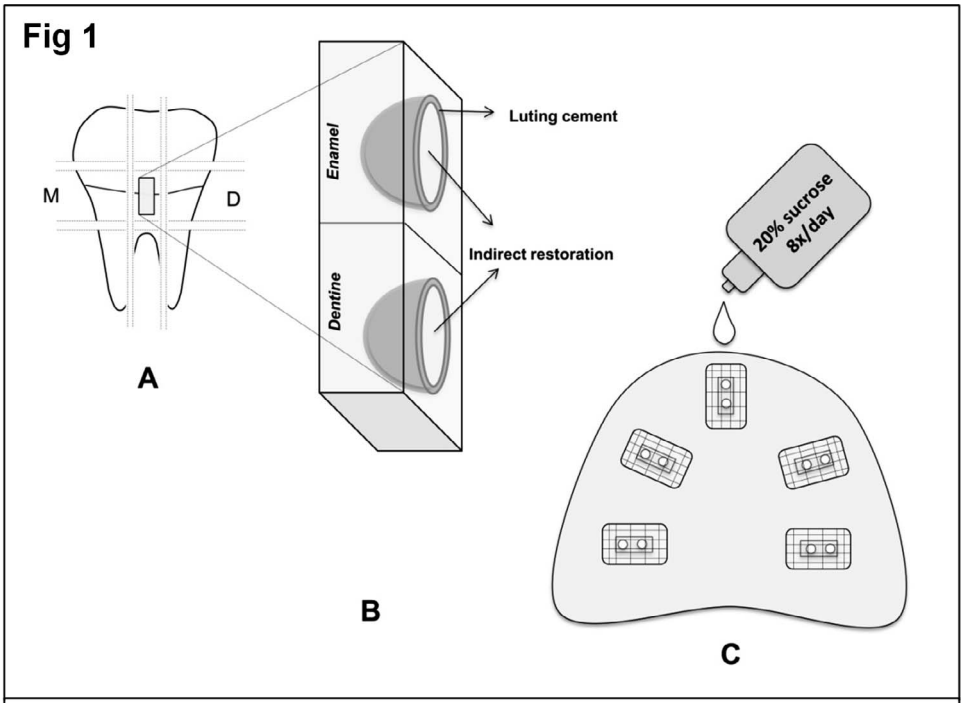

Figure 1. *(A): Schematic illustration of tooth (M, mesial; D, distal) sectioning to obtain blocks. (B): The location of indirect restorations in enamel and dentin. (C): The palatal appliance challenged with 20% sucrose solution.*

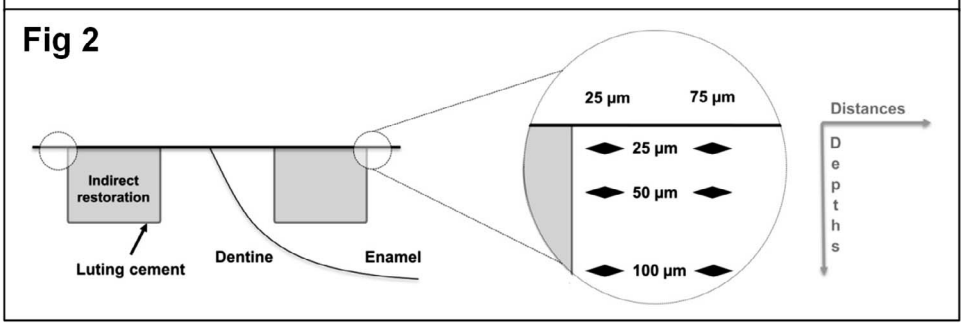

Figure 2. *Schematic representation of indirect restorations and the location of Knoop hardness indentations at two distances (25 and 75 μm) from the preparation margins and three depths (25, 50, and 100 μm) from the outer surface in enamel and dentin.*

ping a 20% sucrose solution eight times per day on each block at predetermined times. After five minutes, the appliance was reinserted in the mouth (Figure 1).[15,22,23]

Following the *in situ* phase, the blocks were laterally included in an epoxy resin and manually polished to expose the inner part of the blocks for Knoop microhardness testing. Polishing was performed under water cooling with #1200 and #2000 SiC sandpaper followed by polishing with a 0.25-micron and a 1-micron diamond paste. The blocks were cleaned with deionized water in an ultrasonic bath for 10 minutes before each sandpaper and paste and after the last polishing.

The Knoop microhardness of the enamel and dentin was measured with a microhardness tester (HMV-2T, Shimadzu Corp, Tokyo, Japan) set at 10*g* for 15 seconds for dentin and 25*g* for 15 seconds for enamel. Indentations were made at 25- and 75-μm distances from the preparation margin and at 25-, 50-, and 100-μm depths from the outer enamel and dentin surface (Figure 2).[20]

Analysis of Fluoride Release

The materials were inserted into Teflon matrices (11 mm in diameter and 1.5 mm in thickness) and pressed between two Mylar strips and glass slides on top and bottom surfaces. The resin cements were light cured on both surfaces for 40 seconds with a light-curing device (Elipar Free Light II, 3M ESPE) and monitored to 1000-mW/cm^2 irradiance every five specimens. Glass ionomer cement was placed on the matrix, pressed for one minute with two glass slides, and allowed to cure for seven minutes. The specimens were removed from the matrices, and excess material was carefully removed by using a surgical blade. After setting time, the GIC specimens were protected with a protective varnish. The materials were inserted in the matrix together with a cotton rope that later kept the specimens suspended inside the tubes.

Table 2: *Mean Values of Knoop Microhardness of Enamel at Different Distances (DI) and Depths (DE)*[a]

DI	DE	GIC	SeT	Max	Smart	Unicem2
25	25	215.37 (7.86) ABCa	203.14 (15.74) ABab	191.57 (7.88) ABCb	158.50 (9.56) Ac	155.19 (14.61) Ac
25	50	221.50 (7.61) ABa	208.00 (7.59) Aab	198.93 (8.03) ABb	162.57 (9.33) Ac	161.27 (14.98) Ac
25	100	222.02 (7.70) Aa	205.21 (11.32) Ab	202.00 (6.40) Ab	169.43 (9.04) Ac	166.55 (14.43) Ac
75	25	200.69 (7.95) Ca	189.50 (12.94) Bab	178.50 (6.31) Cb	157.93 (9.95) Ac	155.44 (12.99) Ac
75	50	206.96 (7.81) BCa	194.36 (13.56) ABab	180.71 (7.09) Cb	160.07 (9.53) Ac	159.74 (12.54) Ac
75	100	208.01 (6.68) ABCa	195.14 (13.28) ABab	185.79 (6.35) BCb	165.07 (8.54) Ac	163.05 (13.44) Ac

[a] *Standard deviations are in parentheses. Mean values with different letters differ statistically ($p<0.05$) in columns (uppercase), comparing distances and depths in the same material, and rows (lowercase), comparing the different materials in the same distances and depths.*

All samples were kept in an oven at 37°C for 24 hours under relative humidity conditions. The GIC discs were then finished and polished (Sof-Lex Finishing and Polishing System, 3M ESPE) under water cooling to remove the varnish layer.[24]

Six specimens from each material were individually immersed in 3 mL of deionized water (ISO FDIS 4049:1999), while six other such specimens were individually immersed in 3 mL of 5 nM lactic acid (pH 5), which accounts for 70% of the total acid found in human dental plaque.[25,26] Specimens were incubated in plastic tubes for 15 days under constant stirring at a temperature of 23°C ± 1°C. The immersing solutions were replaced daily, and the discs were washed in deionized water and dried with paper before changing tubes. The solutions from days 1, 2, 3, 4, 7, 9, 11, 14, and 15 were stored at 4°C in sealed plastic tubes, and those from days 5, 6, 8, 10, 12, and 13 were discarded.

The fluoride concentration of the stored solutions was analyzed in triplicate by adding equal volume of total ionic strength adjustment buffer (TISAB II) solution (1.0 M acetate buffer, pH 5.0, containing 1.0 M NaCl and 0.4% 2-[(2-[bis(carboxymethyl)amino]cyclohexyl)-(carboxymethyl)amino] acetic acid [CDTA]). The analysis was performed using a specific fluoride electrode (ThermoOrion 96-09, Thermo Scientific Fisher, Carlsbad, CA, USA) coupled to an ion analyzer (ThermOrionStar A211, Thermo Scientific Fisher) and calibrated with standard fluoride solutions (0.016 to 32.0 μg F^-/mL in 50% TISAB II). The fluoride concentrations in the media were measured, and the amount of fluoride release was calculated as a function of area (μg F/cm^2).

Statistical Analysis

Normality and homogeneity of variance of Knoop microhardness data were checked with the Shapiro-Wilk and Levene tests. Data were then analyzed by two-way analysis of variance (ANOVA) (factors: material and position). Multiple comparisons were performed by the Games-Howell test (material) and the Tukey test (position). All tests were performed with the SPSS version 22 statistical package ($\alpha=0.05$).

Two-way ANOVA and the Games-Howell test were used for the statistical analysis of fluoride release ($\alpha=0.05$).

The mean cumulative fluoride release (μg F/cm^2) within the first seven days for each cement and the corresponding mean microhardness were compared by the Spearman correlation test ($\alpha=0.05$).

RESULTS

Enamel and Dentin Microhardness

The mean Knoop microhardness values of enamel and dentin at the different distances and depths from the indirect restoration margins are shown in Tables 2 and 3.

Table 3: *Mean Values of Knoop Microhardness of Root Dentin at Different Distances (DI) and Depths (DE)*[a]

DI	DE	GIC	SeT	Max	Smart	Unicem2
25	25	56.98 (5.23) ABCa	47.64 (5.69) Ab	42.36 (4.97) Bbc	37.57 (3.86) Bc	35.61 (8.38) Bc
25	50	58.28 (5.09) ABa	52.57 (4.65) Aab	48.57 (5.60) ABbc	42.93 (3.10) ABc	43.05 (7.07) ABc
25	100	61.16 (6.75) Aa	55.21 (5.25) Aab	51.14 (4.26) Ab	47.43 (5.26) Ab	47.53 (6.46) Ab
75	25	49.00 (5.08) Ca	48.71 (6.22) Aa	44.50 (6.36) ABab	37.50 (4.69) Bb	36.51 (10.78) Bb
75	50	50.92 (6.00) BCa	48.79 (5.22) Aa	44.36 (4.58) ABab	38.50 (4.24) Bb	37.81 (6.77) Bb
75	100	53.40 (5.12) ABCa	53.43 (5.26) Aa	48.57 (5.96) ABab	42.00 (4.95) ABbc	40.12 (7.93) ABc

[a] *Standard deviations are in parentheses. Mean values with different letters differ statistically ($p<0.05$) in columns (uppercase), comparing distances and depths in the same material, and rows (lowercase), comparing the different materials in the same distances and depths.*

Table 4: *Cumulative Fluoride Release (μg F/cm²) of Luting Cements Immersed in Acid or Water During the First Seven Days and Their Percentage Increase (%) in Release from Water to Acid*[a]

Material	Water	Acid	Water/Acid Ratio
GIC	6.0 (4.5) Aa	19.3 (7.1) Ab	0.310
SeT	1.5 (0.6) Ba	10.7 (4.2) Bb	0.140
Max	1.2 (0.7) Ba	6.0 (3.3) Cb	0.200
Smart	0.5 (0.2) Ca	3.9 (2.6) Db	0.128
Unicem2	0.1 (0.1) Da	2.1 (2.3) Eb	0.047

[a] *Standard deviations are in parentheses. Mean values with different letters differ statistically (p<0.05) in columns (uppercase), when comparing the materials, and rows (lowercase), when comparing the solution of immersion.*

In enamel hardness, GIC was statistically superior ($p<0.05$) to SeT at 25-μm distance and 100-μm depth and to Max at all distances and depths. Both Smart and Unicem2 showed the lowest hardness means for enamel at all sites when compared to the other groups ($p<0.05$). The 25- and 75-μm distances differed at 50-μm depth in GIC and Max and at 100-μm depth in Max ($p<0.05$). No other comparison was statistically significant.

In root dentin hardness, GIC was statistically superior ($p<0.05$) to SeT (at 25-μm distance and 25-μm depth), to Max (all depths at 25-μm distance), and to Smart and Unicem2 in all sites ($p<0.05$). SeT was superior to Smart at 25-μm distance and 25-μm depth and 75-μm distance in all depths and to Unicem2 at all sites except at 25-μm distance and 100-μm depth ($p<0.05$). Max differed from Smart and Unicem2 only at 75-μm distance and 100-μm depth ($p<0.05$). At 25-μm distance, the hardness at 100-μm depth was statistically higher than that at 25-μm depth in Max, Smart, and Unicem2. No other comparison was statistically significant.

Fluoride Release

Table 4 presents the mean cumulative fluoride release (μg F/cm²) in acid and water during the first seven days for each luting cement. The fluoride release was statistically higher in acid than in water ($p<0.05$).

When immersed in water, the GIC showed the highest cumulative fluoride release among the tested materials ($p<0.05$). SeT and Max demonstrated similar amounts of cumulative fluoride release ($p>0.05$) but statistically lower than GIC ($p<0.05$). The lowest rates were observed in Smart and Unicem2, which differed statistically from each other as well as from the other materials ($p<0.05$).When immersed in acid, the cumulative fluoride release of all materials differed from each other ($p<0.05$), with the highest average observed for GIC.

Table 5: *Equations and R^2 Values for the Factor Time, Within Each Level of the Factor Material*

Media	Water		Acid	
Material	Equation	R^2 (%)	Equation	R^2 (%)
GIC	$Y = 11.721\ X^{-0.852}$	92.8	$Y = 28.670\ X^{-0.419}$	93.0
SeT	$Y = 2.381\ X^{-0.562}$	89.9	$Y = 19.039\ X^{-0.644}$	95.2
Max	$Y = 1.871\ X^{-0.510}$	72.4	$Y = 12.205\ X^{-0.833}$	96.1
Smart	$Y = 0.735\ X^{-0.500}$	81.0	$Y = 8.634\ X^{-0.995}$	96.8
Unicem2	$Y = 0.177\ X^{-0.611}$	78.9	$Y = 5.916\ X^{-1.596}$	97.1

The fluoride release of the tested materials was measured on a daily basis, and all of them were observed to follow the same pattern of fluoride release. The highest amounts were detected during the first days, tending to decrease with time (Figures 3 and 4).

The fluoride release curves were adjusted to estimate the mean values of fluoride release over 15 days by using the exponential function $Y = \alpha X^{\beta}$ (Table 5), where Y is the mean cumulative fluoride release at a given time, α and β are constants for each material, and X is the variable "time" in days.

The mean cumulative fluoride release in water and lactic acid during the first seven days for each luting cement correlated positively with the overall mean hardness value of enamel (water: $R=0.82$, $p=0.08$; acid: $R=0.92$, $p=0.02$) and dentin (water: $R=0.86$, $p=0.06$; acid: $R=0.95$, $p=0.01$). In addition, statistically significant and positive correlations were detected between enamel and dentin hardness means ($R=0.99$, $p=0.001$) and between cumulative fluoride release means in water and acid ($R=0.96$, $p=0.008$).

DISCUSSION

The null hypotheses were rejected, as the potential to release fluoride and to inhibit demineralization in enamel and root dentin varied between the luting cements. The tooth/restoration interface affects the uptake of fluoride from restorative materials by increasing its concentration and creating a greater on-site diffusion potential.[3] In the present study, the interface between the indirect restoration and the tooth structure allowed the cement film to release fluoride, which could act through the liquid phase adjacent to the tooth structure, preventing its demineralization.[27]

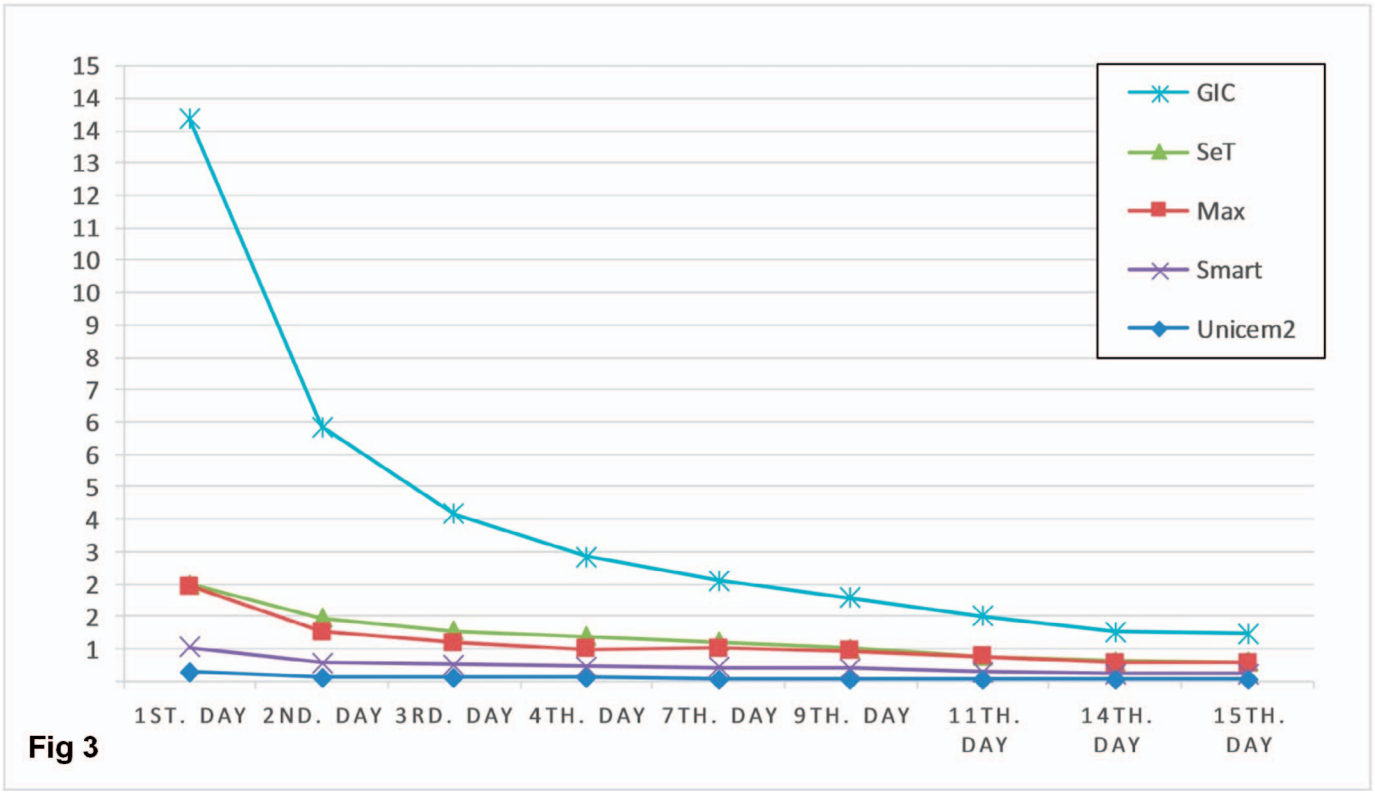

Figure 3. *Daily fluoride release mean (μg F/cm^2) in water.*

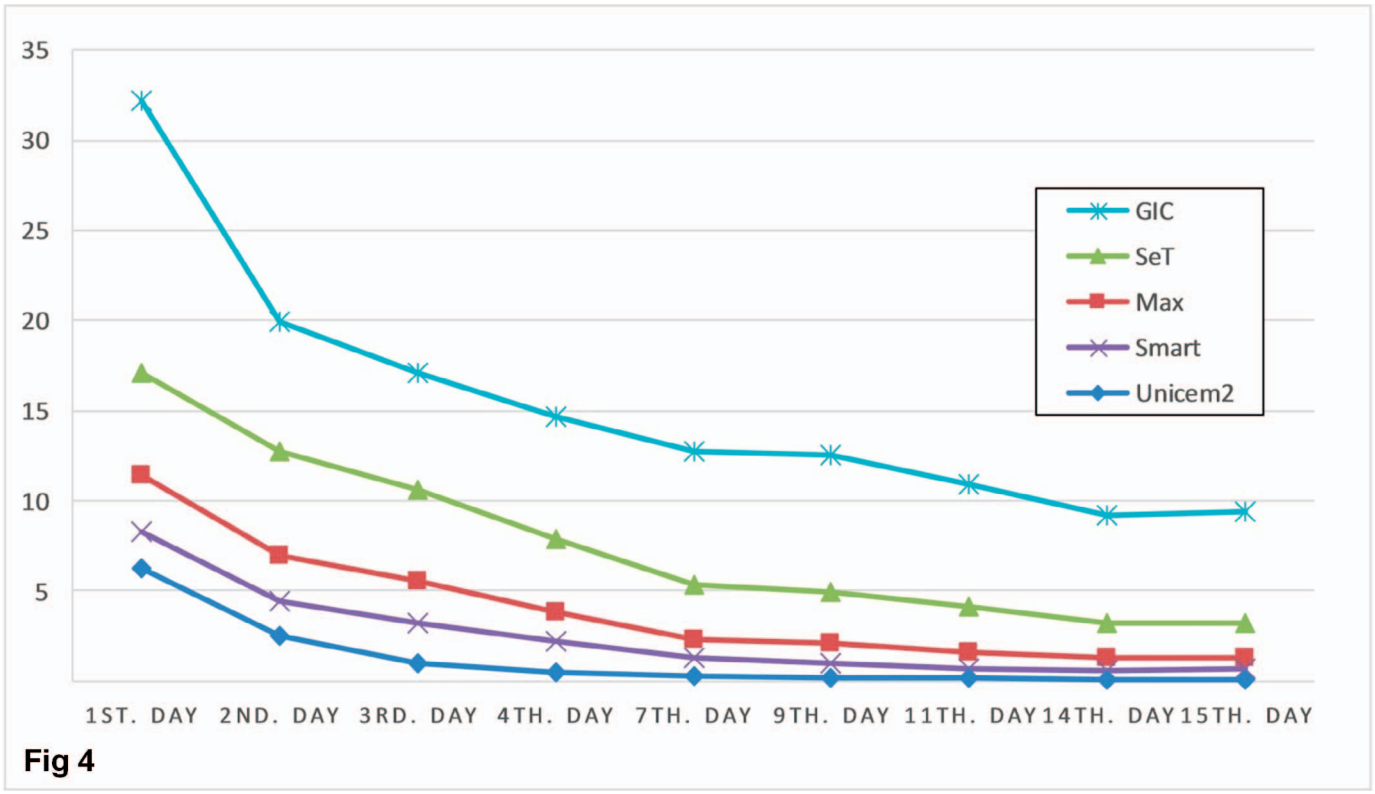

Figure 4. *Daily fluoride release mean (μg F/cm^2) in lactic acid.*

Knoop microhardness was evaluated at two distances from the luting cement interface and three depths from the outer surface of enamel and root dentin. Previous studies have reported the success of microhardness testing in caries inhibition zones adjacent to areas restored with fluoride-releasing materials.[16,28-31] The increase or decrease in hardness reflects the gain (remineralization) or loss (demineralization) of minerals in the dental structure since there is a significant correlation between Knoop hardness and the percentage of mineral.[32] Indeed, the positive correlation between the means of the cumulative fluoride release and the microhardness in enamel and root dentin demonstrates a trend between higher amounts of fluoride release and demineralization inhibition.

The measurement of mineral loss in the oral environment by *in situ* models is useful to study the anticaries potential of fluoride-releasing materials.[14,24] Because mineral loss rate can be twice as fast from the root as it is from enamel,[33] the potential of the luting cements to inhibit demineralization was accessed in both enamel and root dentin. Moreover, the fluoride delivered from dentifrices can overwhelm the anticaries effect of fluoride-releasing materials.[34] In the present study, all volunteers were instructed to use a fluoride toothpaste for their daily oral hygiene. In spite of that, the glass ionomer cement and the self-adhesive resin cements showed distinct potential in preventing demineralization on enamel and root dentin. These results do not agree with previous ones, where zinc phosphate, glass ionomer, and resin cements were not different regarding inhibitory effects on demineralization of enamel and root dentin adjacent to indirect restorations.[9,10] The absence of a high cariogenic challenge in these studies may explain this difference.

The anticaries property of restoratives is associated with the amount of fluoride released and is explained by several mechanisms, including the reduction of demineralization. Two different approaches for the development of fluoride-releasing resin materials are the addition of water-soluble salts (NaF or SnF_2) and fluoride-releasing glass filler systems. In the present study, glass ionomer cement released about 9 to 60 times more fluoride than the SARCs. Indeed, the literature shows that conventional GICs release much higher amounts of fluoride than resin-based materials.[3] A gradual dissolution of the glass particles releases fluoride continuously, creating a direct association between release and material solubility.[35-38] The inorganic filler content by weight of the SARCs declared by the manufacturers is approximately 60% to 70%. However, such range represents the amount of fluoride-containing glass fillers and other non–fluoride-containing particles. In addition, less hydrophilic matrix of resin cements probably reduced the water diffusion and the following release of fluoride from the material in contrast to the aqueous phase of GICs, which enables fluoride ions to diffuse and to be released into the media.[39]

SeT was the only SARC that resulted in enamel and root dentin hardness comparable to that of the GIC at most distances and depths. After SeT, Max resulted in hardness values comparable to GIC, but these equivalences were mainly in dentin depths that were 75 μm distant from the restoration margins. SeT and Max have fluoro-alumino-silicate glass particles that certainly released fluoride into the media following a diffusion movement down the concentration gradient as occurs in GICs. Although Smart and Unicem2 also have glass particles in their composition, these materials failed to achieve enamel and dentin microhardness values comparable to the glass ionomer cement. Smart contains strontium fluoride (SrF_2), which releases fluoride most likely by means of an exchange reaction. Sr is claimed to be beneficial in terms of potentiating the anticaries effect of F, although the optimum concentrations of these elements when combined with each other remain unclear.[40]

Mineral loss rate can be twice as fast from root dentin as it is from enamel.[33] Therefore, fluoride-containing luting resin cements could be beneficial against secondary lesions associated with indirect restorations with margins in dentin. The literature shows that GIC inhibits demineralization of enamel[14] and dentin[15] *in situ* and remineralizes these tissues under cariogenic challenge to a depth of approximately 100 μm.[20] The present study revealed a similar behavior of the materials in both enamel and root dentin. Knoop microhardness values were higher at indentations that were deeper from the outer enamel and dentin surface, probably because the pronounced effect of biofilm on the demineralization of the outer surfaces. Indentations located at a distance of 25 μm revealed higher Knoop hardness numbers than those at distances of 75 μm. Also, three of the four significant pairwise comparisons among the three highest fluoride-releasing materials (GIC, SeT, and Max) occurred at a distance of 25 μm, probably influenced by the proximity of the indentations to the fluoride-releasing cements at the margins of the restorations.

The amount of fluoride released by the glass ionomer and the self-adhesive resin cements increased when they were immersed in acid probably because of their higher dissolution under a lower pH.[41] The water/acid ratio of the materials ranged from a low of 0.047 for Max to a high of 0.310 for GIC. Similarly, a previous study with a pH-cycling regimen showed that Max exhibited higher amounts of fluoride ions in the remineralizing solution than in the demineralizing solution at the first day.[13] Differences in matrix solubility and filler particle sources between the SARCs probably explain their distinct levels of dissolution in water and acid.

Caries is a chronic disease with a daily and continuous progression. Thus, fluoride must be constantly available in the mouth to affect the disease process. An updated systematic review suggested that GIC has a higher caries-preventive effect than amalgam in margins of restorations in permanent teeth.[42] Fluoride release from luting cements may not be as effective as direct restoratives in reducing demineralization of hard tissues due to the small area of the material exposed to the oral environment. The fluoride released by the GIC was higher in the first 24 hours, which agrees with previous studies.[3,5] The initial high release is likely due to the burst of fluoride released from the glass particles that react with polyalkenoate acids during the setting reaction.[3] Then fluoride release from glass ionomers slows down and exhibits a prolonged long-term release,[43] as observed in the present study. Similar to the GIC, the amount of fluoride released from the SARCs in both media underwent a significant reduction during the 15 days of immersion. SeT, MaxCem, and Smart SARCs slow down the fluoride release until the 15th day, and the concentration measured varied from 0.17 μg F/cm^2 (Smart in water, 15th day) to 17.12 μg F/cm^2 (SeT in acid, first day). Very low concentrations of fluoride in water (0.04 μg F/cm^2) and in acid (0.09 μg F/cm^2) were detected for Unicem2 on the last day of cycling, showing that the fluoride source of this material is finite. Similarly, SARCs exposed to a pH cycling showed a gradual decrease in the fluoride release amount with time.[13]

Amounts of fluoride released *in vitro* by GICs range from 5 to 155 ppm in specimens 1 to 1.5 mm thick and 6 mm in diameter.[3] In this study, all materials, except RelyX Unicem 2, released initial amounts of fluoride that exceeded this level. Mean concentration of fluoride release from glass ionomer specimens (1.5 mm thick, 11 mm in diameter) into water/acid ranged from 99.7 to 239.1 ppm (24 hours) to 8.3 to 69.5 ppm (15th day). Fluoride release in water and acid from SeT—the SARC that most released fluoride—ranged from 19.5 to 138.0 ppm (first day) to 3.8 to 25.8 ppm (15th day). Although fluoride-releasing dental materials present the necessary properties to be effective in caries control,[34] none of the materials in the present study showed the potential to inhibit demineralization completely in both hard tissues since the Knoop hardness in all groups was below 272 to 440 KHN in enamel[44] and 55 KHN in dentin.[45] This finding agrees with the knowledge that fluoride alone reduces the development of caries but does not fully prevent its progression.[34]

CONCLUSIONS

Glass ionomer luting cement showed the highest overall potential to minimize demineralization in enamel and dentin around indirect restorations. Only one SARC (SeT) was comparable to the GIC in protecting enamel and dentin against demineralization. The GIC showed the highest cumulative fluoride release in water and acid. For GIC and SARCs, the highest fluoride release occurred on the initial days and tended to decrease with time.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the PUCPR. The approval code for this study is 491524.

Conflict of Interest

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

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Effect of a New Bleaching Gel on Tooth Whitening

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Clinical Relevance

The use of the novel tribarrel hydremide-peroxide bleaching system (KöR) did not offer any advantages in the lightening of bovine teeth compared with a traditional bleaching system (Opalescence) of hydrogen peroxide or carbamide peroxide.

SUMMARY

The purpose of this study was to compare the whitening efficacy of a novel bleaching agent containing a unique tribarrel hydremide-peroxide gel (KöR) with a traditional bleaching system of hydrogen peroxide or carbamide peroxide (Opalescence). Bovine incisors were mounted into a custom resin, arch-shaped mounting device. Four groups of 10 teeth were created using mounting devices containing five teeth each. The in-office and home bleaching gels of KöR and Opalescence were applied to the teeth alone and in trays to simulate a combination of in-office and home bleaching or home bleaching only. Spectrophotometer readings of L* a* b* were performed at baseline, the end of active bleaching (immediate), and three and six months postbleaching. Immediately postbleaching, the use of Opalescence gel resulted in greater change in ΔE* and Δb* (less yellow) for combined and home bleaching techniques compared with KöR. After six months, Opalescence had significantly greater ΔE* and Δb* compared with KöR for home bleaching only. There was no significant difference in ΔL* between Opalescence and KöR at any time period with either technique.

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INTRODUCTION

Much emphasis is placed on outward appearance and often, a person's smile is what meets the eye first. Many people are enamored by whiter enamel, creating a high demand for esthetic bleaching. As such, home and in-office whitening products infiltrated the US marketplace in the late 1980s, and the bleaching empire has exploded ever since.[1] Whitening products include toothpastes, gels, and films, as well as in-office–based systems.[1] In 2005 alone, the in-office and home bleaching market generated $2 billion in sales.[2] With such high demand and potential for profitability, there is a constant race for companies to create the latest and greatest whitening agents while adhering to the former American Dental Association (ADA) safety and efficacy guidelines.[3]

Vital bleaching is a relatively conservative way to achieve whiter teeth vs therapy such as micro-

abrasion, veneers, or crowns. Generally there are two vital bleaching modalities: power bleaching (in-office) with 25%-35% hydrogen peroxide (HP) and home bleaching with 10%-20% carbamide peroxide (CP) or 2%-10% HP in a custom-made mouth guard over two to six weeks.[4,5] The advantages of home whitening include ease of application, reduced chair time and cost, high success rate, and safety of materials.[6] Furthermore, bleaching teeth with 10% CP in a custom-fitted tray has proven to be the safest, most cost-efficient whitening option for a large variety of tooth discoloration conditions.[7] The degree of whitening may be similar for home bleaching and in-office bleaching, but in-office bleaching has more rapid regression.[5,8] Home bleaching may be very successful for a compliant patient because it offers the safest, least expensive way to get whiter teeth with slower regression.

Although the outcomes seem revolutionary, the bleaching process and materials are not new. HP has been a dental workhorse for more than 70 years.[4] Bleaching success is attributed to its ability to penetrate tooth structure and produce free radicals that oxidize organic stains within the tooth.[9] During the bleaching process, CP breaks down into HP and urea, with the HP concentration being approximately one-third of the original CP concentration.[6] Therefore, a 15% CP product is approximately 5% HP. Home bleaching with 10% CP was first reported by Haywood and Heymann[10] in 1989, and although there are many different concentrations of bleaching agents, 10% CP seems to be the gold standard. In fact, 10% CP (specifically, 10% CP Opalescence, Ultradent Products, South Jordan, UT, USA) was the only bleaching product to have earned the ADA seal of approval. If a patient has the time, bleaching with 10% CP can be just as effective, if not more effective, than in-office bleaching with 35% HP. Bernardon and others,[8] in 2010, illustrated such results with a clinical study involving 90 patients who found that home bleaching with 10% CP in a tray was comparable to in-office bleaching with 35% HP. Both home and in-office bleaching have been extremely successful. As such, several manufacturers have attempted to combine the two techniques to develop a more efficacious procedure.[11] Combination bleaching can be described as vital bleaching with 10%-20% CP or 2%-10% HP in a custom tray over two to six weeks with supplemental use of in-office vital bleaching with 25%-35% HP before or after home whitening.[11]

Due to the prevalence of HP and CP in many bleaching products, the dental literature is saturated with research on their respective safety and efficacy. Just recently, a literature review concluded that when manufacturer's instructions are followed, HP- or CP-based tooth whitening is safe and effective.[1] But now there is a new type of bleaching gel. KöR Whitening (Evolve Dental Technology Inc, Irvine, CA, USA) contains a novel dual-activated hydremide-peroxide bleaching gel which the company claims[12] creates a whiteness that is unaffected by consumption of coffee, tea, or red wine with low to no sensitivity. The peroxide gel base is dispensed in a tribarrel system that allows the use of two distinctly different proprietary chemical accelerators. Three separate syringes are mixed together immediately before application. According to the company, KöR 34% hydremide peroxide gel is roughly equivalent to 30% HP and KöR 13% hydremide peroxide is roughly equivalent to 9% HP.[12] Refrigeration is provided throughout all phases of storage and shipping to reduce peroxide degradation. The KöR system also requires a proprietary tray (KöR-Seal trays) that reportedly provides a unique seal of the cervical 1- to 1.25-mm portion of teeth that functions to seal out both saliva and sulcular fluid. The KöR-Seal trays in combination with continuous refrigeration reportedly provide six to 10 hours of whitening activity compared with the typical 25-35 minutes.[12] Other than company articles provided by the inventor, Rod Kurthy, there is no independent research published on the KöR Whitening system.[13,14]

The purpose of this *in vitro* study was to compare the tooth whitening capability of KöR bleaching gels with popular comparable Ultradent Products on bovine incisor crowns. Changes in color of the bovine teeth from the bleaching process were determined using the Commission Internationale de l'Eclairage (CIE) L*a*b* color space. L* indicates lightness (L+ = lightness and L– = darkness), the a* coordinate represents the red/green range (a*+ = redness and a*– = greenness), and the b* coordinate represents for the yellow/blue range (b* + = yellowness and b*– = blueness).[15] The L*a*b* system allows the numeric definition of a color as well as the overall difference between two colors (ΔE). The null hypothesis was that there would be no difference in the change in L* (ΔL), a*, (Δa), b* (Δb), and E* (ΔE*) on the basis of type of bleaching gel per technique over time.

METHODS AND MATERIALS

A total of four groups were created in this study to evaluate the whitening of crowns of bovine incisors using either a combined bleaching technique of in-office and home bleaching (Combined) or home

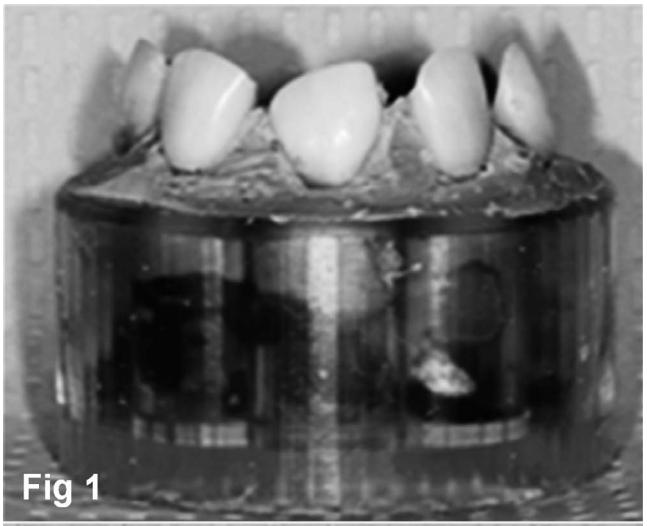
bleaching alone (Home) using KöR and Opalescence bleaching gels.

For the Combined bleaching, two groups were compared using the KöR Max Ultra Kit (Evolve Dental Technology) and Opalescence Boost 40% HP / Opalescence PF 15% CP bleaching gel (Ultradent Products). KöR bleaching kits are sold in three varieties: KöR Standard, KöR Max, and KöR Max Ultra. The KöR Max Ultra Kit was chosen for this study for combined bleaching because it is marketed for use with difficult cases and consists of 13% hydremide peroxide for three 20-minute chairside sessions, 16% CP home bleaching for 28 nights, and 34% hydremide peroxide for three 20-minute chairside sessions.

The Opalescence Boost 40% HP was used with Opalescence PF 15% CP home bleaching gel to create a combined bleaching technique. The Opalescence Boost 40% HP in-office bleaching gel is a dual-barrel system. Two separate syringes are mixed together immediately before application. Two additional groups were created to evaluate the home bleaching technique only. KöR 16% CP was compared with Opalescence PF 15% CP—two home bleaching gels with similar concentrations of CP. The four groups are outlined below:

- Group 1 (Combined): KöR 13% hydremide peroxide and 34% hydremide peroxide (KöR Max Ultra Kit) were used to simulate in-office bleaching, whereas KöR 16% CP was used in custom trays as a home bleaching agent.
- Group 2 (Combined): Opalescence Boost 40% HP was used as an in-office bleaching agent and Opalescence PF 15% CP was used in custom trays to simulate home bleaching.
- Group 3 (Home): KöR 16% CP was used in custom bleaching trays to simulate home bleaching.
- Group 4: (Home): Opalescence PF 15% CP was used in custom bleaching trays to simulate home bleaching.

Forty bovine incisors (Animal Technologies, Tyler, TX, USA) were stored and disinfected in 0.5% chloramine-T (Alfa Chemistry, Stony Brook, NY, USA). The teeth were then examined under a light microscope at 10× magnification (Nikon SMZ-1B, Melville, NY, USA) and discarded if any gaps, cracks, or pigmentation was found that would interfere with the bleaching evaluation. Custom arch-shaped mounting devices were created to provide a mechanism for bleaching tray fabrication and insertion as shown in Figure 1. The custom resin, arch-shaped mounting devices were created using a stereolithography printer



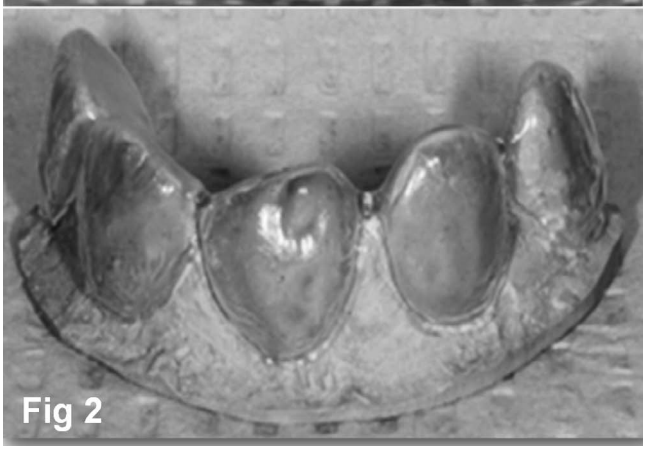


Figure 1. *Teeth mounted in custom resin device.*
Figure 2. *Custom tray fabrication.*

(Viper si2 SLA System, 3D Systems Inc, Darmstadt, Germany) with a clear resin material (Somos WaterShed XC 11122, DMS Functional Materials, Elgin, IL, USA). Each device contained five mounting wells for the bovine teeth. Two mounting devices were printed per group, for a total of 10 teeth per group (n=10). Each tooth was mounted in the wells with vinyl polysiloxane (Regisil PB, Dentsply, York, PA, USA). After all the bovine teeth were mounted in the devices, custom trays were created per the manufacturer's guidelines using each company's proprietary block-out and tray materials, as shown in Figure 2.

KöR-Seal whitening trays were fabricated for use with KöR bleaching products. KöR-Seal trays require several steps and can be accomplished in-office or by mailing impressions to the KöR-Lab. All KöR-Seal trays for this study were fabricated by the research investigators following the manufacturer's instructions. Impressions of the mounted bovine teeth were made using KöR polycarbonate impres-

sion trays. After painting the impression trays with KöR Impression Tray Adhesive, the base impression was made with KöR-Seal VPS Putty. Evolve BLU-grinder burs were used to grind out the base impression. The wash impression was made with KöR Light Body Wash VPS. Impressions were allowed to degas 30-60 minutes, then were sprayed with debubblizer (Smoothex, Whipmix Corporation, Louisville, KY, USA) and poured in dental stone (Denstone, Heraeus Kulzer, South Bend, IN, USA). The model bases were trimmed and blocked out with the same dental stone (0.5- to 1-mm thick, 1 to 1.25 mm from gingival line) per the manufacturer's recommendations. The KöR-Seal trays were made with a soft 0.40-inch ethylene vinyl acetate sheet via a positive-pressure, thermal-forming machine (Biostar, Great Lakes Orthodontics, Tonawanda, NY, USA). To create bleaching trays for home bleaching with Opalescence PF 15% CP, an alginate impression was made of the mounted bovine teeth and poured in dental stone (Die Keen, Heraeus Kulzer, South Bend, IN). Model bases were trimmed and Ultradent LC Block-Out Resin was applied per manufacturer recommendations (0.5-mm thick, 1.5 mm from gingival line). Custom trays were made with 0.035-inch Sof-Tray with the positive-pressure, thermal-forming machine as before.

The superficial enamel of the bovine teeth was cleaned with prophy paste (Nupro, Dentsply, York, PA, USA) using a disposable prophy angle (Nupro Revolv, Dentsply) to remove extrinsic stains. Due to a higher inherent value of bovine teeth, a tea-staining protocol was used as described by D'Arce and others,[16] after mounting the teeth and prior to Home or Combined bleaching. The mounting devices with the bovine teeth were immersed in a solution of black tea for six consecutive days at room temperature in order to create a standardized, stained surface of the enamel.[17] The tea solution was created by soaking one filter bag (Starbucks Awake English Breakfast Tea, Starbucks, Seattle, WA) in eight ounces of boiling water for five minutes. The tea solution was changed every 24 hours. Then, the teeth were stored in synthetic saliva at oral temperature at 37°C for seven days in a laboratory incubator (Model 20GC, Quincy Lab, Chicago, IL, USA) to stabilize the stained surfaces. The synthetic saliva was prepared as described by Lata,[18] using Na_3PO_4, 3.90 mM; $NaCl_2$, 4.29 mM; KCl, 17.98 mM; $CaCl_2$, 1.10 mM; $MgCl_2$, 0.08 mM; H_2SO_4, 0.50 mM; $NaHCO_3$, 3.27 mM; and distilled water, with a pH set to a level of 7.2. A pH meter (Accumet XL50, Fisher Scientific, Waltham, MA, USA) was used to measure pH.

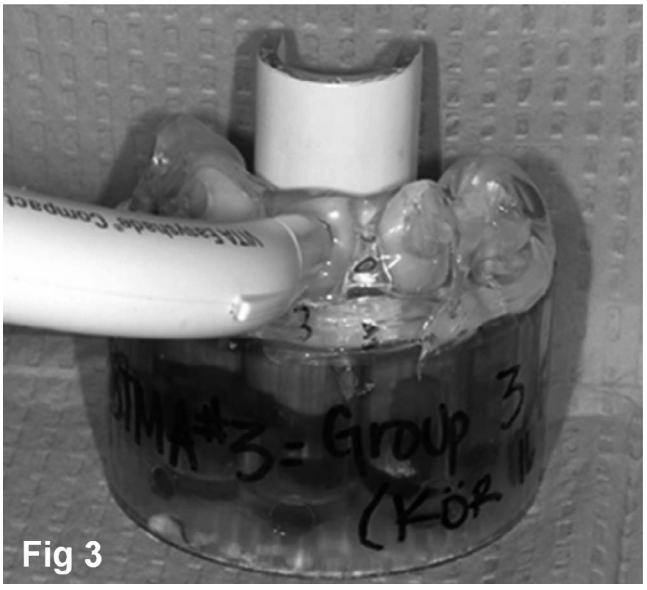


Figure 3. *Clear custom stent to standardize placement of spectrophotometer tip.*

A customized, resin insert was printed using stereolithography and sprayed with white paint. The insert was placed in the mounting device during spectrophotometer readings (Easyshade Compact, VITA, Bad Säckingen, Germany). Teeth were positioned so that the lingual surfaces were parallel to the white insert. A baseline reading was performed using the spectrophotometer. The tip of the spectrophotometer was placed into a customized clear vinyl polysiloxane stent that rested on the teeth to create a standardized position for recording, as shown in Figure 3.

After the baseline reading, the bleaching procedures were performed according to the following four protocols. All bleaching agents were stored in a refrigerator at 1.5°C when not in use (Model HB27AW, Hamilton Beach, Glen Allen, VA, USA), verified with a thermometer (No. 5295, Taylor, North Mankato, MN, USA). KöR bleaching gels arrived from the manufacturer in a refrigerated container.

Group 1 (KöR, Combined)

- Day 1: To simulate in-office bleaching, a 1-mm layer of KöR 13% hydremide-peroxide gel was applied to the teeth using a custom tray for 20 minutes. Bleaching gel was suctioned off the teeth, and a new mix of KöR 13% hydremide-peroxide gel was again applied to the teeth using a custom tray for another 20-minute session. The bleaching gel was suctioned off once again and the teeth were thoroughly rinsed, dried, and stored in synthetic saliva at 37°C.

- Days 1-28: To simulate home bleaching, KöR 16% CP was applied to the teeth for eight hours using a custom tray. The first eight-hour session of home bleaching was the same day as the in-office bleaching. After each daily eight-hour tray bleaching session, the teeth were thoroughly rinsed and dried and stored in synthetic saliva at oral temperature at 37°C. The home bleaching process was repeated every day for 28 days.
- Day 29: To simulate in-office bleaching once again, 1 mm of KöR 34% hydremide-peroxide gel (instead of KöR 13% hydremide-peroxide gel) was applied to the enamel surfaces. The teeth were bleached for three 20-minute sessions instead of two, as recommended by the manufacturer.

Group 2 (Opalescence, Combined)

- Day 1: To simulate in-office bleaching, 1 mm of Opalescence Boost 40% HP gel was applied to the teeth for 20 minutes. The bleaching gel was suctioned off the teeth, and a second 1-mm layer of Opalescence Boost 40% HP gel was applied to the teeth for another 20-minute session. The bleaching gel was suctioned off teeth once again, and a third and final 1-mm layer of Opalescence Boost 40%HP gel was applied to the teeth for the last 20-minute session. At the end of the third bleaching session, bleaching gel was suctioned off and the teeth thoroughly rinsed, dried, and stored in synthetic saliva at 37°C.
- Days 1-4: A four-day resting period was allowed between in-office treatments per manufacturer recommendations.
- Day 5: To simulate in-office bleaching, the Opalescence Boost 40% HP gel was applied to the teeth in the same manner as on Day 1.
- Days 5-8: Resting period. A second four-day resting period was allowed, as after the first in-office treatment. No in-office bleaching gel was applied again until day 9.
- Days 9-30: To simulate home bleaching, 1 mm of Opalescence PF 15% CP was applied to the teeth for six hours using a custom tray following the manufacturer's instructions. After each daily six-hour tray bleaching session, the teeth were thoroughly rinsed, dried, and stored in synthetic saliva at oral temperature at 37°C. The bleaching process was repeated every day for 21 days.

Group 3 (KöR, Home)

- Days 1-21: To simulate home bleaching, KöR 16% CP was applied to the teeth for eight hours with a custom tray following the same technique as described previously with the KöR Combined group.

Group 4 (Opalescence, Home)

- Days 1-21: To simulate home bleaching, Opalescence PF 15% CP was applied to the teeth for six hours using a custom tray using the same technique as described previously with the Opalescence Combined group.

All teeth specimens were stored in artificial saliva at 37°C between active bleaching. The bovine teeth were measured immediately after completion of bleaching and at three and six months postbleaching using a spectrophotometer. L*a*b* values were recorded for each tooth at each time interval. ΔL^*, Δa^*, and Δb^* were calculated by subtracting each reading from baseline per time interval. Baseline values were similar but not the same for all groups. ΔE^* was determined using the following formula: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. Data were analyzed with a repeated-measures analysis of variance (ANOVA) examining the effects of type of bleaching (Home vs Combined) or type of bleaching agent (KöR vs Opalescence) on ΔE^* or Δb^* over time ($\alpha=0.05$). However, significant interactions were found ($p<0.05$). The ΔE^* and Δb^* data were evaluated with a one-way ANOVA per bleaching gel and treatment type and unpaired *t*-tests between bleaching gels and treatment type. A Bonferroni correction was applied because multiple comparison tests were completed ($\alpha=0.01$). Due to the large variability of the data, ΔL^* was analyzed with Kruskal-Wallis and Mann-Whitney U-tests with a Bonferroni correction ($\alpha=0.01$).

RESULTS

Immediately postbleaching, the use of Opalescence gel resulted in greater change in ΔE^* and Δb^* for Combined and Home bleaching techniques compared with KöR ($p<0.01$). After six months, Opalescence had significantly greater ΔE^* and Δb^* compared with KöR for Home bleaching only ($p<0.01$). There was no significant difference in ΔL^* between Opalescence and KöR at any time with either technique ($p>0.01$). Also, there was no significant difference in ΔE^*, b*, or L* for either material or technique on the basis of time ($p>0.01$), as shown in Table 1.

DISCUSSION

The purpose of this *in vitro* study was to evaluate the tooth whitening capability of a new bleaching gel

Table 1: *ΔE*, Δb*, and ΔL* Values for Combined and Home Bleaching Techniques for both Opalescence and KöR Bleaching Gels*[a]

	Combined Bleaching			Home Bleaching		
	Immediate	3 mo	6 mo	Immediate	3 mo	6 mo
ΔE*						
Opalescence	14.5 (5.5) Aa	13.2 (5.2) Aa	13.8 (4.6) Aa	20.4 (7.9) Aa	19.9 (5.5) Aa	20.7 (6.6) Aa
KöR	8.5 (4.0) Ab	11.3 (3.2) Aa	10.4 (3.3) Aa	10.9 (3.2) Ab	14.5 (3.2) Aa	14.0 (3.5) Ab
Δb*						
Opalescence	−11.4 (3.9) Aa	−7.1 (3.1) Aa	−9.5 (3.3) Aa	−17.8 (5.0) Aa	−17.4 (3.0) Aa	−18.7 (4.9) Aa
KöR	−4.7 (3.9) Ab	−6.0 (2.8) Aa	−6.5 (2.1) Aa	−9.6 (3.0) Ab	−12.9 (3.0) Ab	−13.2 (3.2) Ab
ΔL*						
Opalescence	7.9 (4.1) Aa	10.8 (4.0) Aa	9.4 (3.4) Aa	6.9 (8.8) Aa	8.1 (6.3) Aa	6.3 (7.1) Aa
KöR	6.3 (2.9) Aa	9.5 (2.2) Aa	7.9 (2.4) Aa	4.6 (2.6) Aa	5.3 (3.5) Aa	3.5 (3.3) Aa

[a] *Groups with the same uppercase letter per row and treatment or same lowercase letter per column and treatment are not significantly different ($p>0.01$).*

system (KöR) containing a unique hydremide-peroxide formulation. KöR bleaching gels were compared with popular bleaching gels from Ultradent Products on bovine incisor crowns using the CIE L* a* b* color space system.

The null hypothesis was rejected. There was a difference in Δb* and ΔE* on the basis of type of bleaching gel per technique. However, the differences varied over time. For both Combined and Home bleaching, the use of Opalescence gel resulted in a greater change in ΔE* compared with KöR immediately after bleaching. A greater change in ΔE* is associated with an overall greater change in color. However, ΔE* does not provide specific information as to the direction of the color change. Previous literature has shown that whitening from bleaching agents is manifested mainly by a reduction in yellowness (lower b*) and an increase in lightness (higher L*) and to a minor extent, a reduction in redness (lower a*).[19-24] Whereas all values (L* a* b*) were obtained at all intervals of this study, ultimately only ΔE*, Δb*, and ΔL* were reported. Due to the variability and fluctuation of a*, the Δa* values were not analyzed and were deemed singularly noncontributory.[20] These findings are consistent with a study by Lenhard[25] that found that the variance in a* values had only a minor influence on color change.

The use of Opalescence gel resulted in a greater change in Δb* (ie, less yellow) compared with KöR immediately after bleaching for both Combined and Home bleaching. However, after six months post-bleaching, Opalescence had significantly greater Δb* compared with KöR for Home bleaching only. Home bleaching was associated with a greater reduction in yellowness (Δb*) than Combined bleaching; however, there was a tendency for Combined bleaching to result in greater lightness (ΔL*) than Home bleaching, suggesting no overall benefit from either technique. Two recent clinical studies found no statistically significant difference between combined and home bleaching.[11,26] However, more sensitivity was reported with combined bleaching compared with home bleaching.[26] In this study, there was no statistically significant difference in ΔL* between Opalescence and KöR at any time with either technique. Although the use of Opalescence gel may have initially resulted in a greater reduction in yellowness compared with KöR with either Combined or Home bleaching, the difference became less significant over time with Combined bleaching. Although not statistically significant, ΔE* and b* values had a tendency to improve with KöR over the first three months after bleaching and remain more stable with Opalescence. In all groups, bleaching treatment resulted in a significant overall color change (ΔE*) above the limit of visible detectability, which has been reported to be greater than 3.3 units.[27,28] Wiegand and others[20] evaluated the effects of bleaching agents using enamel and dentin bovine segments. After 12 months of storage in artificial saliva, the researchers found no difference in color retention between the different bleaching techniques (home, in-office and walking) using the CIE L* a* b* color system. However, the color change was not stable, with the greatest regression with ΔL*.[20] These results differ from this study, however, where after six months of storage in artificial saliva there was no significant change in ΔE*, b* or L* for either material or technique based on time. Longer storage time may be necessary to demonstrate regression of the whitening effect. Also, in the study by Wiegand and others,[20] the authors did not use artificial

staining prior to the bleaching procedure. Although superficial prestaining of bovine teeth has been used in multiple *in vitro* bleaching studies, it is uncertain to what extent the tea-staining protocol augmented the natural organic discoloration of the bovine teeth.[29-32] Bovine teeth are much more readily available than human anterior teeth, have similar physical chemistry to human teeth, and provide similar results when staining or whitening procedures are evaluated in the laboratory.[16,33-36]

CONCLUSIONS

Within the limitations of this study, the following statements can be made. Immediately after bleaching, the use of Opalescence gel resulted in greater change in ΔE^* and Δb^* (less yellow) compared with KöR for both bleaching techniques. After six months, Opalescence had significantly greater ΔE^* and Δb^* for Home bleaching only. The use of the novel tribarrel hydremide-peroxide bleaching system (KöR) did not offer any advantages in the lightening of bovine teeth compared with a traditional bleaching system (Opalescence) of HP or CP.

Acknowledgement

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Wilford Hall Ambulatory Surgical Center IACUC. The approval code for this study is FWH20150018A.

Disclaimer

The views expressed in this article are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, Uniformed Services University of the Health Sciences, or the United States government.

Conflict of Interest

The authors do not have any financial interest in the companies or products mentioned in this article.

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Retentive Strength of Y-TZP Crowns: Comparison of Different Silica Coating Methods on the Intaglio Surfaces

VF Wandscher • C Prochnow • MP Rippe • LS Dorneles • GL Callegari • P Baldissara • R Scotti • LF Valandro

Clinical Relevance: Tribochemical silica coating and 5-nm thick silica nanofilms conditioning methods used on intaglio surfaces of zirconia crowns improves retention.

doi: http://dx/doi.org/10.2341/16-090-L

Sudden Onset of Subcutaneous Air Emphysema After the Application of Air to a Maxillary Premolar Located in a Nonsurgical Field

EA Van Tubergen • D Tindle • GM Fox

Clinical Relevance: Subcutaneous emphysema commonly develops when compressed air enters through a surgical site. Forced air may inadvertently enter into subcutaneous tissue through intact mucosa. Clinicians must be aware of this possibility to diagnose and manage subcutaneous emphysema during nonsurgical treatment.

doi: http://dx/doi.org/10.2341/15-155-S

Using CAD/CAM–Modified Correlation Mode to Produce Laminate Veneers: A Six-Month Case Report

FSF Siqueira • AFM Cardenas • YL Gruber • C Kose • YM Pupo • GM Gomes • OMM Gomes • JC Gomes

Clinical Relevance: This six-month clinical report demonstrates the use of a new self-etching glass ceramic primer with the CAD/CAM–modified correlation and biogeneric modes in the fabrication of porcelain laminate veneers to achieve esthetic clinical outcomes.

doi: http://dx/doi.org/10.2341/15-300-t

Relationship Between Simulated Gap Wear and Generalized Wear of Resin Luting Cements

A Tsujimoto • WW Barkmeier • T Takamizawa • MA Latta • M Miayazaki

Clinical Relevance: Gap wear and generalized wear simulation models can be used to assess the wear resistance of resin luting cements. In clinical situations, with restricted light exposure conditions, care should be taken to

doi: http://dx/doi.org/10.2341/16-270-L

Extensive Assessment of the Physical, Mechanical, and Adhesion Behavior of a Low-viscosity Bulk Fill Composite and a Traditional Resin Composite in Tooth Cavities

RX Sousa-Lima • LJA Silva • LVF Chaves • S Geraldeli • RCB Alonso • BCD Borges

Clinical Relevance: Integrated analysis of the physical, mechanical, and adhesion behavior of low-viscosity bulk fill resin composite confirms that it presented equal or superior properties when compared with a traditional resin composite, which has been known to be clinically successful.

doi: http://dx/doi.org/10.2341/16-299-L

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Retentive Strength of Y-TZP Crowns: Comparison of Different Silica Coating Methods on the Intaglio Surfaces

VF Wandscher • C Prochnow • MP Rippe • LS Dorneles
GL Callegari • P Baldissara • R Scotti • LF Valandro

Clinical Relevance

Tribochemical silica coating and 5-nm thick silica nanofilm conditioning methods used on intaglio surfaces of zirconia crowns improves retention.

SUMMARY

Objective: To evaluate the effect of different methods of silica deposition on the intaglio surface of yttrium oxide stabilized zirconia polycrystal (Y-TZP) crowns on the retentive strength of the crowns.

Methods: One hundred simplified full-crown preparations produced from fiber-reinforced polymer material were scanned, and 100 Y-TZP crowns with occlusal retentions were milled. Crown/preparation assemblies were randomly allocated into five groups (n=20) according to the treatment of the intaglio surfaces: TBS = tribochemical silica coating via air-abrasion with 30-μm silica-coated alumina particles; GHF1 = application of thin glaze layer + hydrofluoric acid (HF) etching for 1 minute; GHF5 = glaze application + HF for 5 minutes;

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GHF15 = glaze application + HF for 15 minutes; NANO = silica nanofilm deposition (5 nm) via magnetron sputtering. All groups received a silane application. The surfaces of the preparations (polymer) were conditioned with 10% HF for 30 seconds and silanized. The crowns were cemented with resin cement, thermocycled (12,000 cycles; 5°C/55°C), stored for 60 days, and subjected to a retentive strength test (0.5 mm/min until failure). The retention data (MPa) were analyzed using one-way analysis of variance, Tukey tests, and Weibull analysis. Failures were classified as 50C (above 50% of cement in the crown) and 50S (above 50% of cement on the substrate).

Results: The TBS (5.6±1.7 MPa) and NANO groups (5.5±1 MPa) had higher retentive strength than the other groups ($p<0.0001$) and had the highest values of characteristic strength. There was no difference in Weibull modulus, except for the GHF1 group (lower values). The TBS and GHF15 groups, respectively, had 60% and 70% of their failures classified as 50C, while most of the other groups had 50S failures.

Conclusion: Tribochemical silica coating and silica nanofilm deposition on the inner surface of zirconia crowns promoted a higher retentive strength.

INTRODUCTION

Yttrium oxide stabilized zirconia polycrystal (Y-TZP) ceramics have been widely used in dentistry owing to their superior mechanical strength (high fracture toughness)[1,2] and biocompatibility.[3] However, due to their physical, chemical, and microstructural features, the adhesion of Y-TZP ceramics with resin cements is difficult.[4-7] Hydrofluoric acid (HF) etching and silane application can increase the bond strength between vitreous/glass ceramics (based on silica) and composite resins by increasing the surface free energy and wettability[8-13]; in addition, the ceramic primer heat treatment can promote bond improvements.[14-16] However, as zirconia ceramics are dense polycrystalline materials and lack a silicon dioxide (silica) phase, HF treatment fails to produce a microporous surface for bonding.[9,17,18] Thus, surface pretreatment is necessary to modify the topography to increase the mechanical retention and chemical adhesion, thereby enhancing the retention of the crowns to the prosthetic preparation.[19,20] Recently, systematic reviews showed that loss of retention is significantly higher for densely sintered zirconia, compared with all others types of ceramics and metal-ceramics, for both single and multiple crowns.[21,22]

The most commonly used treatment method is air abrasion with silicon oxide particles. This method involves the inclusion/incrustation of silica-coated alumina particles on the cementation surface by air abrasion, followed by application of a silane primer bonding agent.[23-27] These particles increase the roughness of the zirconia surface, while the silane bonding agent promotes adhesion between the abraded surface and the resin matrix of the cement.[28-30] However, studies have shown that air abrasion might create superficial defects and cracks that may initiate fractures.[31,32] Nonetheless, other studies did not find any negative effects when this method was used on Y-TZP ceramics.[33,34] Additionally, clinical failures in Y-TZP crowns (chippings) seem to have no association with the roughness created by the air abrasion of the intaglio surface.[35-38]

Another recently developed technique involves the application of a thin layer of vitreous porcelain (low-fusing porcelain glaze, vitrification or glaze-on) on the ceramic surface. Basically, the glaze is composed by vitreous porcelain (high silica content, amorphous matrix or SiO_2) and pigments (metallic oxides), making a zirconia glazed surface etchable by HF,[39,40] and that can be subjected to silanization or air-particle abrasion.[41] Vitrification is very effective in promoting the adhesion between the Y-TZP ceramic surface and resin cements.[39,42-46] For glass ceramics,[47-49] HF reacts with the silica phase of the porcelain, creating retentive microchannels. Therefore, the effect of longer etching times on the creation of surface irregularities for bonding depends on the microstructure of the ceramic.[8,50,51] Etching increases the contact area between the adhesive agent and the ceramic,[52,53] and the number and size of the irregularities created are associated with the duration of the etching process.[47,54] However, when low-fusing porcelain glazes on zirconia surfaces are treated with HF for too long, the acid etching might completely remove the glaze from the surface. The impact of etching time of porcelain glaze applied on zirconia influences resin bonding strength and crown retention rate has yet to be investigated.

Recently, the deposition of silica nanofilms on the zirconia surface has been studied.[26,55] For this method, a silica nanofilm is deposited on the zirconia surface by plasma processing (reactive magnetron sputtering), making it more chemically reactive. Following up the nanofilm deposition with silanization results in an increase in adhesive strength

Table 1: *Testing Groups and Respective Descriptions of the Surface Conditioning Protocols on the Intaglio Surface*

Group[a] (n=20)	Protocols
TBS[a]	*Tribochemical silica coating:* airborne particle abrasion with silica-coated aluminum oxide particles (30 μm) (Cojet Sand – 3M ESPE), 15 mm distant from Y-TZP crown for 10 seconds at a pressure of 2.8 bar. An adapted device[26] was used to standardize the procedure.
GHF1[b]	*Porcelain glaze + hydrofluoric acid (1 minute):* a single thin layer of low-fusing porcelain glaze (Glaze VITA Akzent, VITA Zahnfabrik, Bad Säckingen, Germany) was applied with a brush (equal proportions of powder and liquid low-fusion porcelains for each five crowns). The crowns were submitted to glaze sintering cycle according to the manufacturer. After, the intaglio surface was conditioned with hydrofluoric acid 10% for 1 minute, washed with air-water spray for 1 minute and dried. The crowns were cleaned via ultrasound (5 minutes in distilled water) and dried.
GHF5 [b]	*Porcelain glaze + hydrofluoric acid (5 minutes):* The procedures were the same as for GHF1, but the surfaces were conditioned for 5 minutes.
GHF15 [b]	*Procelain glaze + hydrofluoric acid (15 minutes):* The procedures were the same that of GHF1, but the surfaces were conditioned for 15 minutes.
NANO	*Silica nanofilm deposition:* A 5 nm SiO_2 nanofilm was deposited by magnetron sputtering. Prior to deposition, the atmosphere inside the chamber was pumped down to $\sim 10^{-7}$ Torr base pressure. During deposition, pressure was kept at 5.2 mTorr using a 20 sccm argon flow rate. The nanofilm thickness was determined by the exposure time of the crowns to the deposition plasma, as the deposition rate is known.[55]

[a] TBS = tribochemical silica coating via air-abrasion with 30-μm silica-coated alumina particles; GHF1 = application of thin glaze layer + hydrofluoric acid (HF) etching for 1 minute; GHF5 = glaze application + HF for 5 minutes; GHF15 = glaze application + HF for 15 minutes; NANO = silica nanofilm deposition (5 nm) via magnetron sputtering.
[b] The groups with glaze application were all made at the same time (n=60). As the technique is inherent to difficulty of standardization, the 60 crowns were randomized in the three glaze groups after the glaze application and sinterization.

without damaging the Y-TZP surface.[26,55,56] Moreover, this technique forms a homogeneous film, improves chemical adhesion to the substratum,[57] and does not promote *m*-phase transformation after the film is applied.[26]

Considering the aforementioned silica deposition methods (low-fusing porcelain glaze application, or silica nanofilm deposition) for improving bonding to zirconia, a question not yet addressed is this: Do these treatments of the intaglio surface of zirconia crowns improve crown retention compared with tribochemical silica coating via air abrasion? Thus, the objectives of this *in vitro* study were: 1) to evaluate the effects of different silica-based coatings of the intaglio surface of zirconia crowns on retentive strength, 2) to compare three different HF etching times for the groups undergoing the vitrification technique, and 3) to evaluate the reliability of the different treatment methods by Weibull modulus. The null hypothesis tested was that there would be no difference among the zirconia surface conditionings.

METHODS AND MATERIALS

Sample-Size Calculation

To determine the number of specimens for group, a sample-size calculation was performed, based on a pilot study, with software from the site "Java applets for power and sample size" (www.stat.uiowa.edu/~rlenth/Power/). With a statistical power of 80%, a standard deviation of the mean of 1.15 MPa, and a detectable difference of 1.38 MPa, it was established that the number of specimens per group should be 20, for a total of 100 specimens divided among five experimental groups (see Table 1).

Thus, the experimental design was based on one factor (zirconia surface treatment) divided into five levels (testing groups, n=20) according to the treatment of the intaglio surface of the Y-TZP crowns (Table 1).

This study was blinded for: crown cementation, aging procedure, retention test, and failure analysis.

Prosthetic Preparation and Crown Production

G10 rods (G10, FR4 Laminate Round Rods Epoxyglass; NEMA grade FR4, Accurate Plastics, Inc, New York, NY, USA) measuring 11 mm in diameter and 1.2 m in length were sectioned in small cylinders of 16 mm height used to obtain 100 identical, simplified, full-crown preparations by computer-aided machining (6 mm in height and a total occlusal convergence angle of 12° with rounded corners; Figure 1a,b).[37,38] Taking into account the fact that all the preparations in G10 had identical dimensions/geometry, just one preparation was impressed using a vinyl polysiloxane impression material (Elite HD + Putty and Light Body Normal Setting, Zhermack, Badia Polesine, Italy), followed by obtaining a model in special plaster (CAM-base, type 4, Dentona AG, Dortmund, Germany).

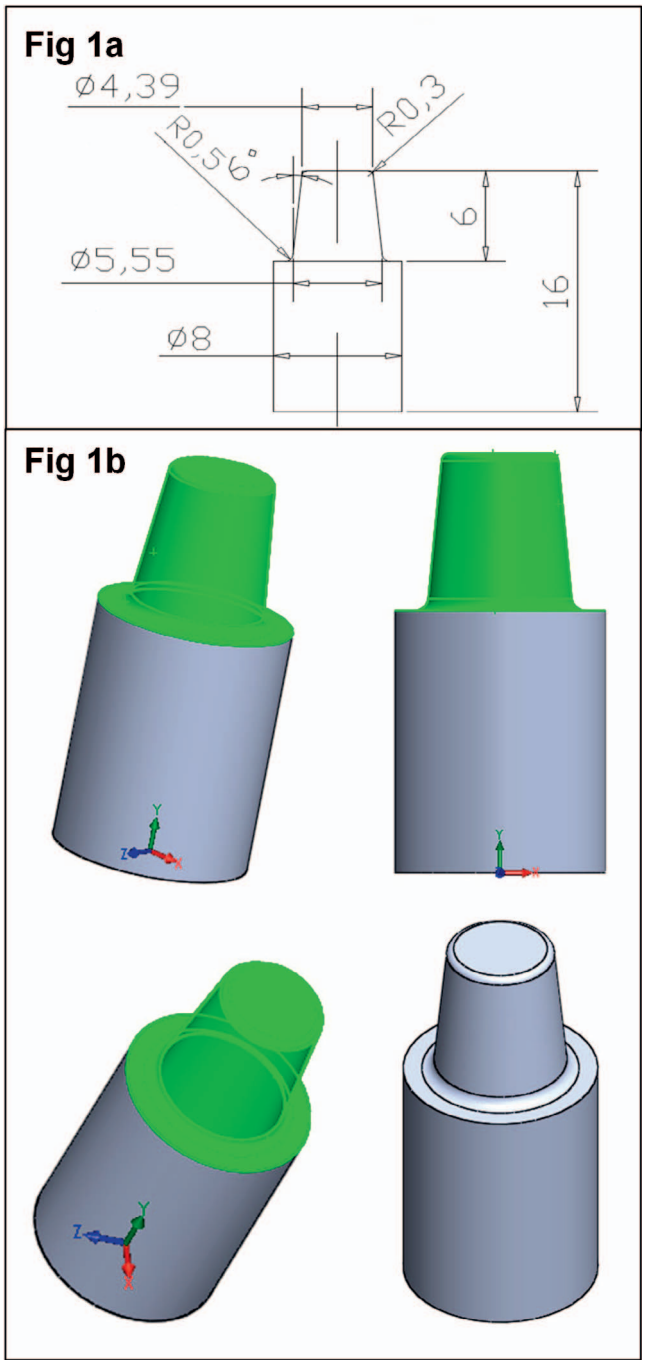

Figure 1. *(a): Schematic drawing of the G10 prosthetic preparations (ø = diameter and R = radius) as a trunked cone. (b): Lateral and frontal views of the G10 preparation in Solid Works software. The green area corresponds to the adhesive area.*

The master die was then scanned (inEos Blue, Sirona, Bensheim, Germany), and the image was transferred to the inLab software (version 3.60, Sirona). Equal crowns (N=100) with occlusal retentions were designed considering a resin cement space of 30 μm, followed by milling the Y-TZP crowns (Cerec InLab MC XL, Sirona; Figure 2a; VITA In-Ceram YZ, YZ-40/19 cubes with dimension of 15.5 × 19 × 40 mm^3, VITA, Bad Säckingen, Germany).

Sintering was performed according to the manufacturer's instructions (Zircomat oven, VITA). The crowns were checked on their preparations for adaptation (Carbono Arti-Spray, Bausch, Bausch Articulating Papers, Inc, Nashua, NH, USA) and cleaned with an ultrasonic device (1440 D – Odontrobras, Ribeirao Preto, Brazil) with distilled water for 10 minutes.

Crown Cementation

After each treatment of the inner surfaces of the Y-TZP crowns (Table 1), methacryloxypropyltrimethoxy-silane (ESPE-Sil Silane, 3M ESPE, Seefeld, Germany) was applied with a microbrush, followed by a 5-minute wait for solvent evaporation.

The preparation surfaces (G10) were conditioned with 10% HF for 30 seconds, cleaned for the same amount of time, and then received an application of silane-based primer (ESPE-Sil, 3M-ESPE), with a 5-minute wait time.[59] The resin cement (RelyX ARC, 3M ESPE) was manipulated according to the manufacturer's instructions and applied to the intaglio surface of the crowns, which were positioned on the preparation. With an adapted surveyor (B2, BioArt, Sao Carlos, Brazil), a load of 750 g was applied to the crowns, the cement excess was removed, and photo activation was performed for 20 seconds on each surface (1200 mW/cm^2, Radii-Cal, SDI, Bayswater, Australia). The specimens were stored in distilled water (37°C) for 24 hours before aging.

Aging: Thermocycling + Storage

All specimens were subjected to thermocycling (12,000 cycles; 5°C to 55°C; 30 seconds per bath and 5 seconds between baths; Ethik Technology, Vargem Grande Paulista, Brazil)[19,20,60,61] followed by storage for 60 days in a wet environment at 37°C.

Retentive Strength Test

After thermocycling, part of the cemented crown was embedded in self-curing acrylic resin (VIPI Flash, VIPI, Pirassununga, Brazil) until total coverage of the retentive part of the crowns was achieved. This process was carried out with the aid of an adapted surveyor (B2, BioArt), which maintained a vertical embedding axis. Retention areas were made apical to the preparations, and the same embedding proce-

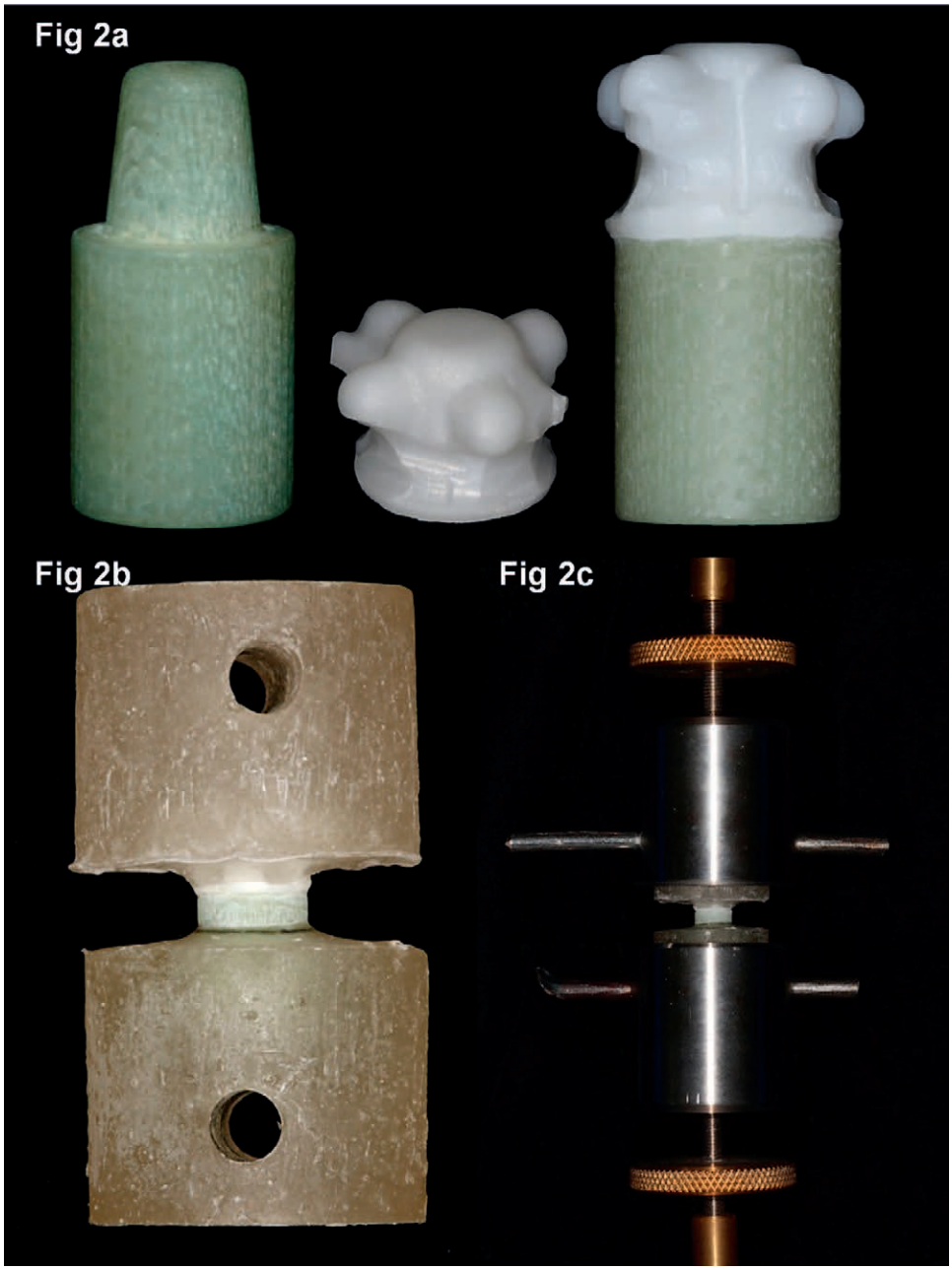

Figure 2. *(a): G10 preparation after milling, zirconia crown with occlusal retention and cemented crown on the G10 preparation. (b): Specimen embedded for the tensile test. (c): Specimen attached on the universal machine test.*

dure was performed, keeping the adhesive interface free to the test. The embedding was necessary to make possible the retention test (Figure 2b).

For the retentive strength test, the superior part of the assembly (the crown) was fixed to the movable axle of a universal testing machine (DL-1000, Emic, São José dos Pinhais, Brazil), which was attached to the load cell (1000 N), while the inferior part (the preparation) was positioned at the fixed base of the testing machine (Figure 2c). The retentive strength test was performed until fracture (decementation) at a speed of 0.5 mm/min.[19,20]

Adhesive Area Calculation

The cementation area was calculated by the SolidWorks software (DS SolidWorks Corporation, Waltham, MA, USA) according to the dimensions presented in Figure 1a, resulting in an adhesive area of 130 mm^2 (Figure 1b). The retentive strength was calculated using the formula: $R = F_{max}/A$, where

Table 2: *One-Way Analysis of Variance Results for Retentive Strength Data*

	Sum of Squares	*df*	Mean Square	F	Significance
Between groups	168.816	4	42.204	30.402	.000
Within groups	131.876	95	1.388		
Total	300.692	99			

Abbreviation: df, degrees of freedom.

R = retentive strength, F_{max} = maximum force for failure (decementation), and A = adhesive area.

Failure Analysis

The tested assemblies were analyzed under a stereomicroscope (Discovery V20, Carl-Zeiss, Gottingen, Germany) to evaluate the type of fracture. The fractures were classified according to the localization of the largest portion of cement: 50C (more than 50% of the cement on the crown), or 50S (more than 50% of the cement on the preparation). Representative images were taken with a scanning electronic microscope (SEM; JSM 5400, Jeol Ltd, Tokyo, Japan). This classification was adapted from Amaral and others[19] and Rippe and others.[20]

Micromorphologic Analysis (Zirconia Blocks and G10 Polymer Surfaces)

Extra zirconia samples (small blocks) were produced from VITA In-Ceram YZ blocks (VITA) in a cutting machine (IsoMet 1000; Buehler, Lake Bluff, IL, USA). The standardization of the analysis surface was performed with Sof-Lex disks (3M ESPE) and polishing with 1200-grit sandpaper. After sintering, the blocks presented an analysis area of 5 × 5 mm, which was conditioned with the aforementioned surface methods.

Etched and non-etched axial surfaces of the G10 preparations (10% HF for 30 seconds) were also analysed.

Surface analyses of both tested strategies and G10 surfaces were performed under an SEM (JSM 5400, Jeol Ltd) to verify the topography and superficial alterations of the Y-TZP ceramic and G10 preparations.

Data Analysis

The nominal values of retentive strength were tabulated and statistical analysis was performed with the SPSS software (version 21, IBM, Chicago, IL, USA). The normality and homoscedasticity were verified and the data were subjected to one-way analysis of variance (ANOVA) and post hoc Tukey test. A Weibull analysis was also performed (Weibull modulus [m]: reliability of retention values represents the variation of strength data and expresses the size distribution of the flaw population in a structure; characteristic strength [σ_0] indicates the strength value at which 63.2% of the specimens survive).

RESULTS

One-way ANOVA (Table 2) showed a statistical difference between the retention values of the groups ($p<0.0001$). Tukey's test showed that TBS and NANO groups presented the highest strength values in relation to the other groups. The GHF15 and GHF5 groups presented intermediate values, and the GHF1 group had the lowest values (Table 3). The Weibull modulus was lower for the GHF1 group, and

Table 3: *Mean ± Standard Deviation Retentive Strength (MPa), Weibull Parameters and Failure Modes of the Zirconia Crowns*[a]

Group[b]	Retentive Strength	Weibull Parameters[c]				Failures[d]	
		σ_0	CI	*m*	CI	50S (%)	50C (%)
GHF1	2.1±0.95 Z	2.55 y	1.76-3.66	1.49 Y	0.94-2	20 (100)	—
GHF5	3.75±1.06 Y	4.14 y	3.59-4.76	3.86 X	2.44-5.19	19 (95)	1 (5)
GHF15	3.78±1.06 Y	4.22 y	3.6-4.9	3.61 X	2.28-4.85	6 (30)	14 (70)
TBS	5.6±1.68 X	6.18 x	5.36-7.1	3.9 X	2.46-5.24	8 (40)	12 (60)
NANO	5.5±0.98 X	5.91 x	5.43-6.41	6.55 X	4.14-8.81	16 (80)	4 (20)

[a] *Different letters in the same column indicate a significant difference.*
[b] *GHF1 = application of thin glaze layer + hydrofluoric acid (HF) etching for 1 minute; GHF5 = glaze application + HF for 5 minutes; GHF15 = glaze application + HF for 15 minutes; TBS = tribochemical silica coating via air-abrasion with 30-μm silica-coated alumina particles; NANO = silica nanofilm deposition (5 nm) via magnetron sputtering.*
[c] *Weibull analysis for retention values: σ_0 = characteristic resistance in MPa, and m = Weibull modulus (95% confidence intervals [Cia]). Lowercase letters were used for values and capital letters for m values.*
[dc] *50S = more than 50% of cement adhered on the substratum; 50C = more than 50% of cement adhered on the crown.*

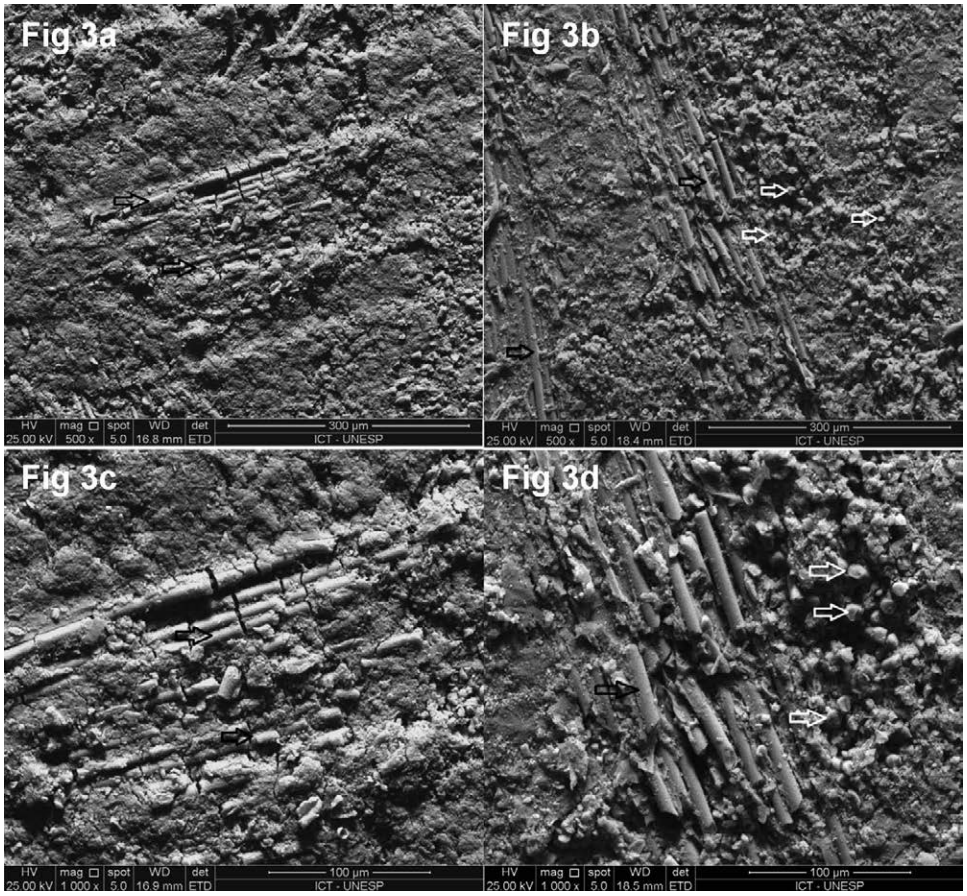

Figure 3. *Representative micrographics of the G10 surfaces with and without HF etching. (a and b): G10 axial surface nonetched and etched, respectively (×500). (c and d): G10 axial surface nonetched and etched (×1000), respectively. G10 analog material presents glass fiber in different senses; it is possible to observe in the nonetched surfaces (a and c) little exposed fiber in only one horizontal direction (black arrow), while the etched surface presents more exposed glass fiber in different directions (black and white arrows).*

the characteristic strength was increased for the TBS and NANO groups.

Figure 3a through d shows the micromorphologic differences between nonetched and etched surfaces of the polymeric material (G10).

We observed the differences between the surface morphologic patterns of the zirconia blocks treated with the different strategies (Figure 4A through M). Compared with the untreated surface (Figure 4L,M), all the groups presented topographic alterations, except the NANO group (Figure 4I,J). The etching time affected the topographic pattern of the glaze groups (the longer the etching time, the greater the presence of pores) (GHF1: Figure 4A,B; GHF5: Figure 4C,D; and GHF15: Figure 4E,F). The TBS group presented irregularities promoted by air abrasion (Figure 4G,H).

In terms of fracture type, the TBS and GHF15 groups presented a higher percentage of 50C failures compared with the other groups (Table 3, Figure 5a, 5b through e).

DISCUSSION

The null hypothesis was rejected: the TBS and NANO groups presented the highest retentive strength values, followed by the GHF15, GHF5, and GHF1 groups.

The silica nanofilm application and tribochemical silica coating presented the highest retention and characteristic strength values (Table 3) compared with other silica deposition methods. It has been shown that silica coating via air abrasion followed by silanization improves the bond strength for silica-based,[9,62] glass-infiltrated alumina,[9,63] and zirconium ceramics.[9,23,61,64-66] Using a shear bond strength test, some authors showed that silica nanofilms deposited by magnetron sputtering reached values of adhesion similar to air abrasion with silica[26] or alumina particles.[56] Despite a similar Weibull modulus (m), the NANO group had a 40% higher value of m than the TBS group, which suggests a high reliability of the silica deposition technique (Table 3). Compared with the vitrification method, deposition of the silica nanofilm via sputtering is

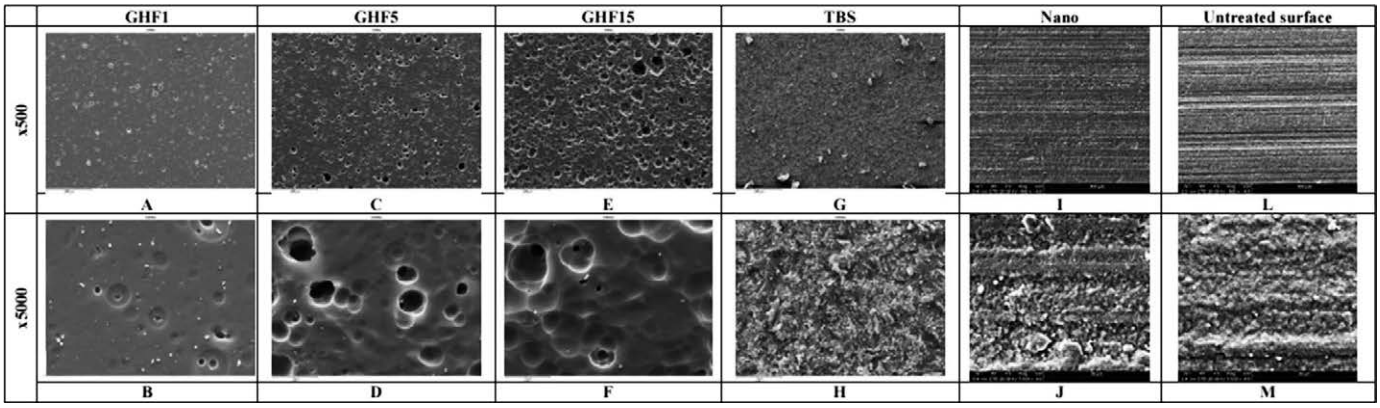

Figure 4. *Representative scanning electron micrographs of the zirconia blocks treated with the same treatments utilized in the intaglio surface of zirconia crowns. (A): GHF1 ×500. (B): GHF1 ×5000). (C): GHF5 ×500. (D): GHF5 ×5000; (E): GHF15 ×500. (F): GHF15 ×5000. (G): TBS ×500 (H): TBS ×5000. (I): NANO ×500. (J): NANO ×5000. (L): Zirconia surface without treatment ×500. (M): Zirconia surface without treatment ×5000. It is possible to observe in glaze groups the greater etching time and the greater quantity and size of pores compared with the air-abraded surface, which presents irregularities promoted by impact of silica-coated alumina particles, and the NANO group, which presents characteristic of the untreated surface (Sof-Lex disks and 1200-grit sandpaper), possibly because the nanofilm presents a thickness of 5 nm.*

rapid, and the thickness and chemical composition of the film can be controlled. Also, it does not subject the ceramic to high temperatures, and avoids the damage associated with sandblasting. However, it requires costly, specialized equipment, and specific training for use.[26]

The NANO group generated mostly 50S (more than 50% of the cement on the G10 substratum surface) failures (80%) compared with the TBS group, which presented 60% 50C (more than 50% of the cement in the crown) failures. According to studies conducted on the magnetron sputtering methods of silica deposition, the coating layer is homogeneous and shows a controlled thickness.[57,58] While the magnetron sputtering creates a 5-nm thick homogeneous silica deposition film (NANO group), the 30-μm diameter silica-coated alumina particles (TBS group) penetrate approximately 15 μm into the Y-TZP crowns.[67] Therefore, despite the fact that both methods promote silica deposition and make the surface more reactive, air abrasion caused more irregularities, thus favoring micromechanical retention between the zirconia and cement. This can be seen on the micrographs (Figure 4G through J)[28-30] and it is the cause of the 50C failure mode. In terms of damage effect of the particle air abrasion on zirconia materials, the literature is controversial.[31-34,68]

Among the studied silica deposition methods, the vitrification method (or glaze-on)[42] seems to be a promising technique. *In vitro* shear bond strength studies showed that the application of a thin layer of low-fusing porcelain glaze on the zirconia surface followed by HF etching generated similar or higher adhesion values compared with conventional surface-treatment methods. However, these studies conditioned the glazed surface for different durations and used different acid concentrations.[39,42-46]

In the current study, we aimed to determine the best acid-etching time of the glazed zirconia surface by means of a crown pull-out test, which evaluates not only the bond strength but also all the complex forces involved, including shear and friction. Our results partially agree with other studies that used a similar methodology (vitrification technique + pull-out crown test).[19,20] Rippe and others[20] verified the effect of the surface treatment and type of cement on the retention of Y-TZP crowns on composite cores. They also showed that the vitrification technique presented higher retention values than the group without treatment but similar to those of a tribosilicatization group using a 2-hydroxylethyl methacrylate (HEMA)-based resin cement. However, when a bisphenol A-glycidyl methacrylate (BIS-GMA)-based cement was used, there was no difference between the groups. In addition, Amaral and others[19] evaluated the retention of Y-TZP crowns with different inner treatments and different substrate types and found no difference among the treatments when a dentin substrate was used; however, when the crowns were cemented on composite cores, tribosilicatization and vitrification significantly increased the retention force in comparison with the control group (no treatment). In the current study, the GHF1 group (Figure 4A,B) presented lower values of retention than the silicatized group (TBS) (Figure 4G,H), possibly because the conditioning time (1 minute) was not enough to

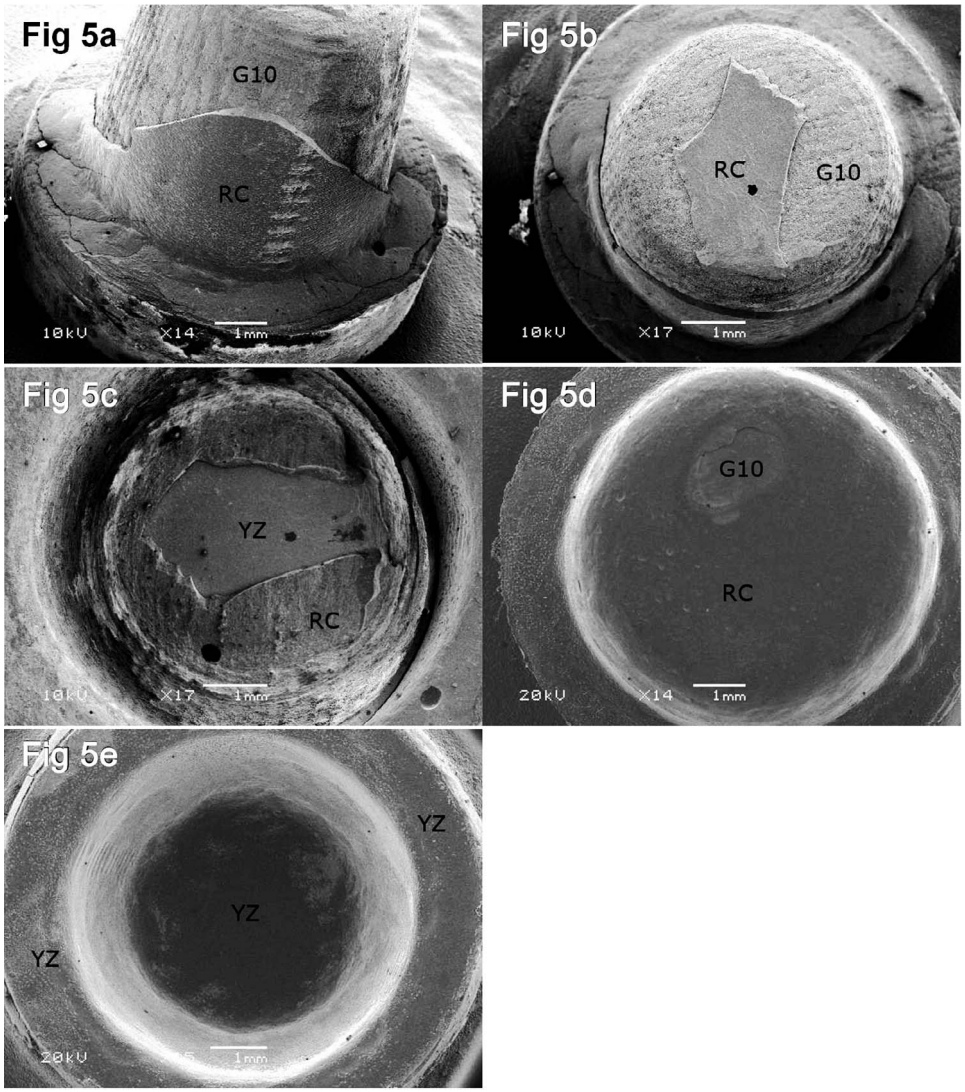

Figure 5. *(A, B, and C): 50C failure of the GH15 group specimen (A and B correspond to G10 preparation and C to intaglio surface of zirconia crown). (D and E): 50S failure of the GH1 group specimen (D corresponds to G10 peparation, and E corresponds to the intaglio surface of the zirconia crown). G10, G10 analog material; RC, resin cement; YZ, zirconia crown.*

generate significant morphologic changes on the glazed surface compared with the air-abraded surface.

Among the studied strategies, the GHF5 and GHF15 groups presented statistically similar and intermediate values of retentive strength (Table 3), as well as characteristic strength. An increase in acid-etching time improved the retention values (GHF5 and GHF15 groups) compared with the GHF1 group. However, the GHF15 group showed a higher number of 50C failures, indicating a greater adhesive interaction with the crown, similar only to the TBS group. This could have been caused by the topographic alterations (Figure 4E,F) promoted by the increased etching time[49] of the glazed surface and consequently a greater surface area for bonding, which facilitates resin cement penetration into the microretentive ceramic surface.[69] The glazing procedure is quick, is performed after the clinical verification of the fit of the crown, being applied by the dental technician. Thus, the clinician receives a zirconia crown with an etchable intaglio surface. But, standardization of the thickness of the low-fusing porcelain glaze is difficult, thus it might increase the marginal gap.[45] At the same time, space for the glaze and cement layer could be produced by computer-aided design/computer-sided manufacturing systems, thus ensuring an adequate fit.[39] Since the thickness of the glass film was approximately 31.8 μm for Ntala and others[39] and 12 μm for Bottino and others,[25] the effect on the marginal fit could be not negligible, considering that the clinically recommended maximum misfit is around 120 μm.[45,70,71] Even though the vitrification/glaze-on technique has its advantages, we believe technical improvements are needed, primarily in the standardization of the

thickness of the glaze layer, in order to prevent any negative effects on the marginal fit.

It is important to highlight that the glaze composition (monophase) may have affected the retention values for glaze groups. If a multiphasic glaze material had been used, maybe better results of crown retention could have been obtained.[39,40] In this sense, the development of multiphasic glaze materials might achieve improvements.

In the current study, we utilized a woven glass-fiber-filled epoxy material (G10) as a dentin analog, in order to standardize the substrate and the preparation. This material showed excellent mechanical and adhesive properties,[59,72,73] and we understand that standardization of the substrate is crucial to verify the pretreatment factor alone in zirconia crowns. Nevertheless, Kelly and others[59] showed that resin cement bond strength to dentin was slightly lower than to the dry and wet analog material (40%-50%), which shows that caution is needed when using the G10 in adhesion tests. Up to now, we know of no other studies that compared the bond strength of dentin and G10 to demonstrate adhesion similarity of these substrates. In this sense, when adhesion studies using G10 are planned, it is very important to evaluate if the G10 as a dentin analog influences the results and interpretation. In the current study, we evaluated the zirconia/resin cement interface and most of the failures were 50S; thus, we believe that it was possible to observe the effect of the zirconia surface treatments. Figure 3a through d shows that etched G10 surfaces present more glass fiber exposed for bonding with the silane couple agent. Additionally, the use of copings in the complex adhesion/retention trial (retentive strength of crowns) reflects the clinical reality more properly, especially due to the axial loads exerted during the test.

The present study had some limitations. It is difficult to compare our results with the current literature because most studies did not present similar geometries of the preparation.[19,20,60,61,70] Furthermore, those studies that utilized zirconia crowns had a large number of variables: the substrate, preparation angle, cement type (with or without monomer phosphate), and application of primers. Mechanical cycling or fatigue experiments were not carried out in these studies either, and such test conditions should be employed in the future.[72,74-76] These new silica deposition methods should be included in prospective clinical trials to evaluate the bond effectiveness and the effect on retention of zirconia crowns under clinical situations.

CONCLUSIONS

1. Tribochemical silica coating (via particle air abrasion) and silica nanofilm deposition (via magnetron sputtering) as pretreatments of zirconia crowns in combination with RelyX ARC luting cement promote higher crown retention compared with the low-fusing porcelain glaze applications (apart from acid etching time).
2. Application of a thin layer of low-fusing porcelain glaze showed variable retention results depending on the etching time; thus, further studies about glaze coated polycrystalline zirconium oxide ceramics should be performed.

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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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Sudden Onset of Subcutaneous Air Emphysema After the Application of Air to a Maxillary Premolar Located in a Nonsurgical Field

EA Van Tubergen • D Tindle • GM Fox

Clinical Relevance

Subcutaneous emphysema commonly develops when compressed air enters through a surgical site. Forced air may inadvertently enter into subcutaneous tissue through intact mucosa. Clinicians must be aware of this possibility to diagnose and manage subcutaneous emphysema during nonsurgical treatment.

SUMMARY

Although rare, subcutaneous air emphysema can occur during dental procedures such as endodontic treatment, surgical extractions, and preparing a tooth for an indirect or direct dental restoration. We report the development of a subcutaneous air emphysema that was introduced through the periodontal ligament of an untreated premolar after the use of an air syringe to dry the tooth.

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INTRODUCTION

Subcutaneous air emphysema, when air is introduced into subcutaneous tissues under forced pressure, is not a common occurrence during dental treatment. However, the use of high-speed air turbine handpieces and air syringes can lead to the introduction of air into the soft tissue spaces. Common routes for entry of air are through endodontically treated teeth, extraction sites, periodontal treatment sites, and during restorative procedures such as class V preparations and crown preparations.[1,2] Trauma to facial areas can also lead to the introduction of air into soft tissues spaces.[3] No case has yet reported the development of a subcutaneous air emphysema in which the air was introduced through a nonsurgical site, which also was not involved in any other hard or soft tissue procedure. We describe a case in which a subcutaneous air emphysema developed by the application of air to the untreated opposing dentition of a tooth being prepared for an onlay.

CASE REPORT

The following case was seen at the University of Michigan School of Dentistry. A 53-year-old man

presented for a Cerec, (Sirona Dental Systems, Inc, Long Island City, NY, USA) porcelain onlay on tooth number 19. His health history was unremarkable other than he was being treated for hypertension with lisinopril and high cholesterol with welchol and over the counter CholestOff. The patient's dentition was previously treated with four quadrants scaling and root planning and was periodontally stable except for a localized deep pocketing in the maxillary right quadrant. Flap surgery was performed on teeth numbers 2 to 5 about one week prior to being seen for onlay preparation on tooth number 19. Before the Cerec onlay preparation, the mandibular left quadrant was anesthetized by a nerve block of the left inferior alveolar nerve with 1.1 mL lidocaine with 1:100,000 epinephrine. The Cerec computer-assisted-design/computer-assisted manufacture (CAD/CAM) system allows for same-day impression and delivery of an onlay. This requires a digital impression of both the preparation and the opposing teeth to mill the onlay onsite with the CAD/CAM machine. Prior to obtaining a digital impression, it is important to evaluate the occlusion to ensure adequate occlusal reduction of the onlay preparation and to thoroughly dry the teeth. After drying the mandibular quadrant, the operator applied air to the opposing maxillary left quadrant with the air syringe, and the patient immediately reported a "lightning bolt" sensation that went from the premolars and traveled posteriorly to the maxillary molars. The patient immediately noticed a large facial swelling on the outer aspect of the left ramus. Oral Medicine was called for a consultation. At initial viewing of the patient, the left side of the patient was noticeably swollen compared with his right side (Figure 1). On examination, the swelling extended six inches in length up the ramus and was about two to three inches across. The affected tissue did not have the crepitus (crackling) feeling on palpation, but was firm to the touch. Trismus was not present with the jaw joints. The airway was not obstructed, and the patient reported that the swelling was not painful but slightly uncomfortable. Swelling was limited to the exterior portion of the ramus, and swelling was not observed on the medial aspect of the ramus. Initial diagnosis by Oral Medicine was a subcutaneous air emphysema but given the proximity to the airway, Oral Surgery was paged to evaluate the swelling. They agreed with the Oral Medicine diagnosis. At the time of the incident, probing depths in the area, the maxillary left quadrant, were unremarkable and ranged from 2 to 3 mm. Prior to scaling and root planing procedures, the probing depths on the buccal of tooth number 12 read from mesial to distal, 8, 3, and 6 mm and for number 13 read 6, 3, and 5 mm. Lingual probing depths on numbers 12 and 13 ranged from 6 to 3 mm. Initial periodontal stability had been established eight weeks after the initial scaling and root planning visit, which was more than 15 years before the development of this subcutaneous air emphysema. Five days prior to the incident, the patient had received periodontal surgery on the maxillary right quadrant for an isolated pocket that developed near tooth number 3. He was also still taking a seven-day course of 500 mg amoxicillin, three times a day, which had been prescribed at that visit; therefore, he was not placed on an additional prophylactic course of antibiotics. Later the same day, the patient was seen in the Graduate Periodontal Clinic to have sutures removed from the maxillary right quadrant. Oral Medicine followed up with the patient while he was waiting for his periodontal appointment. He reported that the swelling felt and looked smaller.

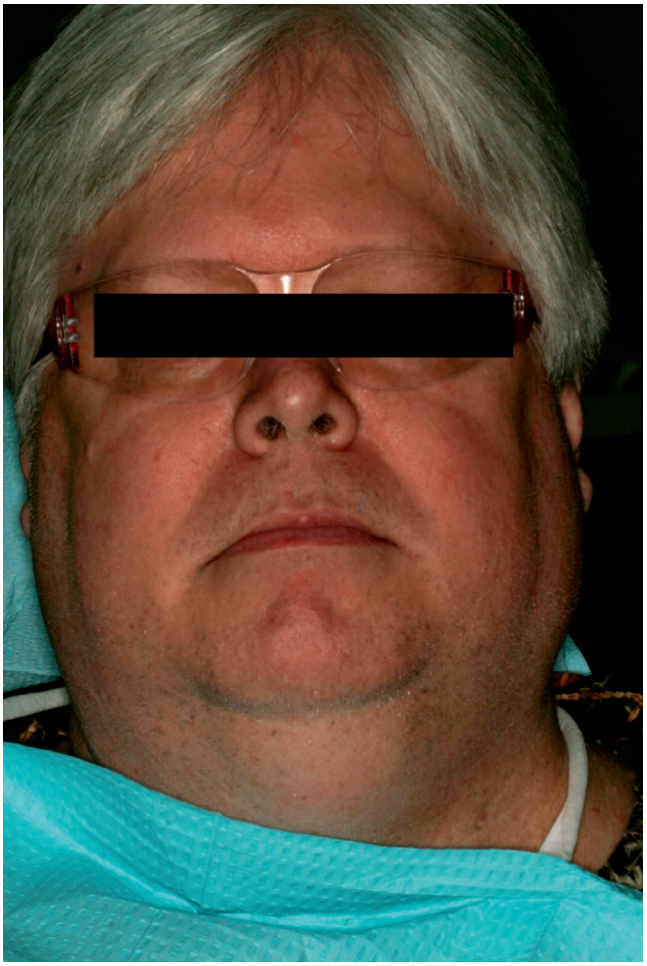

Figure 1. *Image of patient's face after onset of subcutaneous air emphysema affecting the left portion of the patient's ramus.*

This was confirmed by visual examination. Follow-ups via telephone were performed, and the patient reported that the swelling went down after two days. He has not had any further complications in the affected areas.

DISCUSSION

Subcutaneous emphysema related to dental treatment was first described by Turnbull at the beginning of the 20th century.[4] In 1995, 74 cases occurring between 1960 and 1993 were reviewed by Heyman and Babayof.[5] More recently, in 2009, McKenzie and Rosenberg published a review of an additional 32 episodes of subcutaneous emphysema occurring from 1993 to 2008.[1] Most reported cases have been associated with the use of air turbine handpieces during dental surgeries and nonsurgical restorative procedures.[1,5]

In our review of the literature, we found only two cases of subcutaneous emphysema related to the use of a dental air syringe.[2,6] In one case described by Uehara and others, the emphysema was believed to have resulted from air being directed into the gingival sulcus to dry teeth that were endodontically treated for caries.[6] Likewise, in the case described here, the subcutaneous emphysema followed the use of an air syringe in a nonsurgical field. We also believe the gingival sulcus to be the likely conduit for the entrance of the air into the subcutaneous tissue, given the fact that the maxillary teeth had not had any restorative work performed on them at the time of the emphysema.

Subcutaneous cervicofacial emphysema can be a serious and life-threatening event. The subcutaneous introduction of air into the buccal or sublingual spaces has the potential of further spread through the submandibular space and into the fascial spaces of the neck.[7] Pneumomediastinum may readily result following spread of air from the cervical spaces into the mediastinum.[3,8] The signs and symptoms of pneumomediastinum include dyspnea, chest and back pain, a crunching sound with each heartbeat known as Haman's sign, and positive radiographic findings.[9] In addition, nonspecific electrocardiographic changes in the ST-T interval may be seen. Treatment for pneumomediastinum is usually supportive, with close observation for development of further complications such as cardiac tamponade, simple and tension pneumothorax, pneumoperitoneum, mediastinitis, or airway obstruction.[9,10] The differential diagnosis must include allergic reaction along with hematoma arising as a complication of local anesthetic injection.[11] In the present case, signs of an allergic reaction, such as skin erythema and firmness to palapation, were not present. Trismus and intraoral swelling, features of an injection hematoma, were also absent. Crepitus on palpation, a feature commonly associated with subcutaneous emphysema, could not be detected. However, this feature may not be evident until several hours after introduction of the subcutaneous air.[10]

Although neither airway compromise nor cervical swelling was present in this case, patients manifesting such symptoms should be evaluated immediately via imaging to determine the extent of airway compromise and mediastinal involvement.[11-13] Subcutaneous emphysema usually resolves spontaneously over a few days, but more severe cases, especially those with airway compromise, may require hospital admission and monitoring.[13,14] The administration of antibiotics is advocated in severe cases to counter the possible introduction of bacteria along with the air into subcutaneous spaces. Oral flora being the most likely bacterial contaminants, amoxicillin or its equivalent is an appropriate choice. In the case we presented, the patient was on a seven-day course of amoxicillin for periodontal flap surgery and was on day 5 of his regimen and therefore was not prescribed an additional prophylactic antibiotic.

Cases reported in the literature also demonstrate that the passage of air from one fascial space to another fascial space does not always follow a predictable route. Some cases reported that air forced in subcutaneously moved from mandibular spaces to maxillary spaces. For instance, Uyanik and others[15] reported on the case of a patient undergoing root canal treatment on a mandibular anterior tooth that demonstrated that an air emphysema does not necessarily localize at the site where air was introduced. During the placement of the last endodontic file on a mandibular anterior incisor, a swelling appeared in the maxillary canine fossa without compromising the airway.[1,5,15] Likewise, another endodontically treated mandibular molar developed an infraorbital swelling with the application of the air syringe.[16] In the case we presented, the air introduced in the maxilla by an air syringe moved from the canine space (also known as the infraorbital space) posteriorly through the buccal space and finally to the posterior submasseteric space.

Although the introduction of air emphysema with an air syringe is uncommon, it should be understood that it is a risk in dental treatment. It is most likely that the air syringe introduced forced air through

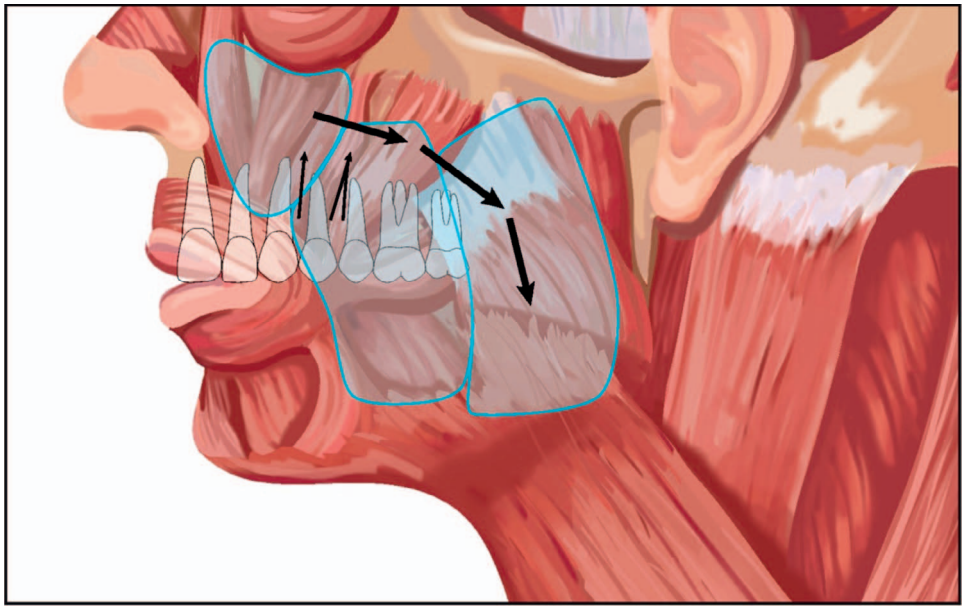

Figure 2. *Schematic of fascial spaces that the air traveled through once entering the subcutaneous tissues through the periodontal ligament of tooth number 12 or 13.*

the periodontal ligament at the site of either tooth number 12 or 13 and subsequently traveled through the canine and buccal fascial spaces before settling in the submasseteric space (Figure 2). This pathway is consistent with activation of the posterior superior alveolar nerve (and also possibly the middle superior alveolar nerve), as reported by the patient. The presence of the middle superior alveolar nerve is highly variable and mostly likely innervates the premolars by means of a nerve plexus.[17-19] The posterior superior alveolar nerve typically innervates the molars, participates in the plexus innervating the premolars, and innervates the periosteum and gingiva surrounding the premolars and molars.[20,21] Given that some cases have reported on the introduction of air emphysema via air syringes, it is important to use the air syringe sensibly to prevent such an event from happening. In the event of an iatrogenic air emphysema, it is important to recognize the risks of airway restriction and / or the presence of facial swelling. A patient with developing airway restriction should be referred to a hospital for computed tomography and further management. If only facial swelling without airway restriction is present, the patient should be followed closely for resolution with consideration given to the administration of prophylactic antibiotics.[16,22]

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Michigan School of Dentistry.

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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Using CAD/CAM–Modified Correlation Mode to Produce Laminate Veneers: A Six-Month Case Report

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YM Pupo • GM Gomes • OMM Gomes • JC Gomes

Clinical Relevance

This six-month clinical report demonstrates the use of a new self-etching glass ceramic primer with the CAD/CAM–modified correlation and biogeneric modes in the fabrication of porcelain laminate veneers to achieve esthetic clinical outcomes.

SUMMARY

The expectation of an esthetically harmonious smile increases the level of difficulty when treating patients. Laminate veneers stand out as a treatment option for cosmetic rehabilitation in clinical practice, as they are a more conservative procedure and mimic dental structures. These laminate veneers are generally made with different techniques; the most common requires an impression of the prepared tooth, an impression antagonist, fabrication models, and extensive laboratory time. The computer-aided design/computer-aided manufacturing (CAD/CAM) system optimizes the fabrication of prosthetic structures, reducing chairside time and promoting good esthetic results. Thus, the purpose of this case report is to present the esthetic result of multiple CAD/CAM manufactured laminate veneers using a new self-etching glass ceramic primer

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with a lithium disilicate ceramic, using the modified correlation and biogeneric modes.

INTRODUCTION

Techniques and materials for indirect dental restorations have evolved quickly. Laminate ceramic veneers are becoming a more popular conservative treatment to achieve esthetic demands.[1] Restorative treatment using porcelain laminate veneers is done with different techniques, all of which typically require an impression of the prepared teeth, impression of the opposing arch, preparation of casts, and extensive laboratory time.[2-4]

Recently, manufacturing materials, techniques, and office time have been simplified by digital dentistry and the introduction of stronger glass-infiltrated ceramics. With the introduction of these ceramics, especially pressed lithium disilicate (LD), laminate veneers can be made using either the conventional lost-wax technique or by designing a prosthetic structure on a computer, followed by its manufacture in a milling machine. This technique is referred to as *computer-aided design/computer-aided manufacturing* (CAD/CAM).[5,6]

The use of LD in a pressed technique and its applications in the dental clinic were introduced by Brodkin and others in 1998.[7] The material is composed of 65% LD in the form of crystalline structures,[8] resulting in a relatively strong ceramic.[9] The LD ceramic has a flexural strength of about 400 MPa, a fracture toughness of 3.3 MPa, and good translucency. In addition to all these, the LD glass portion can be etched using a 5% to 10% hydrofluoric acid (HF) solution and can be bonded to enamel and dentin. The HF etching makes a microporous surface, which increases the bond strength.[10] Different clinical applications are suggested for LD, including veneers, anterior and posterior single crowns, and anterior or posterior fixed prostheses of up to three elements.[11]

The LD glass ceramic (IPS e.max CAD, Ivoclar-Vivadent, Barueri, SP, Brazil) was designed for the CAD/CAM processing technology. In the presintered state, the CAD/CAM block exhibits a flexural strength of 130-150 MPa, allowing for small intraoral adjustments. After sintering at 850°C, LD has a flexural strength of 360 MPa.[12] Because of the translucency and variety of colors, LD can be used for full anatomical restorations (monolithic) with subsequent surface color characterization.[12]

Because of differences in professional judgment regarding the number of steps for cementation, manufacturers have released more versatile self-etching glass-ceramic primers to shorten the pretreatment of dental restorations, which includes providing the etching and silanization steps in only one bottle.

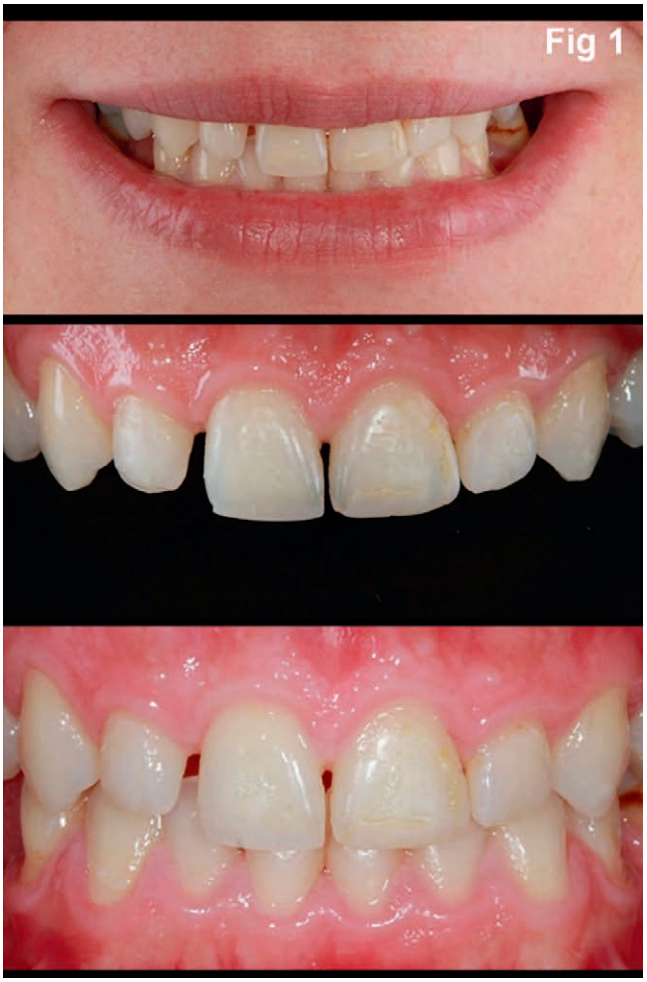


Figure 1. *Baseline aspect of smile.*

There is not enough information in the literature regarding the performance of this new class of universal primers and the development of a high reactivity between the glass ceramic and the primer.

Digital technology is rapidly emerging and has introduced many new possibilities in dental practice because of the different advantages that the system offers. Therefore, the aim of this case report is to present the esthetic results of multiple LD ceramic veneers manufactured with CAD/CAM using the correlation mode associated with the biogeneric mode and a new self-etching glass ceramic primer.

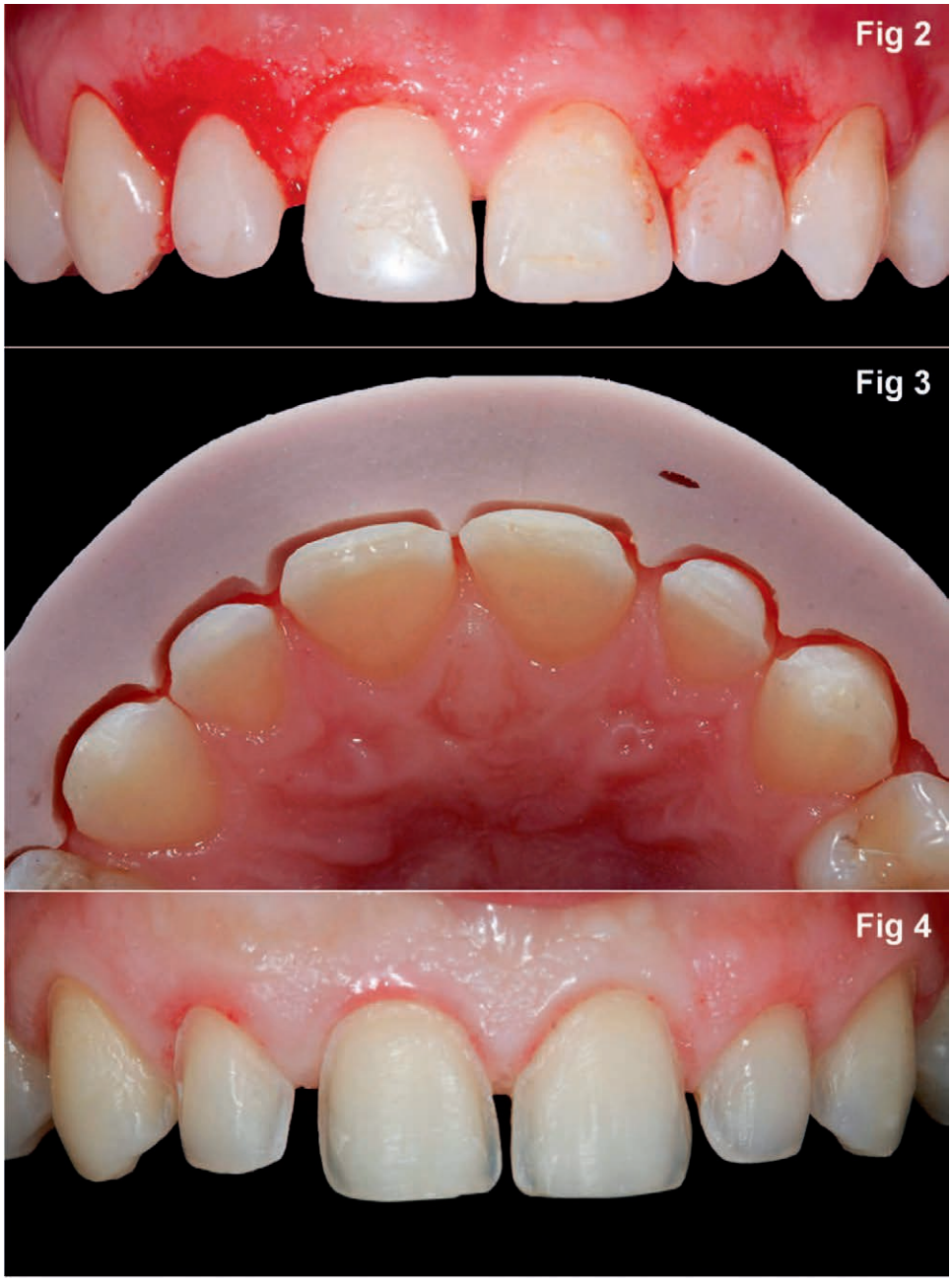


Figure 2. *Immediate result of gingivectomy.*
Figure 3. *Preparations made following the wear tab for guidance.*
Figure 4. *Aspect before polishing and finishing preparations.*

TECHNIQUE DESCRIPTION

A 36-year-old female patient presented to the School of Dentistry at the State University of Ponta Grossa for esthetic treatment. Her main complaint was the presence of clinical diastemas between her maxillary anterior teeth after orthodontic treatment. These anterior maxillary teeth were restored using composite resin five years previously, presenting several fractures and staining of the resin composite (Figure 1).

In addition, the patient was not satisfied with the color and shape of her natural teeth. After clinical and radiographic examinations and obtaining initial impressions, it was determined that the patient had a low caries risk and did not have active caries lesions or signs of periodontal disease.

The esthetic treatment plan alternatives were discussed with the patient, including dental bleaching and gingivectomy, followed by direct composite resin or ceramic veneers. After careful evaluation of the patient's expectations regarding her smile, a dental bleaching, gingivectomy, and ceramic veneers option was selected. Dental bleaching provides the

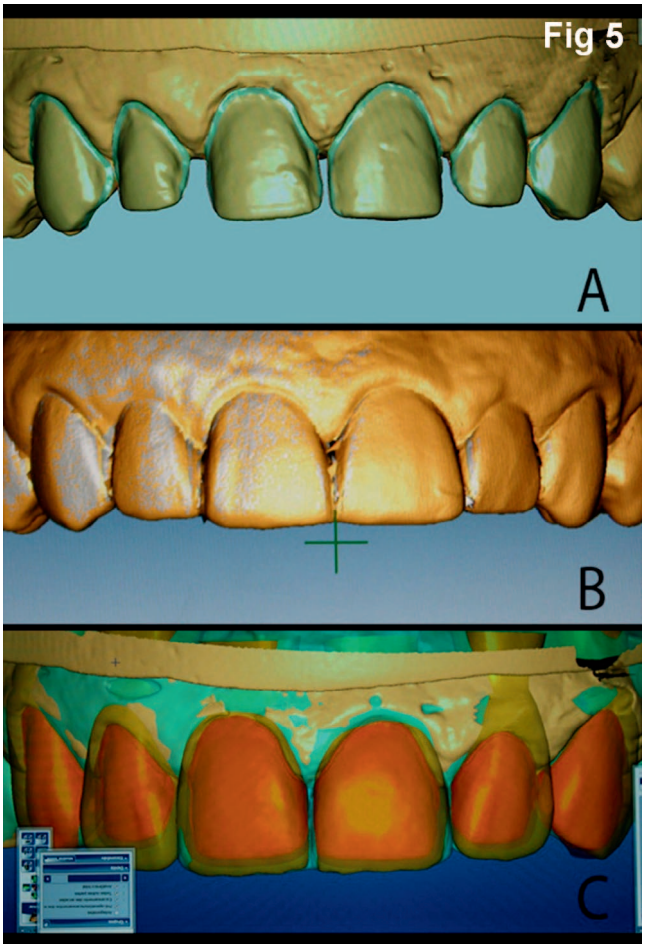


Figure 5. *(A): Digitally planning the correlation mode through the digitalization wax-up model. (B): Scanning model with the prepared teeth. (C): Supplement to the biogeneric order to improve and adapt shape and contour.*

dentist the opportunity to use a more translucent glass ceramic, preventing opaque teeth and ensuring greater translucency, better esthetics, and a more natural look. Minor soft-tissue crown lengthening was recommended to the patient before veneer preparations to correct soft-tissue asymmetry. Ceramic laminate veneers are considered a conservative treatment with excellent results.

In-office dental bleaching was performed using 35% hydrogen peroxide (35% Whiteness HP Maxx, FGM, Joinville, SC, Brazil) for 40 minutes at two sessions separated by a one-week interval (Whiteness HP Blue, FGM). The initial shade of the patient's teeth was A2 (Vita-Zahnfabrik, Spitalgasse, BS, Germany); after bleaching, the shade was A1.

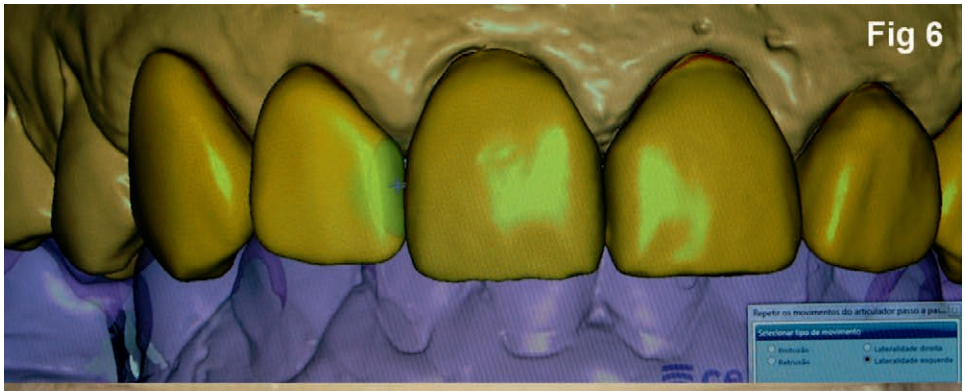
One week after dental bleaching, the patient underwent an esthetic periodontal surgery. (Figure 2). Soft-tissue recontouring (gingivectomy) was indicated in this case to remove the excess gingival tissue because the cementoenamel junction was more than 2 mm below the free gingival margin. An external bevel incision was performed for teeth 7, 8, 9, and 10, and a collar of marginal gingiva was removed to expose more of the crown of the tooth. After waiting for a healing period of 21 days, an initial impression was made using addition silicone (Virtual, Ivoclar-Vivadent) to prepare stone models and a diagnostic wax-up. A mock-up was also prepared to make the procedure more predictable.

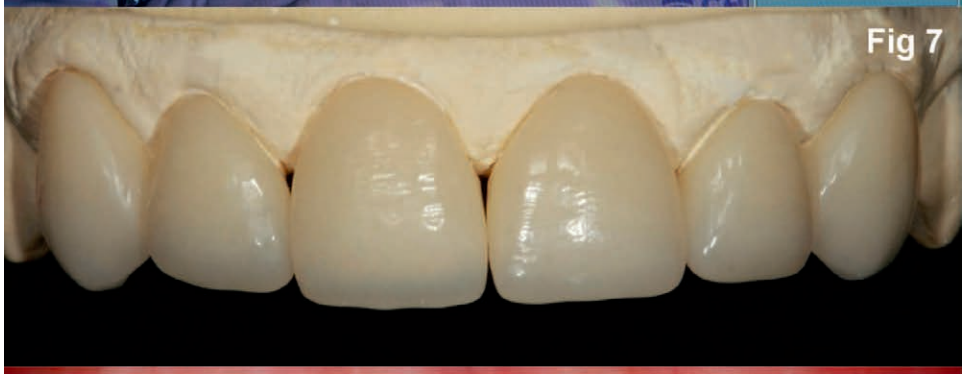
The maxillary anterior teeth were prepared following the silicone preparation guides made from the diagnostic wax-up model (Figure 3). The preparation started with a butt-joint diamond bur No. 1013 (KG Sorensen, Cotia, SP, Brazil). Guides from 0.5 to 0.7 mm in depth were prepared following the slope of the buccal surface of the tooth with a rounded-tip, diamond bur No. 2135. Reductions of 1.5 to 2.0 mm were also performed at the incisal surface. The dental preparation was refined using a No. 2135 FF diamond bur (KG Sorensen). After that, the preparations were finished and polished using Sof-lex discs (3M ESPE, Sumaré, SP, Brazil; Figure 4).

An impression of the prepared teeth was carried out using a double-cord technique with addition silicone (Virtual, Ivoclar-Vivadent), where the first single-cord Ultrapack No. 000 (Ultradent Products Inc., Indaiatuba, SP, Brazil) was inserted into the gingival sulcus and the second single-cord Ultrapack No. 0 (Ultradent Products Inc.) was positioned to enlarge the gingival sulcus. At the time of impressing, the second single cord was removed and the first single cord remained within the gingival sulcus.

To make the provisional veneers, A1 color Ceremill TEMP resin multilayer blocks (AmannGirrbach, Curitiba, PR, Brazil) were used with transitioning from the dentin to the incisal areas. The provisional veneers were designed using Ceramill Mind CAD software (AmannGirrbach) and milled under water cooling using the Ceramill Motion 2 system (AmannGirrbach). Excellent provisionals were made at the initial session to provide for adequate oral hygiene and to approximate the final restoration design, where the patient had the option to experience the esthetic result and determine possible changes to the final restorations.

This case was planned using the correlation mode,[13] but as the patient did not have an ideal shape of the contours of natural teeth to be intraorally scanned, the wax-up model was used for this purpose. A spray contrast was applied to the waxed





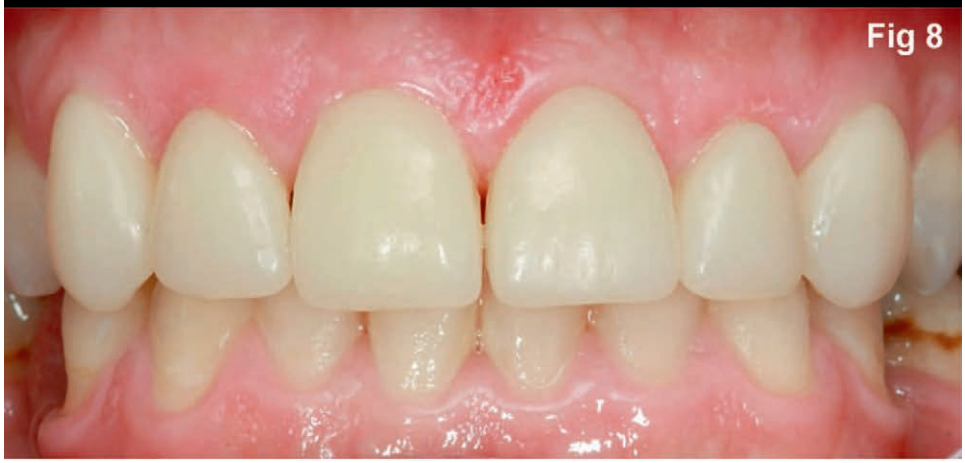


Figure 6. *Ceramill Mind computer-aided design software overlays digital information obtained from the upper and bottom model, scanning to form a record of the virtual bite of joint.*
Figure 7. *Provisional veneers made in Ceramill TEMP resin.*
Figure 8. *Cemented provisional veneers.*

model (Vita Cerec powder; Vita-Zahnfabrik, Spitalgasse, BS, Germany) to obtain the digital information. When necessary to aid and improve the contours, this procedure was complemented using the biogeneric mode to improve and adapt shape and contour (Figures 5A-C).

The CAD/CAM-software, Ceramill Mind (AmannGirrbach), superimposed the digital information obtained from scanning the maxillary and mandibular models to form a record of the virtual occlusal bite (Figure 6). Ceramill Map400 (AmannGirrbach) was used to manufacture provisional veneers using the digital information of the patient wax-up and the biogeneric model (Figure 7).

Provisional veneers were tried in for marginal integrity, functionality, occlusion, esthetics, and patient satisfaction. The provisionals were temporarily cemented (Figure 8) using an acid-etched point technique (midfacial) and bonded using the flowable composite resin, Filtek Z350 (3M ESPE) in the A2 color. Excess composite was removed, and the restorations were light cured with a LED device (Radii Plus, SDI, Bayswater, VIC, Australia) for 40 seconds. The occlusion was checked and adjusted. The provisional veneers were reevaluated after one week following the patient's evaluation of form, function, and esthetics. The patient was allowed to request modifications, which were performed, and

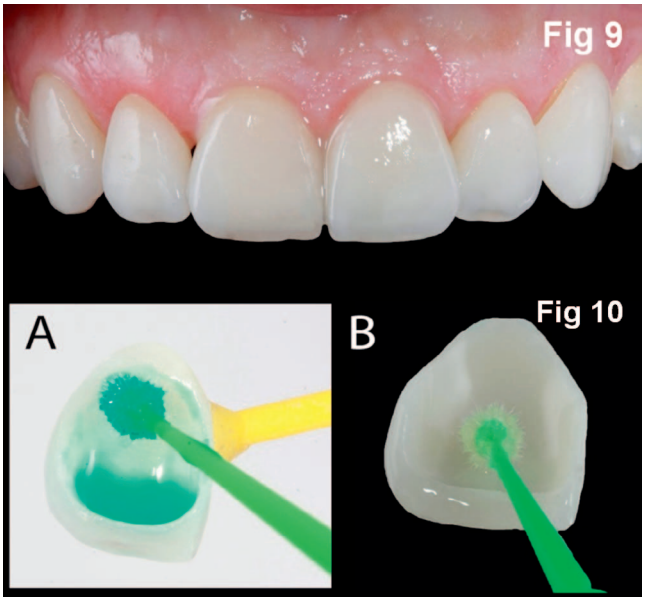


Figure 9. *Try-in test to select the color of the resin cement.*
Figure 10. *(A): Individual laminate veneers etched for 40 seconds with Monobond Etch & Primer. (B): Application of the Excite adhesive system.*

the ceramic laminate veneers were made using LD IPS e.max CAD HT–A1 (Ivoclar-Vivadent).

Prior to cementation, the marginal adaptation, interproximal contacts, and occlusion of the laminate veneers were checked and modified using a No. 2135 FF diamond bur (KG Sorensen) and polished using the Dialite LD ceramic polishing kit (Brasseler Dental EUA, Savannah, GA, USA). The color evaluation was tested with low-, medium-, and high-value try-in paste of the Variolink Veneer cement (Ivoclar-Vivadent; Figure 9).

The internal areas of the laminate veneers were conditioned using Monobond Etch & Prime (Ivoclar-Vivadent Inc., Amherst, NY, USA) by scrubbing for 20 seconds and allowing the primer to react on the surface for 40 seconds, according to the respective manufacturers' instructions. This single ceramic primer allows for etching and silanization of the glass ceramic surface. This combination is effective and shortens the pretreatment for glass-ceramic restorations as compared with the conventional method, making the process easy and reducing the risk of error. After conditioning, the surfaces were washed with an air-water spray to remove the Monobond Etch & Prime (Ivoclar-Vivadent Inc.) and dried prior to the application of the Excite adhesive system (Ivoclar-Vivadent; Figure 10A, B).

The adjacent teeth were protected using a Teflon strip, and the prepared tooth surfaces were etched with 37% Total Etch phosphoric acid (Ivoclar-Vivadent) and rinsed after 15 seconds with an air-water spray. Excess water was removed by gently blowing air. An Excite light-cure adhesive system (Ivoclar-Vivadent) was applied in two coats to the prepared surfaces. Gently blown air was used for solvent evaporation. The adhesive was not light cured. Then, the light-cured Variolink Veneer resin cement (Ivoclar-Vivadent) was placed on the intaglio surface of the veneer and seated into position. The excess resin cement was removed, and the veneers were light cured for 40 seconds from each of the dental surfaces (Figure 11A-D).

After cementation, the cervical edges of the indirect restorations and the gingiva were checked, and excess cement was removed using a No. 12

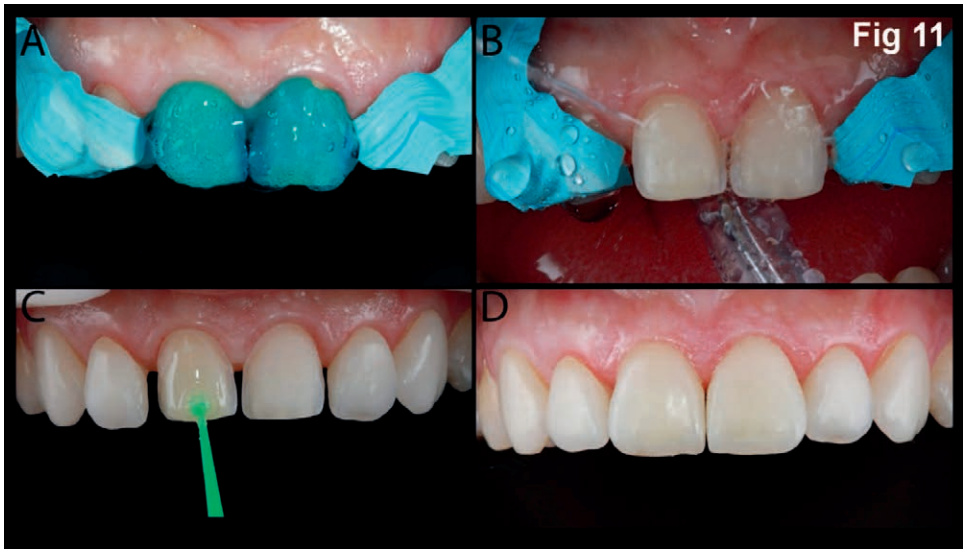


Figure 11. *(A): Etching of the teeth with phosphoric acid for 15 seconds. (B): Rinsed with an air-water spray. (C): Application of Excite adhesive system. (D): Cementation of the laminate veneers of teeth 11 and 21.*

Figure 12. *Appearance immediately after cementation.*
Figure 13. *Outcome after six months.*

scalpel blade for better hygiene and prevention of biofilm retention in the cervical region.

The immediate outcome of the restorative procedure can be seen in Figure 12 and the controlled outcome after six months in Figure 13. The patient was instructed on proper oral hygiene, and the case will be periodically evaluated at six-month intervals.

DISCUSSION

In 2005, an improved material consisting of pressable glass-ceramic LD, IPS e.max Press, was introduced. The chemical basis of this material is the same as IPS Empress 2 (Li_2O-$2SiO_2$) but with properties altered by using a different burning process.[14] When compared with IPS Empress 2, IPS e.max Press shows significant improvement in physical properties and provides a high translucency[15]; therefore, it was the material chosen for the present case report.

The use of CAD/CAM to design a dental restoration is efficient and highly precise.[16-19] Both the fatigue and tensile strength of e.max Press indicate that it is an excellent material for use with CAD/CAM.[20,21] Furthermore, the use of these systems to manufacture porcelain veneers allows the dentist to control the time as well as color, contour, and form. This treatment plan has the advantage of being conservative while providing great optical and esthetic results.[22]

The first method for designing a restoration using CAD/CAM is the biogeneric mode, which uses a database of hundreds of teeth that have been imported as a "library of shapes and forms." The shape and form are chosen by the dentist to best serve the patient's case. The second method of design is the correlation mode. This mode is recommended for duplicating a tooth in the patient's mouth, to provide the ideal contours of the teeth to be scanned intraorally before preparation. In this case report, we opted for the correlation mode with modification because it involved a wax-up model, since the patient had no contour and optimal shape in her natural teeth. When necessary, the projection of the model was improved by analysis of the library teeth (biogeneric model).[22,23] Although there are advantages of fabricating porcelain veneers using CAD/CAM in the laboratory, all the clinical procedures (ie, teeth preparation, cementation) are similar to the conventional fabrication of porcelain veneers.

In this case report, multiceramic laminate veneers were fabricated using an all-digital workflow. This technique provides the opportunity to produce precise provisional restorations with an as-proposed contour for the final laminate veneers, which could improve the esthetic results of the restorations to the patient's liking before cementation, allowing the patient to evaluate the provisional veneers critically for a period of time, providing more security for the patient and the final restoration.[24] This method also

allows for the opportunity to further customize the final restorations based on feedback from the patient regarding his or her desires and expectations. The only disadvantage associated with this technique is the increased manufacturing costs of the provisional veneers.[4]

Moreover, a system that allows for the simultaneous conditioning and silanization of the ceramic was used in this case report. The Monobond Etch & Primer contains trimethoxypropyl methacrylate (methacrylate silane) for silanization and 15%-25% ammonium polyfluoride for conditioning the surface. In addition, this primer contains 75%-85% alcohol and water by weight as solvents and less than 1% of colorant by weight. This material creates a roughness pattern that is less pronounced than HF but is as efficient for bonding.[25-28] The silanization reaction with this primer is similar to silanization with Monobond S or Monobond Plus.[29]

CAD/CAM technology is emerging and there is a learning curve involving software, dentists, and dental lab technicians. With more experience, this innovative technology will yield further esthetic improvements in dentistry, ensuring better patient satisfaction.

CONCLUSION

This article presented a technique for producing laminate veneers using CAD/CAM technology.

Acknowledgements

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of State University of Ponta Grossa, Paraná, Brazil.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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Relationship Between Simulated Gap Wear and Generalized Wear of Resin Luting Cements

A Tsujimoto • WW Barkmeier • T Takamizawa • MA Latta • M Miayazaki

Clinical Relevance

Gap wear and generalized wear simulation models can be used to assess the wear resistance of resin luting cements. In clinical situations, with restricted light exposure conditions, care should be taken to choose a resin luting cement that can be effectively cured in the chemical-cure mode.

SUMMARY

Objective: The relationship between the simulated gap wear and generalized wear of resin luting cements was investigated.

Methods: Five resin luting cements, G-Cem LinkForce (GL), Multilink Automix (MA), NX3 Nexus, Panavia V5 (PV), and RelyX Ultimate were evaluated and subsequently subjected to a wear challenge in a Leinfelder-Suzuki (Alabama) wear simulation device. Half of the specimens from each resin luting cement were photo-cured for 40 seconds and the other half were not photo-cured. The simulated gap and generalized wear were generated using a flat-ended stainless steel antagonist. Wear testing was performed in a water slurry of polymethyl methacrylate beads, and the simulated gap and generalized wear were determined using a noncontact profilometer (Proscan 2100) in conjunction with the Proscan and AnSur 3D software.

Results: A strong relationship was found between the gap wear and generalized wear simulation models. The simulated gap wear and generalized wear of the resin luting cements followed similar trends in terms of both volume loss and mean depth of wear facets with each curing method. Unlike the simulated gap wear and generalized wear of GL and PV, those of MA, NX, and RU were influenced by the curing method.

Conclusion: The results of this study indicate that simulated gap wear of resin luting cements is very similar to simulated generalized wear. In most cases, dual curing appears to ensure greater wear resistance of resin luting cements than chemical curing alone. The wear resistance of some resin luting cements appears to be material dependent and is not influenced by the curing method.

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INTRODUCTION

The increasing esthetic demands in clinical dentistry have resulted in the continued development of esthetic restorative materials such as resin composites, glass ceramic and zirconia.[1] These developments include intracoronal and extracoronal restorations which use esthetic restorative materials requiring the use of a luting cement.[2] Several issues associated with the fit and marginal adaptation of esthetic restorations appear to significantly influence their durability.[3] Thus, the selection of a luting cement is an important consideration in determining the long-term clinical success of esthetic restorations.[4] The stability of such restorations has been increased using a resin luting cement with high mechanical and low solubility properties.[5] Despite their improved physical properties, interfacial and/or marginal defects have been clinically observed to occur frequently around bonded esthetic restorations.[6] This is particularly important for inlay and onlay restorations of posterior teeth, where the cement margin is exposed on the occlusal surface. Therefore, good physical properties (eg, surface hardness and wear resistance) are required for resin luting cements.[7] Although the wear resistance of esthetic restorative materials have been extensively examined, limited research has been reported in the area of resin luting cements.

In a previous study, Shinkai and others[8] examined the wear resistance of different types of resin luting cements and a glass ionomer cement in terms of marginal gap formation. A resin luting cement with micro fillers was found to provide the greatest wear resistance, whereas a glass ionomer cement exhibited the lowest wear resistance. In addition, these investigators reported significant relationships among the gap width, amount of wear, and type of cement. Furthermore, Kawai and others[9] examined the relationship between the gap wear and the filler particle size of a resin luting cement and found a strong relationship (R^2>0.9) between the vertical wear loss and the gap width for the three resin luting cements examined in their study. They also found that a resin luting cement having submicron fillers offered greater wear resistance when compared with those including hybrid fillers.

One of the primary areas of concern when developing luting cements is noncontact generalized wear or erosion.[10] Noncontact generalized erosion is one of the common wear mechanisms responsible for the interfacial and/or marginal breakdown of a luting cement. Whereas the intrinsic wear of a luting cement is important, interfacial and/or marginal gap wear is also a critical concern. Gap wear is influenced by both material properties and the unique environment of the cement.[11] With the objective of evaluating the potential loss of luting cements at the interface, a new model was developed for gap wear simulation. This model uses a thin gap (300 μm wide, 3 mm long) in a stainless steel custom fixture.

The purpose of this laboratory study was to evaluate the relationship between the simulated gap wear and generalized wear of resin luting cements. The null hypotheses to be tested were that 1) there would be no relationship between the simulated gap wear and generalized wear of resin luting cements; and 2) the simulated gap wear and generalized wear of resin luting cements would not be influenced by the type of material or curing method.

METHODS AND MATERIALS

Study Materials

Five resin luting cements were evaluated in this study: G-Cem LinkForce (GL; GC, Tokyo, Japan); Multilink Automix (MA; Ivoclar Vivadent, Schaan, Lichtenstein); NX3 Nexus (NX; Kerr, Orange, CA, USA); Panavia V5 (PV; Kuraray Noritake Dental, Tokyo, Japan); and RelyX Ultimate (RU; 3M ESPE, St Paul, MN, USA). The resin luting cements used in this study are listed in Table 1 along with their associated lot numbers, components, and percentages of filler loading, which were provided by the manufacturers.

Specimen Preparation for Simulated Gap Wear

A total of 40 wear specimens were prepared for each of the five resin luting cements for simulated gap wear testing. A stainless steel custom fixture was designed to examine the resin luting cement wear, with a thin gap for simulated gap wear testing. The two-piece fixture was designed to have a gap (300 μm wide, 3 mm long, and 4 mm deep) for simulated gap wear testing. Half the specimens (20 specimens, designated as the dual-cure group) of each resin luting cement were photo-cured for 40 seconds at a standardized distance of 2 mm with a quartz-tungsten halogen unit (Spectrum 800 Curing Unit, Dentsply Caulk, Milford, DE, USA) set at 600 mW/cm^2. The other half (20 specimens, designated as the chemical-cure group) were not photo-cured. After 24 hours, the cement surfaces were polished flat to 4000 grit using a sequence of silicon carbide papers (Struers, Cleveland, OH, USA) and a grinder-polisher (Ecomet 4, Buehler, Lake Bluff, IL, USA).

Table 1: *Resin Luting Cements*

Resin Luting Cement (Code, Shade)	Main Components (Filler Content)	Manufacturer (Lot No.)
G-CEM LinkForce (GL, A2)	Dimethacrylate, silica filler, initiators, stabilizers, pigments (63.0 wt%, 38.0 vol%)	GC, Tokyo, Japan (1407281)
Multilink Automix (MA, yellow)	Dimethacrylate, HEMA, barium glass filler, silica filler, ytterbium trifluoride, initiators, stabilizers, pigments (68.5 wt%, 42.5 vol%)	Ivoclar Vivadent, Schaan, Lichtenstein (150317)
NX3 Nexus (NX, yellow)	Methacrylate ester monomer, mineral fillers, initiators, stabilizers, pigments, radiopaque agent (66.5 wt%, 43.3 vol%)	Kerr, Orange, CA, USA (13394)
Panavia V5 (PV, Universal)	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, barium glass filler, fluoroaluminosilicate glass, silica filler, initiators, stabilizers, pigments (61.0 wt%, 38.0 vol%)	Kuraray Noritake Dental, Tokyo, Japan (A90026)
RelyX Ultimate (RU, A1)	Methacrylate monomer, alkaline filler, initiator components, stabilizers, pigments, rheological additives, fluorescence dye (67.0 wt%, 43.0 vol%)	3M ESPE, St Paul, MN, USA (588835)

Specimen Preparation for Simulated Generalized Wear

The fixture size for the generalized wear simulations was the same as the custom stainless steel fixture for simulated gap wear, with the exception of the cavity. The custom stainless steel fixtures were machined for simulated generalized wear testing with a cylindrical cavity (4.5 mm in diameter and 4.0 mm deep). Twenty specimens from each of the five resin luting cements were made for both dual-cure and chemical-cure groups.

Wear Simulation

A Leinfelder-Suzuki (Alabama) device was used for wear simulation (Figure 1). The simulator has a plastic water bath, and the custom-wear fixtures were mounted inside the four-station bath. A brass cylinder was then placed around each fixture in the bath to serve as a reservoir for the abrasive medium (ie, a water slurry of unplasticized polymethyl methacrylate [PMMA] with an average particle size of 44 μm). The medium was placed inside the brass cylinders to cover the surface of the resin luting cement in the custom fixtures. The PMMA water slurry inside the brass cylinders was approximately 6.0 mm thick over the surface of the resin cement.

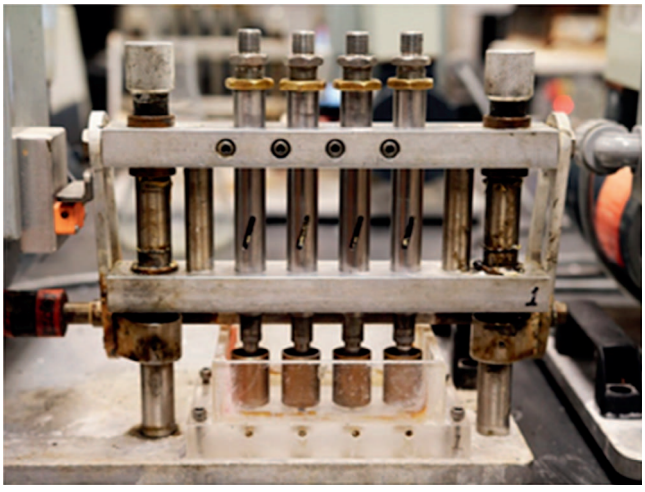

Figure 1. *Leinfelder-Suzuki (Alabama) wear simulator device.*

A stainless steel cylinder (6.5 mm in diameter) with a flat-end stylus tip was used as the antagonist for both the gap wear and generalized wear simulations. The antagonist tips were mounted on spring-loaded pistons to deliver the wear challenges. During load applications, the antagonists rotated approximately 30° as the maximum force was reached (ie, maximum load of 78.5 N at a rate of 2 Hz) and then counterrotated back to the original starting position as the load was relaxed to complete the cycle. Each set of specimens was exposed to 400,000 cycles during the gap and generalized wear simulations.

Wear Measurements

Prior to the wear simulation, each resin luting cement specimen was profiled using a Proscan 2100 noncontact optical profilometer (Scantron Industrial Products, Taunton, England) with the Proscan software. These profiles provided the pretest digitized surface contours.

After wear simulation, the specimens were ultrasonically cleaned (L&R T-14B solid state ultrasonic cleaner, L&R Manufacturing Company, South Orange, NJ, USA) in distilled water for three

minutes and then profiled again with the Proscan 2100 unit. The x-, y-, and z-coordinates of the "before" and "after" scans were exported from the Proscan software to another computer for analysis using AnSur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA).

The facet depth and volume loss of the specimens were determined from the differences between the before and after data sets. A computerized fit of the data sets was first completed with the AnSur 3D software. The volume losses (VLs, mm^3) and mean depths (MDs, μm) of the wear facets were subsequently determined for both simulated gap wear and generalized wear for each of the five resin luting cements polymerized in the dual-cure and chemical-cure modes.

Scanning Electron Microscopy Observations of Resin Luting Cement Surfaces

Ultrastructural observations were conducted on the polished surfaces of the resin luting cements after argon ion etching. Three specimens per test group were observed using field-emission scanning electron microscopy (SEM; ERA 8800FE, Elionix, Tokyo, Japan).

The surfaces of the resin luting cements were polished flat to 4000 grit using a sequence of silicon carbide papers and a grinder-polisher. The surfaces were subsequently polished using abrasive discs (Fuji Star Type DDC, Sankyo-Rikagaku, Saitama, Japan) followed by a series of diamond pastes down to a particle size of 0.25 μm (DP-Paste, Struers) to bring the surfaces to a high-gloss state. The polished surface specimens intended for SEM observation were dehydrated upon immersion in aqueous tert-butanol solutions with increasing concentrations (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for two hours) and were subsequently transferred to a critical-point dryer (Model ID-3, Elionix) for 30 minutes. These polished surfaces (with the objective of enhancing the visibility of the filler particles) were etched for 30 seconds with argon ion beams (EIS-200ER, Elionix) perpendicular to their surfaces at an accelerating voltage of 1.0 kV and with an ion current density of 0.4 mA/cm^2. The surfaces were then coated with a thin film of gold in a vacuum evaporator (Quick Coater SC-701, Sanyu Electron, Tokyo, Japan). The SEM observations were performed using an operating voltage of 10 kV.

Table 2: *Regression Analysis: Gap Wear and Generalized Wear Simulation Models*

Curing Method	Volume Loss (VL)		Mean Depth (MD)	
	R^2	*p*	R^2	*p*
Dual-cure group	0.994	0.000	0.913	0.011
Chemical-cure group	0.860	0.023	0.875	0.020

SEM Observations of Wear Facets After Gap Wear Simulation

The ultrastructural observations were conducted on representative wear facets of the resin luting cements from both the dual-cure and chemical-cure groups for both gap wear and generalized wear simulations using a SEM (TM3000 tabletop microscope, Hitachi-High Technologies, Tokyo, Japan). Three specimens of each resin luting cement test group were observed after gap wear and generalized wear simulations. After the wear analysis, representative specimens were coated with a thin film of gold-palladium in a vacuum evaporator (Emitech SC7620 Mini Sputter Coater, Quorum Technologies, Ashford, UK). The SEM observations were performed using an operating voltage of 15 kV.

Statistical Analyses

The VLs and MDs for gap and generalized wear of the resin luting cements were analyzed using a commercial statistical software package (SPSS Statistics Base, IBM Corp, Armonk, NY, USA). A linear regression analysis between gap wear and generalized wear on the VLs and MDs was conducted. After confirming that the distribution was normal using the Kolmogorov-Smirnov test, a two-way analysis of variance (ANOVA) and Tukey post hoc test were used to analyze each data set, with a significance level of $\alpha = 0.05$.

RESULTS

Regression Analysis

The results of the regression analysis performed on the VLs and MDs following simulated gap wear and generalized wear are shown in Table 2. For the dual-cure groups, a strong positive relationship was evident between the gap wear and generalized wear simulation models for both the VL (R^2=0.994) and MD (R^2=0.913). In addition, a strong relationship was also observed between the gap wear and the generalized wear simulation models for both the VL (R^2=0.860) and MD (R^2=0.875) of the chemical-cure groups. All of these correlations were statistically significant at a level of $\alpha = 0.05$.

Table 3: *Simulated Gap Wear of Resin Luting Cements*[a]

Volume Loss (VL)

Resin Luting Cement	Volume Loss (mm^3)		
	Dual-Cure Group	Chemical-Cure Group	% Difference
GL	0.023 (0.007) a,A	0.028 (0.006) a,A	21.7
MA	0.026 (0.010) a,A	0.039 (0.008) b,B	50.0
NX	0.028 (0.007) a,A	0.042 (0.009) b,B	50.0
PV	0.029 (0.014) a,A	0.033 (0.008) a,b,A	13.8
RU	0.030 (0.007) a,A	0.044 (0.006) b,B	46.7

Abbreviations: GL, G-CEM LinkForce; MA, Multilink Automix; NX, NX3 Nexus; PV, Panavia V5; RU, RelyX Ultimate.
[a] Same lowercase letter in same vertical column indicates no significant difference (p>0.05). Same capital letter within individual rows indicates no significant difference (p>0.05).

Gap Wear Simulation

The VL and MD results for the wear facets of the resin luting cements after the gap wear simulations are shown in Tables 3 and 4, respectively. The two-way ANOVA results indicated that the material type ($p<0.001$), curing method ($p<0.001$), and interaction between the material type and curing method ($p<0.001$) significantly affected the VLs and MDs of the five resin luting cements. Significant differences were evident among the materials for both the dual-cure and chemical-cure groups. The trend in the VLs and MDs of the dual-cure (GL-MA-NX-PV-RU) and chemical-cure (GL-PV-MA-NX-RU) groups following the gap wear simulations were similar. Unlike MA, NX, and RU, the VL and MD of the wear facets of GL and PV were not influenced by the curing method.

Generalized Wear Simulation

The VL and MD results for the resin luting cements after generalized wear simulation are shown in Tables 5 and 6, respectively. The two-way ANOVA results revealed a significant effect for the material type ($p<0.001$), curing method ($p<0.001$), and interaction between the material type and curing method ($p<0.001$) for the VLs and MDs of the five resin luting cements. Significant differences were evident among the materials for both the dual- and chemical-cure groups. The trends in wear for the VLs and MDs of the dual- (GL-MA-NX-PV-RU) and chemical- (GL-PV-MA-NX-RU) cure groups, following generalized wear simulation are similar. Unlike MA, NX, and RU, the VLs and MDs of the wear facets of GL and PV were not influenced by the curing method.

Table 4: *Simulated Gap Wear of Resin Luting Cements*

Mean Depth (MD)

Resin Luting Cement	Mean Depth (μm)		
	Dual-Cure Group	Chemical-Cure Group	% Difference
GL	35.4 (9.2) a,A	44.4 (11.7) a,A	25.4
MA	38.2 (13.0) a,b,A	62.7 (11.9) c,B	64.1
NX	40.4 (8.3) a,b,A	64.3 (9.7) b,B	59.2
PV	42.9 (7.2) a,b,A	52.3 (9.9) a,c,A	21.9
RU	45.2 (9.6) b,A	69.5 (10.4) b,B	53.8

Abbreviations: GL, G-CEM LinkForce; MA, Multilink Automix; NX, NX3 Nexus; PV, Panavia V5; RU, RelyX Ultimate.
[a] Values in parentheses are standard deviations. Same small case letter in same vertical column indicates no significant difference (p>0.05). Same capital case letter within individual rows indicates no significant difference (p>0.05).

Table 5: *Simulated Generalized Wear of Resin Luting Cements*[a]

Volume Loss (VL)

Resin Luting Cement	Volume Loss (mm^3)		
	Dual-Cure Group	Chemical-Cure Group	% Difference
GL	0.426 (0.090) a,A	0.498 (0.096) a,A	16.9
MA	0.525 (0.088) a,A	0.721 (0.102) b,B	37.3
NX	0.579 (0.140) b,A	0.764 (0.142) c,B	31.9
PV	0.619 (0.092) b,A	0.673 (0.120) b,A	8.7
RU	0.664 (0.122) b,A	0.960 (0.106) d,B	44.6

Abbreviations: GL, G-CEM LinkForce; MA, Multilink Automix; NX, NX3 Nexus; PV, Panavia V5; RU, RelyX Ultimate.
[a] Values in parentheses are standard deviations. Same small case letter in same vertical column indicates no significant difference (p>0.05). Same capital case letter within individual rows indicates no significant difference (p>0.05).

SEM Observations of Resin Luting Cement Surfaces

Representative SEM images of the polished resin luting cement surfaces are shown in Figure 2. The

Table 6: *Simulated Generalized Wear of Resin Luting Cements*[a]

Mean Depth (MD)

Resin Luting Cement	Mean Depth (μm)		
	Dual-Cure Group	Chemical-Cure Group	% Difference
GL	35.3 (6.9) a,A	39.4 (8.4) a,A	11.6
MA	43.5 (6.6) b,A	56.3 (9.3) b,B	29.4
NX	50.3 (7.2) c,A	66.7 (10.6) c,B	32.6
PV	51.0 (9.9) c,A	55.8 (9.1) a,b,A	9.4
RU	54.3 (9.4) c,A	73.2 (11.5) c,B	34.8

Abbreviations: GL, G-CEM LinkForce; MA, Multilink Automix; NX, NX3 Nexus; PV, Panavia V5; RU, RelyX Ultimate.
[a] Values in parenthesis are standard deviations. Same small case letter in same vertical column indicates no significant difference (p>0.05). Same capital case letter within individual rows indicates no significant difference (p>0.05).

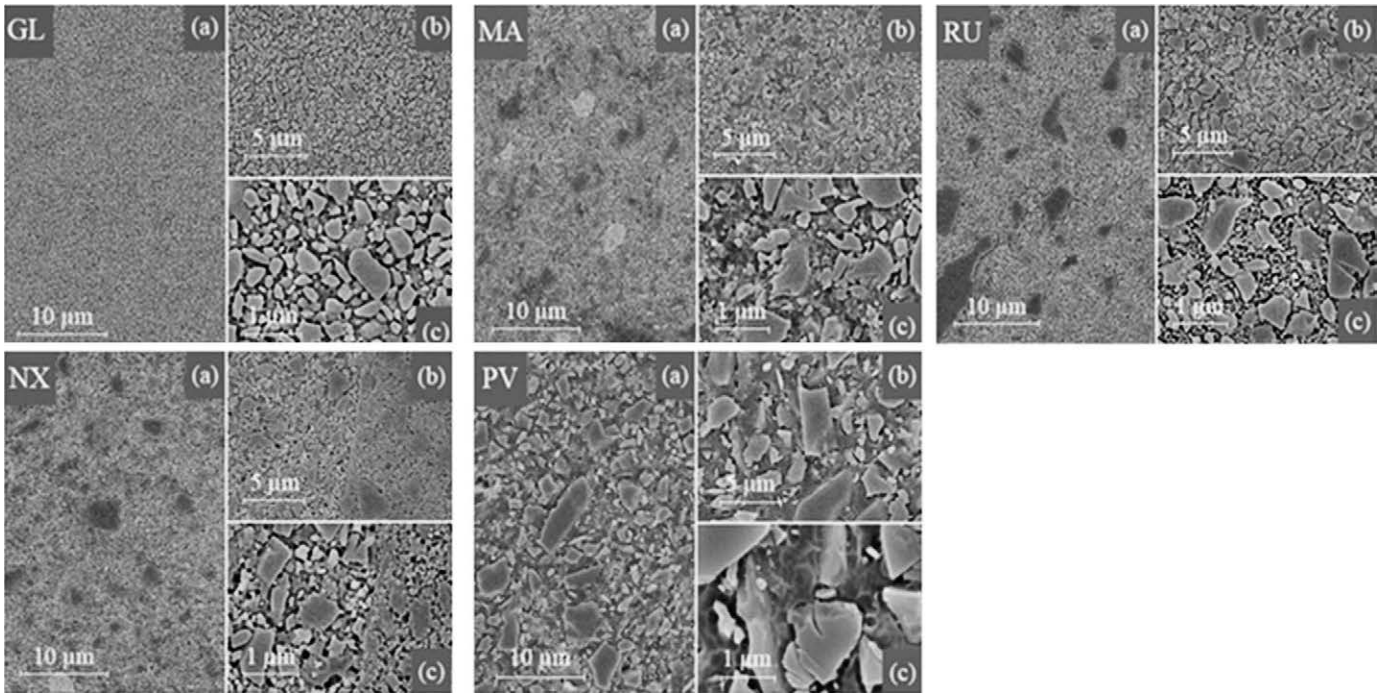

Figure 2. *Representative scanning electron microscope images of polished resin luting cement surfaces at (a): 2500× magnification, (b): 10,000× magnification, and (c): 30,000× magnification. The sizes and distributions of fillers in the different types of resin luting cements vary depending on the material: GL, wide size range (0.1-1.0 μm) of small irregular particles; MA, wide size range (1-8 μm) of irregular particles; NX, wide size range (0.1-10 μm) of irregular particles; PV, wide size range (0.1-15 μm) of irregular particles; RU, wide size range (0.1-20 μm) of irregular particles.*

argon ion etching revealed clear differences in the filler particle size, shape, and distribution of the specimens studied. The resin luting cement specimens exhibited a wide variety of filler particle sizes and shapes.

The SEM images of polished GL surfaces, with argon ion etching, showed the presence of small irregular particles with a wide size distribution (0.1-1.0 μm). The SEM images of polished MA surfaces, with argon ion etching, revealed the presence of irregular particles with wide size distributions (0.1-8 μm). For the NX, PV, and RU specimens, the SEM images also revealed the presence of irregular particles with wide size distributions (0.1-10 μm, 0.1-15 μm, and 0.1-20 μm, respectively).

SEM Observations of Wear Facets After Gap Wear Simulation

Representative SEM images of the wear facets that were obtained after conducting the gap wear simulation of the dual- and chemical-cure groups are shown in Figure 3. The SEM images of the worn surfaces of the MA, NX, and RU, within the chemical-cure group after performing the gap wear simulations, showed clearer gap formations at lower magnification as compared with those of the dual-cure group. The higher magnification micrographs appeared to indicate a greater extent of filler particle plucking for the chemical-cure group as compared with the dual-cure group. On the other hand, the SEM images obtained after the gap wear simulation of the worn surfaces of the GL and PV, which were polymerized using the two different curing methods, did not show any clear differences.

SEM Observations of Wear Facets After Generalized Wear Simulation

Representative SEM images of wear facets of the dual-cure and chemical-cure groups after generalized wear simulation are shown in Figure 4. The SEM images obtained for the worn surfaces of the MA, NX, and RU, within the chemical-cure group after the generalized wear simulations, appeared to have a greater degree of filler particle plucking compared with the dual-cure group. The SEM images obtained after the generalized wear simulation for the worn surfaces of the GL and PV, which were cured using the two different methods, did not show any clear differences.

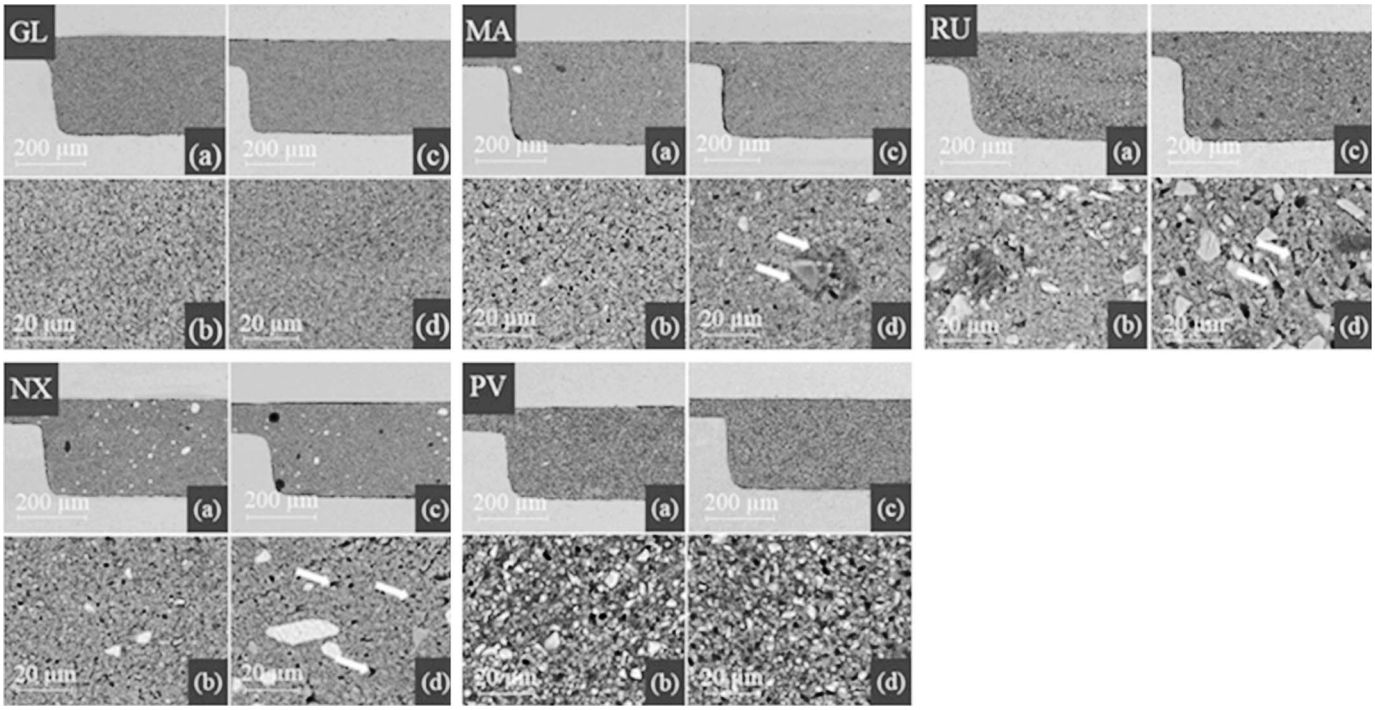

Figure 3. *Representative scanning electron microscope (SEM) images of the wear facets after the simulated gap wear of resin luting cements within the dual-cure group at (a): 250× magnification and (b): 2500× magnification and within the chemical-cure group at (c): 250× magnification and (d): 2500× magnification. The SEM images of the worn surfaces of MA, NX, and RU within the chemical-cure group show a greater degree of filler particle plucking (white arrows, higher magnifications) than the dual-cure group. On the other hand, the SEM images obtained after simulated gap wear for the worn surfaces of GL and PV samples cured using different methods do not show any clear differences.*

DISCUSSION

Two wear simulation models were used in this laboratory study to assess the relative wear resistances of five resin luting cements. The simulated wear results for the resin luting cements generated using a new gap wear model were compared with those obtained with a more commonly used generalized wear simulation model. In both models, a flat-ended stainless-steel antagonist was used to induce wear via an abrasive medium composed of a water slurry of PMMA beads. The dual-cure and chemical-cure groups for the five resin luting cements were assessed using the two wear simulation models along with SEM observations.

In a previous study,[11] simulated wear of self-adhesive resin cements was investigated using both a gap (for toothbrush abrasion) and the Academisch Centrum for Tandheelkunde Amsterdam (ACTA) wear simulation models. The investigators reported that the self-adhesive resin cements showed good wear resistance against toothbrush abrasion but not against ACTA wear. Thus, there was no correlation (R^2=0.0567) between the two wear models. However, in the present study, after comparing simulated gap wear and generalized wear of five resin luting cements, strong positive correlations were found between the two wear models for both the dual-cure (VL [R^2=0.994] and MD [R^2=0.913]) and chemical-cure (VL [R^2=0.860] and MD [R^2=0.875]) groups.

Barkmeier and others[12,13] conducted several wear simulation studies with a frequently used generalized wear simulation model to evaluate the wear resistance of resin-based materials. In addition, they also reported good agreement between simulated generalized wear and clinical wear of resin composite materials.[14] A new gap wear simulation model was used in the present study in an effort to assess the wear resistance of resin luting cements occurring at the marginal areas of cemented clinical restorations. The new gap wear simulation model delivers wear challenges with the same stainless steel antagonist tip used in the generalized wear simulation model. The primary difference is the area of resin luting cement being exposed to the wear process. Overall, the correlation between the wear characteristics (in terms of VL and MD) of the resin luting cements in the two wear simulation models was excellent. Although the objective of using the new gap wear simulation model was to more closely replicate the type of cement abrasion that may occur

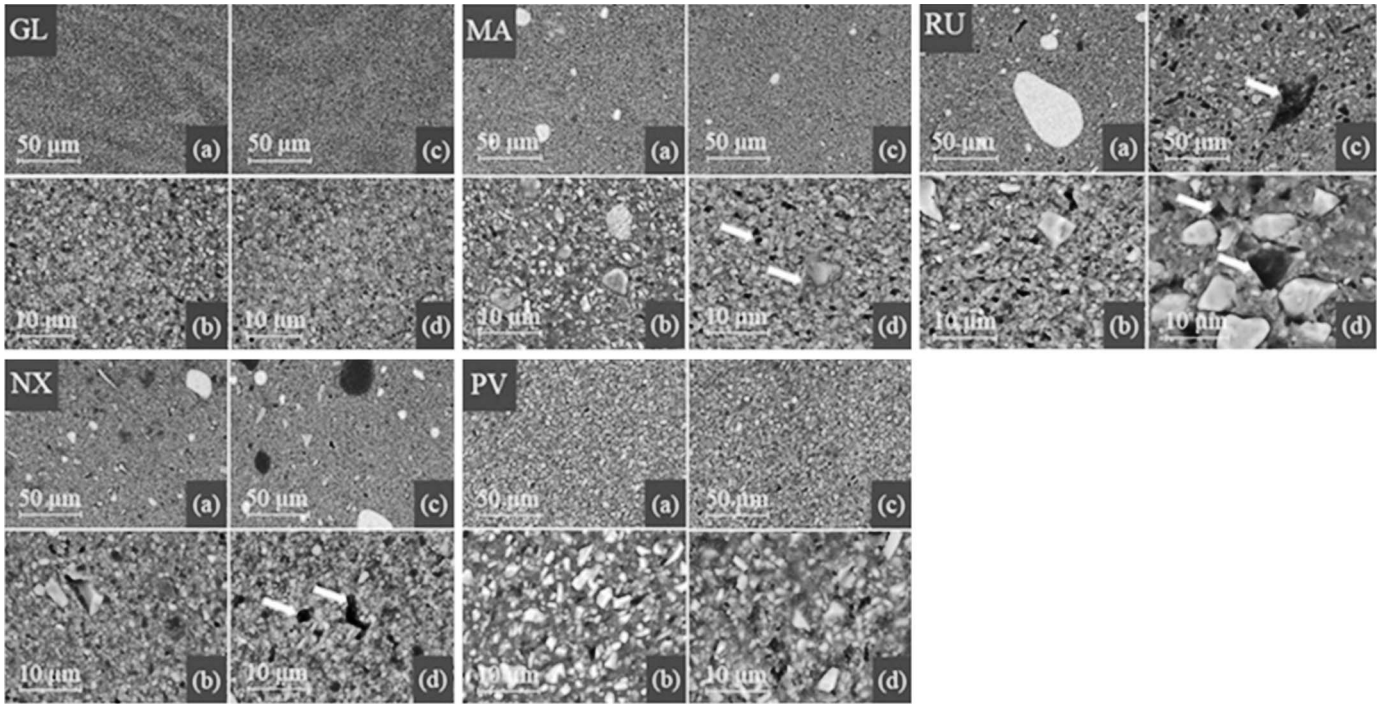

Figure 4. *Representative SEM images of the wear facets after simulated generalized wear of resin luting cements within the dual-cure group at (a): 1000× magnification and (b): 5000× magnification and within the chemical-cure group at (c): 1000× magnification and (d): 5000× magnification. The SEM images of the worn surfaces of MA, NX, and RU within the chemical-cure group after the generalized wear simulations seemed to have higher extents of filler particle plucking (white arrows) than the dual-cure group. The SEM images obtained after generalized wear for the worn surfaces of GL and PV samples cured using different methods do not show any clear differences.*

in the oral cavity, the results indicated that the wear resistance of resin luting cements can be assessed with the standard generalized wear simulation model. However, the new gap wear simulation model does appear to mimic the type of wear challenges encountered at marginal areas of cemented restorations in the oral cavity. The results of the new gap wear simulation model reaffirmed that thin film abrasive wear of resin luting cement in marginal closure areas remains an issue for the long-term clinical success of cemented restorations.

Simulated gap wear and generalized wear of resin luting cements of the dual-cure and chemical-cure groups clearly showed significant differences among the different materials. However, the observed trends (dual-cure group: GL-MA-NX-PV-RU; chemical-cure group: GL-PV-MA-NX-RU) in the simulated wear characteristics of VL and MD for the five resin luting cements were the same in the two wear simulation models, regardless of the curing method (Tables 3-6).

It has been reported that the filler load plays a particularly important role in the wear resistance of resin-based materials, with higher filler loads reducing the level of wear.[15] The filler load values provided by the manufacturers varied greatly among the resin luting cements (Table 1), although a clear relationship between the simulated wear and the filler load was not evident in the present study.

Conversely, a clear relationship between the simulated wear and the filler size was found. The filler particle size trend among the polished resin luting cements (GL-MA-NX-PV-RU), as determined from the SEM observations using argon ion etching, was the same as the trend for both simulated gap wear and generalized wear models within the dual-cure group. It has been reported that the filler particle size affects the simulated wear of resin composites.[16] That is, resin composites with fillers having low particle sizes showed lower simulated wear. These results for resin cements are consistent with the earlier results for restorative resin composites.[17] GL from the dual-cure group showed smaller filler particles and thus lower simulated wear. On the other hand, RU, which had larger filler particles and a broad size distribution, exhibited greater wear regardless of the wear simulation model. Therefore, both the simulated gap and generalized wear of the resin luting cements were influenced by the filler particle size, as in the case of resin composites. The

differences between the wear resistances of different types of resin luting cements have been hypothesized to result from the lower interparticle spacing between small filler particles. Because small filler particles are more closely packed, the resin matrix between them is protected from further wear.[18] On the other hand, when larger filler particles are removed from the resin composite surface, a void is produced, thereby exposing the underlying resin matrix to wear.[19] Also, the removed particles can cause further abrasion of the surfaces of resin-based materials, resulting in increased wear.[19]

In addition, although different types of resin luting cements having similar shades should ideally be used for a comparative study, the types of resin luting cement used herein showed slight differences in shade (Table 1) due to the limited availability of shades from the manufacturers. It has been reported that the shades and translucencies of resin-based materials significantly influence their degrees of conversion.[20] Therefore, the differences between the shades and translucencies of the types of resin luting cements used herein may have contributed to the material differences in observed wear in simulated gap wear and generalized wear.

The simulated gap and generalized wear of the resin luting cements differed significantly depending on the curing method. The trend observed for the simulated gap wear and generalized wear of the chemical-cure group (GL-PV-MA-NX-RU) was different than that of the dual-cure group (GL-MA-NX-PV-RU) (Tables 3-6). Previous studies comparing the influence of the curing method on the degree of conversion of resin luting cements have generally found that photo-curing produces a significantly higher degree of conversion than does chemical curing alone for dual-cure resin luting cements.[21,22] In addition, the wear resistance and mechanical properties of resin-based materials were reported to increase by improving the degree of conversion.[23,24] The ability of dual-cure resin luting cement to cure effectively in the chemical-curing mode is key to the long-term clinical success of cemented restorations, particularly when photo-curing is not possible or is limited. Therefore, based on the results of this study, clinicians should pay more attention to the different setting characteristics of dual-cure resin luting cement when selecting these materials for clinical use.

The VLs and MDs in the gap wear facets of MA, NX, and RU significantly increased (VL: 46.7%-50.0%; MD: 53.8%-64.1%) for the chemical-cure group as compared with the dual-cure group. Similarly, the VLs and MDs in the generalized wear facets of MA, NX, and RU significantly increased (VL: 31.6%-44.6%; MD: 29.4%-34.8%) for the chemical-cure group samples as compared with the dual-cure group. On the other hand, the VLs and MDs of GL and PV showed similar values ($p>0.05$) for both gap wear and generalized wear simulation models and both the dual- and chemical-cured groups.

The SEM images of the worn surfaces (gap wear and generalized wear simulations) of the MA, NX, and RU, within the chemical-cure group, appeared to show a greater degree of cracking and filler particle plucking than the dual-cure group. On the other hand, the GL and PV wear simulation specimens cured using the different polymerization methods did not show any clear differences. These findings might be explained by the different compositions of the materials. Polymerization of dual-cure resin luting cement can be activated either by inducing a photo initiator (eg, camphorquinone) or breaking the molecules of a chemical initiator (eg, benzoyl peroxide) such that free radicals are formed to initiate polymerization reactions.[25,26] The contents of the initiators of photo- and chemical-polymerization in resin luting cement differ depending on the material.[27] For instance, the benzoyl peroxide content of some types of resin luting cements are double those of other materials. Some cements are overly dependent on photo polymerization, which may lead to inadequate polymerization and performance in clinical situations.[28] Unfortunately, detailed information from the manufacturers about the contents of photo- and chemical-initiators in resin luting cement is very limited; thus, further comparisons are difficult.

On the basis of the results of this study, the first null hypothesis that there would be no relationship between the simulated gap wear and the generalized wear of different types of resin luting cement was not rejected. On the other hand, the second null hypothesis that the simulated gap wear and generalized wear of different types of resin luting cement are not influenced by the type of material or curing method was rejected.

CONCLUSION

The results of this study indicate that the simulated wear of resin luting cements generated using a new gap model is very similar to that produced by a commonly used generalized wear simulation model. Thus, both models can be used to assess the wear resistance of resin luting cements. In addition, dual

curing appears to result in greater wear resistance of resin luting cement compared with chemical curing in most cases, but some types of resin luting cements showed no significant differences between the two curing methods. Therefore, the influence of the curing method on resin luting cements seems to be material-dependent.

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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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Extensive Assessment of the Physical, Mechanical, and Adhesion Behavior of a Low-viscosity Bulk Fill Composite and a Traditional Resin Composite in Tooth Cavities

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Clinical Relevance

Integrated analysis of the physical, mechanical, and adhesion behavior of low-viscosity bulk fill resin composite confirms that it presented equal or superior properties when compared with a traditional resin composite, which has been known to be clinically successful.

SUMMARY

Objectives: To compare the degree of conversion (DC), depth of polymerization (DP), shrinkage stress (SS), flexural strength (FS), elastic modulus (EM), and bond strength (BS) of a low-viscosity bulk fill resin composite and a paste-like traditional composite.

Methods: Tetric Evo-Flow Bulk Fill (TBF) and Empress Direct (ED; Ivoclar Vivadent) composites were used. DC (%) and FS/EM (MPa/GPa) were evaluated in bar specimens (7×2×1 mm; n=10) using Fourier-transform infrared spectroscopy and a three-point bending test in a universal testing machine (UTM), respectively. For DP and BS tests, conical cavities (n=10) were prepared in bovine dentin and restored with the composites. DP was analyzed by calculating the bottom-to-top surface microhardness ratio (BTHR), and BS (MPa) was determined by push-out testing in the UTM. SS (MPa) was measured for one increment of

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TBF and two increments of ED in a UTM attached to an extensometer (n=5). Data were analyzed using Student *t*-test and analysis of variance (α=0.05).

Results: TBF presented higher values than ED for DC (85.7±6.6% vs 54.2±4.9%) and BS (0.95±0.70 MPa vs 0.35±0.15 MPa). TBF values were lower than ED values for FS (76.6±16.8 MPa vs 144.9±24.1 MPa) and maximum SS (0.77±0.07 MPa vs 1.07±0.15 MPa). TBF and ED values were similar for BTHR (0.83±0.16 vs 0.84±0.08) and EM (11.5±2.8 GPa vs 12.5±2.6 GPa).

Conclusions: The physical and mechanical properties of TBF, a bulk fill resin composite, were similar or superior to those of ED, a conventional composite, with the exception of FS measurements.

INTRODUCTION

A major objective in restorative dentistry is the development of resin composites that allow rapid filling of cavities and provide acceptable clinical longevity. Current clinical protocols recommend the application of traditional resin composites in increments of no more than 2-mm thickness.[1] However, this technique increases chair time, favors the incorporation of voids in the restoration body, increases the risk of contamination by moisture between composite layers, and makes working in small cavities more difficult.[2,3]

Recent advances in resin composites have led to the emergence of bulk fill materials,[4] which can be inserted in a dental cavity in a single 4-5–mm layer[5,6] and are commercially available in low and medium viscosity (paste-like consistency).[7] Both of them generally have decreased filler loads and increased filler sizes to enhance the depth of cure. They also may contain photoinitiators able to provide effective depth of cure at 4-5 mm, as well as monomers with low double-bond concentrations and, in some cases, monomers that cleave during polymerization.[8,9]

Most traditional flowable composites have reduced percentages of inorganic filler particles (44%-55% of total volume) and greater amounts of resinous components, in comparison with traditional paste-like composites. Flowable composites have low elastic moduli that compete with stress development, potentially helping to maintain the marginal seal of the restoration.[10] Moreover, flowable composites are readily workable and adaptable to cavity walls, and their use can reduce marginal defects in restorations.[11]

Many studies have demonstrated the favorable properties of low-viscosity bulk fill composites, such as a suitable degree of conversion (DC)[2,6,12] and bond strength similar to that of traditional paste-like composites.[13] However, a limited number of studies have evaluated the physical, mechanical, and adhesion behaviors of low-viscosity bulk fill composites using methods capable of reproducing clinical conditions, such as the filling of dentin cavities that are deep (4 mm) and have high C-factor.

The aim of this study was to compare the DC, depth of cure, shrinkage stress, flexural strength, elastic modulus, and bond strength of a low-viscosity bulk fill composite and a conventional resin composite in dentin cavities with high C-factor. The null hypothesis was that there is no significant difference between materials tested.

METHODS AND MATERIALS

Experimental Design

This laboratory study was conducted to examine the response variables DC, depth of polymerization, maximum shrinkage stress, flexural strength, elastic modulus, and bond strength in a low-viscosity bulk fill resin composite (Tetric Evo-Flow Bulk Fill [TBF]; Ivoclar Vivadent, Schaan, Liechtenstein) and a conventional resin composite (Empress Direct [ED]; Ivoclar Vivadent). For DC, depth of cure, flexural strength, elastic modulus, and bond strength, the factor under study was resin composite (TBF vs ED). For the evaluation of maximum shrinkage stress, resin composite (TBF vs ED) and increment (one 4-mm increment of TBF, first and second 2-mm increments of ED, and sum of first and second ED increments) were the factors under study. The compositions of the materials are listed in Table 1.

Degree of Conversion, Flexural Strength, and Elastic Modulus Tests

For these tests, bars (7×2×1 mm) of the two resin materials (n=10 each) were prepared using a polyvinyl siloxane mold (Express XT; 3M ESPE, St Paul, MN, USA).[14] Both resin materials were inserted in single increments. A polyester strip and a 1-mm glass plate covered each specimen, and final photoactivation was performed for 20 seconds using a polywave light-emitting diode (LED) light-curing unit (Bluephase G2; Ivoclar Vivadent) at 1150 mW/cm^2 (measured by using a computer-controlled

Table 1: *Materials Used in this Study*[a]

Material	Manufacturer	Chemical Composition (wt%)	Lot No.
Tetric Evo Flow Bulk Fill (TBF)	Ivoclar Vivadent	Ethoxylated bisphenol A dimethacrylate (10-25%), Bis-GMA (3-7%), ytterbium trifluoride (3-5%), tricyclodecane dimethanol dimethacrylate (1-3%)	TM0056
Empress Direct (ED)	Ivoclar Vivadent	urethane dimethacrylate (10-25%), Bis-GMA (2,5-3%), ytterbium trifluoride (3-10%), tricyclodecane dimethanol dimethacrylate (3-10%)	T28435
Stae	SDI	Acetone (50-55%), acrylic monomers (20-40%)	141141

Abbreviation: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate.
[a] *Data obtained from Material Safety Data Sheet (MSDS).*

spectrometer - USB2000, Ocean Optics, Dunedin, FL, USA). The samples were stored in an oven at 37°C for 24 hours before testing.

First, DC was measured using Fourier-transform infrared spectroscopy (IRTracer-100; Shimadzu, Kyoto, Japan) coupled to a total attenuated reflectance device. The absorption spectra of nonpolymerized and polymerized materials were determined between 1800 and 1500 cm^{-1}, with 32 scans taken at 4-cm^{-1} intervals. As both composites contain aromatic vinyl bonds of bisphenol and aliphatic bonds of the methacrylate functional group, the aliphatic carbon-to-carbon double-bond absorbance peak intensity (located at 1638 cm^{-1}) and that of the aromatic component (located at 1608 cm^{-1}; reference peak) were measured. The DC was calculated using the following equation: $DC\ (\%) = 100 \times [1-(R_{polymerized}/R_{nonpolymerized})]$, where R represents the ratio between the absorbance peaks at 1638 cm^{-1} and 1608 cm^{-1}.

Flexural strength (in MPa) and elastic modulus (in GPa) were then evaluated using a universal testing machine (model 4111; Instron Corporation, Dayton, OH, USA) with a three-point bending design, a crosshead speed of 0.5 mm/min, and a 50 N load cell until fracture. The distance between supports was 5 mm. Bluehill 2 software (Illinois Tool Works Inc, Glenview, IL, USA) was used to calculate flexural strength and elastic modulus, considering the dimensions of the specimens.

Depth of Polymerization and Push-out Bond Strength Tests

Figure 1 is a schematic representation of the specimen preparation and methods used to evaluate the depth of polymerization and push-out bond strength. Twenty bovine incisors with no enamel cracks or structural defects were selected and decontaminated in an aqueous solution of thymol (0.1%) at 4°C for one week. The roots were removed at the cementoenamel junction (CEJ) with a water-cooled diamond saw using a precise cutting machine (Isomet 1000; Buehler, Lake Forest, IL, USA). A transverse mesiodistal cut was made 4 mm from the CEJ to obtain a 4-mm-thick slice with a central void corresponding to the pulp cavity. Standardized conical cavities (4.8-mm top diameter×2.8-mm bottom diameter×4-mm height) were prepared with Maxicut burs (Komet Inc, Lemgo, Germany) using a hand piece under air-water cooling. The bur was replaced every 10 preparations. The cavities had Configuration-factors of 2.5.

After cavity preparation, bonding procedures were performed. Dentin was acid-etched for 15 seconds with phosphoric acid (Super Etch; SDI, Victoria, Australia) and rinsed thoroughly with water for 15 seconds. Excess water was blotted with absorbent paper, leaving the dentin surface visibly moist (wet bonding). The Stae adhesive system (SDI) was applied; photoactivation was performed for 10 seconds with an LED unit (Bluephase G2; Ivoclar Vivadent). Each specimen was placed over a glass slide, and the composite was inserted into the cavity. TBF was inserted in one increment, completely filling the cavity; a polyester strip was placed on the top surface and photoactivated for 20 seconds. ED was inserted incrementally; the first 2-mm increment was inserted and photoactivated for 20 seconds, then the second 2-mm increment was inserted and photoactivated for 20 seconds under a polyester strip. After 24 hours, the restorations were finished/polished with Sof-Lex Pop On aluminum oxide discs (3M ESPE) and stored in water for 24 hours at 37°C before evaluation of the depth of polymerization and bond strength.

Depth of polymerization was analyzed using the bottom-to-top hardness ratio.[15] Knoop indentations were made on the top and bottom surfaces of each restoration with an indenter (HMV-2; Shimadzu), using a 50-*g* load for 30 seconds. Three readings were taken on each surface, and the mean Knoop hardness numbers and the bottom-to-top hardness ratio were calculated.

Then, push-out bond strength was evaluated. An acrylic device with a central hole was adapted to the

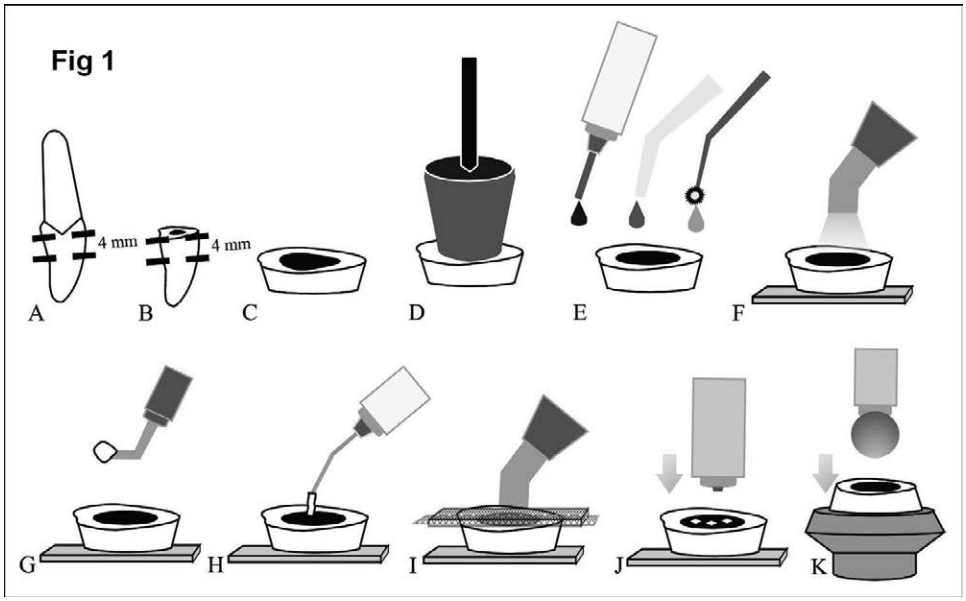

Figure 1. *Schematic representation of the polymerization depth and push-out bond strength analyses. Crowns of bovine incisors were removed from the roots at the cementoenamel junction (CEJ) and then cut 4 mm from the CEJ (A,B) to create three-dimensional 4-mm dentin cavities (C). Cavities were prepared using a Maxicut bur (D). Cavities were acid-etched and washed with water, and adhesive systems were applied to moistened dentin (E), which was photoactivated (F). Two increments of conventional composite (G) and a single increment of flowable bulk fill composite (H) were applied and photoactivated (I) under a Mylar strip and glass plate. Knoop hardness was measured on the top and bottom surfaces (J), and push-out bond strength was evaluated (K).*

base of a universal testing machine (model 4411; Instron, Buckinghamshire, UK). The central hole was used to position each specimen with the preparation bottom (the end with the smallest diameter) facing up. A rounded probe was adapted to the testing machine and a compressive force was applied with a crosshead speed of 0.5 mm/min to the bottom surface of the restoration to induce rupture of the tooth composite bonding area. The results (in Kgf) were divided by the bonded area (22.65 mm^2) and transformed to MPa. After the test, failure mode was examined using a dissecting microscope (Stereozoom; Bausch & Lomb, Rochester, NY, USA), using the following classification: adhesive (type 1), adhesive/cohesive in composite (mixed 1; type 2), adhesive/cohesive in composite and dentin (mixed 2; type 3), cohesive in composite (type 4), and cohesive in dentin (type 5).

Shrinkage Stress

Shrinkage stress was characterized as described in a previous study.[16] Briefly, the flat surfaces of poly(-methyl methacrylate) rods (6-mm diameter, 13- and 28-mm lengths) were sandblasted to produce a layer of methyl methacrylate, followed by two coats of a bonding agent (ScotchBond Multi-purpose Plus Adhesive; 3M ESPE). The unfilled resins were light-cured with an LED unit (Bluephase G2; Ivoclar Vivadent) for 10 seconds. The rods were attached to the opposing clamps of a universal testing machine (model 3342; Instron) with the treated surfaces facing each other at distances of 4 mm for TBF and 2 mm for ED. The composite was inserted into the empty space and shaped into a cylinder according to the rod perimeters. An extensometer (model 2630-101, 0.1-mm resolution; Instron) was attached to the rods to monitor specimen height and to provide feedback to the testing machine to keep the height constant. The load cell provided the corresponding force necessary to counteract the polymerization shrinkage force to maintain the specimen's initial height. The short rod was attached to the testing machine through a hollow stainless-steel fixture with a lateral slot that allowed the tip of the LED guide to be positioned in contact with the polished end surface of the rod. Force development was monitored for 20 minutes from the beginning of photoactivation, and nominal stress was calculated by dividing the maximum force value by the cross-sectional area of the rod. For TBF, stress was calculated only for 4-mm bulk increments. For ED, stress was calculated for 2-mm thicknesses placed directly between the rods (first increment), 2-mm thicknesses placed between the rod and a prepolymerized 2-mm increment (second increment), and for the sum of the first and second increments (n=5). The maximum shrinkage stress of photoactivation (expressed in MPa) was obtained from stress vs time curves.

Statistical Analysis

The Student *t*-test was used to analyze the DC, flexural strength, elastic modulus, depth of cure, and push-out bond strength (α=0.05). Maximum shrinkage stress was evaluated using one-way analysis of variance (ANOVA) with Tukey tests ($p<0.05$). IBM

Table 2: *Means ± Standard Deviations of Degree of Conversion (DC; %), Flexural Strength (FS; MPa), Elastic Modulus (EM; GPa), Bond Strength (BS; MPa), and Hardness Bottom-to-top Ratio (HB/T) According to the Composite Resins Analyzed*[a]

	DC	FS	EM	BS	HB/T
TBF	85.7 ± 6.6 A	76.6 ± 16.8 B	11.5 ± 2.8 A	0.94 ± 0.70 A	0.83 ± 0.16 A
ED	54.2 ± 4.9 B	144.9 ± 24.1 A	12.5 ± 2.6 A	0.35 ± 0.15 B	0.84 ± 0.08 A
p-value	0.001	0.001	0.415	0.026	0.944

Abbreviations: ED, Empress Direct; TBF, Tetric Evo Flow Bulk Fill.
[a] *Different letters indicate statistically significant differences among the resins according to T test, considering each evaluated property (each column).*

SPSS Statistics for Windows software (version 20.0; IBM Corporation, Armonk, NY, USA) was used to perform statistical analyses.

RESULTS

The DC, flexural strength, elastic modulus, depth of polymerization, and bond strength results are presented in Table 2. The DC and bond strength were significantly greater for TBF than for ED. Flexural strength was significantly greater for ED (144.9±24.1 MPa) than for TBF (76.61±16.8 MPa). TBF and ED had similar elastic moduli (11.5±2.8 GPa and 12.5±2.6 GPa, respectively) and depths of cure/bottom-to-top hardness ratio (0.83±0.16 and 0.84±0.08, respectively). All samples showed adhesive (type 1) failures between the adhesive and dentin.

The shrinkage stress results are presented in Table 3. ANOVA showed significant differences in maximum shrinkage stress according to material and increment (all p=0.001). The highest maximum shrinkage stress mean was obtained for the sum of the first and second increments of ED, while the lowest maximum shrinkage stress mean was obtained for the second increment of ED.

DISCUSSION

The null hypothesis that there was no significant difference between materials was rejected, since the resin composites showed only similar elastic moduli and depths of polymerization. The conventional material showed greater flexural strength than did the bulk fill, and the latter showed less shrinkage stress and greater bond strength and DC than did the former.

Intrinsic characteristics of the chemical components of resin composites, such as the amount of filler particles, type and concentration of the photoinitiator system, type and amount of monomers, color, and translucency, are related directly to the DC.[10,17] The bulk fill material tested has a comparable amount of filler particles to the conventional one. However, the former contains a germanium-based photoinitiator, Ivocerin, which can absorb more light in the range of 400-450 nm. Photoactivation of Ivocerin forms at least two radicals that initiate radical polymerization; in contrast, only one radical is formed in camphorquinone/amine systems, present in the conventional material, resulting in less efficient initiation of polymerization.[18] In addition, the bulk fill material has more low-viscosity monomers and is more translucent than is the conventional one. Low-viscosity monomers increase the DC of resin-based materials as a result of their greater mobility during polymerization;[19] indeed, translucent materials favor higher light transmittance and photoinitiator excitation.[20] These properties explain the higher DC observed for the bulk fill in comparison with conventional material.

A uniform DC along the composite thickness can guarantee a satisfactory depth of polymerization. In this study, the top-to-bottom hardness ratio was used to determine the polymerization depth of resin composites.[15] Bulk fill and conventional materials had similar bottom-to-top hardness ratios, despite differences in incremental application and photoactivation. The presence of a more reactive photoinitiator system, more low-viscosity monomers, and increased translucency in the bulk fill material explain this similarity in polymerization depth, despite the thickness of its increment. The high

Table 3: *Means ± Standard Deviations of Maximum Shrinkage Stress (MPa) in Accordance with the Type of Increment/Resin Composite Analyzed*[a]

Type of Increment/Resin Composite	Maximum
4-mm increment of TBF	0.77 ± 0.07 B
First 2-mm increment of ED	0.69 ± 0.03 B
Second 2-mm increment of ED	0.37 ± 0.13 C
Sum of first and second increments of ED	1.07 ± 0.15 A
p-value	0.001

Abbreviations: ED, Empress Direct; TBF, Tetric Evo Flow Bulk Fill.
[a] *Different letters indicate statistically significant differences among the resins.*

radiance emitted by the curing light used in this study likely favored satisfactory excitation of photoinitiators from the tops to the bottoms of the specimens, resulting in bottom-to-top hardness ratios > 0.8, considered to be satisfactory.[21] As cavity filling with a single 4-mm composite resin increment, instead of two 2-mm increments, reduces chair time, the use of a low-viscosity bulk fill composite can simplify restorative procedures under clinical conditions.

The DC, presence of crosslinking high-molecular-weight monomers, and percentage, size, and shape of filler particles are related to the elastic moduli of resin composites.[22] Increased amounts of filler particles and crosslinking high-molecular-weight monomers decrease the DC, as they promote light scattering and decreased mobility during polymerization, respectively, although they favor an increased elastic modulus of resin composites.[1] The bulk fill material presented a higher DC, whereas the conventional one contains more crosslinking high-molecular-weight monomers and filler particles, explaining the similar elastic moduli observed for these two materials in this investigation. On the other hand, the conventional material had greater flexural strength than did the bulk fill. However, the latter is a flowable material that requires a covering layer of high-viscosity resin composite in tooth restorations;[22,23] in this way, when the manufacturer's recommendations are followed, it is possible that the reduced flexural strength of the bulk fill material should not be a problem since the covering material would have increased flexural strength. However, further analysis should be performed to confirm this assumption.

Recent research has focused on the influence of resin matrix chemistry on the mechanical properties of resin composites. Factors such as the type and amount of filler particles, transference of stress between the filler particles and resin matrix, adhesion between these components, and the chemical composition of the matrix have been associated with differences in flexural strength.[22-25] The greater flexural strength of the conventional material in this study can be explained by the high percentage of inorganic fillers in this composite.[26]

Shrinkage stress occurs as a result of the development of covalent bonds between the polymer chains already formed, causing contraction, and is defined by a combination of factors such as matrix composition, amount of charge, photoactivation standard, DC, elastic modulus, and shape of the cavity into which the composite is inserted.[27] In this study, shrinkage stress of the conventional material was measured in two separate steps to better simulate the incremental insertion protocols of conventional resin composites. Shrinkage stress was significantly higher for the conventional than for the bulk fill material, meaning that the latter was better able to dissipate the generated tension. This can be appointed as significant advantage of the low-viscosity bulk fill resin composite, because even with application in a single 4-mm increment, this material showed less shrinkage stress than did a traditional paste-like composite.

The flow of stresses may be associated with the pre-gelation phase time, and this can be attributed to the modified polymer chains of bulk fill flowables, which are very flexible in the pre-gelation phase. This highly stress-relieving internal monomer may delay the gel point, which could allow more time to compensate for shrinkage; consequently, shrinkage stress would be reduced. This may have been a factor that caused a better dissipation of tensions by the bulk fill material.[28,29]

Another strategy that may be related to decreased shrinkage is the replacement of high-molecular-weight with lower-weight monomers.[28] The bulk fill material contains urethane dimethacrylate (UDMA), and the conventional one contains ethoxylated bisphenol A dimethacrylate (Bis-EMA); although both are considered to be high-weight monomers, Bis-EMA has a lower weight (362.41 g/mol) than does UDMA (470.56 g/mol); this difference may explain the lower tension generated by this material.

In general, shrinkage stress was greater for the first increment of the conventional composite than for the second increment. However, shrinkage stress may have been reduced in the second increment because it had to be placed over a previously applied 2-mm increment because of methodological aspects, which can absorb stress.

In push-out bond strength tests, stress generated by composite polymerization is transferred directly to the adhesive interface, as the composite shrinks into the cavity. Although the bond strength of bulk fill composites can also be analyzed using microtensile tests after the filling of Class I and Class II cavities,[30,31] specimen preparation using diamond saws can apply external stress to the tooth/composite interface, leading to underestimation of bond strength values. In contrast, the push-out method allows the measurement of bond strength without this external stress, so that differences between materials are related to their intrinsic characteris-

tics. Thus, the lower maximum shrinkage stress of the bulk fill material in comparison with the conventional one explains the greater bond strength of the former.

Further studies involving physical, mechanical, and adhesion analyses of other low-viscosity and paste-like bulk fill composites in high–C-factor dentin cavities should be performed in an attempt to obtain information regarding materials from other manufacturers. Moreover, dentin bond strength analysis after aging methods should also be investigated further to evaluate dentin bond durability of low-viscosity bulk fill and conventional resin composites in high–C-factor dentin cavities.

CONCLUSIONS

The physical and mechanical properties of Tetric Evo Flow Bulk Fill, a low-viscosity bulk fill resin composite, were similar or superior to those of Empress Direct, a conventional resin composite, with the exception of flexural strength.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Rio Grande do Norte (UFRN).

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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