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Guest Editorial

COVID-19 Pandemic Is Here, Airplanes Crash, and the Profession Has an Educational Dilemma

J. Martin Anderson, DDS, FACD, FICD

It has been said that "one should beware when the camel's nose is beneath the tent." The camel's nose is a metaphor, of course, for a situation where the permitting of a small, seemingly innocuous act will open the door for larger, clearly undesirable actions.

Recently (January 2018), the American Dental Association (ADA) took action to support its policy calling for the elimination of patients from the dental licensure examination process. The ADA requested that the Council on Dental Education and Licensure direct the development of a Dental Licensure Objective Structured Clinical Examination (DLOSCE).

The ADA DLOSCE would be a written exam (much like the OSCE-administered exam in Canada) that excludes the assessment of clinical skills completed on a live patient. Written tests are an effective way to find out what level of knowledge has been acquired. No part of the OSCE assesses the applicant's clinical skills or patient management.

The Western Regional Examining Board (WREB) dental examination is a highly developed and respected clinical examination that is already accepted by about 44 states for licensure. Most dental schools also test students for clinical competency with patients at some point. The issue, however, is whether schools' testing and determination regarding candidate readiness is sufficient to eliminate a third-party, high-fidelity, patient-based assessment. There is a small but consistent number of candidates who, despite graduating from an accredited program, are still unable to demonstrate acceptable competence when working independently. These students are unable to perform a simple restoration on a patient even after multiple attempts. Dental schools are either (a) knowingly graduating a small number of non-ready candidates, relying on an independent, third-party clinical exam to "apprehend" them, or (b) unknowingly allowing this small number of candidates to get through via an insufficient assessment of minimal competency... which is more disturbing. It is especially disturbing this year, as the COVID-19 pandemic has caused most dental schools (just weeks before graduation) to close classroom lectures and provide only limited care to patients. Clinical competency examinations may be severely compromised or eliminated entirely. These issues make existing clinical board exams very important in keeping a small number of candidates (about 3%) from practicing until they are able to demonstrate minimal clinical competence on a patient.

The WREB operative dentistry clinical examination includes the following topics: local anesthesia and pain management, moisture control and soft tissue management, communication and patient management, disease management and removal, instrumentation with material handling skills, and anatomic/functional skills. If dental schools are not testing students with live patient clinical examinations, then who can be trusted to protect the public? One might even ask: What is the point of having examinations at all, especially if the number of failures is so small? Consider the following: most people trying to board airplanes are not terrorists, but the millions of people trying to board go through elaborate security procedures every day. Are these procedures 100% foolproof and perfect? No, but

scrapping them for no screening at all would reduce public safety. And if the COVID-19 pandemic means dental students are unable to complete their clinical requirements or their clinical competency examinations, how will dental schools certify student readiness to practice dentistry? The educational dilemma is worsened because outside testing agencies (like the WREB) will likewise have similar difficulties giving clinical examinations if the COVID-19 pandemic persists.

In education, most will agree that "trust is the coin of the realm." The public would like to trust our educators, and the following questions might help with understanding the educational dilemma:

1. Would the public trust teenagers and others to be given a driver's license without having passed a drivers' test in a vehicle?
2. Would the public trust pilots to fly airplanes without having passed an Federal Aviation Administration flight exam or trust airplane manufacturers to produce safe airplanes?

The ADA is an immensely powerful and influential lobby. The deans of dental schools have a vested interest in getting their students a dental license to practice and will undoubtedly also have great influence. Without a doubt, both will argue vigorously for the development and implementation of OSCEs. That said, the public should be wary. Poorly tested pilots, poorly tested airplanes, and dentists who are inadequately tested on live patients can be hugely dangerous to the public.

These are strange and challenging times for dental educators.

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Clinical Technique

Molar Incisor Hypomineralization: Etiology, Clinical Aspects, and a Restorative Treatment Case Report

D Sundfeld • LMS da Silva • OJ Kluppel • GC Santin
RCG de Oliveira • RR Pacheco • NIP Pini

Clinical Relevance

Total-etch adhesive systems and resin composite are clinically viable dental materials for esthetic restorations in teeth presenting white/yellow/brown hypomineralization stains.

SUMMARY

Molar-incisor hypomineralization (MIH) is a condition that negatively affects enamel and dentin, especially the first molars and permanent incisors, causing esthetic and functional problems. The present clinical case report presents and discusses the etiology and clinical characteristics of MIH and describes a restorative protocol for MIH-affected teeth.

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INTRODUCTION

Among the pathologies that may negatively affect the smile and dental structures, molar incisor hypomineralization (MIH) is defined as a systemic hypomineralization of qualitative character that directly affects the enamel and dentin of the first molars, with or without the involvement of the incisors.[1] Less frequently, MIH-like defects have been reported in permanent canines, premolars, and primary second molars.[2-4] As result of an altered (or disturbed) matrix production, secretion, arrangement, crystal formation, or matrix resorption, a compromised enamel structure may be observed.[5] Defects during calcification/maturation stages usually lead to normal volumes of enamel with insufficient mineralization (hypomineralization) and, consequently, altered translucency. MIH, amelogenesis imperfecta, and dental fluorosis are examples of such qualitative alterations.[4,6,7] On the other hand,

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quantitative defects (hypoplasia) are caused by a disturbance during the amelogenesis matrix secretion phase.[8,9]

MIH-affected teeth usually present the enamel with opaque-white, yellow, or brown colorations, with or without posteruptive degradation (PED), varying according to its severity.[1,5,10-12] The mild form of MIH is associated with delimited opaque areas, varying from white to brown, in nonstress areas on first permanent molars, with no structural loss and no dental sensitivity.[10,13-15] Moderate (with slight or no dental sensitivity)[15] and severe forms of MIH are associated with opaque areas at the occlusal/incisal third of teeth. This area occasionally undergoes PED due to reduced hardness and high porosity of enamel, leading to functional and esthetic complications and dental sensitivity.[15-17] PED commonly leads to surfaces that are more susceptible to biofilm accumulation and development of carious lesions.[18] In severe cases, PED may lead to dental sensitivity due to dentin exposure.[1,19] MIH usually presents asymmetrically, affecting two-thirds of the crowns of molars and incisors. One group of teeth may be more affected than the other.[20]

There are multiple treatment options for MIH-affected teeth, which include preventive, desensitizing, and remineralizing products; calcium and vitamin supplements; resin infiltration; fissure sealants; enamel microabrasion; direct or indirect restorations; extractions; and orthodontic alignment.[4,19] The choice and indication of treatment depend on the severity, patient age, socioeconomic factors, and treatment expectations.[21,22] Restorative procedures are great treatment options for teeth with structure loss, requiring restoration of function and esthetics. Some materials and procedures may be considered, including resin composite direct restorations, ceramic indirect restorations, and prefabricated metallic crowns (posterior teeth).[23] However, adhesive procedures on MIH-affected teeth are critical and may affect the bond strength and longevity of these restorations.[16,24-26] More invasive procedures, such as extractions, are adopted for teeth with major structural impairment, aiming for a future orthodontic/prosthetic rehabilitation after dental surgery.[22]

Therefore, the aim of the present clinical case report is to demonstrate an esthetic restorative procedure performed on MIH-affected teeth. The 18-month clinical follow-up and articles concerning MIH are presented and discussed.

CLINICAL CASE REPORT

A 17-year-old female patient presented to the Restorative Dentistry Clinic of Ingá University Center–UNINGÁ (Maringá, PR, Brazil) with a chief complaint of poor esthetics. The patient presented with stains on the maxillary and mandibular anterior teeth (Figures 1 and 2). During anamnesis, the patient's guardian reported that she was born with low birth weight and was hospitalized due to a severe condition of anemia during childhood (when she was about 1 year old) and had antianemic injections, recommended in cases of iron deficiency. The use of amoxicillin was also frequent because of respiratory tract infections (chronic bronchitis) that affected her during infancy (18 months old until 8 years old), and associated with the use of nebulizers to help the pulmonary air ventilation as well.

During dental examination, white, yellow, and brown stains were observed on the buccal surfaces of the maxillary canines, laterals, and central incisors (Figures 1 and 2). On the mandibular teeth, white/"creamy" stains were found on the facial surface of the left lateral incisor and in both central incisors (Figures 1 and 2A). Milder enamel stains were observed on both mandibular canines and on the right lateral incisor. Residual bonding materials were found after bracket debonding on the right maxillary lateral incisor and canine and on the maxillary left lateral incisor. The guardian also reported that the patient's first mandibular permanent molars presented severe structure loss and were submitted to surgical extractions when the patient was 10 years old. The depths of each stain were assessed by positioning the tip of a light-emitted diode (LED) light-curing unit from the palatal/lingual surfaces (Figure 3). Considering both clinical and medical history, MIH was diagnosed. The clinicians proposed the following treatment plan, which was accepted by the patient and guardian: 1) at-home vital dental bleaching; 2) enamel microabrasion on teeth #6, #7, and #10; and 3) direct composite resin restorations on teeth #8, #9, and #11.

At-home dental bleaching was performed by using 10% carbamide peroxide (Opalescence PF, Ultradent Inc, South Jordan, UT, USA) for 2 hours per day, for 20 days. Alginate impressions of the maxillary and mandibular arches were made, and stone models were poured and used to fabricate the custom acetate bleaching trays. The patient was instructed to place a small drop of bleaching product into each tooth section and to contact the clinician if any discomfort or sensitivity occurred. The bleaching treatment

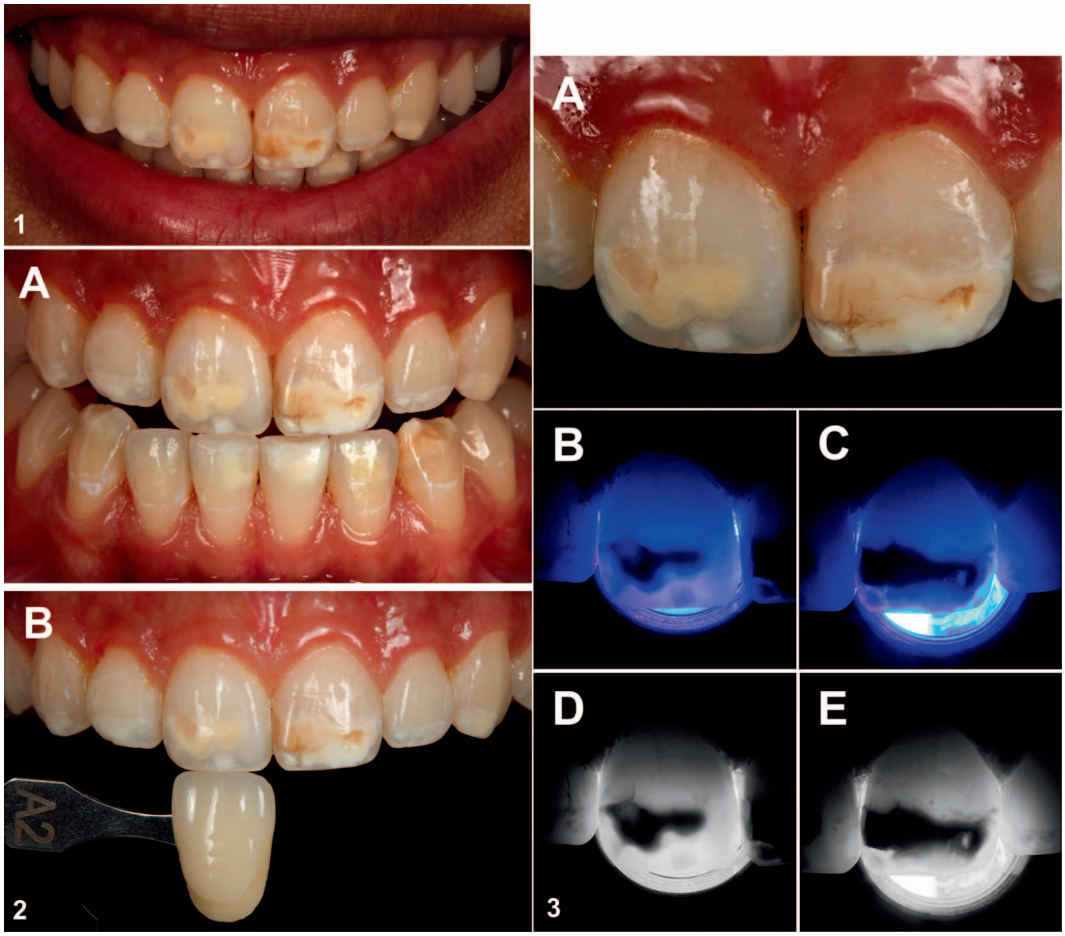

Figure 1. *Initial clinical aspect showing white/yellow/brown stains in maxillary and mandibular anterior teeth.*

Figure 2. *(A): Intraoral view during mandibular protrusion (note the white/yellow/brown stains in maxillary and mandibular anterior teeth). (B) Initial teeth shade evaluation (color A2 using Ivoclar shade guide).*

Figure 3. *(A): Close-up view of stained central incisors. Transillumination assessment positioning the tip of the light-curing unit at the palatal surface on the right (B and D) and left (C and E) maxillary central incisors. Dark and intense color indicates deep stains.*

resulted in a color alteration from A2 to B1 (Ivoclar Shade Guide, Vita Shade System) using a visual assessment method (Figure 4).

After 1 month, the restorative procedures with resin composite were performed. The chromatic mapping was made by placing small resin composite increments (Vit-l-escence, Ultradent Inc) on the buccal surface of the maxillary incisors (Figure 5). After rubber dam isolation, the hypomineralized stains were removed using a spherical diamond bur (#1014, KG Sorensen, Cotia, SP, Brazil) coupled to a high-speed handpiece with water irrigation (Figure 6). After complete removal, the enamel was etched for 30 seconds and dentin for 15 seconds using 35% phosphoric acid (Ultra-Etch, Ultradent Inc; Figure 7). Then, the primer (step 2) of a total-etch three-step adhesive system (Adper Scotchbond Multi-Purpose, 3M ESPE, St Paul, MN, USA) was applied only to dentin. Solvent from the primer was allowed to evaporate using air from the syringe for 15 seconds, followed by the unfilled adhesive resin application (adhesive, step 3; Figure 8). The adhesive was light activated for 10 seconds using a polywave LED light-curing unit (Valo Cordless, Ultradent Inc) in the regular mode (1,000 mW/cm^2). Resin composite restoration (Vit-l-escence) was made using an incremental technique, respecting the original dental anatomy (Figure 9).[27]

The enamel microabrasion was performed for residual orthodontic bonding materials, intrinsic stain removal, and regularization of the enamel surface (Figure 10).[28,29] The surface layers of the stained enamels located on the buccal surface of both maxillary lateral incisors and right maxillary canine

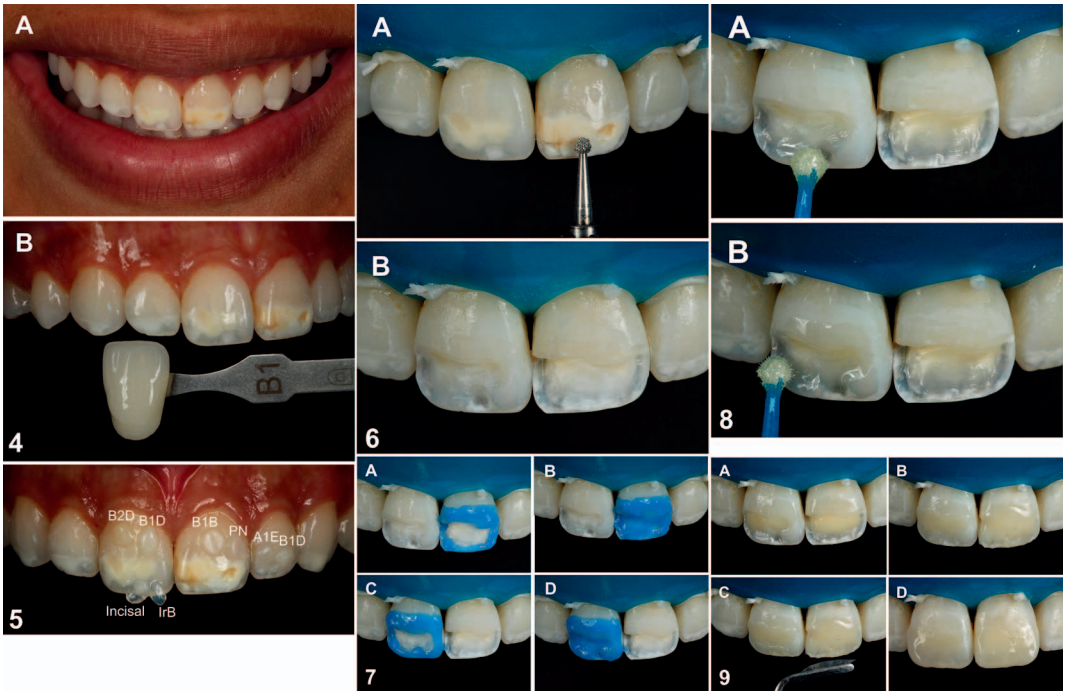

Figure 4. *(A): Clinical aspect after at-home dental bleaching. (B) Teeth shade selection using a visual color scale (color B1 using Ivoclar shade guide).*

Figure 5. *Resin composite (Vit-l-escence, Ultradent Inc) color mapping prior to direct restorations: [B2D] Dentin, B2; [B1D] Dentin, B1; [PN] Enamel, Pearl Neutral; [IrB] Incisal, Iridescent Blue; [A1E] Enamel, A1; [B1D] Dentin, B1; [Incisal] Incisal shade (Forma, Ultradent Inc.).*

Figure 6. *Complete removal of hypomineralized stains using a spherical diamond bur (#1014, KG Sorensen) on the buccal surface of both maxillary central incisors.*

Figure 7. *(A, C): Acid etching with 35% phosphoric acid for 30 seconds on enamel. (B, D): Acid etching with 35% phosphoric acid for 15 seconds on dentin.*

Figure 8. *(A): Application of primer (step 2) on dentin only. (B): Application of the unfilled adhesive (step 3) on the entire preparation. Same protocol was adopted for the left maxillary central incisor.*

Figure 9. *(A): Increment of B2 Dentin resin composite. (B): Increment of B1 Dentin resin composite. (C): Increment of Iridescent Blue at the incisal third. (D): Increment of Pearl Neutral Enamel shade as the last increment.*

were removed using a super-fine (macroabrasion) tapered bur (#3195FF, KG Sorensen) under copious water irrigation attached to a high-speed handpiece (Figure 10A,C). A microabrasive product (Opalustre, Ultradent Inc) was used to remove the remaining stains, using a specially designed rubber cup (Opal-Cups, Ultradent Inc) with a low-speed handpiece (Figure 10B,D). After two weeks, it was observed that the incisal stains were not completely removed on teeth #6 and #7 since they were deeper than the enamel microabrasion was able to remove. Thus, direct resin composite restorations were made on both maxillary lateral incisor and right maxillary canine (Figure 11) following the technique described above for both maxillary central incisors.

Finishing and polishing procedures were performed using an aluminum oxide disc (Sof-Lex, 3M ESPE), followed by abrasive rubber discs (Jiffy, Ultradent Inc; Figure 12). Later, a silicon-carbide–impregnated brush (Jiffy Brush, Ultradent Inc) was used, followed by the application of a diamond paste (Diamond Polish, Ultradent Inc) using a goat-hair brush. The patient was satisfied with the treatment (Figure 13) and did not want to remove the stains located in the mandibular teeth. Figure 14 represents the 18-month follow-up of the procedures.

DISCUSSION

The present clinical case report describes the esthetic restorative treatment of MIH-affected teeth using a combination of dental bleaching, direct resin composite restorations, and enamel microabrasion. The last procedure was performed to remove resid-

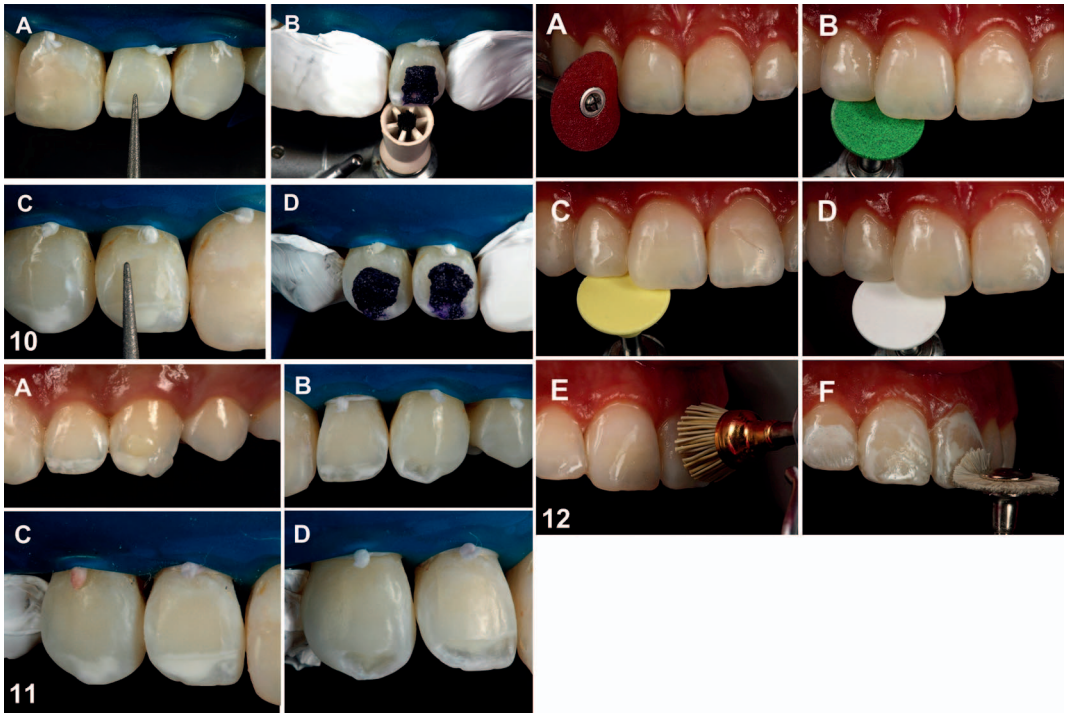

Figure 10. *(A, C): Macroabrasion using a super-fine tapered diamond bur (#3195, KG Sorensen) on the buccal surface of both maxillary lateral incisors and right maxillary canine. (B): Application of the microabrasive product (Opalustre, Ultradent Inc) using the specific rubber cup (Opalcup, Ultradent Inc).*

Figure 11. *(A): Resin composite shade mapping on the left maxillary canine before direct resin composite restoration ([B2D] Dentin, B2 and Pearl Neutral Enamel shade). (B): Complete removal of the stained opacity at the incisal third. (C): Stained buccal surface at the incisal surface. (D): Removed stains prior to direct resin composite restoration. Although the stain is evident in the left lateral incisor during rubber damn isolation, it was not necessary to perform a direct restoration since the stain disappears when hydrated in saliva.*

Figure 12. *(A): Finishing using aluminum oxide disc. (B-D): Abrasive rubber discs with progressive reduction of abrasive size. (E): Impregnated silicon carbide brush. (F): Use of a diamond paste associated with a goat-hair brush.*

ual orthodontic bracket bonding materials and white remineralized stains around them and, consequently, smoothening the enamel surface after bracket debonding.[29] The patient was satisfied with the clinical esthetic outcomes from the described techniques.

Disturbance (ie, illness) during the late enamel maturation phase of amelogenesis may negatively affect the function of ameloblasts (which are very sensitive to changes in their surrounding environment),[30] leading to the qualitative defects found in MIH.[31] The MIH diagnosis was based on medical and dental history. The best age for a correct diagnosis of MIH is about eight years, as the maxillary and mandibular permanent incisors and first molars are fully erupted.[32] The patient's guardian reported that the patient presented with severe anemia during early childhood. This information is of great value, since the etiology of MIH may be related to some systemic factors and changes/problems during the prenatal (ie, hypocalcemia and/or diabetes), perinatal (ie, premature birth or prolonged delivery), and/or postnatal periods (ie, antibiotic use and/or nutrition problems).[33] The severe anemia reported was framed as one of the possible causes of MIH.[34] The etiology of MIH is not fully elucidated,[5,33,35] and it is difficult to obtain scientific evidence on the origins/causes of MIH development because of the lack of standardized measurement protocols/guidelines. Therefore, well-designed future cohort studies are required.[36-38]

Scientific reports[37,39-41] found that premature birth and low birth weight are associated with MIH. Moreover, there is considerable evidence of an association between early childhood illness (fever, asthma, and pneumonia, up to three or four years of age) and MIH.[38,42] A positive correlation was reported between respiratory disease and a severe variant of MIH with incisor involvement,[43] including bronchitis,[39,44] and ear, nose, and throat diseases.[11] A genetic

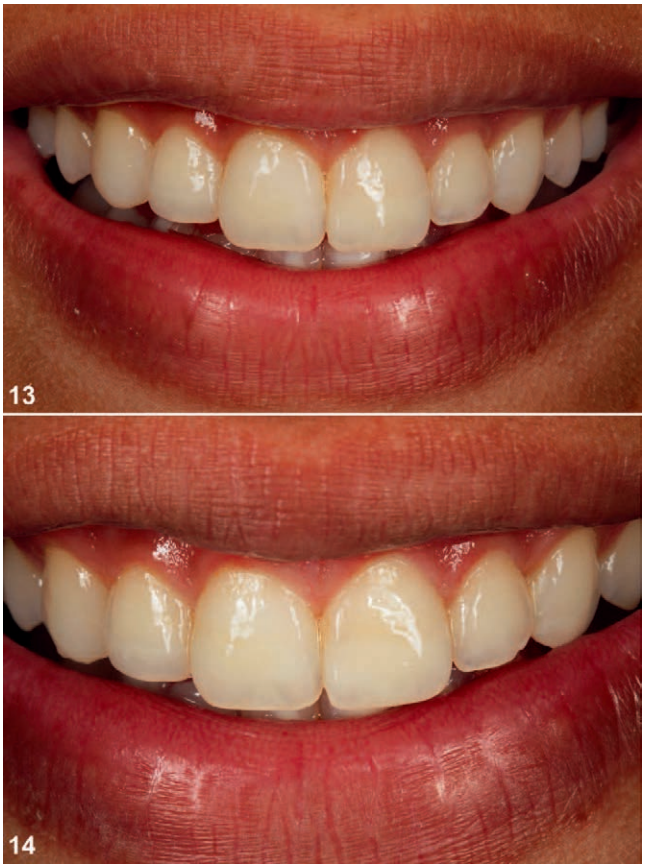

Figure 13. *Clinical aspect after 1 week.*

Figure 14. *Clinical aspect after 18 months (resin composite restorations were repolished).*

component from multifactorial pathogenesis was also hypothesized.[37,42,45] A systematic review[42] reported several studies indicating that chickenpox, renal disease, measles, gastrointestinal disease, tonsillitis, otitis, and adenoiditis might be related to the etiology of MIH. Medication intake (eg, antibiotics such as amoxicillin or penicillin) is strongly associated with MIH etiology and development.[11,46-48] Corticosteroids and bronchodilators (asthma drugs) were related to enamel defects as well.[49] It is noteworthy to state that the mineralization of the first permanent molars usually starts at birth and is fully completed at four to five years of age.[50] The early use of those drugs may have a negative influence on the amelogenesis process. Many of those factors may have influenced or increased the odds of MIH development for the patient presented in this case report, including low birth weight, bronchitis, anemia, and antibiotic intake.

The human dental enamel, in its normal state, has a mineral content of 95%, which leads to a high hardness that can withstand occlusal forces and chewing. The other 5% consists of water (4%) and traces of organic content.[51,52] Generally, the MIH-affected enamel presents a reduction in the quantity and quality of mineral content compared with normal enamel. Thus, MIH-affected enamel has a reduced content of calcium and phosphate, and consequently, there is a reduction in the hardness and elasticity, increased porosity,[31,53-56] increased carbon and carbonate concentrations, and greater protein content,[31,45,54,55,57] which hamper the growth of hydroxyapatite crystals.[58,59] Also, the MIH-affected enamel is not organized into hydroxyapatite crystals, usually presenting loosely packed crystals, less-dense prismatic structure, partial loss of the prismatic pattern, less distinct prism borders, and more evident interprismatic space.[53,55,57,60,61] Studies[54-57] have demonstrated that MIH lesions start at the enamel-dentin junction and end at the enamel surface; thus, MIH lesions are located throughout the whole enamel thickness. All of this information attests to the fact that hypomineralized enamel is more fragile than sound enamel.

The degree of lesion opacities is directly related to the degree of porosity: creamy/white lesions and those without posteruptive breakdown are less porous when compared with yellow/brown–colored enamel.[55,56,62] Thus, yellow/brown opacities are more prone to evolve into PED when compared with white/creamy opacities.[12] Dentin may also be negatively affected by MIH, presenting lower mineral density when compared with nonaffected dentin, at the cervical region.[56] Heijs and colleagues[63] reported that MIH-affected dentin under MIH-affected enamel presents few morphological alterations, of which the most significant is the increased interglobular dentin compared with a normal dentin. However, no structural differences were verified when compared with nonaffected enamel.

In MIH-affected teeth, the restoration stage is problematic because of the micromorphological changes found in the hard tissues, which may adversely affect the adhesion between the restorative materials and these substrates.[24,25] A significantly higher bond strength to sound enamel when compared with MIH-affected enamel has been reported, regardless of the type of adhesive system used (total-etch or self-etch systems).[23,25] The explanation is that a conventional phosphoric etching pattern on MIH enamel is much less pronounced, and this etching also exposes porosities and voids resulting in a "weak link for enamel-resin bond."[25] An abnormal etching pattern in MIH-affected enam-

el compared with sound enamel was found in other studies.[54,61] Moreover, total-etch adhesive systems demonstrated higher bond strength to MIH-affected enamel when compared with self-etch adhesive systems.[25] On the other hand, dentin adhesion beneath hypomineralized enamel seems not to be negatively affected and may be performed using either total-etch or self-etch adhesive systems.[25]

Considering all of this information, all MIH-affected enamel was removed so as not to jeopardize the bonding quality between the adhesive system and enamel. This procedure ensured that margins were in sound enamel since the cavosurface margins in hypomineralized enamel show less bonding capability[16] and may be more susceptible to marginal breakdown. An *in vivo* study[26] found increased restoration failures in first permanent molars when the hypomineralized tissue was left surrounding the cavities, which means that the complete removal of hypomineralized enamel significantly increases the success of MIH-affected teeth restored with resin composite compared with a noninvasive/conservative group (hypomineralized tissue was left in the cavity). Similarly, Fagrell and others[64] also recommended the need for removal of MIH-affected areas up to healthy enamel to ensure adequate adhesion and less chance of bacterial invasion,[5,64] but with more tooth structure loss.

For anterior teeth, the removal of all white/yellow/brown stains is also reasonable because of esthetic concerns. Sönmez and others[26] recommended a more conservative technique by treating the hypomineralized enamel with NaOCl instead of removing all the MIH-affected enamel. However, leaving white/yellow/brown stains around the margins of the preparation may affect the esthetic quality, especially on the facial surfaces of incisors. Future studies should describe and standardize guidelines for assessing the causes of MIH and surface treatments prior to bonding, in order to achieve more conservative dental preparations for resin composite restorations in MIH-affected teeth.

CONCLUSIONS

Regardless the cause of MIH, the restorative treatment should comprise the removal of all MIH-affected dental hard tissues (enamel and dentin) prior to bonding procedures for resin composite restorations. After 18 months, satisfactory bonding to MIH-affected teeth using a total-etch adhesive system was observed. Preventive treatments (topical fluoride application, glass-ionomer restorations) should be adopted as soon as the MIH is diagnosed in order to avoid PED, development of caries lesions and pain, and, consequently, invasive treatments such as root canal treatments or extractions.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ingá University Center–UNINGÁ, Brazil.

Conflict of Interest

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

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Two-year Follow-up of Ceramic Veneers and a Full Crown Treated With Self-etching Ceramic Primer: A Case Report

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Clinical Relevance

The Monobond Etch & Prime seems to be an efficient option for adhesive cementation of ultrathin veneers and full crown ceramic with good properties after two years of clinical follow-up.

SUMMARY

The use of the self-etching ceramic primer combines the stages of acid conditioning and silanization in cementation procedures of ceramic restorations. The protocol is a simpler and safer alternative to the conventional protocol for surface treatment of silica-based ceramics. This case report describes the steps of an esthetic rehabilitation with ultrathin veneers and full crown based on lithium disilicate treated with a ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent, Schaan, Liechtenstein). After two years of clinical follow-up, the restorations presented satisfactory esthetic and functional performance, color stability, surface and marginal integrity, and absence of cracks and debonding. More research is needed to investigate the clinical performance and longevity of the ceramic restorations treated with self-etching ceramic primers.

INTRODUCTION

Adhesion is a key factor for the long-term success of ceramic restorations.[1] For certain restoration types, such as ultrathin veneers, retention to the tooth surface depends solely on the micromechanical and

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Table 1: *Material, Brand, Manufacturers, and Chemical Composition of the Materials Used in This Clinical Case*

Material	Brand	Manufacturer	Composition
Lithium dissilicate ceramic	IPS e.max Press	Ivoclar Vivadent	SiO, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, and other oxides and ceramic pigments
Fluorapatite ceramic	IPS e.max Ceram	Ivoclar Vivadent	SiO_2, Al_2O_3, Na_2O, ZnO, CaO, P_2O_5, F, other oxides, amd pigments
Self-etch ceramic primer	Monobond Etch & Prime	Ivoclar Vivadent	Butanol, tetrabutylammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, bis(triethoxysilyl)ethane, silane methacrylate, colourant, ethanol, water
Phosphoric acid 35%	Ultra-Etch,	Ultradent Products Inc	Phosphoric acid, cobalt aluminate blue spinel, and siloxane
Adhesive system	Excite F	Ivoclar Vivadent	Bisphenol A-glycidyl methacrylate (Bis-GMA), ethanol. 2-hydroxyethyl methacrylate, phosphonic acid acrylate, urethane dimethacrylate, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide, potassium fluoride
Resin cement	Variolink Esthetic LC	Ivoclar Vivadent	ytterbium trifluoride, urethane dimethacrylate, glycerin-1.3-dimethacrylate, 1,10-decandiol dimethacrylate

chemical retention between the dental substrate, resin cement, and ceramic veneer.[1,2] The treatment of the internal ceramic surface is fundamental for a higher and stable bond through the creation of micro-retention, increase of surface energy, and formation of a chemically active surface for the adhesive and resin cement.[3,4]

The gold standard for the surface treatment of silica-based ceramics is the application of hydrofluoric acid (HF) and silane.[5-7] Silica-based ceramics are acid sensitive because they have a high amount of glass phase, which is susceptible to a selective dissolution from acid conditioning. This feature, combined with the excellent optical properties, makes glass ceramics the first choice for esthetic restorations.[8] However, despite its wide use and proven efficacy,[9,10] the conventional protocol with HF and silane has some disadvantages such as the high toxicity of HF[11] and the weakening of the ceramic veneer in excessively long acid applications,[12-14] which can affect mechanical resistance.

The self-etching ceramic primer Monobond Etch & Prime (MEP, Ivoclar Vivadent, Schaan, Liechtenstein) has recently been launched as an alternative for the surface treatment of silica-based ceramics to overcome the limitations of conventional procedures. Its composition includes ammonium polyfluoride, which acts as a conditioning agent, and silane methacrylate, which acts as a bonding agent, thus joining the conditioning and silanization procedures into a single step[15] (Table 1). The advantages of this product are the simplification of the technique, single application method for the different types of ceramic systems, prevention of excessive conditioning even after a prolonged application time, and the greater safety and biocompatibility.[16]

The performance of MEP has been investigated by several laboratory studies. The conditioning pattern generated by MEP is more superficial and less evident than that produced by HF.[10,17-21] Despite these findings, the bond strength of ceramic specimens treated with the self-etching primer is similar to that observed with the conventional protocol.[15,17,22-29] However, some authors have reported higher bond strength of specimens treated with HF and silane.[10,21,30,31] The fatigue strength of lithium disilicate–based ceramic samples treated with MEP was lower than that of samples conditioned with 5% HF and silane.[19]

However, there is no clinical trial evaluating the long-term performance of MEP. One case report described an oral rehabilitation with ceramic restorations with follow-up of six months.[22] Considering the scarcity of clinical studies and the promising results of the few laboratory studies with the self-etching primer, the aim of this work was to describe a case report of an esthetic treatment with lithium disilicate–based ceramic restorations treated with MEP and the clinical performance of the restorations in a two-year follow-up.

CASE REPORT

A 22-year-old male patient sought specialized dental care for a restoration fracture on tooth 9. The following characteristics were observed through clinical examination: midline deviation, extensive restoration with moderate color change in the labial surface of tooth 9, diastemas between the central

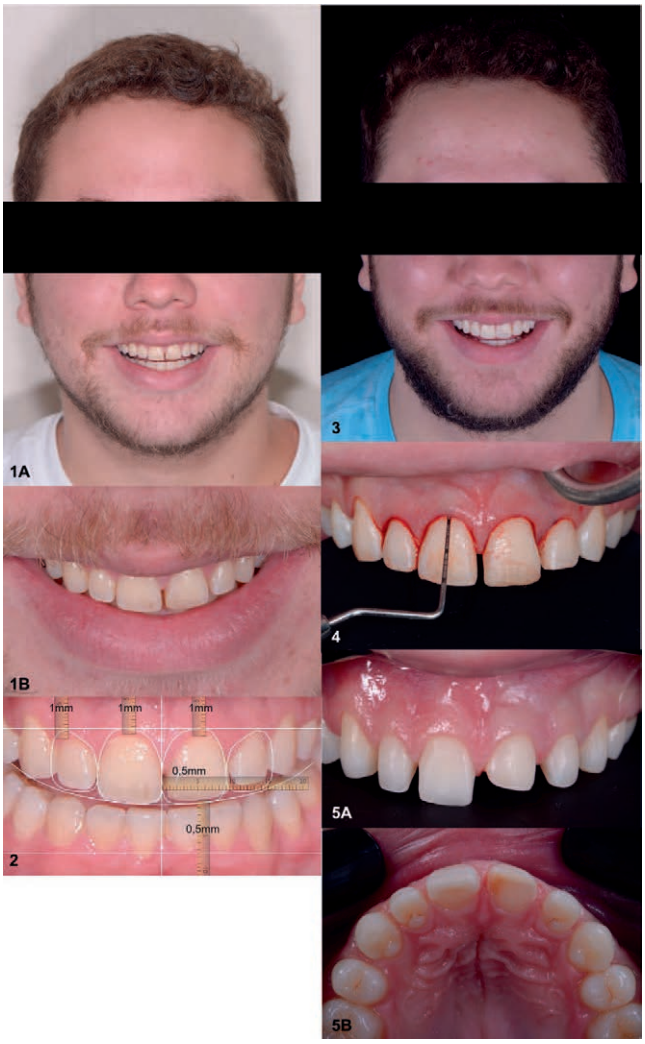

Figure 1. *(A) Initial photographs of the face and (B) smile of the patient showing the restoration in tooth 9 with moderate color change, diastemas and deficient dental proportion.*

Figure 2. *Digital analysis of the smile showing midline deviation, planning of the gingivoplasty and dental proportion correction.*

Figure 3. *Evaluation of the mockup planning.*

Figure 4. *Gingivoplasty for regularization of gingival zenith and increase of clinical crown.*

Figure 5. *(A) Minimally invasive preparation for full crown in tooth 9 and for ultrathin veneers in the other teeth. (B) Occlusal view of the preparations of teeth 6 to 11.*

and lateral incisors, unfavorable dental proportion, and gingival zenith with a negative impact on gingival harmony (Figure 1A,B).

After digital smile planning, the correction of the gingival contour by clinical crown increase of the central and lateral incisors (Figure 2) was proposed. An orthodontic treatment was also suggested for a better distribution of the interdental spaces, thus increasing the predictability of the case. Afterward, study models were created from impressions made with vinyl polysiloxane/silicone (Express XT commercially available in the United States as Express VPS, 3M ESPE, St Paul, MN, USA) and sent to the laboratory for diagnostic wax-up for teeth 6 to 11. The mockup was performed with Protemp bis-acryl resin A1 (3M ESPE) (Figure 3). In this phase, the occlusal contacts and eccentric movements were evaluated clinically with metallic articulating film (Arti-fol 12 μm, Bausch Articulating Papers Inc, Nashua, NH, USA). After esthetic and functional approval by the patient, gingivoplasty was recommended for the regularization of gingival zenith (Figure 4).

Sixty days after the surgical procedure, the prosthetic procedures for the veneers were started. The restoration on tooth 9 was removed, and the tooth was prepared for full crown; the remaining teeth from 6 to 11 were prepared to receive ultrathin veneers (Figure 5A,B). Tooth preparations were minimally invasive and done with a diamond bur (#8862.314.012, Komet, Lemgo, Germany). All angles were rounded, and the margins were chamfered, continuous, and well defined.[32] The preparations were finished and polished with a multilaminated bur and an Arkansas polisher (#H48L.314.012 and #649.314.420, Komet) mounted on a multiplier contra-angle (Kavo, Biberach, Germany). Preparations were guided by an index made with condensation silicone[32] (Zetaplus/Zhermack, São Paulo, SP, Brazil) (Figure 6). Impressions of the preparations were made by the two-impression technique with vinyl polysiloxane/silicone (Express XT) and sent to the laboratory. Temporary veneers were then made with Protemp Bis acryl resin A1 (Figure 7A,B).

The cast models were scanned for fabrication of the lithium disilicate veneers (IPS e.max Press, Ivoclar Vivadent) in a computer-aided design and computer-aided manufacturing (CAD/CAM) system (Amann Girrbach, Koblach, Austria). For better esthetics, the incisal and labial surfaces of the ceramic restorations were stratified with fluorapatite ceramics (IPS e.max Ceram, Ivoclar Vivadent) (Figure 8A-C). After dry-testing to check the marginal fit, the color of the resin cement was selected (Variolink Veneer Try-In, Ivoclar Vivadent) (Figure 9). The pieces were then washed and dried with air jets.

The surfaces of the veneers were treated with MEP (Ivoclar Vivadent), which was applied and rubbed onto the surfaces for 20 seconds with a microbrush (SDI Limited, Baywater, VIC, Australia)

Figure 6. *Silicone index for teeth preparations based on diagnostic wax-up.*

Figure 7. *(A) The papillae were covered with Teflon strips to avoid gingival compression and maintain the space for dental hygiene. (B) Temporary restorations made with bis-acryl composite resin in shade A1 (Protemp-4/3M ESPE).*

Figure 8. *(A) Frontal view of lithium disilicate ceramic restorations (IPS e.max Press, Ivoclar Vivadent) and stratification of the buccal and incisal surfaces with fluorapatite ceramic (e.max Ceram, Ivoclar Vivadent). (B) Final view of ceramic restorations and (C) cervical view.*

Figure 9. *Selection of the neutral cement color with the try-in paste (Variolink Esthetic LC Try-In, Ivoclar Vivadent).*

Figure 10. *(A) Application of Monobond Etch & Prime (Ivoclar Vivadent) followed by (B) washing. (C) Appearance of the inner surface of ceramic restorations after surface treatment.*

Figure 11. *Application of the resin cement and adaptation to the prepared tooth.*

and allowed to act for another 40 seconds. The surfaces were then rinsed with water and dried with air jets (Figure 10A-C). Pumice and water prophylaxis was performed on the prepared teeth with a Robinson mini brush (ICBrush, Ultradent Products Inc, South Jordan, UT, USA) and then washed with water and dried with air jets. Teeth were then conditioned with 35% phosphoric acid (Ultra-Etch, Ultradent Products Inc) for 20 seconds, rinsed, air jet dried, and treated with an adhesive system (Excite F, Ivoclar Vivadent).

Neutral color Variolink Esthetic resin cement (Ivoclar Vivadent) was used for cementation of the ceramic veneers (Figure 11). The excess cement was removed with a brush,[33] followed by light curing for 40 seconds on each surface (Radii Plus, SDI Limited, 1200 mW/cm^2). Glycerin gel (Liquid Strip, Ivoclar Vivadent) was applied on the cervical (crown and

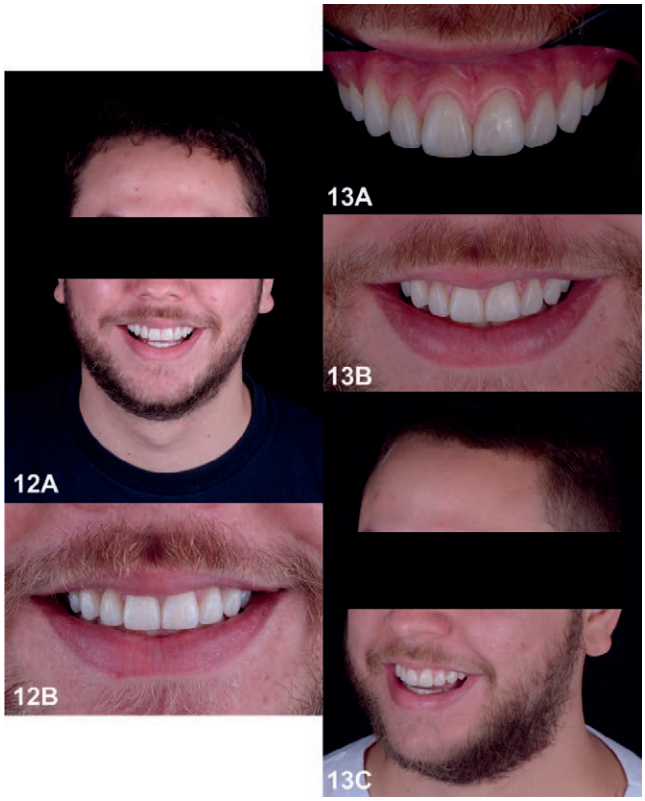

Figure 12. *(A) Facial appearance and (B) frontal view of the smile after cementation.*

Figure 13. *(A) Final view of ceramic restorations and (B) smile after two years of follow-up; (C) lateral view of the facial appearance.*

veneers) and incisal (veneers) regions, and another curing cycle was performed. After polymerization, the excess cement was removed with a #12 scalpel blade. A protective splint was manufactured for the patient to prevent tooth wear during sleep. The occlusal splint has been used daily by the patient during these two years of follow-up. The veneers and the crown can be seen in Figures 12A,B and 13A-C, following cementation and after two years of follow-up, respectively.

DISCUSSION

The clinical success of ceramic restorations depends on an adequate adhesion between the dental substrate and the restorative material. This adhesion is composed of two interfaces: resin-cement and ceramic-cement.[34] Therefore, an appropriate surface treatment of the restoration is necessary. In the present clinical case, the restorations treated with self-etching ceramic primer showed color stability, ceramic and margin integrity, absence of cracks, fractures, discoloration, and debonding after the two-year follow-up. One previous clinical case also

reported the application of MEP for the surface treatment of veneers and full crown of lithium disilicate–based ceramics. After the six-month follow-up period, the authors did not detect signs of failure, and the esthetic and functional characteristics of the restorations were considered excellent.[22]

MEP was developed for the treatment of the internal surface of silica-based ceramics for resin cementation.[16] The mechanism of action involves the conditioning of the ceramic by ammonium polyfluoride, causing the increase of roughness, contact area, and surface energy. The residues are removed by washing with water, and after drying, the silane methacrylate initiates the chemical reaction, leaving a thin layer of silane on the ceramic surface, which will react with the resinous monomers.[16,19]

Several *in vitro* studies have been developed to evaluate the effect of MEP on adhesion and mechanical properties of glass ceramics. Concerning topographic changes, studies found that the conditioning pattern generated by MEP is less evident, with lower values of surface roughness[10,17-21] and higher contact angle[21] compared with HF. The conditioning depth of the samples of glass and hybrid ceramics treated with MEP was lower than the samples treated with 5% and 10% HF for 20 and 60 seconds, and the concentration and application time of HF presented a linear relationship with the dissolution depth of the glass matrix.[20] Considering the current use of extremely thin ceramic restorations, such as in the present case, the use of less aggressive conditioning agents can benefit the mechanical properties and longevity of the pieces.[10,20]

MEP adhesion was evaluated by tensile bond strength or shear strength tests. The bond strength was similar to the treatment with HF and silane in most published articles.[15,17,22-29] Some studies performed the aging of the samples to simulate clinical degradation, especially at the adhesive interface, which is important to indicate the durability of adhesion between materials.[25] After aging, the tensile bond strength obtained with the self-conditioning primer was similar to HF and silane,[17,25,27-29] whereas the shear bond strength was higher in the samples treated with the conventional protocol.[10,21] In general, adhesion stability was negatively influenced by aging, regardless of the type of surface treatment.[10,17,25,27,28] The exception was the study by Prado and others,[21] in which the shear bond strength of the glass ceramic samples treated with MEP did not differ between before and after aging (storage in water for 70 days and thermocycling for 12,000 cycles), indicating a greater adhesion stability.

Scherer and others[19] tested fatigue resistance in lithium disilicate discs bonded to a dentin analogue using 5% HF and silane or MEP with and without aging and found higher values for HF. Although microstructural changes caused by HF are more aggressive, the micromechanical and chemical bonding with the resin cement appears to compensate and improve the strength of the samples. Despite these findings, the authors argue that MEP is a good option for surface treatment, given its ability to cause topographic changes in glass ceramics without the use of the highly toxic HF using a simple application method. Moreover, they point out that the resistance to fatigue achieved appears to be sufficient to withstand the masticatory loads, with an exception for high bite forces recorded during sleep bruxism.[19,35-37]

Despite the satisfactory clinical performance in the present clinical case and the favorable results of laboratory studies, many questions about MEP are still unanswered. Controlled and randomized clinical trials are essential to evaluate the longevity of adhesive restorations treated with this product. In addition, little has been investigated *in vitro* about the influence of MEP on the mechanical properties of glass ceramic, as well as the effect of different application protocols. Therefore, it is critical that more research be developed before MEP can be used in clinical practice with safety and predictable long-term performance.

CONCLUSION

The satisfactory clinical performance of ceramic restorations treated with MEP after two years of follow-up supports the promising results showed by laboratory studies. However, further research is needed to investigate the clinical performance and longevity of MEP and to fully recommend it as a safe and reliable alternative for the surface treatment of glass ceramic restorations.

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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Clinical Research

Pure Ormocer vs Methacrylate Composites on Posterior Teeth: A Double-blinded Randomized Clinical Trial

CRG Torres • MG Augusto • IF Mathias-Santamaria • R Di Nicoló • AB Borges

Clinical Relevance

The clinical behavior of pure ormocer composite is reliable when used in class II restorations after 24 months of evaluation.

SUMMARY

Objective: The aim of this study was to evaluate the clinical performance of class II restorations made using pure ormocer and methacrylate composites in a period of 24 months, using a split-mouth double-blinded randomized design.

Methods and Materials: Thirty patients received two class II restorations (n=60) performed with different composites: GrandioSO (methacrylate, nanohybrid) and Admira Fusion (pure ormocer, nanohybrid). The universal adhesive system (Futurabond M+) was applied in all restorations using the self-etching mode. The composites were placed by the incremental technique. The restorations were evaluated using the FDI World Dental Federation criteria after 7 days and 6, 12, and 24 months postoperatively.

Results: After 24 months, 23 patients attended the recall and 46 restorations were evaluated. Fisher's statistical analysis (5%) showed no difference between the materials. One pure ormocer restoration and one methacrylate restoration presented small fractures. Only one tooth suffered a fracture of the remaining tooth structure. Admira Fusion presented, respectively, 100%, 95.66%, and 100% of accept-

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able performance in general scores for esthetic, functional, and biological properties. GrandioSO presented, respectively, 100%, 91.31%, and 95.66% of acceptable performance in the same scores.

Conclusion: After 24-month follow-up, nonsignificant differences between the tested composites was detected. Both materials provided acceptable clinical performance in class II restorations.

INTRODUCTION

The demand for dental materials with high esthetics and longevity led to the research and development of new composites. Composition, size, shape, distribution, and content of filler particles are paramount for the composite's properties.[1,2] Great efforts in recent years have been made to improve the filler technology, increasing the mechanical and esthetic properties of these materials, resulting in the current nanohybrid and nanoparticle containing composites.[3] However, few changes were performed in relation to the organic matrix, and many traditional dimethacrylate monomers are still in use.

Bis-GMA (bisphenol A-glycidyl methacrylate) has been the main monomer used in composite formulations since its development in 1956 by Bowen.[4] Due to its high viscosity, it is necessary to add low molecular weight monomers in the blend to achieve the appropriate viscosity on the final formulation for clinical use.[5] However, these diluent monomers increase the polymerization shrinkage and water sorption of the composites.[6] In addition, unreacted monomers are eluted from the cured material, increasing its cytotoxicity to the pulp cells.[7] Thus, aiming to improve the properties of the composite restorative materials, new monomers have been investigated.

Ormocer is the acronym for organically modified ceramic. They are produced by hydrolysis and polycondensation reactions (sol–gel processing) to form a molecule with a long inorganic silica chain backbone and organic lateral chains.[8] Compared with Bis-GMA, the ormocer molecule has more methacrylate groups available to set bonds.[9] The composites with ormocer are expected to demonstrate higher degree of conversion and increased wear resistance and toughness due to the formation of a polymer network more highly crosslinked.[10] Another advantage of ormocer would be higher biocompatibility, because the increased number of chemical bonds between the methacrylate groups would reduce the amount of unreacted free monomers in the polymer network.[11]

However, the first generation of ormocer composites contained, besides the ormocer molecules, regular low molecular weight dimethacrylate monomers acting as diluents. The presence of such diluents may have hampered the expected results, and no clear advantages were observed when using the first-generation ormocer-based fillings in comparison with conventional composites.[12,13] Recently, a pure ormocer composite was developed. According to the manufacturer, there is no diluent methacrylate monomer in the composition, because special ormocer molecules of various viscosities were created.

In comparison with methacrylate composites, the new ormocer material may offer advantages of lower polymerization shrinkage and water sorption.[14,15] In addition, this new material was reported to present higher microhardness and degree of conversion.[16] However, there is still a lack of information regarding its clinical performance. Thus, the aim of this study was to investigate the clinical performance of a pure ormocer and a methacrylate-based resin composite in class II restorations. The null hypothesis tested was that the monomer composition (ormocer × methacrylate) does not influence the restoration clinical behavior in relation to esthetic, functional, and biological properties.

METHODS AND MATERIALS

The description of the experimental design followed the Consolidated Standards of Reporting Trials (CONSORT) statement.[17]

Trial Design, Settings, and Location of Data Collection

This was an equivalence, split-mouth, double-blind (patients and examiner), randomized clinical trial. The study was carried out in the clinics of the School of Dentistry at the local University from August 2014 to January 2017.

The PICO question was stated, and the parameters were defined: P, adult patients presenting two class II cavities; I, restoration performed with pure ormocer composite; C, restoration with methacrylate composite; and O, clinical performance according to FDI World Dental Federation criteria. The research question analyzed was as follows: Do composite class II restorations made with pure ormocer composite present better clinical performance than restorations

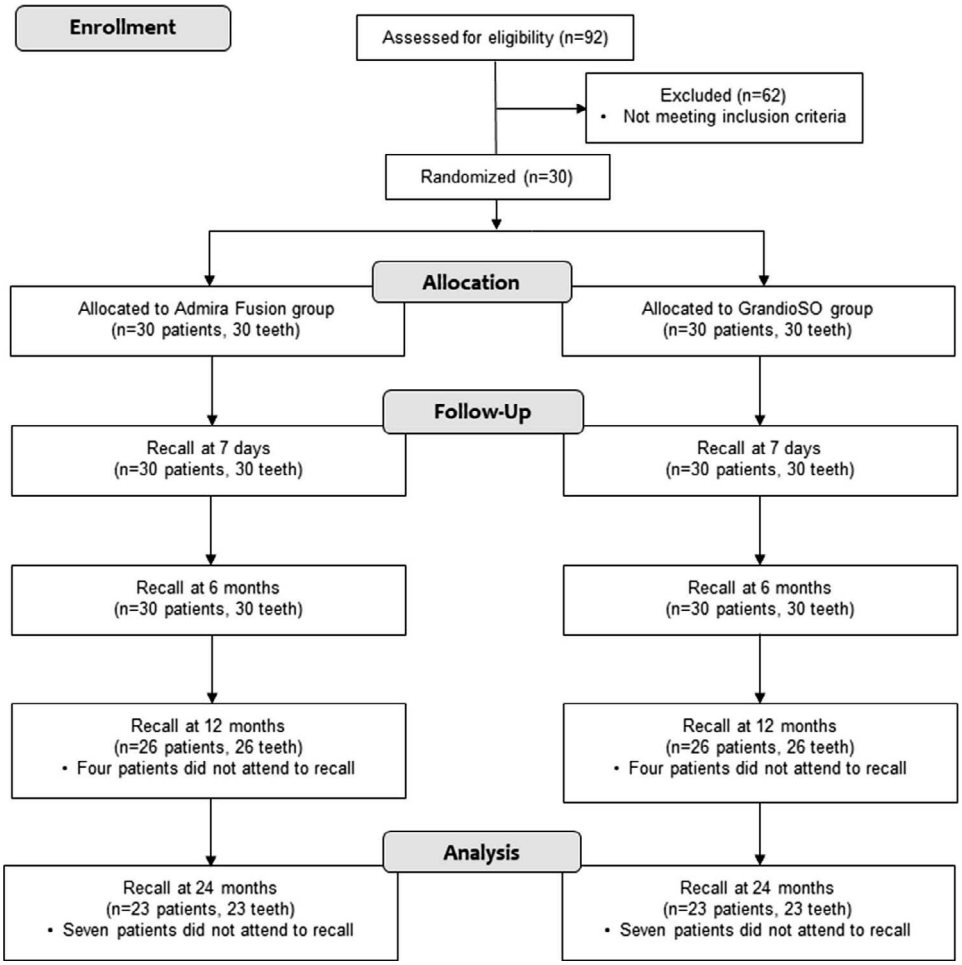

Figure 1. *CONSORT 2010 flow diagram.*

made with methacrylate composite according to FDI criteria?

Recruitment

The patients were selected as they searched for treatment in the local university. No advertisement was made for participant recruitment, forming a sample of convenience.[18]

Eligibility Criteria

A total of 92 participants were examined to form a group with 30 patients that attended to the inclusion and exclusion criteria (Figure 1). Patients were required to have good general health, be older than 18 years old, and present at least 20 teeth in occlusion. Patients with extremely poor oral hygiene, severe or chronic periodontitis, or heavy bruxism habits were excluded from the study. Patients had to present at least two teeth with class II cavities to be restored (two molars or two premolars). The cavities had to present size of the isthmus being no more than two-thirds of the intercuspal distance; the antagonist and the adjacent tooth had to make contact; there needed to be vital pulp; and there needed to be an absence of painful symptoms.

Sample Size Calculation

The sample size calculation was based on the clinical success rate (97.5% at 24 months) of posterior class II composite restorations observed in a previous study.[19] Using a significance level of 0.05, power of 80%, and equivalence limit of 15%, the sample size required per group was 23 teeth. Considering the possible dropouts, a total of 30 patients were selected, totaling 60 restorations, 30 for each group.

Random Sequence Generation and Allocation Concealment

The randomization was performed using online software (www.sealedenvelope.com). A blocked list was generated, and a randomization code was performed according to two treatment possibilities

Table 1: *Materials Used in the Restorative Procedures*

Material	Manufacturer	Composition		
Futurabond M+	VOCO	UDMA, HEMA, 10-MDP, camphorquinone, BHT, ethanol, and water		
GrandioSO	VOCO	Organic matrix: Bis-GMA, Bis-EMA, TEGDMA	Inorganic fillers: barium aluminum borosilicate glass ceramic filler, silicon dioxide nanoparticles (0.02-1 μm)	Filler content: 87% w/w
Admira Fusion	VOCO	Organic matrix: organically modified ceramic (Ormocer)	Inorganic fillers: barium aluminum borosilicate glass ceramic filler, silicon dioxide nanoparticles (0.02-1 μm)	Filler content: 84% w/w

Abbreviations: UDMA - urethane dimethyl methacrylate, HEMA – Hydroxyethylmethacrylate, 10-MDP – 10-methacryloyloxydecyl dihydrogen phosphate, Bis-GMA – bisphenol A-glycidyl methacrylate, TEGDMA – triethylene glycol methyl ether methacrylate.

(GrandioSO or Admira Fusion). Thus, the operator started at the first quadrant to be restored and then chose between two opaque sealed envelopes containing the randomization code; the envelopes were prepared by a staff member not involved in any of the phases of the clinical trial.

Interventions: Restorative Procedure

Three operators, each with at least three years of clinical experience, performed all interventions. Shade selection was performed using a VITA Classical shade guide (VITA Zahnfrabrik, Bad Säckingen, Germany). All patients received local anesthesia before the tooth preparation, which was performed using a high-speed handpiece fitted with a round diamond bur under water cooling. When present, carious dentin was removed with a round carbide bur at low speed. The outline shape of the preparations was limited to the removal of caries/defective restoration, without beveling. Isolation with rubber dam and clamps was performed. Each patient received at least two restorations, one using a pure ormocer composite and another using a methacrylate composite, subjecting the different materials to the same clinical conditions and enabling the comparison between them. Table 1 presents the specifications of the materials used.

In deep preparations, a glass-ionomer cement liner (Meron R, Voco, Cuxhaven, Germany) was applied on the pulpal wall. In very deep preparations, calcium hydroxide cement (Dycal, Dentsply, Rio de Janeiro, Brazil) was applied, followed by a thin layer of glass ionomer cement.

Futurabond M+ (Voco) was used in self-etching mode according to manufacturer's instructions for all preparations. A thin layer of adhesive was actively applied for 20 seconds, followed by a gentle blow of air for five seconds and light curing for 10 seconds. The light curing was performed with an LED device having an emittance of 700 mW/cm^2 (Emitter A, Schuster, Santa Maria, RS, Brazil).

Restorative procedures were performed using a precurved metallic sectional matrix (Unimatrix System, TDV, Pomerode, SC, Brazil), associated with a separating ring and a wooden wedge. The composite was applied using an incremental oblique technique. Each increment of 2 mm was light cured for 20 seconds. Finishing was performed with fine-grain diamond burs (KG Sorensen, São Paulo, SP, Brazil). At the proximal surface, any excess was removed with abrasive strips (3M ESPE, St Paul, MN, USA). After seven days, the polishing procedures were performed with abrasive silicone tips (Dimanto, VOCO, Cuxhaven, Germany) and at the proximal surfaces with fine-grained strips.

Calibration Procedures for Clinical Evaluation

Two examiners who did not participate in restoration placement were trained for restoration evaluation with an online calibration tool (www.e-calib.info) in August 2014. Intraexaminer and interexaminer agreement of at least 85% was necessary before beginning the evaluation.[20]

Blinding

The study was classified as double-blind, because participants and the examiners (who were not involved with the restoration procedures) were blinded to the intervention.

Clinical Evaluation

Intraoral photographs were taken at baseline and at the recall appointments. Digital images were obtained using a Canon T3i camera with a Macro lens (Canon, Ota, Tokyo, Japan).

Restorations were evaluated after 7 days and 6, 12, and 24 months according to the FDI criteria.[21,22] Visual assessment of restorations was performed

Table 2: *Number of Lesions According to Sex and Age of Patients*

Characteristics of Patients	Number of Lesions
Sex	
Female	48
Male	12
Age (years)	
20-29	4
30-39	4
40-49	10
50-59	8
≥60	4

under dental unit overhead light without magnification. Cotton roll isolation was used to ensure a dry field. The marginal adaptation was analyzed with special probes. The marginal gap width was classified using two special probes (Deppeler, Rolle, Switzerland) with tip diameters of 150 and 250 μm. The firmness of the contact was first checked with a waxed dental floss. If it was considered weak, metal blades of increasing thicknesses (25, 50, and 100 μm) were inserted into the interdental space, determining the thickest one that could enter the interproximal area. For each of the evaluated parameters, one of the following scores was assigned: clinically very good; clinically good; clinically sufficient/satisfactory; clinically unsatisfactory; and clinically poor. Disagreements between examiners were discussed to reach a consensus.

Statistical Analysis

The statistical analyses followed the intention-to-treat protocol according to the CONSORT recommendation.[17] This protocol includes all subjects in their originally randomized groups, even those that were not able to keep the scheduled recall visits. This approach is considered more conservative and less open to bias.

Descriptive statistics were used to describe the distributions of the evaluated criteria. Statistical analysis for each individual item was performed, as well as for each overall parameter. The differences in the ratings of the two groups after 24 months were tested with the Fisher's exact test, with a significance level of 5%.

RESULTS

The restorative procedures were implemented exactly as planned, and no modification was performed. Sixty-two of 92 subjects were not enrolled in the study because they did not fulfill the inclusion criteria. Thus, 30 subjects were selected. Details regarding the characteristics of the research subjects and the restored cavities are shown in Tables 2 and 3.

Table 3: *Characteristics of Restored Cavities*

Characteristics of Restored Tooth	Number of Lesions
Tooth distribution	
Premolars	36
Molars	24
Dental arch distribution	
Maxillary	38
Mandibular	22
Presence of antagonist	
Yes	60
No	0
Pulp protection	
Yes	53
No	7
Faces involved	
OM	15
OD	27
MOD	18
Width	
Small	17
Medium	18
Large	25
Depth	
Shallow	9
Medium	28
Deep	23
Reason for restoration	
Caries	3
Fracture	14
Caries and fracture	3
Esthetic	26

The percentage of patients that attended recall evaluations was as follows: 100% (7 days), 100% (6 months), 87% (12 months), and 77% (24 months). The qualitative evaluation according the FDI guidelines is presented in Table 4. The overall Cohen's κ statistics showed excellent agreement between the examiners in the 7-day (0.93), 6-month (0.96), 12-month (0.96), and 24-month (0.91) follow-up.

After 24 months, only one restoration made with pure ormocer composite and one made with methacrylate composite presented small fractures, which did not indicate the need for replacement of the restoration. Only one tooth presented fracture of the remaining dental structure (one nonsupporting

Table 4: *Number of Evaluated Restorations and Classification According to the FDI Criteria*

FDI Criteria	Score	7 Days		6 Months		12 Months		24 Months	
		OR	MA	OR	MA	OR	MA	OR	MA
Esthetic properties									
1. Surface luster	Clinically excellent	30	30	27	28	21	22	18	16
	Clinically good	—	—	3	2	5	4	5	7
2. Staining									
a) Surface	Clinically excellent	30	30	27	26	22	19	20	17
	Clinically good	—	—	3	4	4	7	3	6
b) Marginal	Clinically excellent	30	30	28	27	19	‘8	14	13
	Clinically good	—	—	2	3	7	8	9	10
3. Color match and translucency	Clinically excellent	26	28	26	28	22	23	19	18
	Clinically good	4	2	4	2	4	3	4	5
4. Esthetic anatomical form	Clinically excellent	30	30	30	30	26	26	23	23
Functional properties									
5. Fracture and retention	Clinically excellent	30	30	30	30	25	26	20	21
	Clinically good	—	—	—	—	1	—	1	1
	Clinically satisfactory	—	—	—	—	—	—	1	—
	Clinically unsatisfactory	—	—	—	—	—	—	1	1
6. Marginal adaptation	Clinically excellent	30	30	27	28	23	24	18	18
	Clinically good	—	—	3	2	3	2	4	3
	Clinically satisfactory	—	—	—	—	—	—	1	2
7. Occlusal contour and wear									
a) Qualitatively	Clinically excellent	30	30	30	30	26	26	23	23
b) Quantitatively	Clinically excellent	30	30	30	30	26	26	23	23
8. Proximal anatomic form									
a) Contact point	Clinically excellent	24	23	24	24	21	20	19	18
	Clinically good	2	2	2	1	1	1	—	1
	Clinically satisfactory	4	5	4	5	4	5	4	4
b) Contour	Clinically excellent	30	30	30	30	26	26	23	23
9. Patient’s view	Clinically excellent	30	30	30	30	26	25	23	22
	Clinically good	—	—	—	—	—	1	—	—
	Clinically satisfactory	—	—	—	—	—	—	—	—
	Clinically unsatisfactory	—	—	—	—	—	—	—	1
Biological properties									
10. Postoperative sensitivity	Clinically excellent	25	22	28	27	25	24	21	21
	Clinically good	5	7	2	3	1	2	1	1
	Clinically satisfactory	—	1	—	—	—	—	1	1
11. Recurrence of caries, erosion and abfraction	Clinically excellent	30	30	30	30	25	25	23	23
	Clinically good	—	—	—	—	1	1	—	—
12. Tooth integrity	Clinically excellent	30	30	30	30	26	26	23	22
	Clinically good	–	–	–	–	–	–	–	–
	Clinically satisfactory	–	–	–	–	–	–	–	–
	Clinically unsatisfactory	–	–	–	–	–	–	–	–
	Clinically poor	–	–	–	–	–	–	–	1
13. Periodontal response	Clinically excellent	25	24	27	28	24	24	22	22
	Clinically good	5	6	3	2	2	2	1	1
14. Adjacent mucosa	Clinically excellent	29	28	30	30	26	26	22	22
	Clinically good	1	2	–	–	–	–	1	1
15. Oral and general health	Clinically excellent	30	30	30	30	24	24	21	21
	Clinically good	–	–	–	–	1	1	1	1
	Clinically satisfactory	–	–	–	–	1	1	1	1

Abbreviations: MA, methacrylate composite; OR, pure ormocer composite.

cusp). The fractured areas were restored with the same technique and materials applied at the baseline. Fisher's exact test detected no significant difference ($p>0.05$) among evaluated FDI criteria for the two materials after 24 months.

In general scores for esthetic, functional, and biological properties, the pure ormocer composite presented, respectively, 100%, 95.66%, and 100% of clinically acceptable scores and the methacrylate composite presented, respectively, 100%, 91.31%, and 95.66%.

DISCUSSION

The results of this study showed that the different composites did not influence the clinical performance of class II restorations after 24 months. Thus, the null hypothesis tested was accepted.

Minimizing polymerization shrinkage is still the main goal of composite science development, because it remains as the principal reason for restorations failure. Shrinkage occurs during polymerization as weak van der Walls forces are converted into covalent bonds, reducing the distance between monomer molecules while forming a polymer.[23] The shrinkage stress at the tooth-restoration interface can produce cuspal deflection, fracture of the remaining tooth structure, and formation of marginal gaps, which have been associated with marginal staining and postoperative sensitivity.[23] A previous study found lower polymerization shrinkage in the latest version of ormocer composites compared with traditional methacrylate-based materials.[14] This may be due to the fact that ormocer molecules are larger than the Bis-GMA,[9] promoting a lower volumetric reduction of the material. However, the results of the current study showed no significant differences between the pure ormocer and the conventional methacrylate composite, regarding the marginal stain and postoperative sensitivity. This may be due to the incremental technique that may have reduced the effects of the polymerization shrinkage[24] and masked the differences between the composites.

Nonsignificant differences were detected between the composites in the parameters related to the esthetic properties. They presented only "clinically excellent" and "clinically good" scores. Both composites tested contain the same nanohybrid particles and similar filler content (w/w). The filler size and distribution are the main determining factors for surface properties such as roughness and gloss after polishing.[25] In addition, the nanoparticles present size below the wavelength of visible light (0.1-100 nm) that provides translucency and opalescence to these materials, enhancing their esthetic properties.[26]

Nonsignificant differences were detected between the composites in the parameters related to the functional properties. After 24 months, only one restoration made with pure ormocer and one made with methacrylate composite presented small fractures with partial loss (less than half of the restoration). A previous review showed that the incidence of fractures is higher in the first three years after the placement of the restoration.[27] Thus, we may speculate that in the next follow-ups, the fracture rate of the restorations will tend to remain low, indicating an excellent performance of the composites.

Both composites showed similar biological properties results. No caries recurrence was observed in the present study, which may be an indication that the restorations with both composites present an excellent polishability, reducing the biofilm retention. However, it should be highlighted that secondary caries trends occur later, being detected in longer-term follow-up periods.[27] Thus, a longer follow-up time may be necessary to confirm the observed results.

Soft tissue response to dental materials may be detected by effects on the periodontium and adjacent mucosa. These effects may be related to presence of residual unreacted monomers, roughness, and biofilm accumulation.[21] A previous *in vitro* study showed that the methacrylate and the ormocer composite tested present a low surface roughness, even after abrasive episodes.[16] The Bis-GMA molecule has two polymerizable units, whereas the ormocer has numerous organic polymerizable ones that increases the probability of interaction and chemical bonding with neighbor molecules and reduces free monomers after curing.[9] Thus, pure ormocer composites are expected to present better biological properties. However, within the limits of this study, it was not possible to demonstrate better clinical behavior of the improved ormocer restorative material in relation to the conventional nano-hybrid composite over two years. A longer period of evaluation may be necessary to show potential relevant differences.[28]

Previous studies demonstrated the role of the adhesive system on the clinical performance of a dental restoration, mainly related to postoperative sensitivity, marginal staining, retention, and sec-

ondary caries.[28,29] In the current study, all restorations were performed with a self-etching adhesive containing 10-MDP acidic monomer, which is incorporated in different adhesive formulations by various manufacturers. Besides micromechanical retention provided by the hybrid layer and tag formation, 10-MDP showed chemical bonding to the tooth structure, contributing to the bonding durability.[30,31] The adhesive system used was compatible with the pure ormocer restorative material formulation. According to the manufacturer, the adhesive system tested can be used with or without total or selective acid etching. They claim that the formulation is capable of effectively etching enamel and dentin without previous acid treatment, which was demonstrated in previous *in vitro* studies.[32,33] Therefore, it was decided to test the clinical performance of the material in association with ormocer material in its more challenging situation, based only on its self-etching properties. Previous clinical trials demonstrated adequate performance of this adhesive in the self-etching mode only, in association with methacrylate-based composites.[19,34]

According to the guidelines proposed by the American Dental Association, adhesive-based materials can be considered clinically acceptable if, after a six-month follow-up, they present less than 5% failure rate.[35,36] For full acceptance, the restoration losses cannot exceed 10% after a period of 18 months. Thus, the materials tested in this study could obtain complete acceptance, because the fracture rates of the pure ormocer and the methacrylate composites were both 3.33% (one failure in 30 restorations). The advantages of the ormocer technology that has been demonstrated *in vitro*[14-16,37] offers potential clinical advantages that may be proven with continued clinical studies.

CONCLUSION

After a 24-month follow-up, ormocer and methacrylate-based composites showed acceptable clinical performance in class II restorations. Nonsignificant differences were detected between both materials.

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Regulatory Statement

This study was approved by the local Institutional Review Board (no. 924.098) and registered in a National Clinical Trial Registry System under protocol RBR-9W5P8Q. The participants were informed about the nature and objectives of the study, and a consent form was signed by each one.

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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Assessment of Peroxide in Saliva During and After At-home Bleaching With 10% Carbamide and Hydrogen Peroxide Gels: A Clinical Crossover Trial

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Clinical Relevance

This study suggests that at-home bleaching systems are safe in relation to toxicity based on peroxide levels in saliva since the amount of peroxide potentially ingested is much lower than the estimated toxic dose.

SUMMARY

Objectives: This study evaluated the presence of peroxide in saliva using at-home bleaching systems containing hydrogen peroxide (HP) and carbamide peroxide (CP) with a prefilled tray (PT) or conventional tray (CT).

Methods and Materials: Participants received bleaching treatments after the sequence randomization (n=10): PT-HP/Opalescence-Go10%; CT-HP/WhiteClass10%; and CT-CP/OpalescencePF10%. Saliva was collected at the following times: baseline; at 1, 5, 15, and 30 minutes after administration; and at 3, 5, and 8 minutes after the tray was removed. Colorimetric analysis using analytic spectrophotometry was performed. The salivary flow (SF) was monitored during use of trays. Data about peroxide concentration (PC) were submitted to repeated-measures analysis of variance and Tukey tests (5%), and toxic dose was calculated based on body weight. The relation between SF and PC was verified with the Pearson correlation test.

Results: There was a significant difference for bleaching (p=0.0001) and time (p=0.0003) but

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not for interaction (p=0.3121). PC was lower for CT-CP in relation to PT-HP and CT-HP. After tray removal, expectoration, of the remaining gel, and mouth rinsing, no peroxide was detected in saliva. Correlation between SF and PC was considered weak (r=0.3379). The overall mean SF was 50.44% during tray use. In general, PC in saliva was 68.72% lower than the estimated toxic dose (0.26 mg/kg/day) considering all the bleaching systems.

Conclusions: Higher peroxide levels were detected in saliva with 10% HP gels. Nevertheless, they were below estimated toxic dose and were considered safe in relation to toxicity.

INTRODUCTION

Tooth bleaching is an esthetic treatment frequently performed in dental offices as it is minimally invasive and has proven clinical efficacy.[1,2] The most common active ingredient in bleaching gels is hydrogen peroxide, used in its original compound or produced by chemical reaction of its precursor carbamide peroxide. Due to its low molecular weight and high instability, hydrogen peroxide is able to diffuse into dental tissues, decomposing in free radicals that act on chromophores.[3,4] As a result of the oxidative reaction, the chromophores are converted into less complex structures, resulting in alteration of the optical properties of tooth substrate, increasing its reflectance and consequently resulting in a visual impression of a whiter tooth.[1,3,5]

Bleaching treatment can be used for teeth with intrinsic or extrinsic discoloration, as well as vital and nonvital teeth.[3,6,7] For vital teeth, three techniques can be performed: in office; at home; and using over-the-counter products.[8,9] The at-home bleaching technique was first described by Haywood and Heymann[10] in 1989 using 10% carbamide peroxide overnight. Several bleaching systems are available in the market with different peroxide concentrations, presentations, and suggested protocols of use. The more concentrated gels (up to 10% hydrogen peroxide) are marketed as at-home bleaching treatment alternatives to reduce the time of tray use during the day compared with the conventional at-home bleaching protocol.[2,6,10]

Although bleaching treatment is considered safe and effective,[1,2,11] potential adverse effects caused by peroxide have been described in literature. The most relevant negative consequences related to tooth bleaching are tooth sensitivity during and after bleaching procedures and gingival irritation. Additionally, peroxide release in saliva and potential gel ingestion have also been described.[3,6,12,13]

The potential toxicologic effect of hydrogen peroxide in contact with soft tissues or swallowed is a subject of concern by researchers and clinicians.[14] A safe hydrogen peroxide exposure level (no observed effect level) was determined in a previous study with catalase-deficient mice, reporting a maximum dose of 26 mg/kg/d.[15] This corresponds to the dose level in humans, considering the conventional uncertainty factor of 100-fold, of 0.26 mg/kg/d.[16,17] Peroxide release in the oral environment can be related to bleaching gel composition, viscosity, hydrogen peroxide concentration, amount of gel dispensed in the tray, type of tray, and adaptation in the dental arches.[16,18,19] Although peroxide levels in saliva are usually considered safe with lower-concentrated products, the more concentrated products may reach levels above the estimated toxic dose.[18] Even though many products are already available in the dental market with higher peroxide concentrations and different gel viscosity and tray designs, their safety regarding peroxide released in saliva has not been fully established.

This randomized crossover clinical trial aimed to evaluate the amount of peroxide released in saliva during tray use with different bleaching systems and compare it with the estimated toxic dose. Additionally, salivary flow during tray use was measured. The following null hypotheses were tested: 1) there is no difference among bleaching systems in relation to presence of peroxide in saliva, 2) there is no increase in salivary flow of participants during tray use, and 3) there is no difference in salivary flow during the use of the different bleaching systems.

METHODS AND MATERIALS

Ethical Aspects

This study was approved by the committee for the protection of human participants of the local university, and it was registered at the ReBEC website (Brazilian Clinical Trials Registry virtual platform). The structure of this article followed the protocol established by the Consolidated Standards of Reporting Trials Statement (CONSORT).[20]

Study Design

This study followed a factorial 3×8 design, considering the following as experimental factors: bleaching systems at three levels: prefilled trays with 10% hydrogen peroxide (PT-HP) (Opalescence Go 10%, Ultradent Products, Inc. South Jordan, UT, USA),

Table 1: *Eligibility Criteria*

Inclusion Criteria	Exclusion Criteria
Good general health	Smoking or alcoholic-dependent patients
> 18 years old	Pregnancy
Absence of noncarious cervical lesions, active caries, gingival recession, or periodontal disease	Tooth sensitivity
Do not use orthodontics appliance or removable prosthesis	Bruxism habits
Presence of all teeth from first molar to first molar (upper and lower) without restorations	Periapical alterations
Availability of recurring returns	Use of medicaments that alter salivary flow

customized trays with 10% hydrogen peroxide (CT-HP) (White Class 10%, FGM Produtos Odontológicos, Joinville, SC, Brazil), and customized trays with 10% carbamide peroxide (CT-CP) (Opalescence PF 10%, Ultradent Products) and time at eight levels (baseline; 1, 5, 15, and 30 minutes; and 3, 5, and 8 minutes after the tray was removed). These factors were tested in a prospective, randomized, and crossover clinical trial with three phases involving participants selected according to preestablished criteria (Table 1). To exclude a reduced salivary flow (SF) condition (lower than 2 mL/min), the participants were submitted to an SF analysis based on collection of unstimulated saliva during 5 minutes. The SF was calculated considering volume of saliva (mL) divided by time (minutes).

Participants were submitted to tooth bleaching treatment using prefilled trays or customized trays. Peroxide concentration in saliva measured at different times was the main dependent variable. The treatment sequence was randomized. The PICO (problem/patient/population, intervention/indicator, comparison, outcome) question was stated, and the parameters were defined: P = adult patients with discolored teeth; I = 10% hydrogen peroxide in prefilled or customized trays; C = 10% peroxide carbamide in customized trays; and O = peroxide concentration in saliva. The main research question was: Does 10% hydrogen peroxide in prefilled or customized trays present similar peroxide concentration in saliva compared with 10% carbamide peroxide in customized trays? Peroxide detected in saliva was compared with the estimated toxic dose ingestion. Additionally, as secondary outcomes, salivary flow during tray use and comparison among treatments were determined.

Sample-size Calculation

The sample size was determined using a power of 80% and alpha of 5%, considering means and standard deviations from a previous study.[21] The calculated effect size was 0.9, which resulted in a sample size of eight participants. Then, 10 participants were selected considering possible dropouts. The data were obtained using the G-Power 3.1 software.[22]

Group Division and Randomization Process

The participants underwent the bleaching treatments: 10% hydrogen peroxide in prefilled tray (PT-HP/Opalescence Go), 10% hydrogen peroxide in customized tray (CT-HP/White Class), and 10% carbamide peroxide in customized tray (CT-CP/Opalescence PF). The composition and manufacturers of each bleaching gel are presented in Table 2. The sequence of treatments that each participant received was generated using the software RANDOM.ORG True Random Number Service (www.random.org).

Table 2: *Composition of the Bleaching Gels Tested*

Bleaching Gel	Composition	Peroxide Concentration	Manufacturer
Opalescence Go	Glycerin, 20% water; carbomer; 10% hydrogen peroxide; sodium hydroxide; PVP (polyvinylpyrrolidone); silica; xylitol; disodium phosphate; 3% potassium nitrate; sucralose; flavor; 0.25% sodium fluoride; EDTA (ethylenediamine tetraacetic acid); sodium lauryl sulfate.	10% Hydrogen peroxide	Ultradent Products, Inc. South Jordan, UT,/USA
White Class	10% hydrogen peroxide; neutralized carbopol; 5% potassium nitrate; sodium fluoride; aloe vera; calcium gluconate; stabilizer; humectant; deionized water.	10% Hydrogen peroxide	FGM Produtos Odontológicos, Joinville, SC/ Brazil
Opalescence PF	Glycerin; 20% water; xylitol; 10% carbamide peroxide; carbomer; propylene glycol-300; polyacrylic acid; sodium hydroxide; EDTA (ethylenediamine tetraacetic acid); 0.5% potassium nitrate; 0.25% sodium fluoride.	10% Carbamide peroxide	Ultradent Products, Inc. South Jordan, UT,/USA

Blinding

The participants and operator were not blinded for the procedures since the type of tray used cannot be masked. Nevertheless, the examiner was blinded for chemical analysis.

Laboratory Procedures

Alginate impressions of maxillary and mandibular dental arches of patients were taken to obtain casts to produce the customized trays. Reservoirs 1 mm (±0.1 mm) thick were created on the facial surfaces of anterior teeth, including the first premolars in both arches, applying a light-cured resin (Ultradent LC Block-Out Resin) on the casts. The resin layer thickness was standardized using a thickness gauge with blunt tips that was positioned on the thirds of the buccal surface of each tooth before and after the resin application. Then, customized trays were fabricated with 0.9-mm-thick vinyl acetate sheets (Sof-Tray Regular, Ultradent Products) using the thermoforming process. Trays were precisely trimmed/scalloped completely involving tooth surface (1 mm incisal or occlusal to gingival margin), and its adaptation was verified on the casts. A small and controlled flame was gently applied to the edges to ensure maximum fit on cervical area, according to manufacturer's instructions. The trays were placed over teeth to verify the adaptation in the participants' mouths.

Study Intervention

The bleaching procedures were performed in three phases, according to the treatment randomization sequence. The participant underwent each of the three bleaching systems on a different day, during the morning, with a washout period of 1 week between treatments. The operator dispensed a standardized quantity (0.350 g for maxillary arches and 0.250 g for mandibular arches) of the bleaching gel inside the tray. This amount was calculated based on the average of three participants in a pilot study because the amount of gel present in the prefilled trays (0.799g±0.023g for maxillary and 0.766g±0.023g for mandibular) was too high and clinically impracticable to be applied in the customized trays. Any excess overflow of gel was carefully removed with gauze swabs. The prefilled trays were removed from the package and placed on the dental arches following manufacturer's instructions. Participants used the bleaching systems for 30 minutes.

Detecting Peroxide in Saliva

A spectrophotometry method based on the reaction of 4-aminoantipyrine and phenol with hydrogen peroxide was adopted. This method allows the oxidation of peroxide by peroxidase enzyme, resulting in solution color change from transparent to pink.[21] To detect hydrogen peroxide, an enzymatic reagent was prepared. The amount of hydrogen peroxide in saliva was quantified using an analytic spectrophotometer (Bioespectro SP-22, Curitiba, PR, Brazil), which related light absorbance with peroxide concentration of each sample, according to the Beer-Lambert law.

An aqueous solution containing aminophenazone (4-aminoantipyrine; 4 mmol/l), phenol (24 mmol/l), and peroxidase (0.4 U/mL) dissolved in 0.1 M phosphate buffer at pH 7.0 was used as the enzyme reagent. The reagents were stored at 4°C.[23] Before the analysis, a calibration curve with a standard hydrogen peroxide solution of known concentration was determined to obtain accurate measurement. The concentration of hydrogen peroxide solution was verified with potassium permanganate titration using a potentiometric titrator (HI 902, Hanna Instruments, Woonsocket, RI, USA). This curve is defined by absorbance values (optical density) of solution in relation to hydrogen peroxide concentration.

Participants were advised not to eat or drink for 2 hours before the analysis. During the analysis, they were instructed to swallow dry in order to collect whole saliva.[16] Thus, they should avoid swallowing consciously even when they desired to do it, since they needed to expectorate their saliva in graduated tubes during the study. At the beginning of the experiment, before insertion of the trays, participants expectorated saliva in the graduated tubes for 5 minutes. Then, the bleaching trays were inserted in the mouth, and saliva samples were collected again at 1, 5, 15, and 30 minutes.

After the tray was removed, the remaining gel was removed from tooth surfaces with a toothbrush without toothpaste for 30 seconds followed by expectoration; and the mouth was rinsed abundantly for 1 minute. Two minutes after the tray was removed, saliva samples were again collected. Subsequently, saliva was collected after 5 and 15 minutes, that is, 33, 38, and 48 minutes after the beginning of the procedure. For each analysis, 1000 μL of the collected saliva was added inside acryl-cuvettes (Sarstedt, Nümbrecht, Germany) with 1000 μL of enzymatic reagent, resulting in different

pinkish solutions, depending on the amount of peroxide present. After 48 minutes, total salivary flow was measured.

Comparison With Estimated Toxic Dose

This measurement was performed based on body weight (kg). Each participant was weighed to determine individual safe dose. The values were compared to safe daily dose, determined in previous studies (0.26 mg/kg/day).[15,16] To calculate the daily dose of peroxide, the values were expressed in milligrams of hydrogen peroxide per kilogram of body weight.

Statistical Analysis

To analyze data distribution, the Kolmogorov-Smirnov normality test was applied (α=0.05), and for homoscedasticity calculation, the Levene test was used (α=0.05). The relation between salivary flow and peroxide released in saliva was determined.

For comparison of peroxide presence in saliva and salivary flow, according to the bleaching systems, repeated measures analysis of variance and post hoc Tukey test were applied (5%); the bleaching treatments were the fixed factor, and the participants were the repeated factor. The relation between salivary flow and presence of peroxide in saliva was verified with the Pearson correlation test. The tests were performed using Statistica for Windows (StatSoft, Tulsa, Oklahoma, USA) and Graphpad Prism (Graphpad Prism Software, La Jolla, CA, USA).

RESULTS

There was no loss of participants or missed appointments during the study. The flow chart shows the distribution of participants among the groups (Figure 1). Eight participants were women and two were men, with average age of 27.2 ± 2.75 years. The average weight of participants was 57.0 ± 11.81 kg.

Peroxide Concentration in Saliva

Two-way repeated measures analysis of variance showed significant differences for bleaching system ($p<0.0001$) and time factors ($p=0.0003$) but not for interaction ($p=0.3121$). Hydrogen peroxide concentration in saliva was lower for CT-CP/OpalescencePF than for PT-HP/OpalescenceGo and CT-HP/White Class. When compared to the daily estimated toxic dose (0.26 mg/kg/d), the peroxide concentration in saliva was 64.83% lower for PT-HP/OpalescenceGo, 68.38% for CT-HP/White Class, and 72.97% for CT-CP/OpalescencePF (Figure 2). After tray removal, no peroxide was detected in saliva. Hydrogen peroxide concentration in saliva for each bleaching system compared with estimated toxic dose is shown in Figure 3.

Salivary Flow

Comparison of salivary flow before and after bleaching procedures showed significant differences for all groups ($p=0.0001$). Nevertheless, no differences were observed among bleaching systems at the same time ($p=0.0342$). The correlation between salivary flow and peroxide concentration was weak for all bleaching systems ($r=0.3775$ for PT-HP/OpalescenceGo; $r=0.3322$ for CT-HP/White Class; and $r=0.0162$ for CT-CP/OpalescencePF). Salivary flow increased by 42.38% for PT-HP/OpalescenceGo, 54.60% for CT-HP/White Class, and 54.34% for CT-CP/OpalescencePF from baseline to during treatment (Figure 4).

DISCUSSION

Considering the results, the first and second null hypotheses were rejected as there was a significant difference in presence of hydrogen peroxide in saliva among the bleaching systems, and salivary flow increased during tray use. The third null hypothesis was accepted because there was no significant difference in salivary flow during the study among the groups. The 10 participants received three different types of treatment as appropriate for a crossover study. The participants were 20% men and 80% women Although there is a discrepancy in participants' gender, the quality of saliva samples were not supposed to be affected as salivary flow and proteome profile are not influenced by sex.[24,25] This model allows for spectrophotometric analysis of peroxide in the same person, thereby decreasing the influence of variables related to oral environment such as pH, flow, and salivary content. The 1-week washout period was chosen to ensure that peroxide was eliminated from saliva and teeth, since it has been previously shown that residual oxygen may remain in the tooth structure after bleaching procedures.[26,27]

Considering possible toxicity caused by ingestion of peroxide,[2,12,18,28] this study evaluated the presence of hydrogen peroxide in saliva with a spectrophotometric method, which is considered accurate for this investigation.[21] This analysis was based on measuring light absorption by the dye generated by the reaction between hydrogen peroxide and the

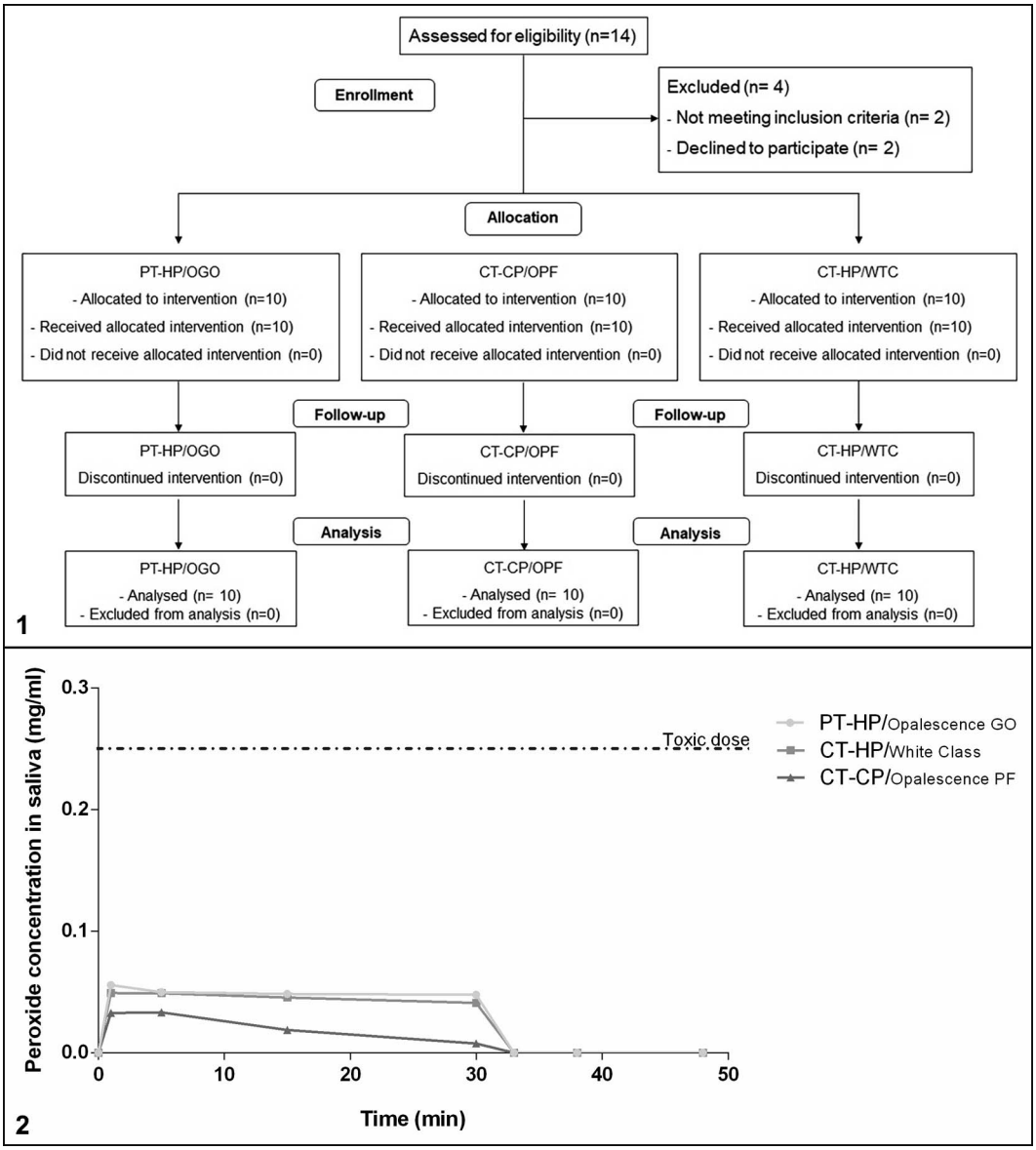

Figure 1. *Flow chart of the study (CONSORT).*

Figure 2. *Peroxide concentration in saliva in relation to time in comparison to toxic dose.*

chemical components of the solution, using an analytical spectrophotometer and considering the Beer-Lambert law. Thus, the light absorption by the molecules in a solution is directly proportional to their absorbance, path length (how long the light needs to overcome the solution) and concentration.[29,30]

The higher concentrations of peroxide in saliva were detected in bleaching systems with 10% hydrogen peroxide, despite type of tray used. Although in a lower concentration, hydrogen peroxide released by carbamide peroxide gel was also detected in the first minutes of the study, but it decreased during the analyzed period. It is important to highlight that after the tray removal, gel removal with toothbrush, and rinse, no peroxide was detected in saliva with all bleaching systems. This result was also observed in a previous study with 10% carbamide peroxide gels dispensed in customized trays.[16] The presence of peroxide in saliva was measured at 30 minutes because this is the time of use usually recommended by the manufacturers of 10% hydrogen peroxide gels.

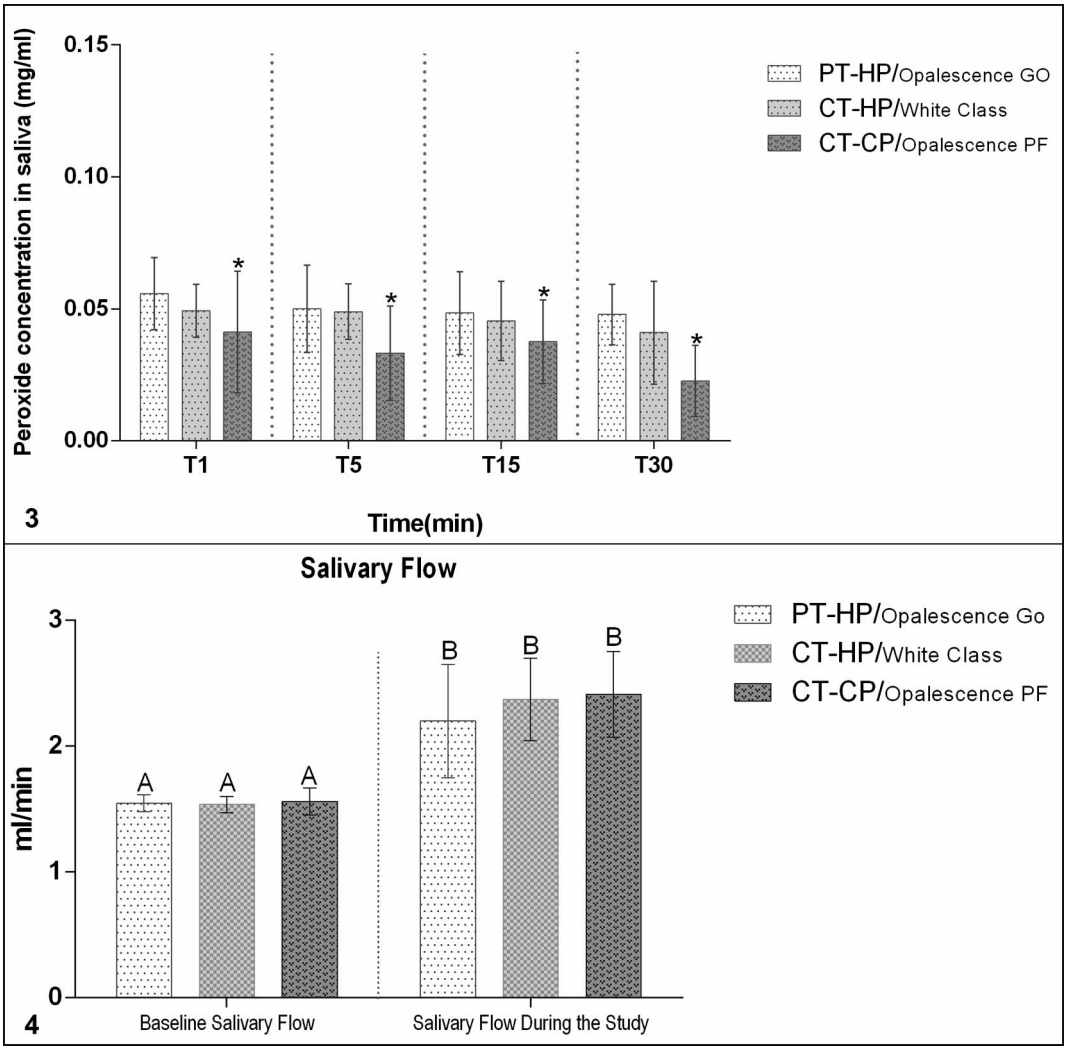

Figure 3. *Hydrogen peroxide concentration in saliva for each bleaching system according to time (asterisk shows significantly lower peroxide concentration in saliva inn each time,* $p<0.05$*).*

Figure 4. *Mean baseline salivary flow and salivary flow during the study (mL/min) for different bleaching systems and results of the Tukey test (different letters mean significant differences, 5%).*

Carbamide peroxide decomposes, generating approximately 3.5 parts of hydrogen peroxide and 6.5 parts of urea,[14] resulting in a real concentration of 3.5% of hydrogen peroxide versus the 10% hydrogen peroxide of the other two products tested. In this way, it was in fact expected that hydrogen peroxide detected in saliva would be lower than the 10% carbamide peroxide gel.

Human saliva is considered an effective defense against possible toxic effects due to the presence of enzymes such as peroxidase and catalase, which degrade peroxide.[14,16] Additionally, salivary flow can be influenced by several factors, including drugs, taste sensations, olfactory stimulus, circadian cycle, as well as movements and tactile stimulation of mucosa.[31] The trays are considered a tactile stimulation inside the mouth, and this is related to the overall 50% increase in salivary flow of participants in this study compared with initial salivary flow (before the use of trays). The relation between bleaching system and salivary flow was considered weak. Although prefilled trays are adaptable, they cannot accurately copy a patient's dental arch compared with customized trays; however, this did not influence salivary flow change in participants.

Even though peroxide was detected in participants' saliva, the peroxide concentration was considered much lower than the estimated toxic dose. Different protective mechanisms are related to peroxide degradation in organisms, such as oxida-

tion reaction by salivary peroxidases, catalase reaction from bacteria, consumption of peroxide due to its antimicrobial action, and the diffusion of peroxide through the tooth.[21] Bleaching gels also have different viscosities, which can favor or not the retention of gels inside the trays.[18] Nevertheless, bleaching systems containing the same hydrogen peroxide concentration did not show differences regarding the presence of peroxide in saliva, even with prefilled trays containing a higher amount of bleaching agent compared with customized trays. Although the prefilled tray is universally designed and presents a higher amount of bleaching gel, the gel is more viscous and is concentrated in the incisal/occlusal region of the tooth, which does not favor its overflow to saliva. Moreover, the tray extends beyond marginal gingiva and therefore acts as an external barrier, reducing the eventual contact between saliva and bleaching gel.

In this study, peroxide concentration in saliva was considered low and safe in relation to estimated toxic dose, even using at-home gels with higher concentrations of peroxide. Thus, at-home bleaching procedures are not expected to produce significant risk to human health under present conditions tested. Care should be taken to ensure these favorable results. When customized trays are used, they should be properly manufactured, and trimmed, and the adaptation to dental arches should be checked. Also, the patient must be oriented regarding the correct gel application, avoiding excessive amounts that may promote overflow. It is important to highlight that even though reservoirs may not interfere with bleaching efficacy and gel degradation,[19,32] when they are not present, more gel could be lost to the oral environment, thereby increasing the risk of peroxide contact with soft tissues, release to saliva, and, consequently, ingestion.[32] Additionally, patients must be instructed to rinse the mouth abundantly and expectorate after tray removal as our findings indicate no detectable presence of peroxide in saliva after these procedures.

Considering that the peroxide concentration in saliva within the tested parameters was extremely low, it can be inferred that the bleaching agent is retained within the tray, suggesting that the amount of gel does not decrease significantly during the time of use, maintaining the bioavailability of the bleaching agent to interact with the tooth surface without compromising bleaching efficacy. Indeed, in additional studies conducted by our research group, whitening efficacy was also not influenced by the type of tray used or the gel degradation pattern.[33,34]

CONCLUSIONS

Higher peroxide levels were detected in saliva with 10% hydrogen peroxide at-home bleaching gel compared with 10% carbamide peroxide gel, despite the type of tray used. Nevertheless, a sufficiently large margin of safety was observed with all bleaching systems tested compared with the estimated toxic dose. Salivary flow increased around 50% during tray use, independent of the bleaching system tested.

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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 Institute of Science and Technology committee for the protection of human participants. The approval code issued for this study is No. RBR-34F49C.

Conflict of Interest

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

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Laboratory Research

Influence of Different Cordless Light-emitting-diode Units and Battery Levels on Chemical, Mechanical, and Physical Properties of Composite Resin

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Clinical Relevance

Irradiance may decrease as the light-emitting diode (LED) is discharged. Therefore, the LED must be charged carefully to prevent the possibility of influencing the chemical, mechanical, and physical properties of composite resin.

SUMMARY

The aim of this study was to evaluate the influence of different light-emitting diode (LED) curing units and battery levels on the chemical, mechanical, and physical properties of composite resins. The irradiance for each cycle from full to completely discharged battery level was evaluated, for five different new cordless LED units: Optilight Color (Gnatus), Bluephase (Ivoclar), Valo (Ultradent), Radii Plus (SDI), and Radii Xpert (SDI). After the irradiance evaluation, composite resin specimens were prepared and light cured, while varying the battery level for each LED unit: high level (HL, 100%), medium level (ML, 50%), and low level (LL, 10%). The degree of conversion, diametral tensile strength, sorption, and solubility were also evaluated. Data were checked for homoscedasticity and submitted to two-way and three-way analysis of variance, depending on the test performed, followed by the Tukey test with a significance level of 95%. A negative correlation was found between irradiance and cycles of light curing, which was checked by the Pearson correlation test. Valo and Radii Xpert were not influenced by the battery level in any test performed. How-

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ever, different battery levels for some LED units can influence the degree of conversion, diametral tensile strength, sorption, and solubility of composite resins.

INTRODUCTION

Composite resins are widely used in clinical dentistry and have several indications for use, including direct restorations, sealants, inlays, onlays, crowns, luting agents, and orthodontic devices.[1] Composite resin restorations are esthetic, have good mechanical properties, and achieve success rates of up to 81.5% at 10 years.[1–3] The evolution of composite resins has been extensive over the past decades with regards to inorganic fillers, the organic matrix, and the form of activation.[4] The development of light-curing units (LCUs) was an additional milestone for adhesive dentistry, facilitating clinical practice, reducing working time, and improving the prognosis of restorations.[5]

Different LCUs can be used for light curing, which include quartz-tungsten halogen, plasma arc, argon laser, and light-emitting diode (LED), with LED being the most popular. LED curing units are currently the standard devices in most modern dental practices.[4] The light emitted by LCUs stimulates the initiator present in the composites and starts the polymerization reaction.[6,7] The polymerization reaction involves chemical reactions of organic oligomers to form a new polymeric material with an increase in molecular weight, resulting in the conversion of monomers into a polymer.[8] Thus, the light spectrum emitted by the LCU must stimulate the photoinitiator present in the restorative material.[6]

The most commonly used term to indicate the intensity of the light emitted by an LCU is *irradiance* (mW/cm^2), which is the value of the power divided by the area of the LCU tip.[9] Irradiance represents the light that reaches the target, not necessarily the light coming out of the LCU, and is maximized when the tip is pointing directly at the place to be polymerized.[10]

Composite resins need adequate light curing for maximum mechanical and aesthetic properties of the restorations, achieving a predictable clinical long-term outcome.[11,12] One of the most important factors related to the clinical performance of composites is the degree of conversion (DC), which is associated with excellent physical, mechanical, and biological properties of these composites. The DC may also be associated with irradiance, appropriate light spectrum, and the photoinitiator used.[13]

Even after light curing, composite resins remain unstable and can interact with the environment, indicating that these materials can absorb and release water and chemicals in the oral cavity.[14] This phenomenon is called *sorption and solubility*, and it can be a precursor of several physical and chemical processes that lead to deleterious effects on the mechanical properties of composite resins, which can cause functional damage and compromise the longevity of the restoration.[14,15]

For convenience, cordless LEDs are more commonly used than devices that need to be plugged in. Although a lithium battery is found in most cordless LCUs in dentistry, there are few studies regarding its influence on the performance of the resin materials. Previous studies have concluded that the battery level can influence the quality of restorations, showing that as some LED curing units are discharged, the irradiance also decreases, compromising the properties of the materials being cured.[16,17] However, it is still unclear what properties and how much the battery charge may be relevant to the final mechanical properties of the composite resin, considering that each device may behave differently.[18]

Thus, the aim of this study was to evaluate how different battery levels of different cordless LED curing units affect irradiance and its influence on the DC, diametral tensile strength, sorption, and solubility of a nanohybrid composite resin. The tested null hypothesis was that different battery levels and different cordless LED curing units do not influence the performance of the equipment and the properties of the composite resin.

METHODS AND MATERIALS

Irradiance Measurement

To determine the power percentage corresponding to each battery level, five new cordless LED curing units were selected: Optilight Color (GNATUS, Ribeirão Preto, Brazil), Bluephase N/G2 (Ivoclar Vivadent, Schaan, Liechtenstein), Valo (Ultradent, Salt Lake City, UT, USA), Radii Plus (SDI, Bayswater, Australia) and Radi Xpert (SDI). The curing units were fully charged as recommended by the manufacturer and used until completely unloaded. The maximum number of completed cycles of 20 seconds with a full battery (100%) was determined. Based on that data, the number of cycles corresponding to 50% and 10% battery levels was set.[16,19]

The higher power (mW) of the cordless LED units during the cycle was individually checked for all light cycles using a power meter (Nova, Ophir Spiricon, Logan, UT, USA), and was then divided by the tip area (cm^2), calculated from the optical diameter, as measured with a digital caliper (CD6 CS, Mitutoyo, Kanagawa, Japan), to obtain the irradiance (mW/cm^2).[7]

Irradiance as a function of battery discharge was measured in real time, and the radiant power output from each LCU was characterized over one full battery discharge cycle. Lights were used until either the battery ran out or the LCU ceased to function. The lights were allowed to rest for 30 seconds after each exposure to allow the units to cool down, and the operator had a 15-minute break after the 50th light exposure, and then after every hundred exposures, as needed.[16]

Specimen Preparation

Nanohybrid composite resin specimens (Aura, shade DC1, SDI) were prepared in a stainless-steel matrix (5 mm in diameter and 2 mm in depth) for all tests. Discs were light cured through a Mylar strip for 20 seconds using the cordless LED curing unit, with the center of the tip positioned in the center of the sample at a distance of 1 mm with different battery levels according to the experimental groups: high battery level (HL, 100%), medium battery level (ML, 50%), and low battery level (LL, 10%).

Degree of Conversion

The DC of the composite resin specimens (n=10) was evaluated using a Fourier transform infrared spectroscopy unit (FTIR) (Tensor 27, Bruker, Ettlingen, Germany) and measured on the bottom of the specimens. The number of remaining carbon double bonds was determined, which was calculated by comparing the percentage of aliphatic C=C (vinyl) (1638 cm^{-1}) and aromatic C=C absorption (1608 cm^{-1}) between cured and uncured specimens. The spectra of the cured and uncured specimens were obtained using 32 scans at a resolution of 4 cm^{-1}, within the range from 1000 to 6000 cm^{-1}. The spectra were subtracted from the background spectra using the software provided with the FTIR unit (OMNIC 6.1, Nicolet Instrument Corp, Madison, WI, USA). The acquired spectra were expanded and analyzed in the region of interest from 1560 to 1670 cm^{-1}. The DC was calculated using the standard baseline technique and a comparison between the peak area at 1639 cm^{-1} (aliphatic C=C) and the internal standard peak at 1609 cm^{-1} (aromatic C=C). Then, the DC was calculated using the following equation: DC (%) = [1 – (Cured aliphatic / Aromatic ratio) / (Uncured aliphatic / Aromatic ratio)] × 100.

Sorption and Solubility

The sorption and solubility of the composite resin were verified for each experimental group using new specimens (n=10). After preparation, the specimens were stored in a desiccator with silica gel and maintained in an oven at 37°C for 24 hours. Then, the specimens were weighed on an analytical balance with 0.01 mg accuracy (AG200, Gehaka, São Paulo, Brazil) at 24-hour intervals until a constant weight was obtained, which was considered *m1*. After the weighing procedures, the specimens were immersed in an artificial saliva medium and kept in an oven at 37°C. After 7 days,[20] the specimens were removed from storage, the excess liquid was removed using absorbent papers, and the specimens were weighed to obtain *m2*. Afterward, the specimens were taken to the desiccator with silica gel at 37°C to eliminate the absorbed saliva and were weighed daily until reaching a constant mass, which was considered *m3*. The major and minor diameters and thickness of the specimens were measured at four points using a digital caliper (CD6 CS, Mitutoyo) after the final drying for *m*1. These measures were used to obtain the volume (V) of each specimen (mm^3) and to calculate the sorption (Sor) and solubility (Sol) rates, based on the following formulae: $Sor = (m2 - m3)/V$ and $Sol = (m1 - m3)/V$; where *m1* is the mass of the specimen (μg) before the immersion in liquid medium, *m2* is the mass of the specimen (μg) after immersion in liquid medium over 7 days, *m3* is the mass of the specimen (μg) after desiccation until reaching constant mass, and V is the volume of the specimen (mm^3).[21]

Diametral Tensile Strength

A diametral tensile strength test was performed on the specimens before obtaining DC (n=10) and sorption and solubility (n=10) using a mechanical testing machine (DL 2000, EMIC, São José dos Pinhais, Brazil). Specimens subjected to sorption and solubility testing were compared with samples that were not subjected to any treatment. Specimens were positioned vertically on a testing machine between a stainless steel flat tip and a base. Then, a compressive load was applied vertically on the lateral portion of the cylinder at a crosshead speed of 0.5 mm/min, producing tensile stresses perpendicular to the vertical plane and passing through the

Table 1: *Tip Diameter, Power (mW), and Irradiance (mW/cm^2) for Different Battery Levels*

	Bluephase	Optilight Color	Raddi Plus	Valo	Radii Xpert
Tip diameter (cm)	0.93	0.70	0.60	0.95	0.78
Tip area (cm^2)	0.67	0.38	0.28	0.7	0.48
Battery level power (mW)					
HL (100%)	1237	1054	490	1103	769
ML (50%)	1182	644	487	1054	755
LL (10%)	1159	528	485	1054	744
Irradiance (mW/cm^2)					
HL (100%)	1846	2773	1750	1575	1611
ML (50%)	1749	1694	1739	1505	1581
LL (10%)	1731	1381	1732	1505	1557

Abbreviations: HL, high level; LL, low level; ML, medium level.

center of the specimen, until failure. After each compressive test, the fracture load (F) was recorded in newtons (N), and the diametral tensile strength (σt) was calculated (MPa) as follows: $\sigma t = 2F / \pi dh$, where, d is the diameter (5 mm) of specimens, h is the height (2 mm) of specimens, and the constant π is 3.1416.

Statistical Analyses

The data were checked for homoscedasticity and submitted to analysis of variance (ANOVA) testing; The influence of battery level and LCU on water sorption, solubility, DC, and interaction between them were analyzed using two-way ANOVA; the influence of battery level, LCU, subjecting the specimens to water sorption and solubility challenge, and interaction between them were analyzed using three-way ANOVA. Both forms of ANOVA testing were submitted to the Tukey test. Correlations between irradiance and cycles were checked using the Pearson correlation test. SigmaPlot software (SigmaPlot 12.0, Systat Software, San, Jose, CA, USA) was used to conduct the tests, with a significance level set at 95%.

RESULTS

The power and irradiance of each LED curing unit are shown in Table 1. The results for DC, sorption, solubility, and diametral tensile strength are presented in Tables 2 through 5, respectively. There was a negative correlation between irradiance and cycles of light curing (Figure 1). The LEDs lasted 190 (Valo), 191 (Bluephase), 447 (Radii Xpert), 528 (Optilight Color), and 642 (Radii Plus) cycles of 20 seconds until they were completely discharged.

For DC testing, both the LCU ($p \leq 0.001$) and battery level ($p=0.010$) showed statistical differences. Optilight Color and Radii Plus were significantly influenced by the battery level, with the LL group presenting significantly inferior results (53.21% and 49.28%) compared with the HL (57.93% and 53.23%) and ML (54.93% and 52.17%) groups, which presented similar results. Optilight Color presented the highest statistical performance in the HL group. In the LL group, Valo presented similar results to Optilight Color and Radii Plus (Table 2).

In the sorption test, there was a significant difference in the battery level only for Optilight Color ($p=0.003$). Optilight Color at HL presented significantly better performance compared with ML and LL. In the comparison between devices, Bluephase presented significantly superior sorption results at all battery levels (Table 3).

For the solubility results, Valo and Radii Xpert showed no influence based on the battery level. There were no differences among the five LED

Table 2: *Mean ± Standard Deviation for the Degree of Conversion (%) of Composite Resins With Different Light-emitting Diodes at Different Battery Levels (n=10)*[a]

	Optilight Color	Bluephase	Valo	Radii Plus	Radii Xpert
HL (100%)	57.93 ± 2.98 Aa	51.39 ± 3.75 Ab	52.92 ± 3.17 Ab	53.23 ± 2.80 Ab	52.89 ± 3.81 Ab
ML (50%)	54.93 ± 2.92 ABa	52.29 ± 2.75 Aa	51.73 ± 5.15 Aa	52.17 ± 3.22 ABa	52.17 ± 3.20 Aa
LL (10%)	53.21 ± 2.65 Ba	48.95 ± 2.5 Ab	52.85 ± 2.47 Aab	49.28 ± 2.21 Bab	50.56 ± 6.23 Aab

Abbreviations: HL, high level; LL, low level; ML, medium level.
[a] *Different letters represent significant differences ($p<0.05$). Capital letters compare battery level (column), and lowercase letters compare devices (row).*

Table 3: *Mean ± Standard Deviation for Sorption (μg) of Composite Resins With Different Light-emitting Diodes at Different Battery Levels (n=10)*[a]

	Optilight Color	Bluephase	Valo	Radii Plus	Radii Xpert
HL (100%)	4.68 ± 6.71 Bc	29.72 ± 8.36 Aa	14.26 ± 7.84 Ab	15.02 ± 3.47 Ab	11.68 ± 4.92 Abc
ML (50%)	12.75 ± 4.23 Ab	33.57 ± 7.65 Aa	19.55 ± 8.81 Ab	12.75 ± 7.10 Ab	13.23 ± 6.54 Ab
LL (10%)	17.01 ± 6.83 Ab	33.16 ± 7.40 Aa	17.55 ± 8.31 Ab	12.21 ± 4.13 Ab	18.12 ± 6.00 Ab

Abbreviations: HL, high level; LL, low level; ML, medium level.
[a] *Different letters represent significant differences (p<0.05). Uppercase letters compare battery level (column), and lowercase letters compare devices (row).*

curing units in the HL and ML groups. In LL, Radii Plus was significantly superior to Valo, which was similar to Bluephase and significantly superior to Optilight Color (Table 4).

The sorption and solubility challenge did not influence the result of the diametral tensile strength analysis ($p = 0.871$). Only Optilight Color was influenced by the battery level, while also presenting higher results than the other groups for HL (46.66 and 46.09 MPa). In ML, Valo presented higher values (41.41 and 42.26 MPa) than the other groups, which were similar to each other. In LL, Optilight Color and Valo were significantly superior to the other groups, which were similar to each other (Table 5).

DISCUSSION

The battery level and the different LED curing units influenced the irradiance, DC, diametral tensile strength, sorption, and solubility of the composite resin. Thus, the null hypothesis was rejected.

The light curing of dental materials has facilitated clinical practice and is used worldwide; it is uncommon for an office to not have an LCU.[22] Several studies have related the light emitted by LCUs with the properties of the composite resin[13,23–25]; however, few studies have related the influence of the battery charge of cordless LCUs with the properties of composite resins.[16,19] Based on the prevalence of cordless LCUs, verifying their operation at full power has become even more important. The use of an LCU with a partially or an almost completely discharged battery may result in incomplete and unsatisfactory polymerization of restorative materials.[26] Although most devices use a lithium battery, the LCUs may behave differently as their batteries are discharged.[27]

The properties of composite resin are dependent on several factors, including composition, insertion technique, thickness, and method of light curing.[28,29] During polymerization of the composite, it is ideal for a greater quantity of monomers to be converted into polymeric chains.[30] Polymerization involves a free-radical reaction in which the state of a material is transformed from viscous to rigid. During this process for composite resins, the terminal aliphatic C=C bonds are broken and converted to primary C-C covalent bonds between methacrylate monomers. As polymerization progresses, however, the diffusion rate of the propagating free radicals reduces. Thus, monomer conversion is not complete at the end of the reaction; therefore, part of the monomers remain as pendant double bonds or trapped unreacted monomers.[31]

The DC is directly related to the physical and mechanical properties of a composite resin.[32] Lower DC is related to impaired mechanical properties, greater discoloration, and degradation, resulting in reduced strength and longevity.[33,34] The conversion readings of this study were performed on the bottom surface of the samples in order to evaluate the DC in the region that received less irradiance, which has a lower conversion of monomers into polymers.[35]

Table 4: *Mean ± Standard Deviation for Solubility (μg) of Composite Resins With Different Light-emitting Diodes at Different Battery Levels (n=10)*[a]

	Optilight Color	Bluephase	Valo	Radii Plus	Radii Xpert
HL (100%)	8.51 ± 5.77 Ba	4.93 ± 8.3 Ba	4.94 ± 8.85 Aa	12.65 ± 1.27 Aa	9.38 ± 6.63 Aa
ML (50%)	10.1 ± 6.98 Ba	9.44 ± 8.12 ABa	10.55 ± 9.63 Aa	13.03 ± 1.30 Aa	12.78 ± 8.24 Aa
LL (10%)	21.32 ± 5.81 Aa	13.7 ± 5.52 Aab	9.99 ± 7.72 Ab	−9.56 ± 7.08 Bc	15.54 ± 4.99 Aab

Abbreviations: HL, high level; LL, low level; ML, medium level.
[a] *Different letters represent significant differences (p<0.05). Uppercase letters compare battery level (column), and lowercase letters compare devices (row).*

Table 5: *Mean ± Standard Deviation for the Diametral Tensile Strength (MPa) of Composite Resins With Different Light-emitting Diodes at Different Battery Levels (n=10)*[a]

	Optilight Color		Bluephase		Valo	
	N/SS	SS	N/SS	SS	N/SS	SS
HL (100%)	46.66 ± 5.34 Aa	46.09 ± 4.89 Aa	33.03 ± 5.13 Ac	33.77 ± 3.98 Ac	40.02 ± .65 Ab	42.13 ± 3.10 Ab
ML (50%)	38.84 ± 3.03 Bb	37.58 ± 4.95 Bb	31.74 ± 2.56 Ac	30.88 ± 2.62 Ac	41.41 ± 4.53 Aa	42.26 ± 3.08 Aa
LL (10%)	40.34 ± 2.81 Ba	37.69 ± 3.27 Ba	32.20 ± 3.78 Ab	32.98 ± 3.69 Ab	39.49 ± 40.07 Aa	39.17 ± 2.62 Aa

Abbreviations: HL, high level; LL, low level; ML, medium level; N/SS, group that did not pass the sorption and solubility challenge; SS, group that passed the sorption and solubility challenge,
[a] *Different letters represent significant differences ($p<0.05$). Uppercase letters compare battery level (column), lowercase letters compare the devices (row). There was no significant difference between the SS and N/SS groups.*

The distance from the tip and the thickness of the resin, especially when greater than 2 mm, considerably reduces the irradiance[6] and may influence the properties of the resin, according to the DC, strength, sorption, and solubility results of this study. Therefore, the irradiance was found to influence the DC of composite resins.[37]

The ISO 10650:2015 standard for measuring the output from dental LCUs recommends using a laboratory-grade power meter to measure the total radiant power output.[38] This power value is then divided by the tip area to produce an irradiance value for the light. Therefore, it is possible to increase the irradiance in two ways: by increasing the power or reducing the area of the tip. The irradiance value can only represent an average output value across the light tip.[27] The light output from many LCUs is not uniform, and some areas of the tip may contain points of very high irradiance, while others have practically no light, but the only value considered and disclosed is the average. Ideally, the beam profile should be homogeneous; however, the central part of the tip of most LCUs has a higher irradiance and the peripheral areas have a reduced irradiance.[9,39,40] In an LED curing unit with an irradiance of 868 mW/cm^2, the tip will likely present about 40% of the area emitting less than 500 mW/cm^2, while the central region presents more than 2500 mW/cm^2.[7]

This study used a new device from each manufacturer. Although all LCUs in the present study featured a lithium battery, with the exception of Valo, which uses an iron phosphate lithium battery, the present results cannot be extrapolated to all LCUs, even though most cordless LCUs use a lithium battery, as the performance of each product may be different.[19]

In the oral environment, composite resins can absorb water and other substances from saliva, food, and drinks; which may have some influence on their degradation.[14] Sorption is influenced by the polarity of the molecular structure, the presence of hydroxyl groups capable of forming hydrogen bonds with water, and the degree of cross linking in the continuous matrix.[41] Water sorption is associated with solubility, which is the release of residual products as unreacted monomers. These products alter the matrix microstructure, creating voids suitable for the formation of microfractures.[42,43] Sorption and solubility in composite resins have a significant effect on the clinical success of restorative materials, influencing the esthetic appearance, integrity, and surface properties.[41] During and after the polymerization process, free monomers remain present in the restorative material and can detach from the composite resin and be incorporated in the oral fluids.[44,45] The level of battery and different LEDs can influence the sorption and solubility, as shown in the results of this study. The negative value of the Radii Plus in the LL group of the solubility test can be explained by the fact that the fluid absorbed during the sorption process is confined and included as part of the polymeric structure of the material.[19,46]

Valo and Radii Xpert were not influenced by battery level in any test and presented satisfactory results compared with the other devices. The Optilight Color presented excellent results in the tests, but it was most influenced by the battery level; even with the irradiance and test results decreasing as the battery was discharged, it showed good results even at the 10% battery level. Bluephase and Valo emitted greater light power, therefore, the number of cycles until the battery was totally discharged was smaller compared with other groups that used less power. While Valo and Bluephase were discharged at about 190 cycles, Radii Plus required 642 20-second cycles.

Table 5: *Mean ± Standard Deviation for the Diametral Tensile Strength (MPa) of Composite Resins With Different Light-emitting Diodes at Different Battery Levels (n=10)[a] (ext.)*

	Radii Plus		Radii Xpert	
	N/SS	SS	N/SS	SS
HL (100%)	36.52 ± 3.26 Ac	35.87 ± 3.47 Ac	34.15 ± 4.01 Ac	33.00 ± 2.85 Ac
ML (50%)	34.50 ± 3.67 Abc	36.63 ± 3.36 Abc	33.03 ± 4.02 Ac	32.10 ± 2.11 Ac
LL (10%)	33.61 ± 4.40 Ab	34.98 ± 3.17 Ab	32.57 ± 2.44 Ab	32.65 ± 2.66 Ab

A previous study evaluated the influence of the battery level of the LED curing unit Coltolux (Coltene, Feldwiesenstrasse, Switzerland) on the DC, sorption, solubility, and diametral tensile strength. The lowest battery level (10%) negatively affected all analyzed mechanical properties.[19] Another study found a decrease in radiant exposure of Bluephase (~4%), which was similar to the present study (~6%).[16] On the other hand, no influence of the battery level on the irradiance emitted by Bluephase was found in another study.[18] The difference between these two results may be explained by the way irradiance was measured (Radiometer, Kerr, Orange, CA, USA), which was not performed according to the specifications of ISO 10650: 2015.[38]

Another factor that may influence the DC and the other mechanical properties is the light spectrum emitted by the LED and the light spectrum that stimulates the initiators present in the composite resin.[47] Valo and Bluephase are polywave LCUs, delivering a light output with two or more wavelength peaks, which has the possibility of stimulating initiators with a different activating wavelength from camphorquinone (468 nm).[48] The manufacturer of the composite resin tested in this study did not provide information about which initiator is present.

This study was laboratory-based and has certain limitations. The LED curing units tested are from internationally renowned companies in dentistry, therefore, lower-quality LCUs may be detrimental to restorations.[7] In clinical practice, the deleterious effects should actually be worse because the distance from the tip of the LCU to the resin was minimal in the present study, separated only by a Mylar strip. Results tend to get worse when increasing the distance to the tip, reaching up to less than 25% of the irradiance emitted by the LED at only 9 mm of distance.[10]

In the present study, the analyzed samples had a 5-mm diameter and all LED tips were larger in diameter even LEDs with smaller tips like Radii Plus, Radii Xpert and Optilight Color. For devices that do not have a homogeneous beam profile, the center provides more irradiance compared with the periphery of the tip, which may have influenced the results of the study and the performance of the devices.[9] The DC was tested only at the center of the samples; therefore, LEDs that do not have a homogeneous distribution were favored in this experiment. On the other hand, the resistance test was less influenced because it is relevant that the polymerization occurs in the whole sample for better resistance.

Another limitation was found regarding the oral environment. During the process of restoration, restorative materials may be in close contact with adjacent gingival tissue, saliva, and gingival fluid before polymerization is complete, which may last about 24 hours.[49,50] The samples tested in the present study were immersed in artificial saliva 24 hours after light curing, which is different from what happens in the oral environment. Clinically, the oral environment is subjected to different kinds of foods, beverages, and pH changes due to disorders such as bulimia or gastroesophageal reflux disease; alternatively, the samples in the current study were only submitted to an artificial saliva challenge, meaning that the clinical results will likely be different.[51]

Therefore, clinicians should know the features of LED curing units and select the best LCU possible for their clinic. The battery level may influence the properties of the composite resin; therefore, the LCU should be fully charged to avoid any failures. The beam profile may not be homogeneous throughout the tip area; therefore, photopolymerization should be carried out in more than one location to ensure that the entire area receives sufficient irradiance to obtain optimum physical and mechanical properties. Further clinical and laboratory studies should be performed with different devices and different light-activated materials to prove the

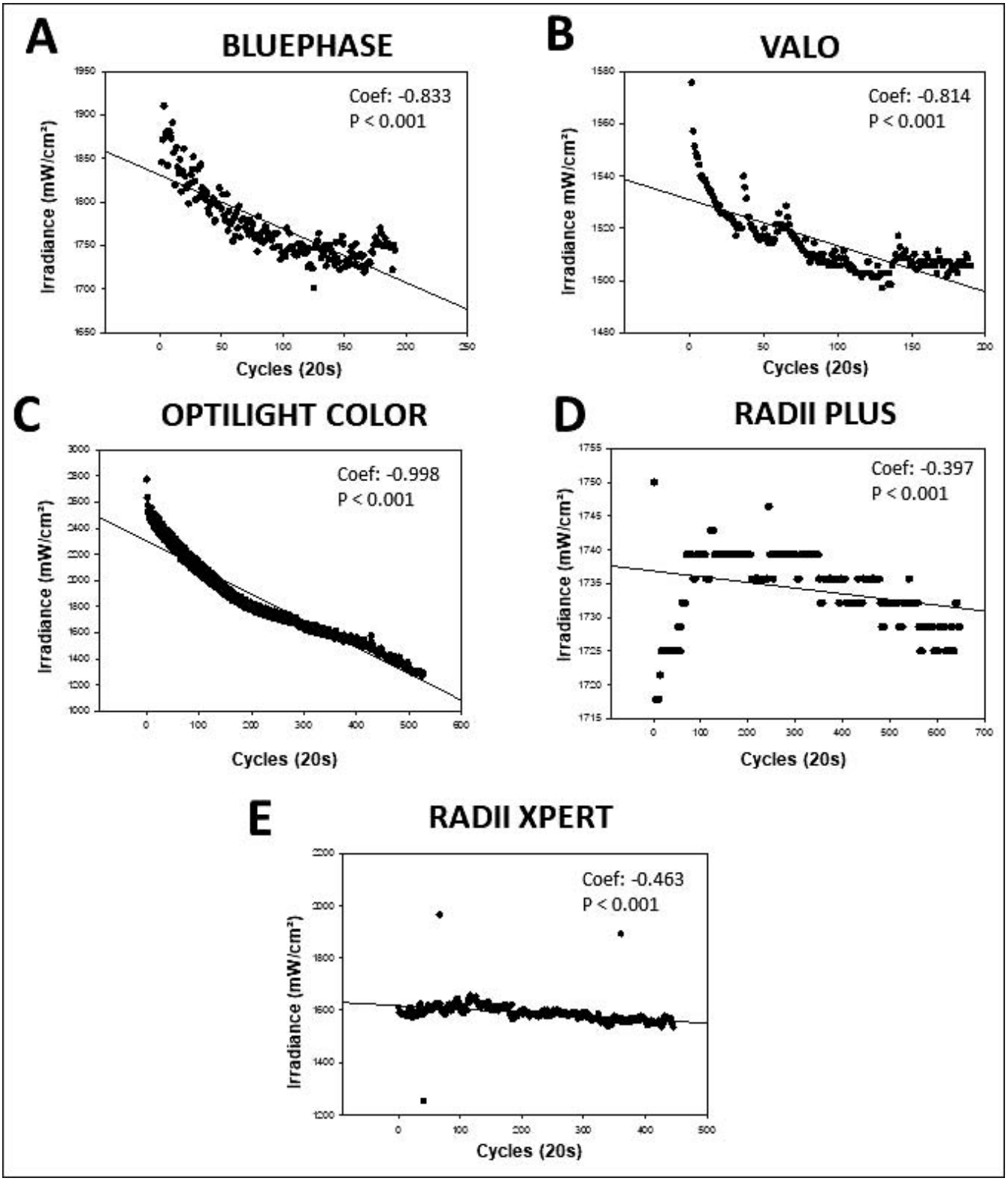

Figure 1. *Correlation graphs of irradiance (mW/cm^2) and cycles (20 seconds) of LCU. (A): Bluephase, (B): Valo, (C): Optilight Color, (D): Radii Plus, and (E): Radii Xpert.*

influence of the LCU and battery level on the properties of these materials.

CONCLUSION

Within the limitations of the present study, some LEDs at different battery levels were found to significantly decrease the DC, diametral tensile strength, sorption, and solubility of composite resins. Valo and Radii Xpert were not influenced by the battery levels in any test performed.

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 Preheating and Fatiguing on Mechanical Properties of Bulk-fill and Conventional Composite Resin

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Clinical Relevance

Bulk-fill composite resins may have comparable mechanical properties to conventional composite resin. Preheating does not reduce the mechanical properties of composite resins.

SUMMARY

Statement of Problem: Bulk-fill composite resins are increasingly used for direct restorations. Preheating high-viscosity versions of these composites has been advocated to increase flowability and adaptability. It is not known what changes preheating may cause on the mechanical properties of these composite resins. Moreover, the mechanical properties of these composites after mastication simulation is lacking.

Purpose: The purpose of this study was to evaluate the effect of fatiguing and preheating on the mechanical properties of bulk-fill composite resin in comparison to its conventional counterpart.

Methods and Materials: One hundred eighty specimens of Filtek One Bulk Fill Restorative (FOBR; Bulk-Fill, 3M ESPE) and Filtek Supreme Ultra (FSU; Conventional, 3M ESPE) were prepared for each of the following tests: fracture toughness (International Organization for Standardization, ISO 6872), diametral tensile strength (No. 27 of ANSI/ADA), flexural strength, and elastic modulus (ISO Standard 4049). Specimens in the preheated group were heated to 68°C for 10 minutes and in the fatiguing group were cyclically loaded and thermocycled for 600,000 cycles and then tested. Two-/one-way analysis of variance followed by Tukey Honest Significant Difference (HSD) *post hoc* test was used to analyze data for statistical significance (α=0.05).

Results: Preheating and fatiguing had a significant effect on the properties of both FSU and FOBR. Fracture toughness increased for FOBR specimens when preheated and decreased when fatigued (p=0.016). FOBR had higher fracture toughness value than FSU. Diametral tensile strength decreased significantly after fatiguing for FSU (p=0.0001). FOBR had a lower diametral tensile strength baseline value compared with FSU (p=0.004). Fatiguing

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significantly reduced the flexural strength of both FSU and FOBR (p=0.011). Preheating had no effect on the flexural strength of either FSU or FOBR. Preheating and fatiguing significantly decreased the elastic modulus of both composite resins equally (p>0.05).

Conclusions: Preheating and fatiguing influenced the mechanical properties of composite resins. Both composites displayed similar mechanical properties. Preheating did not yield a major negative effect on their mechanical properties; the clinical implications are yet to be determined.

INTRODUCTION

Composite resin was introduced to the dental world by Rafael Bowen in 1957.[1] Composite resins have changed the way dentistry is practiced and have become one of the most important dental materials. Approximately 261 million composite resin restorations are placed around the world each year.[2] However, polymerization shrinkage and its resultant shrinkage stress is one of the major shortcomings of conventional composite resin.[3] The resultant polymerization shrinkage stress manifests clinically with several clinical complications such as cusp deflection, micro-cracking and fracture of enamel margins, microleakage, debonding, postoperative sensitivity, and pulpal irritation.[4-13]

To reduce shrinkage stresses, an incremental technique for composite resin placement was introduced. This technique has insured proper light polymerization of the resin composite and reduced the polymerization shrinkage stress.[14,15] However, incremental placement of conventional composite resin is a technique sensitive procedure that requires clinical skills and special instruments, and it is also time consuming. As a result, less technique-sensitive and more efficient approaches for composite resin placement were required.

Manufacturers realized desirability of a material that is simply and rapidly placed with reduced polymerization shrinkage. Their efforts resulted in the introduction of bulk-fill composite resins, a wide range of materials that can be placed and photopolymerized in a single layer of 4-8 mm thickness. Several changes in the chemistry of monomers, particle size, and shape were required to allow those materials to be used in bulk. Bulk-fill composite resins can be classified into low-viscosity and high-viscosity materials. Low-viscosity bulk-fills are meant to serve as dentin replacement and therefore need to be capped with conventional materials due to their poor physical and mechanical properties.[16] High-viscosity bulk-fill materials on the other hand have good physical and mechanical properties that allow them to restore an entire cavity without the need for a capping layer.[17,18]

Laboratory testing of the mechanical properties of composite resin materials is a common method for determining their properties. The clinical relevancy of those tests is established to some extent.[19] Mechanical properties testing can help identify materials with a high likelihood of premature failure due to fracture and their wear characteristics. Fatiguing of composite resins prior to mechanical testing by cyclic loading and simultaneous thermocycling is highly recommended to increase the clinical relevance of the results.[20] Chewing simulation is one of the methods that can be used to fatigue specimens at a reasonable cost and time.

There are multiple protocols and techniques that dentists use when they place composite resins in their practice. Preheating composite resin is a relatively common technique that is thought to increase flowability and reduce film thickness.[21] Preheating composite resin may maximize polymerization, reduce shrinkage forces, and increase surface hardness.[22-24] However, this effect on mechanical properties, wear, and clinical performance has yet to be investigated.

The purpose of the present study was to test the effect of fatiguing and preheating on the mechanical properties of a high-viscosity bulk-fill composite resin and compare it to its conventional counterpart. The null hypotheses were that fatiguing and preheating yield no significant effect on the mechanical properties of the tested materials. Also, there is no significant difference in the mechanical properties between high-viscosity bulk-fill and conventional composite resin.

METHODS AND MATERIALS

Two composite resin types were used in this study (Table 1): Filtek One Bulk Fill Restorative (FOBFR; 3M ESPE, St Paul, MN, USA) and Filtek Supreme Ultra (FSU; 3M ESPE).

Specimen Distribution and Group Descriptions

Specimens (N=180) were prepared and distributed into six groups (n=10) for each test performed: group 1, FOBR baseline; group 2, FSU baseline; group 3,

Table 1: *Composite Resins Used in This Study*

Product	Type	Manufacturer	Matrix Composition	Filler Type	Filler Size (nm)	Filler Load (weight %)
Filtek Supreme Ultra, A2	Conventional nanofilled composite resin	3M ESPE	Bis-GMA, UDMA, TEGDMA, bis-EMA	Ytterbium trifluoride, nonaggregated silica, nonaggregated zirconia, zirconia/silica clusters	4-20	78.5
Filtek One Bulk Fill Restorative,A2	Bulk-fill nanofilled composite resin	3M ESPE	AFM, AUDMA, UDMA, and DDDMA	Ytterbium trifluoride, nonaggregated silica, nonaggregated zirconia, zirconia/silica clusters	4-20	76.5

Abbreviations: AFM, addition fragmentation monomer; AUDMA, aromatic urethane dimethacrylate; bis-EMA, Ethoxylated bisphenol-A dimethacrylate; bis-GMA, bisphenol-A glycidyldimethacrylate; DDDMA, 1, 12-Dodecanediol dimethacrylate; TEGDMA, Triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

FOBR preheated; group 4, FSU preheated; group 5: FOBR fatigued; group 6, FSU fatigued.

Specimen preparation and photo-polymerization were done according to manufacturer's instructions at room temperature (23±1°C), except for groups 3 and 4 that were preheated to a temperature of (68±1°C) for 10 minutes before photo-polymerization by using a composite warmer (HeatSync, Bioclear, Seattle, WA, USA).

Specimens of groups 5 and 6 were subjected to fatiguing. This was achieved by cyclic loading the specimens under 50 N load with a steatite antagonist using a chewing simulator (CS-4.8, SD Mechatronik, Feldkirchen-Westerham, Germany) for 600,000 cycles at 1.4 Hz. Thermocycling was simultaneously performed using distilled water at 5°C and 55°C with a 30-second dwell time. Specimens were inspected for premature failure every 12 hours. Each chamber was supplied with a sensor that indicated if a specimen were to fracture at a specific cycle number. Specimens were tested for their mechanical properties.

Fracture Toughness

The single edge V-notched beam (SEVNB) method (ISO 6872)[25] was used to measure the fracture toughness of FOBFR and FSU composite resins. Sixty beam-shaped specimens 21.0 ± 0.1 mm in length, a rectangular cross section of 4.0 ± 0.1 mm in depth, and 3.0 ± 0.1 mm in thickness were prepared for each of the composite resins. Polyvinylsiloxane impression material (PVS) molds were created to the exact dimensions for easy removal after photo-polymerization. Composite resin material was injected into the mold preventing the entrapment of air bubbles. A transparent ethylene film and glass slide were placed with slight pressure over the mold to confine the material and minimize exposure to oxygen from the atmosphere during photo-polymerization. Each specimen was photo-polymerized according to the manufacturer's recommended time of exposure using a visible light curing unit (Elipar DeepCure-S, 3M ESPE, St. Paul, MN USA) with a useable wavelength range of 420-490 nm and mean light irradiance of 1168 mW/cm^2. The wavelength and irradiance of the curing unit were calibrated and confirmed using the MARC Light Collector (BlueLight Analytics, Halifax, Canada). After completion of photo-polymerization, the specimens were examined and any containing voids/defects were excluded from testing. A #15 blade was used to remove excess composite resin from the edges, and a 600-grit silicon-carbide abrasive paper (MicroCut, Buehler, Lake Bluff, IL, USA) was used for final smoothing. Specimens were then stored in deionized water at 37°C for 24 hours. The width (b) and thickness (w) of each specimen were recorded prior to testing using a digital micrometer capable of measurements to ±1 μm accuracy (QuantuMike Micrometer, Mitutoyo Corporation, Kawasaki, Japan). A notch depth of approximately 0.5 mm was cut into the bar specimen using a 150 μm-thick diamond blade. A razor blade impregnated with diamond polishing paste (3.5 μm, Kent Supplies, New York, NY, USA) was positioned in the notch, and a light force (5-10 N) was applied using a gentle back and forth horizontal motion while maintaining a constant pressure. The depths of the V-shaped notches were measured and confirmed from both sides with a calibrated microscope. An acceptable notch depth measured between 0.8 and 1.2 mm. For groups 5 and 6, the V-shaped notch was made after the fatiguing process.

Fracture toughness testing was performed using a four-point bending fixture. The 3-mm-width face with the V-notch was placed down on the testing fixture (tensile side). Specimens were loaded on an Instron Universal Testing Machine (Instron 4411,

Instron, Norwood, MA, USA) with a crosshead speed of 0.5 mm/min until fracture.

The peak fracture load was recorded to three significant figures, and the fracture toughness [K_{IC} (MPa×$m^{1/2}$)] was determined according to the following formula:

$$K_{IC} = F/bw^{½} * L/w * 3\alpha^{½}/2(1-\alpha)^{3/2} * Y;$$

$$Y = 1.9887 - (1.326 * \alpha) - (3.49 - 0.68 * \alpha + 1.35 * \alpha^2) * (\alpha) * (1-\alpha)/(1+\alpha)^2$$

where K_{IC} is the fracture toughness (MPa×$m^{1/2}$); α = average V-notch depth of group; F = fracture load; b = width of specimen; w = thickness of specimen; L = distance between support beams; and Y is the stress intensity shape factor.

Diametral Tensile Strength

The diametral tensile strength (DTS) of FOBFR and FSU was determined under specification (No. 27 of ANSI/ADA, 1993).[26] Sixty cylindrical shaped specimens (6.0±0.1 mm diameter and 3.0±0.1 mm height) were prepared for each composite resin material according to a similar methodology previously described. Specimens were immersed in water at 37°C for 24 hours prior to testing. The cylindrical-shaped specimens were positioned on their side between two compression plate fixtures (Instron 4411, SINTECH, MTS System Corporation). Specimens were loaded at a crosshead speed of 0.5 mm/min until fracture. For groups 5 and 6, specimens were fatigued as described previously prior to loading. The peak load was recorded, and the DTS was determined according to the following formula:

$$DTS = 2F/\pi dt,$$

where F = maximum force applied; d = diameter of specimen; and t = thickness of specimen.

Flexural Strength and Young's Elastic Modulus

The flexural strength of FOBFR and FSU was determined by a three-point bending test according to ISO Standard 4049.[27] Sixty specimens (2.0±0.1 mm thickness, 2.0±0.1 mm width, and 25.0±0.1 mm length) were prepared for each composite resin material according to the previously described methodology. Testing was performed using the three-point bending fixture (Instron 4411, SINTECH, MTS System Corporation) with a crosshead speed of 0.5 mm/min. For groups 5 and 6, specimens were fatigued as described previously prior to loading. Flexural strength was determined according to the following formula:

$$\alpha = 3FL/2wt,$$

where F = maximum force applied; L = distance between support beams; w = width of specimen; and t = thickness of specimen.

The elastic modulus (Young's modulus) was determined using Test Works software (SINTECH, MTS System Corporation, Shakopee, MN, USA). The software required the dimensions of each specimen prior to starting the test. After fracture, the software yielded an elastic modulus based on the dimensions and flexibility of the specimen.

Statistical analysis was performed using analysis of variance (ANOVA) and Tukey's multiple comparison tests for pairwise comparisons at $p<0.05$ and 95% CIs to test significant differences in fracture toughness, flexural strength, elastic modulus, and DTS. The Statistical Package for the Social Sciences (SPSS) version 23.0 (SPSS/IBM, Armonk, NY, USA) was used for statistical analysis.

RESULTS

Fracture Toughness

Mean and SD values of fracture toughness for each group are summarized in Table 2. Preheating and fatiguing had no significant effect on the fracture toughness value of FSU ($p>0.05$). For FOBR, preheating and fatiguing had a significant effect on the fracture toughness value ($p=0.016$). Preheating increased fracture toughness, whereas fatiguing decreased fracture toughness (Figure 1). A pairwise comparison between FSU and FOBR for baseline, preheated, and fatigued showed that FOBR has significantly better fracture toughness values for all conditions ($p<0.05$).

DTS

Mean and SD values of DTS for each group are summarized in Table 3. In the FSU groups, preheating had no significant effect on the DTS ($p>0.05$), whereas fatiguing had a significant effect on DTS ($p=0.0001$; Figure 2). For FOBR, preheating and fatiguing had no significant effect on the DTS ($p>0.05$). A pairwise comparison between FSU and FOBR for baseline, preheated, and fatigued showed that FSU had a significantly higher DTS value for baseline only ($p=0.004$).

Table 2: *Mean and SD of Fracture Toughness (MPa × $m^{1/2}$) of Both Composite Resin Types*[a]

Composite Resin	Baseline	Preheated	Fatigued
Filtek Supreme Ultra	1.53 ± 0.21	1.57 ± 0.13	1.57 ± 0.11
Filtek One Bulk Fill Restorative	1.78 ± 0.13 A	1.94 ± 0.16 Ab	1.66 ± 0.07 Aa

[a] *Uppercase letters indicate statistical significance between types of composite resin (p>0.05). Lowercase letters indicate statistical significance within the same composite resin (p>0.05).*

Flexural Strength

Mean and SD values of flexural strength for each group are summarized in Table 4. For FSU and FOBR, preheating had no significant effect on flexural strength ($p>0.05$), whereas fatiguing had a significant effect on flexural strength ($p=0.011$; Figure 3). A pairwise comparison between FSU and FOBR for baseline, preheated, and fatigued showed that FSU had significantly higher flexural strength in the fatigued group ($p<0.05$), whereas FOBR had significantly higher flexural strength in the preheated group ($p=0.045$).

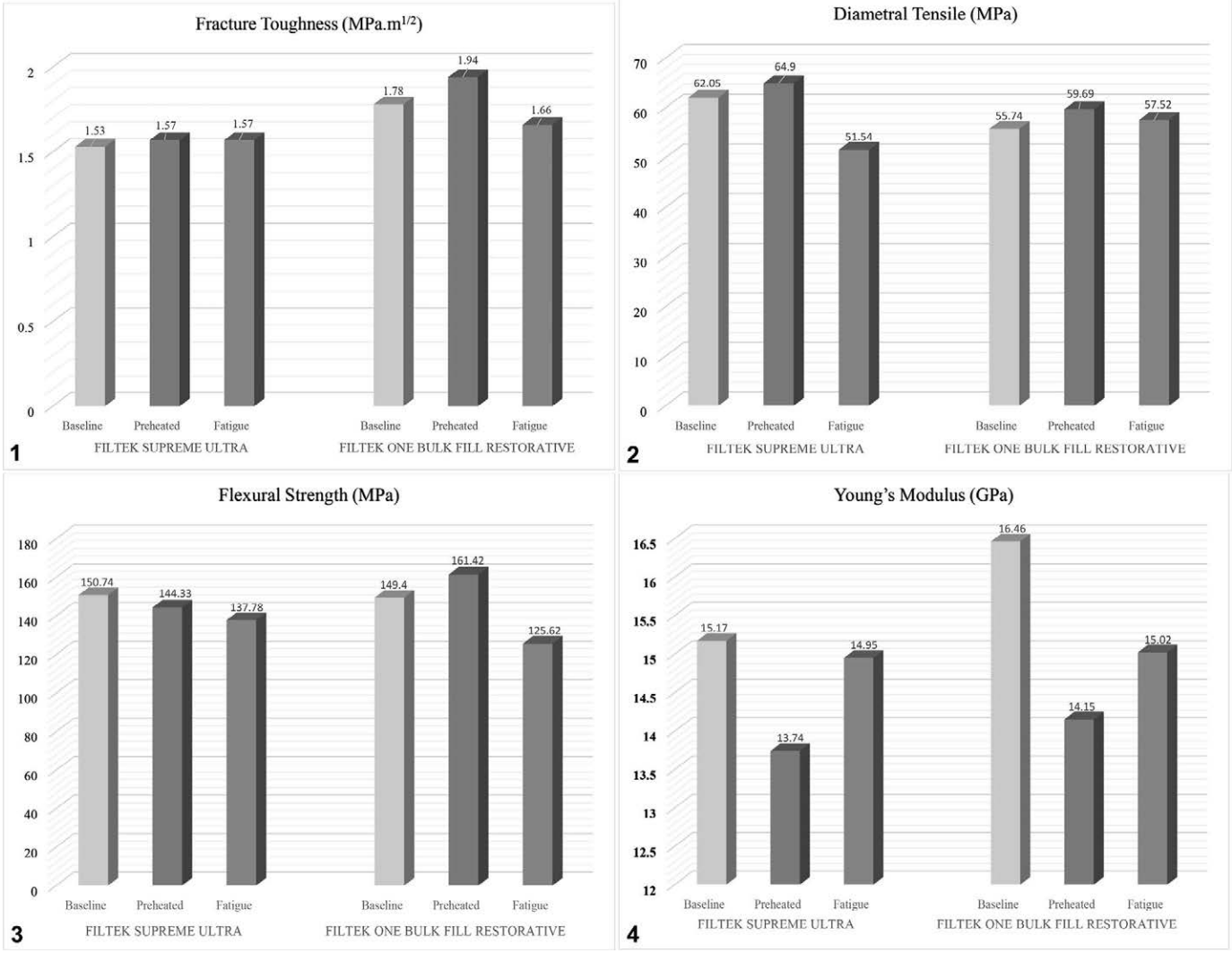

Figure 1. *Mean of fracture toughness of each composite resin type.*
Figure 2. *Mean of diametral tensile strength each composite resin type.*
Figure 3. *Mean of flexural strength each composite resin type.*
Figure 4. *Mean of Young's modulus each composite resin type.*

Table 3: *Mean and SD of Diametral Tensile Strength (MPa) of Both Composite Resin Types*[a]

Composite Resin	Baseline	Preheated	Fatigued
Filtek Supreme Ultra	62.05 ± 5.06 A	64.90 ± 7.74	51.54 ± 7.80 a
Filtek One Bulk Fill Restorative	55.74 ± 3.34	59.69 ± 6.70	57.52 ± 5.08

[a] *Uppercase letters indicate statistical significance between types of composite resin (p>0.05). Lowercase letters indicate statistical significance within the same composite resin (p>0.05).*

Young's Elastic Modulus

Mean and SD values of Young's elastic modulus for each group are summarized in Table 5. Regarding FSU, preheating and fatiguing had a significant effect on Young's modulus ($p<0.05$). For FOBR, preheating and fatiguing had a significant effect on Young's modulus ($p<0.05$; Figure 4). A pairwise comparison between FSU and FOBR for baseline, preheated, and fatigued showed that there is no significant difference in Young's elastic modulus between the two composite resin types ($p>0.05$).

DISCUSSION

The effect of preheating and fatiguing on the mechanical properties of two composite resins, bulk-fill and conventional, was tested in this *in vitro* study. The first null hypothesis was rejected because both preheating and fatiguing yielded a statistically significant difference on some of the mechanical properties of the two tested composite resins. The second null hypothesis was also rejected because there was a statistically significant difference in the mechanical properties between bulk-fill and conventional composite resins. Some differences were found at the baseline and others were found in the preheating and fatiguing groups.

Preheating is a common practice to increase flowability and reduce stickiness of the composite resins. Preheating is especially useful for composite resins with a high percentage of inorganic filler particles that are highly viscous. It has been shown that preheating these composites ensures better adaptation to cavity walls.[28] The two composites that were chosen for this study are highly filled and viscous. The FOBR has 76.5 wt% filler load, whereas FSU has 78.5 wt% filler load. FSU served as a control, and it can be used for the same applications that are indicated for FOBR.

Composite resin restorations may fail over time due to the accumulation of damage produced by cyclic forces (fatiguing).[19] Therefore, laboratory fatiguing of composite resins prior to testing is a valid technique to increase the clinical relevancy of the generated results.[20] This can be done by using simulators that cyclically load specimens with simultaneous thermocycling, in an effort to emulate the intraoral challenge. Hence, both materials were fatigued for 600,000 cycles, which is equivalent to 2.5 years of clinical performance.[19] Loading parameters included a 0.5-mm indentation with steatite indenter, vertical movement, 1.4 Hz, and a load of 49 N. These parameters were selected after multiple pilot tests to determine the best parameters. All samples survived the fatiguing challenge in this study.

Fracture toughness is considered the most important mechanical property in determining resistance to fracture because almost all materials contain flaws.[29] In this study, preheating increased the fracture toughness of both FOBR and FSU, with fracture toughness values of FOBR significantly higher. A possible explanation for this may be due to the different monomer of FOBR (Table 1) that may enhance a better degree of conversion after preheating. Daronch and others[23] clearly showed that preheating significantly enhanced monomer to polymer conversion of composite resin due to increasing mobility of the monomer and filler particles. The enhanced polymerization yielded an increase in the fracture toughness. Fatiguing, on the other hand, had no significant effect on the fracture toughness of FSU, but it decreased the fracture toughness of FOBR. Fatiguing with thermocycling may have led to filler particle loss and surface changes that

Table 4: *Mean and SD of Flexural Strength (MPa) of Both Composite Resin Types*[a]

Composite Resin	Baseline	Preheated	Fatigued
Filtek Supreme Ultra	150.74 ± 11.52	144.33 ± 7.00	137.78 ± 7.27 aA
Filtek One Bulk Fill Restorative	149.40 ± 13.66	161.42 ± 4.40 A	125.62 ± 16.28 a

[a] *Uppercase letters indicate statistical significance between types of composite resin (p>0.05). Lowercase letters indicate statistical significance within the same composite resin (p>0.05).*

Table 5: *Mean and SD of Elastic Modulus (GPa) of Both Composite Resin Types*[a]

Composite Resin	Baseline	Preheated	Fatigued
Filtek Supreme Ultra	15.17 ± 0.73	13.74 ± 1.35 b	14.95 ± 0.60 a
Filtek One Bulk Fill Restorative	16.46 ± 1.43	14.15 ± 2.03 b	15.02 ± 1.05 a

[a] *Uppercase letters indicate statistical significance between types of composite resin ($p>0.05$). Lowercase letters indicate statistical significance within the same composite resin ($p>0.05$).*

decreased fracture toughness. Those changes can be due to chemical breakdown by hydrolysis, stress-induced effects, and chemical composition changes by leaching or loss of strength due to corrosion.[30] Because fracture toughness is a function of microstructure, differences in chemical composition may explain why fatiguing decreased the fracture toughness of FOBR but not that of FSU. Baseline fracture toughness values of both composite types were comparable to other studies.[31,32] Tiba and others[33] investigated the fracture toughness of a range of bulk-fill composite resin materials ranging from 0.8 to 1.7 MPa $\times$ $m^{1/2}$. To the authors' knowledge, no published study has tested the effect of preheating and fatiguing on this property.

Many clinical failures of composite resin restorations are related to tensile stress. The DTS test, which is an indirect method to assess tensile strength, was performed in this study. Baseline DTS of FSU was significantly higher than that of FOBR. Schliebe and others[34] had a similar result in their study where the conventional composite resin had higher DTS than its bulk-fill counterpart. Preheating increased the DTS for both composite resins, but it was not statistically significant. This finding was similar to what was found by Nada and others.[35] This can be attributed to the enhanced polymerization of preheated composite resins. Fatiguing had a significant effect reducing the DTS of FSU (51.54 MPa). No other published studies were found that tested the effect of fatiguing on DTS.

Flexural strength and elastic modulus are two important qualities of composite resin materials. For the material to withstand masticatory forces, it should offer sufficient flexural strength to allow the material to resist fracture. The elastic modulus determines the stiffness of the material. The evaluated composites in all groups had adequate flexural strength according to ISO 4049-2009, which requires a value of at least 80 MPa. The flexural strength of both composites was not affected by preheating but was decreased by fatiguing. The results of this study are in agreement with the findings of Uctasli and others, who concluded that preheating had no effect on flexural strength of composite resins.[36] Although the clinical significance of this is unknown, fatiguing these composite resins seems to affect their mechanical properties. The literature that describes the effect of fatiguing is scarce, but it can be speculated that the microstructure is affected when those materials are fatigued. There was no difference in elastic modulus between the two composites (baseline). Preheating and fatiguing significantly reduced the elastic modulus of both FSU and FOBR. However, the clinical significance of this reduction is unknown, and data cannot be extrapolated to clinical outcomes without highlighting deficiencies in this method, such as flaw distribution and structural reliability of the material.[37,38] Nonetheless, this method is recommended by ISO 4049 for polymer-based materials and is applied for comparative purposes.

A limitation of this study is that only one brand of composite resin material was tested. Thus, the results of study cannot be extrapolated to other brands of composite resin. It is well known that not all brands are similar. Different brands of composite resin offer a wide range of materials with various chemical formulations that may result in different mechanical properties. Another limitation of the study is that the specimens used in this study are flat following ISO standards and do not have anatomical geometry that simulates the clinical scenario. Also, testing at different temperatures, and its effect on the mechanical properties, was not investigated and is encouraged for future research. This laboratory study does not substitute the need for well-conducted randomized controlled clinical trials.

Within the limitations of this laboratory study, the following conclusions were drawn:

- preheating had no major negative effect on the mechanical properties of composite resins;
- laboratory fatiguing yields useful information by emulating the intraoral challenge and predicting the effect of that on the properties of composite resins;

- conventional and bulk-fill composite resins have minimal difference in their mechanical properties; and
- the clinical implication of changes in the mechanical properties yielded by preheating is yet to be determined.

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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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Depth-dependence of Degree of Conversion and Microhardness for Dual-cure and Light-cure Composites

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Clinical Relevance

New dual-cure bulk-fill composites show promise for uniform degree of conversion and microhardness throughout the entire depth of direct restorations.

SUMMARY

Objective: The aim of this study was to evaluate the degree of conversion (DC) through micro-Raman spectroscopy and surface microhardness in Vickers hardness (VHN) of three new dual-cure bulk-fill resin-based composites (RBCs) compared with light-cure bulk-fill and incremental RBCs at two clinically relevant depths and for two light irradiation times.

Methods: Three commercially available restorative dual-cure bulk-fill RBCs (BulkEZ, HyperFIL, and Injectafil) were evaluated and compared with three light-cure RBCs (Filtek Bulk Fill Flowable, Filtek One Bulk Fill, and incremental Filtek Z250) as controls. Specimens were prepared in two different depths (0.5 mm and 5 mm) and were light irradiated for 20 seconds or 40 seconds. Self-cure was also evaluated for the three dual-cure bulk-fill RBCs. Micro-Raman spectroscopic measurements and VHN tests (n=5) were made after 24 hours of dry storage in the dark at room temperature for all test conditions. Data were analyzed using one-way and two-way analyses of variance (α=0.05).

Results: All tested RBCs showed significantly higher DC and VHN values at 0.5-mm depth than at 5-mm depth, with the exception of BulkEZ, which showed similar DC and VHN values at two depths. The three dual-cure bulk-fill RBCs showed significantly higher DC than the three light-cure RBCs under the same curing condition. The three dual-cure RBCs showed much smaller differences in VHN values between the two depths than the three light-cure RBCs. Twenty seconds and 40 seconds of light irradiation did not generate significant difference in DC and VHN values for the three dual-cure bulk-fill RBCs at either depth or for the three light-cure RBCs at the 0.5-mm depth; however, 40 seconds of light irradiation generated significantly higher DC and VHN values for One Bulk Fill and Z250 at the 5-mm depth compared with 20 seconds of light irradiation.

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INTRODUCTION

Most dental resin-based composites (RBCs) are based on methacrylate resins, and the polymerization process is usually activated by visible light. A key way to quantify the effectiveness of polymerization is the degree of conversion (DC), which measures the percentage of carbon-carbon double bond (C=C) to carbon-carbon single bond (C-C) conversion to form the polymeric network. A high DC is vital to enhance the mechanical properties, chemical stability, and longevity of the restoration.[1,2] Incomplete monomer conversion not only causes premature failure of restorations and secondary caries[3–5] but also results in monomer elution, which is suggested to be responsible for undesirable biological responses, such as cytotoxicity and pulp tissue inflammation.[6–8] It has been shown that DC and other properties of light-cured RBCs depend on several factors: power and intensity of the light source, time and distance of irradiation, amount and particle size of the filler, ratio between the organic and inorganic components, and initiator systems.[9–13] Clinically, the most common strategy to maximize DC is to provide sufficient energy to the system by increasing curing time.[14] Several studies focusing on commercially available composites have emphasized the need to apply at least 20 seconds, but more likely 40 seconds, of light irradiation to minimize the amount of eluted monomers, even with the use of high irradiance light-emitting diode (LED) lights.[15–17]

Conventional dental RBCs are applied in increments of 2-mm thickness to allow sufficient photopolymerization.[18,19] However, this is time consuming and inconvenient, especially for deep posterior cavities. The demand for a faster and simpler restorative procedure led to the development of bulk-fill RBCs that can be placed in a single layer of up to 4-5 mm.[20] The improved depth of cure is usually achieved through greater translucency of the material, increased photoinitiator content, or an additional photoinitiator type.[13,21] Even with improved formulations, these light-cure bulk-fill RBCs can still suffer from unsatisfactory polymerization at deep layers due to impeded access or light attenuation,[22] especially for deep cavities where the distance from the top of the highest cusp to the cavity floor can reach up to 8 mm. The presence of very deep cavities has led to the development of chemical-cure RBCs that use chemical initiators to activate the polymerization reaction by mixing two components together.[23] The chemical-curing process avoids the problem of insufficient photopolymerization in deep layers and enables bulk filling in one layer. One of the disadvantages of chemical-cure materials is their fixed working time intervals: a very short one does not allow the practitioner to perfectly place the RBC material in the cavity; conversely, a longer working time interval is more time consuming and not clinically feasible.

To solve these problems, manufacturers introduced dual-cure resins. These products contain both light-cure and chemical-cure components: the light-cure component provides rapid, initial hardening of the top layers of the resins, stabilizing the restorations; then, the deeper layers that receive insufficient light irradiation are polymerized by the slower chemical-curing reaction. Some advantages of dual-cure resins as restorative materials include the possibility of a bulk insertion, clinical time saving, the achievement of polymerization in deep areas due to chemical curing, and the development of lower shrinkage stresses.[24]

There has been great interest in the curing processes of dual-cure resins. The impact of light irradiation on the chemical-curing mechanism of dual-cure resins has not been well established. Previous studies have suggested that it was necessary to light cure dual-cure resins in order to obtain an optimal polymerization of the materials, and when limited to chemical curing or inadequate light exposure, dual-cure resins may not obtain maximum mechanical properties.[25–29]

Manufacturers recently launched new dual-cure bulk-fill RBC products designed for direct restorations, such as BulkEZ (Zest Dental Solutions, Carlsbad, CA, USA), HyperFIL (Parkell Inc, Brentwood, NY, USA), and Injectafil DC (Apex Dental Materials Inc, Lake Zurich, IL, USA). Manufacturers claim that these products have unlimited depth of cure and other desirable properties, such as elimination of flowable liners, better cavity adaptation, and reduced shrinkage stress due to slower chemical curing. Very limited research is available assessing the polymerization and mechanical properties of these dual-cure bulk-fill RBCs. The aim of the present study was to evaluate the DC and Vickers hardness (VHN) values of the three new dual-cure bulk-fill RBCs in comparison with three light-cure RBCs Filtek Bulk Fill Flowable (FBF) , Filtek One Bulk Fill (FOBF) and incremental Filtek Z250 (Z250), all from 3M ESPE Dental Products (St. Paul, MN, USA), at two different depths (0.5 mm and 5 mm) for two different light irradiation times (20 seconds and 40 seconds). The null hypotheses were that 1) there would be no difference in the DC and VHN values among the investigated RBCs, 2)

Table 1: *Chemical Compositions of the Six RBCs*

RBC (Manufacturer)	Shade	Classification	Resin	Filler Content	Particle Size
BulkEZ (Danville Materials)	A2	Dual-cure, bulk-fill RBC	BisEMA, TEGDMA, BisGMA, and UDMA	Barium glass, 50-70 wt%; YbF3, 1-20 wt%	Proprietary
HyperFIL (Parkell, Inc)	universal	Dual-cure, bulk-fill RBC	BisEMA, UDMA, and other dimethacrylate monomers.	Barium glass/silica, 75 wt%	15 nm-3.5 μm
Injectafil (Apex Dental Materials, Inc)	A2	Dual-cure, bulk-fill, RBC	Bis-GMA and other methacrylate resins	Silica glass, 75 wt%	Submicron to 5 μm
FBF (3M ESPE)	A2	Light-cure, bulk-fill RBC	Bis-GMA, UDMA, Bis-EMA, and Procrylat resin	YbF3 and zirconia/silica, 64.5 wt%	0.1-5.0 μm
FOBF (3M ESPE)	A2	Light-cure, bulk-fill RBC	AUDMA, UDMA, diurethane-DMA, and DDDMA	YbF3 and zirconia/silica, 76.5 wt%	4-100 nm and clusters
Z250 (3M ESPE)	A2	Light-cure, incremental filling RBC	Bis-GMA, UDMA, and Bis-EMA resin	Zirconia/silica, 82 wt%	0.01-3.5 μm, Average 0.6 μm

Abbreviations: AUDMA, aromatic dimethacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate ; Bis-EMA, ethoxylated bisphenol-A dimethacrylate; DDDMA, 1,12-dodecanediol dimethacrylate; DMA, dimethacrylate ; FBF, Filtek Bulk Fill Flowable; FOBF, Filtek One Bulk Fill; RBC, resin-based composite; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; YbF3, ytterbium trifluoride; Z250, incremental Filtek Z250.

specimen depth would have no effect on the DC and VHN values of each individual RBC, and 3) light irradiation time would have no effect on the DC and VHN values of each individual RBC.

METHODS AND MATERIALS

Six commercially available restorative dental RBCs were evaluated, including three dual-cure bulk-fill RBCs (BulkEZ, HyperFIL, and Injectafil) and three light-cure RBCs (FBF, FOBF and Z250). The chemical compositions of the six RBCs are listed in Table 1.

Specimen Preparation

Specimens were prepared in two depths (0.5 mm and 5 mm). Commercially available spacers were used as molds for specimen preparation. The 5 mm specimens were prepared in 5 mm × 6.3 mm black spacers (Model 13ME069, Grainger, Lake Forest, IL, USA), and the 0.5 mm specimens were prepared in 0.5 mm × 24 mm black spacers (Model BB-24MM-0.5, Walmart, Bentonville, AR, USA). All the specimens were prepared according to the manufacturers' instructions in a dark room at room temperature. An LED light-curing unit (Elipar S10, 3M ESPE, Seefeld/Germany) with manufacturer rated power density of 1200 mW/cm^2 was used for specimen irradiation. Uncured RBCs were filled into the molds in one increment. The specimen was immediately covered with an unbreakable plastic cover slip (Catalog No. 12-547, Fisher Scientific, Pittsburgh, PA, USA) and light-cured for 20 seconds or 40 seconds with the tip of the curing light in contact with the cover slip. The actual irradiant power density across the cover slip was measured to be 960 mW/cm^2. No light irradiation was used for the self-cure condition. All specimens were stored in the dark at room temperature for 24 hours before measurements. Five specimen replicates were used for each test condition (n=5) and three random locations were measured on each specimen surface.

Micro-Raman Spectroscopy

A LabRam HR 800 Raman spectrometer (Horiba Jobin Yvon, Edison, NJ, USA) with a monochromatic He-Ne laser (632.8 nm) and operating at excitation power of 20 mW was used to collect Raman spectra of the specimens. It was equipped with a confocal microscope (Olympus BX41, Tokyo, Japan), a piezo-electric XYZ stage with a minimum step width of 50 nm, and an air-cooled charge-coupled device detector of 1024 × 256 pixels. The following parameters were used for the Raman spectroscopy: 1800 grating, 400-μm confocal hole, and 150-μm slit width. Spectra were Raman-shift-frequency calibrated with the known peak of silicon at 520.7 cm^{-1}.

Specimen surfaces were scanned through a 100× Olympus objective. To obtain spectra of the uncured materials, the RBCs were placed on the surface of a microscope slide and were immediately scanned before polymerization happened. Raman spectra were acquired over the spectral region of 1350 to 1800 cm^{-1}

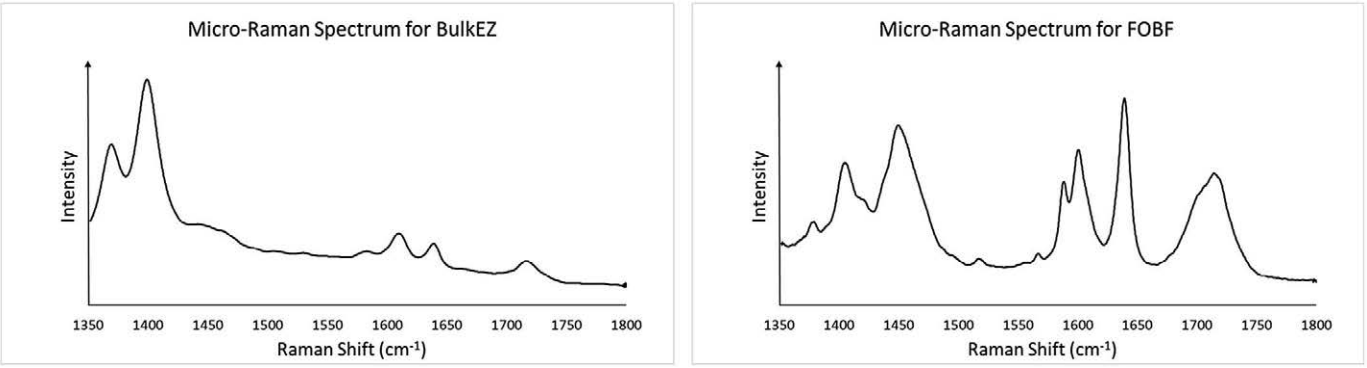

Figure 1. *Representative micro-Raman spectra for BulkEZ (left) and FOBF (right).*

with an acquisition time of 100 seconds. Five specimens with three random locations on each specimen surface were measured for each test condition. Labspec 5 software (Horiba Jobin Yvon) was used to analyze the acquired Raman spectral data. The Raman spectra were processed through cosmic spike removal, spectral smoothing, and two-point baseline adjustment. The Raman peaks at 1450, 1609, and 1637 cm^{-1} were fitted with Gaussian-Lorentzian shapes, and the maximum peak heights at those positions were measured for DC calculation.

Degree of Conversion Calculation

For RBCs containing bisphenol A-glycidyl methacrylate (Bis-GMA)/ethoxylated bisphenol A dimethacrylate (Bis-EMA) (all but FOBF), the peak of 1609 cm^{-1} (aromatic carbon double bonds) was used as an internal reference for normalization. The resin monomer content of FOBF does not contain Bis-GMA or Bis-EMA and therefore lacks the 1609 cm^{-1} peak. For FOBF, the alternative peak of 1450 cm^{-1} (C-H) was used as the internal reference for normalization (Figure 1).

The DC% was calculated by the reduction of normalized peak height at 1637 cm^{-1} (aliphatic carbon=carbon) between uncured and cured material using the following Equation (1):

$$\mathrm{DC}\% = \left\{1 - \frac{(1637\ \mathrm{cm}^{-1}/1609 \text{ or } 1450\ \mathrm{cm}^{-1})_{\text{Peak height before cure}}}{(1637\ \mathrm{cm}^{-1}/1609 \text{ or } 1450\ \mathrm{cm}^{-1})_{\text{Peak height after cure}}}\right\} \times 100.$$

The DC values from a total of 15 measurements of five specimens were used to calculate the mean and standard deviation of DC% for each test condition.

Vickers Hardness Test

The same specimen surfaces analyzed using confocal Raman spectroscopy were also used to determine their VHN values. A microhardness tester (MO Tukon, Wilson Instrument Division, Bridgeport, CT, USA) with a pyramidal diamond indenter was used to indent the specimen surface with a constant load of 100 gf for 15 seconds of dwell time. Once the load was removed, the length of the two indentation diagonals were measured using the microscope of the tester, and the VHN values were calculated automatically by the tester using the following formula:

$$\mathrm{VHN} = 0.102\,\frac{2F\sin\left(\frac{\alpha}{2}\right)}{d^2},$$

where F is the applied load (N), α is apex angle of the indenter (136°), and d is the average length of the diagonal (mm). Three random measurements were made for each specimen surface. The VHNs from a total of 15 measurements of five specimens were used to calculate the mean and standard deviation of VHN for each test condition.

Scanning Electron Microscopy (SEM)

Following VHN testing, specimens were polished using a 0.25-μm diamond polishing compound (Metadi, Buehler USA, Lake Bluff, IL, USA) on a spinning polisher (Buehler USA). Then the polished specimens were coated with a Pd-Au alloy in a sputtering machine (Leica EM SCD050, North Ryde, Australia). The coated specimens were placed inside a field emission scanning electron microscope (FEI-XL30, ESEM-FEG, FEI Company, Hillsboro, OR, USA) using a 15 kV electron beam at a working distance of 10 mm. SEM images were taken at 2000× magnification using a back-scatter detector to assess filler size and distribution for all six RBCs.

Table 2: *DC% Mean (Standard Deviation) for All Test Conditions*

	Composite and Irradiation Time											
	BulkEZ		HyperFIL		Injectafil		FBF		FOBF		Z250	
	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s
Depth, mm												
0.5	74.39 (1.58)	73.03 (0.73)	79.93 (1.41)	78.29 (1.59)	76.35 (0.84)	77.18 (1.04)	60.18 (1.25)	61.21 (1.37)	55.03 (4.22)	55.45 (3.75)	62.06 (1.11)	62.37 (1.27)
5	73.50 (0.65)	72.42 (0.70)	75.14 (1.12)	74.87 (1.77)	70.23 (1.58)	68.05 (1.56)	55.54 (2.34)	56.79 (1.03)	41.34 (4.39)	49.03 (3.25)	44.83 (2.55)	51.95 (3.84)
Self-cure	72.91 (0.65)		74.69 (0.60)		69.4 (1.43)		N/A		N/A		N/A	

Abbreviations: FBF, Filtek Bulk Fill Flowable; FOBF, Filtek One Bulk Fill; Z250, incremental Filtek Z250; N/A, not available.

Statistical Analysis

Statistical analyses were performed using Microsoft Excel 2016 (Microsoft, Redmond, WA, USA) and GraphPad Prism 5 (version 5, GraphPad Software, Inc, San Diego, CA, USA). Normality of data distribution was verified by the Kolmogorov-Smirnov test. All DC and VHN data were analyzed using an unpaired Student *t*-test, and one-way or two-way analysis of variance (ANOVA) with Bonferroni posttests for multiple comparisons ($\alpha=0.05$). Pearson correlation was used for DC and VHN correlation analysis.

RESULTS

Degree of Conversion

The numeric DC results for all test conditions are summarized in Table 2, and the bar chart is illustrated in Figure 2.

Analysis of the DC values of each individual dual-cure bulk-fill RBC at two depths and in the self-cure condition showed that for BulkEZ, the DC values were not significantly different among the two depths and self-cure conditions for either irradiation time ($p>0.05$); for HyperFIL and Injectafil, the DC values were not significantly different between 5 mm and self-cure ($p>0.05$), but they were both significantly lower than the DC values at 0.5 mm ($p<0.001$) for either irradiation time. For all light-cure RBCs, the DC values at 0.5 mm were significantly higher than those at 5 mm for either irradiation time ($p<0.005$). The ratio of mean DC value at 5 mm to mean DC value at 0.5 mm for 20 seconds of irradiation was 0.99 for BulkEZ, 0.94 for HyperFIL, 0.92 for Injectafil, 0.92 for FBF, 0.75 for FOBF, and 0.72 for Z250; the ratio for 40 seconds of irradiation was 0.99 for BulkEZ, 0.96 for HyperFIL, 0.88 for Injectafil, 0.93 for FBF, 0.88 for FOBF, and 0.83 for Z250.

Comparisons of the DC values between the 20 second and 40 second irradiation times at the same depth for each individual RBC using an unpaired Student *t*-test showed that the two different irradiation times did not generate significant differences in DC values for the three dual-cure bulk-fill RBCs at either depth or for the three light-cure RBCs at the 0.5-mm depth or for FBF at the 5-mm depth ($p>0.05$); however, 40 seconds of light irradiation generated significantly higher DC values compared with 20 seconds of light irradiation for FOBF ($p=0.0136$) and Z250 ($p=0.0086$) at the 5-mm depth. Comparisons of the DC values at the same depth for the same irradiation time among the six RBCs showed that the DC values of the three dual-cure bulk-fill RBCs were generally higher than the DC values of the three light-cure RBCs ($p<0.0001$).

Vickers Hardness

The numeric VHN results for all test conditions are summarized in Table 3, and the bar chart is illustrated in Figure 3.

Analysis of the VHN values of each individual dual-cure bulk-fill RBC at two depths and in the self-cure condition showed that for BulkEZ, the VHN values were not significantly different among the two depths and self-cure conditions for either irradiation time ($p>0.05$); for HyperFIL and Injectafil, the VHN values were not significantly different between 5 mm and self-cure ($p>0.05$), but they were both significantly lower than the VHN values at 0.5 mm ($p<0.01$) for either irradiation time. For the light-cure RBCs, the VHN values at 0.5 mm were significantly higher than the VHN values at 5 mm for either irradiation time ($p<0.001$). The ratios of mean VHN at 5 mm to mean VHN at 0.5 mm for 20 seconds of irradiation were 0.88 for HyperFIL, 0.90 for Injectafil, 0.96 for BulkEZ, 0.64 for FBF, 0.55 for FOBF, and 0.57 for Z250; the ratios for 40 seconds of irradiation were 0.86 for HyperFIL, 0.87 for Injecta-

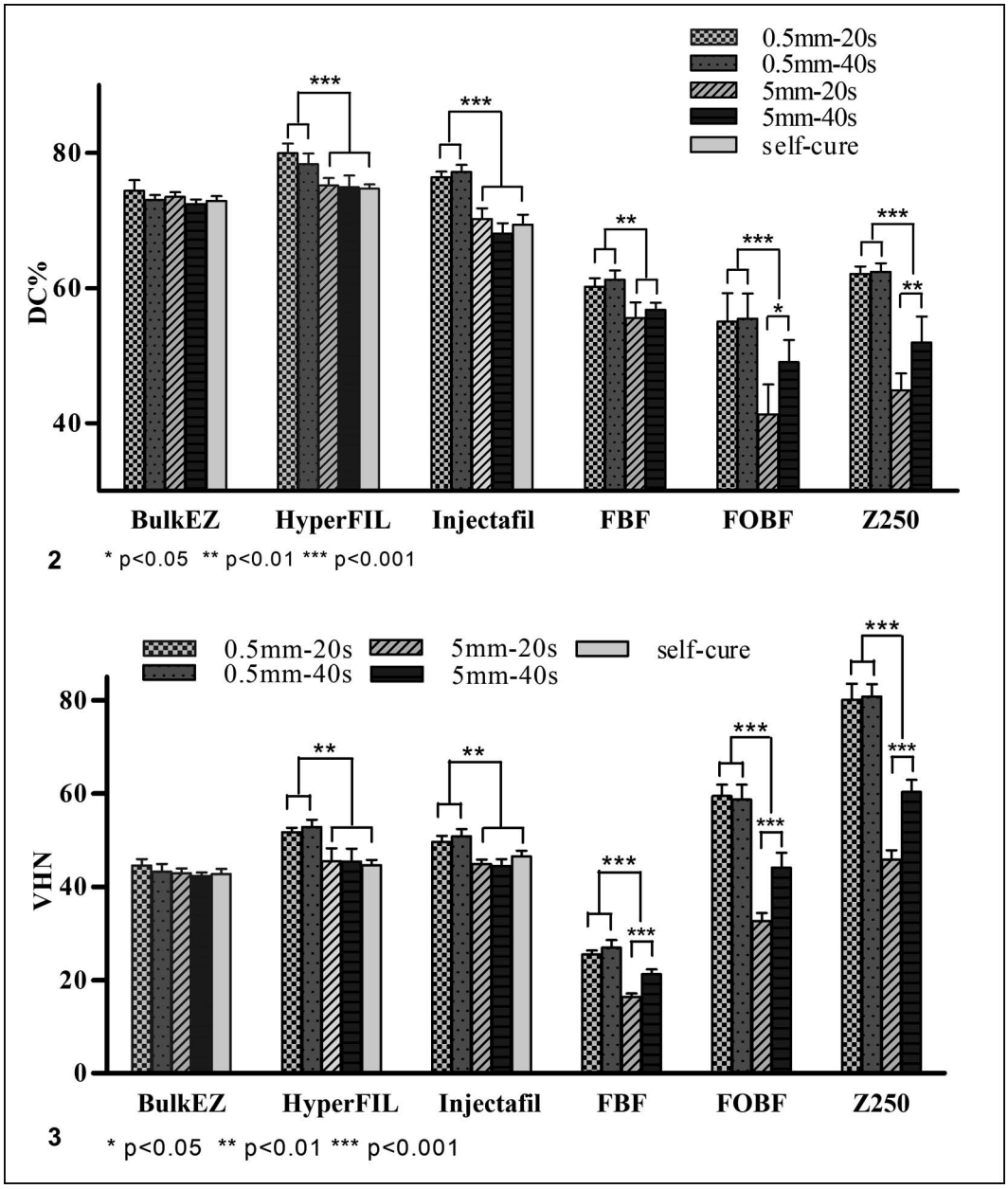

Figure 2. *DC% bar chart for all test conditions.*
Figure 3. *VHN bar chart for all test conditions.*

Table 3: *VHN Mean (Standard Deviation) for All Test Conditions*

	Composite and Irradiation Time											
	BulkEZ		HyperFIL		Injectafil		FBF		FOBF		Z250	
	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s	20 s	40 s
Depth, mm												
0.5	44.53 (1.44)	43.25 (1.65)	51.72 (0.95)	52.87 (1.50	49.63 (1.31)	50.85 (1.61)	25.46 (0.87)	27.01 (1.59)	59.48 (2.45)	58.70 (3.20)	80.09 (3.50)	80.78 (2.70)
5	42.92 (1.01)	42.36 (0.77)	45.42 (2.73)	45.47 (2.76)	44.90 (1.00)	44.45 (1.52)	16.38 (0.76)	21.25 (1.02)	32.66 (1.69)	44.12 (3.21)	45.84 (1.99)	60.38 (2.60)
Self-cure	42.79 (1.12)		44.69 (1.09)		46.52 (1.24)		N/A		N/A		N/A	

Abbreviations: FBF, Filtek Bulk Fill Flowable; FOBF, Filtek One Bulk Fill; Z250, incremental Filtek Z250; N/A, not available.

Table 4: *Analysis Results for Pearson Correlation Between Mean DC and VHN for Six RBCs (α=0.05)*

	Composite					
	BulkEZ	**HyperFIL**	**Injectafil**	**FBFF**	**FOBF**	**Z250**
Pearson *r*	0.9257	0.9382	0.9596	0.9712	0.9912	0.9999
R square	0.857	0.8803	0.9208	0.9433	0.9825	0.9999
P value	0.012	0.0091	0.0048	0.0144	0.0044	< 0.0001
Correlation significant	Yes	Yes	Yes	Yes	Yes	Yes

Abbreviations: RBCs, resin-based composites; FBF, Filtek Bulk Fill Flowable; FOBF, Filtek One Bulk Fill; Z250, incremental Filtek Z250.

fil, 0.98 for BulkEZ, 0.77 for FBF, 0.75 for FOBF, and 0.75 for Z250.

Comparisons of the VHN values between 20 seconds and 40 seconds of irradiation times at the same depth for each individual RBC showed that the two different irradiation times did not generate a significant difference for the three dual-cure bulk-fill RBCs at either depth ($p>0.05$) or for the three light-cure RBCs at the 0.5-mm depth ($p>0.05$); however, 40 seconds of light irradiation generated significantly higher VHN values compared with 20 seconds of light irradiation for FBF, FOBF, and Z250 at the 5-mm depth ($p<0.0001$).

Correlations Between DC and VHN

The mean DC and VHN values for different test conditions for each individual RBC were subjected to Pearson correlation analysis. The results are summarized in Table 4. The DC-VHN correlations for all five test conditions of dual-cure bulk-fill RBCs and all four test conditions of light-cure RBCs are plotted in Figure 4.

All six RBCs showed good correlation between DC and VHN values with the Pearson correlation coefficient $r>0.9$. The three dual-cure bulk-fill RBCs showed similar DC-VHN correlation patterns–high DC range (68%-80%) and medium VHN range (40-53); the three light-cure RBCs showed similar lower DC ranges (40%-63%); Z250 showed the highest VHN range (45-81), FOBF showed a medium VHN range (32-60), and FBF showed the lowest VHN range (16-27).

SEM Images

The SEM images of all six RBCs at 2000× magnification are shown in Figure 5: All three dual-cure bulk-fill RBCs contained irregularly shaped fillers of a wide range of sizes (from sub-micron to a few microns for HyperFIL and up to 10 μm or higher for Injectafil and BulkEZ). FBF and Z250 contained spherical-shaped fillers from sub-microns to a few microns. FOBF contained nanometer-sized fillers and filler clusters of a few microns.

DISCUSSION

Limited research data have been published on the polymerization and mechanical properties of the new dual-cure bulk-fill RBCs. In the present study, the DC and VHN values of three new dual-cure bulk-fill dental restorative RBCs (BulkEZ, HyperFIL, and Injectafil) were evaluated in comparison with three light-cure RBCs (bulk-fill RBCs FBF and FOBF and traditional incremental RBC Z250). The results showed that the DC and VHN values of the six RBCs were material related; therefore, our first null hypothesis was rejected. The DC and VHN values were significantly lower at the 5 mm depth than at the 0.5 mm depth for five RBCs (with the exception of BulkEZ); therefore, our second null hypothesis was largely rejected. Two different light irradiation times produced significantly different DC and VHN values for some test conditions (FOBF and Z250 at the 5 mm depth); therefore, our third null hypothesis was partially rejected.

A major difference between bulk-fill RBCs and conventional RBCs is their depth of cure. As a conventional RBC, Z250 has to be incrementally placed in layers of 2 mm in order to allow sufficient photo-polymerization. The manufacturers claim that the light-cure bulk-fill RBCs FBF and FOBF achieve an improved depth of cure of 4-5 mm. The manufacturers of the three dual-cure bulk-fill RBCs claim that they have unlimited depth of cure. A commonly accepted criteria for determining adequate depth of cure is the 80% bottom/top ratio for DC or hardness.[30–32] In the present study, the data at 0.5 mm could be treated as top surface measurements and the data at 5 mm could be treated as bottom surface measurements. The 5 mm/0.5 mm ratios for DC and VHN were calculated, and similar 80% ratio criteria were used to determine the efficiency of polymerization at the 5-mm depth. The results partially support the claims for the six RBCs. For Z250, the DC and VHN values at 5 mm were <80% of the values at 0.5

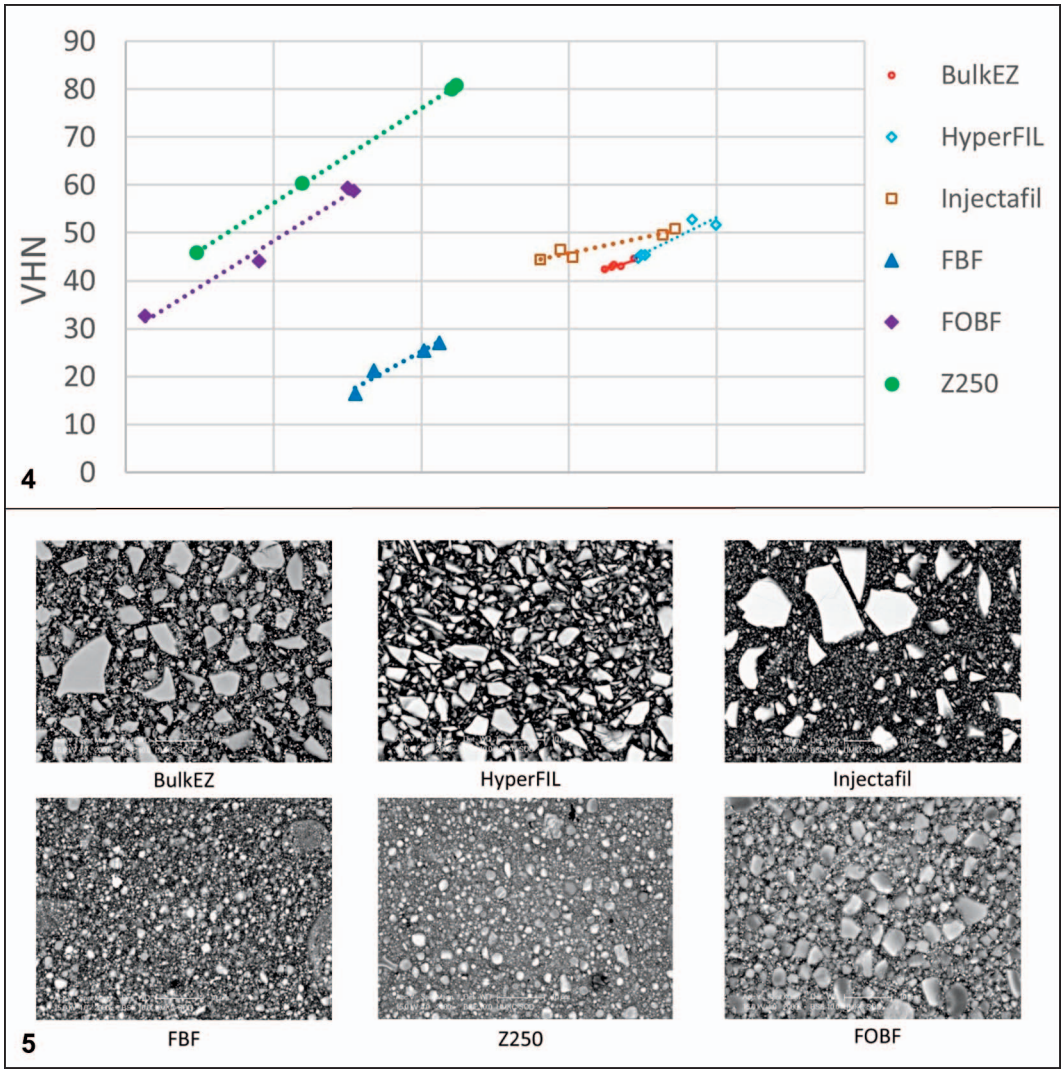

Figure 4. *DC%-VHN correlation chart for six RBCs.*
Figure 5. *SEM images of the six RBCs at 2000× magnification.*

mm for the manufacturer recommended 20 seconds of light irradiation, indicating insufficient polymerization at the 5 mm specimen depth. For FBF, the DC at 5 mm was >90% of the DC at 0.5 mm, but the VHN value at 5 mm was <80% of the values at 0.5 mm for the manufacturer recommended 20 seconds of light irradiation. Similar DC and VHN findings at the 4 mm depth for FBF were reported in previous studies.[33,34] For FOBF, the DC and VHN values at 5 mm were all <80% of the values at 0.5 mm, indicating insufficient polymerization and thus lower hardness at the 5 mm depth and raising questions about the manufacturer's claim of up to 5 mm depth of cure. For the three dual-cure bulk-fill RBCs, the DC and VHN values at the 5 mm depth were the same as the self-cure conditions; both were >5% of the DC and VHN values at the 0.5 mm depth, indicating sufficient polymerization in specimen depth up to 5 mm from chemical curing. Light curing slightly improved the DC and VHN values for HyperFIL and Injectafil in the top layers. BulkEZ showed the same DC and VHN values among all test conditions, indicating that chemical curing is its dominant polymerization process. The results suggest that the three dual-cure bulk-fill RBCs can achieve unlimited depth of cure thanks to their efficient and depth-independent chemical-curing process.

Two clinically relevant light irradiation times—20 seconds and 40 seconds—were assessed for the six RBCs. The results showed that an extended light irradiation time of 40 seconds did not generate any difference in DC or VHN values for the three dual-

cure bulk-fill RBCs at either depth. The explanation could be that the three dual-cure bulk-fill RBCs mainly rely on chemical curing for polymerization and therefore light irradiation time did not affect their DC or VHN values. For the three light-cure RBCs, the two light irradiation times did not generate any difference in DC or VHN values at the 0.5-mm depth; however, 40 seconds of irradiation generated significantly higher DC values for FOBF and Z250 (but not for FBF) and significantly higher VHN values for FBF, FOBF, and Z250 at the 5 mm depth compared with 20 seconds of irradiation. Similar findings were reported by a previous study for FBF and Z250.[34] The explanation could be that in the top layers, where the curing light can reach with sufficient power, 20 seconds of irradiation time was enough to achieve maximum polymerization for those materials; while in the deep layers up to 5 mm, where the curing light was greatly attenuated, extended light irradiation time could help achieve better polymerization and greater hardness for some light-cure RBCs.

Previous studies have indicated that the loading, size and shape of filler particles of light-cure RBCs significantly affected their mechanical properties and light transmittance characteristics, which consequently affected the DC and depth of cure of the RBCs.[35,36] Reduced filler loading can help increase light transmission and depth of cure for bulk-fill light-cure RBCs but at the same time can also weaken their mechanical properties.[37,38] In the present study, FBF, which had the lowest filler loading of 64.5 wt%, showed good depth of cure (>90% 5 mm/0.5 mm DC ratio) but the lowest microhardness level (VHN<30), while the conventional Z250, which had the highest filler loading of 82 wt%, showed the highest microhardness level at 0.5 mm (VHN ~80) but poor depth of cure (<80% 5 mm/0.5 mm DC ratio).

On the other hand, dual-cure bulk-fill RBCs utilize chemical curing mechanisms to achieve depth of cure and therefore are not constrained by the low filler-loading design. With similar filler loading (>70 wt%), the three dual-cure bulk-fill RBCs showed much higher DC values than FOBF. FOBF showed the lowest DC, probably for two reasons: 1) FOBF contains high molecular weight monomers (aromatic dimethacrylate [AUDMA], urethane dimethacrylate [UDMA], and 1,12-dodecanediol dimethacrylate [DDDMA]) with decreased number of reactive carbon=carbon groups in its resin, which consequently results in lower DC[39]; 2) the high viscosity of FOBF could hinder the mobility of its reactive monomer species and reduce the conversion.[40] Compared with FOBF, the three dual-cure bulk-fill RBCs showed lower VHN values at 0.5 mm but higher VHN values at 5 mm, with a smaller difference in VHN values between the two depths. Material viscosity has been shown to be an important factor on the polymerization and clinical performance of RBCs.[41,42] Compared with high-viscosity packable RBCs, low-viscosity bulk-fill RBCs provide better adaptation to the cavity wall and potentially reduce marginal gaps and microleakage.[43,44] With relatively low viscosity and high flowability, the three dual-cure bulk-fill RBCs eliminate the need for flowable liners.

The results from our previous study showed that the polymerization kinetics of HyperFIL and Injectafil were depth dependent—the top layers were characterized by faster light-activated polymerization and the deep layers were characterized by slower chemical-activated polymerization, while the polymerization of BulkEZ was characterized by moderately paced polymerization across the entire depth.[45] These kinetics results are consistent with the DC and VHN results in the present study in which BulkEZ showed depth-independent properties while HyperFIL and Injectafil showed depth-dependent properties. It has been well documented that the rate of polymerization of a dental composite often affects its polymerization contraction stress.[46] The depth-independent, moderately paced polymerization kinetics of BulkEZ is likely to result in more uniform and moderate shrinkage stress; while the depth-dependent polymerization kinetics properties of HyperFIL and Injectafil are likely to result in higher shrinkage stress in the fast-cured top layers and lower shrinkage stress in the slowly cured deep layers. The difference in DC and VHN profiles from the present study and the difference in polymerization kinetics profiles from our previous study for the three dual-cure bulk-fill RBCs may have interesting implications for their physical properties and clinical performance.

Future studies are needed to experimentally measure other polymerization and mechanical properties, such as shrinkage volume and stress of these new dual-cure bulk-fill RBCs and to better understand their overall properties (eg, shrinkage, marginal gaps, microleakage). Research to evaluate the long-term clinical performance of these dual-cure bulk-fill RBCs is also desired.

CONCLUSION

The three dual-cure bulk-fill RBCs can achieve unlimited depth of cure with high degree of conver-

sion and medium microhardness levels. Among them, BulkEZ showed uniformity in degree of conversion and microhardness levels across all depths, while HyperFIL and Injectafil showed slightly higher degree of conversion and microhardness levels in top layers than in deep layers. The difference may have interesting implications for their physical properties and clinical performances.

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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Differences in Radiopacity, Surface Properties, and Plaque Accumulation for CAD/CAM-Fabricated vs Conventionally Processed Polymer-based Temporary Materials

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Clinical Relevance

As temporary materials are often used in prosthetic dentistry, there is need to investigate their behavior in the oral environment. Parameters such as surface roughness and surface free energy correlate to the level of plaque adhesion, which can impact gingival health.

SUMMARY

Objective: To test computer-aided design/computer-aided manufacturing (CAD/CAM)-fabricated and conventionally processed polymer-based temporary materials in terms of radiopacity (RO), surface free energy (SFE), surface roughness (SR), and plaque accumulation (PA).

Methods and Materials: Six temporary materials (n=10/n=10) were tested, including three CAD/CAM-fabricated (CC) materials—Art Bloc Temp (CC-ABT), Telio CAD (CC-TC), and VITA CAD Temp (CC-VCT)—and three conventionally processed (cp) materials: Integrity Multi Cure (cp-IMC), Luxatemp Automix Plus (cp-LAP), and Protemp 4 (cp-PT4). Zirconia acted as the control group (CG, n=10). RO was evaluated according to DIN EN ISO 13116. SFE was investigated using contact angle measurements. SR was measured using a profilometer. The PA tests were performed using three microorganisms: *Streptococcus mutans*, *Actinomyces naeslundii*, and *Veillonella parvu-*

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***la*. Data were analyzed using Kolmogorov-Smirnov, Kruskal-Wallis, Mann-Whitney *U*-, Dunn-Bonferroni, Wilcoxon, Levene, and Pearson tests and one-way analysis of variance with post hoc Scheffé test (α=0.05).**

Results: No radiopacity was observed for any CC material or cp-PT4. CG showed the highest RO, while no differences between cp-IMC and cp-LAP were found. CG showed lower SFE compared to all polymer temporary materials, except in the case of CC-TC. cp-LAP and cp-IMC presented higher SFE than did CC-TC and CG. CC-ABT presented lower initial SR values compared to cp-PT4 and cp-LAP. For cp-LAP, a higher initial SR was measured than for all CAD/CAM materials and cp-IMC. All specimens showed a certain amount of PA after the incubation period. *A naeslundii* and *V parvula* resulted in comparable PA values, whereas the values for *S mutans* were lower by one log level. CAD/CAM materials showed superior results for SR, SFE, and PA, whereas all materials lacked RO.

INTRODUCTION

Prosthetic treatments for fixed dental prostheses (FDPs) such as veneers, crowns, and fixed partial dentures generally depend on indirect fabrication of the definite restoration in the laboratory. Over the period of the laboratory production, which usually takes about seven to 12 days, provisional restoration is necessary to protect the prepared tooth. The fabrication of provisional restorations protects the prepared tooth surfaces from thermal, mechanical, and biological noxae and provides functional, phonetic, diagnostic, esthetic, and stabilizing value.[1–5] There are a great variety of materials and techniques for provisional restorations.[6] Common dental materials used in restorative techniques are tooth-colored polyethylmethacrylates (PEMA) and polymethylmethacrylates (PMMAs), bisphenol A-glycidyl methacrylates (Bis-GMAs), and glass-fiber reinforced composites. Provisional restorations can be produced directly on the prepared tooth or indirectly in the conventional way in the laboratory. These are in contrast to the computer-aided design/computer-aided manufacturing (CAD/CAM) techniques using advanced materials, which enable the fabrication of both provisional and definitive restorations with higher mechanical strength and better clinical outcomes.[7] Provisional restorations may provide short-term function until the definitive restoration has been fabricated or long-term function during a longer course of treatment, such as one involving complete-mouth prosthodontic treatments, oral surgery, or orthodontic or endodontic procedures.[8] The quality of the provisional restoration highly affects the success of treatment outcomes; thus, materials with good mechanical properties as well as dimensional stability and color stability are required. Regarding surface roughness, values of 0.22-1.5 μm are reported for temporary materials.[9–11] In addition, most dental provisional composites are radiolucent and are not visible with standard radiographic techniques, so they challenge good control and easy detection of surplus temporary materials, in contrast to opaque materials such as zirconia. To date, there are very few studies in the literature describing radiopacity of temporary materials; a single study[12] reports no radiopacity (RO) for Telio CAD. In terms of fabrication, a precise marginal fit allows one to maintain gingival health by preventing plaque accumulation around poorly fit margins, which would lead to irritation or inflammation of the periodontal tissues.[1,5,13] Dental plaque, a complex biofilm, is produced by colonization of over 500 bacterial species following a regimented pattern; initial colonizers adhere to the enamel salivary pellicle followed by secondary colonizers showing interbacterial adhesion.[14,15] Plaque development contributes to diseases such as caries, gingivitis, and periodontal disease.[15] Most bacteria in the oral cavity can only survive if they stick to the hard surfaces of teeth, filling materials, prostheses, or dental implants.[16] These different hard surfaces with different chemical characteristics as well as different surface characteristics (such as surface roughness [SR] and surface free energy [SFE] values) can retain varying quantities of bacterial plaque. SR and SFE have a major impact on the initial adhesion and the retention of oral microorganisms.[17] The smaller the SFE or the SR, the lower the plaque accumulation, thereby reducing the risk for periodontal infections.[16] FDPs should generally have highly polished surfaces in order to reduce plaque accumulation (PA) and gingival damage, leading to conditions free from inflammation.[16,18–21] This inflammation could lead to bleeding from the gingiva during the try-in process of the restoration, which affects the quality of adhesive restorative techniques.[16]

In summary, the main goal of all provisional materials should be the fabrication of high-quality and well-fitting temporary restorations with smooth surfaces, enhancing patient health during treatment periods.

Table 1: *Summary of Tested Materials Including Polymer-based Temporary Materials and Control Group Categorized by Type of Material, Product Name, Abbreviation, Manufacturer, Lot No., and Chemical Composition*

Type of Material	Product Name, Shade	Abbreviation	Manufacturer	Lot No.	Chemical Composition, wt%
Polymer-based temporary materials					
Conventionally processed	Integrity Multi Cure, A2 (dual-curing)	cp-IMC	Dentsply Sirona, York, PA, USA	170511	Urethane-modified Bis-GMA, DMA
	Luxatemp Automix Plus, A3.5 (self-curing)	cp-LAP	DMG, Hamburg, Germany	772091	PMMA, SiO_2, UDMA, DMA, Bis-GMA
	Protemp 4, A2 (self-curing)	cp-PT4	3M, Seefeld, Germany	6600623	PUR 10%-20%, silanized SiO_2 5%-10%, DMA 50%-60%, amorphous SiO_2 20%-30%
CAD/CAM fabricated	Art BlocTemp, BL2	CC-ABT	Merz Dental, Lütjenburg, Germany	52808	PMMA, MMA <1%, dibenzoyl peroxide 0%
	VITA CAD Temp, 1M2T/CT40	CC-VCT	VITA Zahnfabrik, Bad Säckingen, Germany	11000	PMMA, SiO_2 14%, pigments
	Telio CAD, LT A2	CC-TC	Ivoclar Vivadent, Schaan, Liechtenstein	R36500	PMMA 99.5%, pigments <1.0%
Zirconia (control group)	Nexx Zr Zirkonoxid	CG	Sagemax Bioceramics, Federal Way, WA, USA	GEMBD	$ZrO_2 \geq$ 89%, Y_2O_3 4%-6%, $HfO_2 \leq$5%, Al_2O_3 <1%

Abbreviations: Al_2O_3, alumina; DMA, dimethacrylate; HfO_2, hafnium dioxide; PUR, polyurethane; SiO_2, silicium dioxide; UDMA, urethane dimethacrylate; Y_2O_3, yttria; ZrO_2, zirconia.

The null hypothesis of this investigation was that no differences in RO, SFE, SR, and PA exist between three CAD/CAM-fabricated (CC) and three conventionally processed (cp) polymer-based temporary materials.

METHODS AND MATERIALS

The RO, SFE, SR, and PA of six polymer-based temporary materials (three CC and three cp materials [Table 1]), were determined and compared with zirconia (Nexx Zr Zirkonoxid, Sagemax Bioceramics, Federal Way, WA, USA), which served as a control group (CG) (Figure 1).

Specimen Preparation

Thirty disc-shaped specimens with a diameter of 10 mm and a thickness of 2 mm were milled (Cerec MCXL, Dentsply Sirona Inc, York, PA, USA) from three polymer-based CC temporary materials (n=10/CC material), namely Art Bloc Temp BL2 (CC-ABT, Merz Dental, Lütjenburg, Germany), Telio CAD LT A2 (CC-TC, Ivoclar Vivadent, Schaan, Liechtenstein), and VITA CAD Temp IM2T/CT40 (CC-VCT, VITA Zahnfabrik, Bad Säckingen, Germany). For the CG, 10 specimens were milled to be "overdimensioned" from zirconia and sintered according to manufacturer's instructions.

Using the CC specimens, standardized silicone molds with a diameter of 10 mm and a thickness of 2 mm were created as templates to produce 30 cp specimens. For this, the polymer-based cp temporary materials (n=10/cp material), namely Integrity Multi Cure A2 (cp-IMC, Dentsply Sirona), Luxatemp Automix Plus A3.5 (cp-LAP, DMG, Hamburg, Germany), and Protemp 4 A2 (cp-PT4, 3M, Seefeld, Germany), were filled into the silicone molds and allowed to polymerize for 10 minutes. The dual-curing cp-IMC was additionally light-cured (20 seconds) (Elipar S10, 3M).

All specimens were polished with a laboratory polishing machine (Abramin, Struers, Ballerup, Denmark) and silicon carbide paper up to P2000 (SiC Foil, Struers) under permanent water-cooling to a final thickness of 1 ± 0.03 mm. For all polymer-based temporary materials, preliminary polishing was performed with a goat-hair brush (diameter of 20 mm, Polirapid, Singen, Germany) and polishing paste (Signum HP Paste, Kulzer, Hanau, Germany); this step was followed by ultrasonic cleaning in distilled water (Ultrasonic T-14, L&R Manufacturing Co, Keamy, NJ, USA) and high-gloss polishing with a linen buffing wheel (Komet Dental, Lemgo, Germany). For zirconia specimens, preliminary and high-gloss polishing was performed using a polisher containing diamond grit particles and a felt wheel with polishing paste (Dia Glace, Yeti Dental, Engen, Germany), respectively. All manual polishing steps were performed according to standardized polishing protocols using a hand piece at a maximum speed of 6000 rpm. After polishing, all specimens were stored

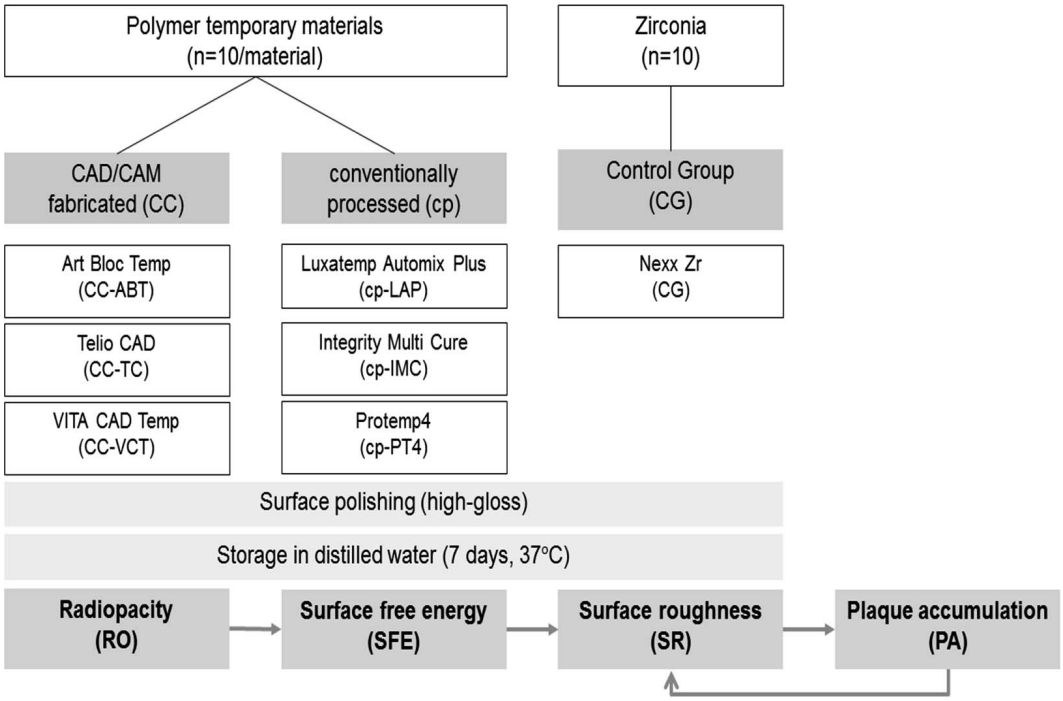

Figure 1. *Study design for testing differences in radiopacity, surface properties, and plaque accumulation for CAD/CAM-fabricated vs conventionally processed polymer-based temporary materials.*

in distilled water for seven days at 37°C (HeraCell 150i, Kulzer).

Analysis of RO

The RO was evaluated according to DIN EN ISO 13116.[17] The specimens of each material were arranged on radiographic films (Insight IP-21, Carestream Dental, Stuttgart, Germany) with an aluminum step wedge with variable thickness (from 0.3 mm to 6.3 mm in 0.3-mm increments). Conventional x-rays were taken by an intraoral x-ray unit (Heliodent DS, Dentsply Sirona) operating at 60 kV, 7 mA, and 0.16 seconds by maintaining the same positioning of the x-ray unit throughout the analysis of RO of all materials. Each group was radiographed three times before the position on the aluminum wedge was adjusted. Three different positions (bottom, middle, and top) on the same aluminum step wedge were examined to obtain a varying reference for the RO of each material. Radiographic films were developed with standard developing solution according to manufacturer's instructions. Images were transferred to a computer and loaded into a picture-editing software (Adobe Photoshop PS4, Adobe Systems, San Jose, CA, USA) for evaluation. After creating a standard curve based on the gray values of the aluminum step wedge, the RO of the specimens was calculated with the measurements report of the picture-editing software corresponding to the respective thickness of aluminum (mm/Al).

SFE Measurements

The SFE was investigated after ultrasonic cleaning in distilled water (Ultrasonic T-14, L&R Manufacturing) by measuring the contact angle (Kruess Easy Pearl, Kruess, Hamburg, Germany) of distilled water as polar and diiodomethane as dispersed test liquid at room temperature for each material (n=10/material). Successively, three drops, with a defined drop volume of each test liquid (6 μL of distilled water, 3 μL of diiodomethane), were positioned on different areas of the specimen surface. For each sessile drop, a picture was taken after five seconds. The baseline was adopted, the drop contour labeled, and the contact angles calculated using DSA 4 software (Drop Shape Analysis, Release 1.0, Kruess). For water the calculation method "Tangent 1" and for diiodomethane the calculation method "Circle" were applied. Finally, SFE was calculated according to the method of Owens-Wendt-Rabel-Kaelble.[18]

SR Measurements

The SR was measured (Mahr Perthometer SD 26, Mahr, Göttingen, Germany) twice, initially and after analysis of plaque accumulation, for each material (n=10/material). For both measurements, six read-

ings, including three horizontal and three vertical lines with a track length of 5.6 mm, were recorded with a distance of 0.25 mm in between. For each specimen, the mean value of the six readings was calculated and assigned as the SR value to the specimen.

Analysis of PA

For analyzing the PA, the specimens (n=10/material) were placed in 48-well plates (Greiner Bio-One, Kremsmünster, Austria) in an upright position and covered with 1.2 mL of brain-heart infusion medium (BD Diagnostics, Heidelberg, Germany). The experiments were performed using three microorganisms, namely *Streptococcus mutans* (*S mutans*, ATCC 25175, DSMZ, Heidelberg, Germany) and *Actinomyces naeslundii* (*A naeslundii*, ATCC 19039, DSMZ) as representatives for caries pathogens as well as *Veillonella parvula* (*V parvula*, ATCC 17745, DSMZ) as an early member of the periodontopathogenic biofilms close to the gingival margin. The strains were grown on Schaedler agar plates (BD Diagnostics) containing vitamin K1 and 5% sheep blood for 48 hours under defined culture conditions (37°C, 5.8% CO_2 for *S mutans* and *A naeslundii*; anaerobic chamber containing 5% H_2, 10% CO_2, 85% N_2 for *V parvula*). Each well, containing one specimen disc, was inoculated with 100 μL of the respective bacterial suspension (*S mutans, A naeslundii,* or *V parvula*) at an optical density of 0.5 measured at 600 nm in 0.9% sodium chloride (NaCl) solution (Varioskan Multiplate Reader, Thermo Fisher Scientific, Waltham, MA, USA). To allow the formation of adult biofilms, the plates were incubated for five days under the same culture conditions as described above. After incubation, the specimens were aseptically removed from the well plate, rinsed twice with 0.9% NaCl, and transferred to 15-mL Falcon tubes (Greiner Bio-One). The tubes were vortexed for 60 seconds to disrupt the biofilm. To determine the number of viable bacteria, a luminescence-based assay was performed (BacTiter-Glo, Promega, Mannheim, Germany). This assay measures the amount of adenosine triphosphate (ATP) present in the respective specimen, which is directly proportional to the number of viable bacterial cells. To each well of a 96-well plate 100 μL of the assay reagent was added and mixed with 10 μL of the bacterial sample. After an incubation time of five minutes, the luminescence was recorded by a luminescence reader (GloMax Navigator System, Promega). The measurement was performed in duplicate for each specimen (n=10/material).

Statistical Analysis

The assumption of normal distribution was tested using the Kolmogorov-Smirnov test. To investigate possible differences between the variances, the Levene test was performed. To determine differences between the tested materials nonparametric analyses, such as Kruskal-Wallis and Mann-Whitney *U*- and Wilcoxon tests were calculated for SFE, SR, and PA values. RO was analyzed using one-way analysis of variance with post hoc Scheffé test. Correlations were tested using the Pearson test. All *p*-values below 0.05 were construed as statistically significant. All statistical tests were performed using SPSS V5 statistics software (IBM, Armonk, NY, USA).

RESULTS

No radiopacity was detected for any CC polymer temporary material (CC-ABT, CC-VCT, CC-TC) or cp-PT4, as no x-ray shadow could be observed in the area of the specimens on the x-ray films. RO was only observed for cp-IMC, cp-LAP, and CG (Figure 2; Table 2). CG showed the highest values of RO ($p<0.001$), while no differences between cp-IMC and cp-LAP were found ($p>0.05$).

The CG showed lower SFE values compared to all polymer temporary materials, except CC-TC ($p<0.001$). Conventionally processed cp-LAP and cp-IMC presented higher SFE than did CC-TC and CG ($p<0.001$). The remaining materials showed no differences ($p>0.05$).

CC-ABT presented lower initial SR values compared to cp-PT4 and cp-LAP ($p<0.001$). For cp-LAP, a higher initial SR was measured than for all CC polymer temporary materials (CC-VCT, CC-TC, and CC-ABT) and the conventionally processed cp-IMC ($p<0.001$). After analysis of PA and cleaning, CC-TC showed lower SR compared with conventionally processed cp-PT4 and pc-LAP ($p<0.001$). Furthermore, an increase of SR values was observed for cp-PT4, CC-ABT and CC-VCT, and CG (Table 2).

Regarding PA, all specimens showed a certain amount of bacterial colonization after the incubation period. *A naeslundii* and *V parvula* resulted in comparable bacterial number values, whereas the values for *S mutans* were lower by one log level (Figure 3). Within all groups, homogeneous distribution of the variances for all bacterial species tested was found. Within the *S mutans* and *A naeslundii* groups, no significant differences for the number of viable bacteria could be found between the materials tested. Within *V parvula*, significant differences

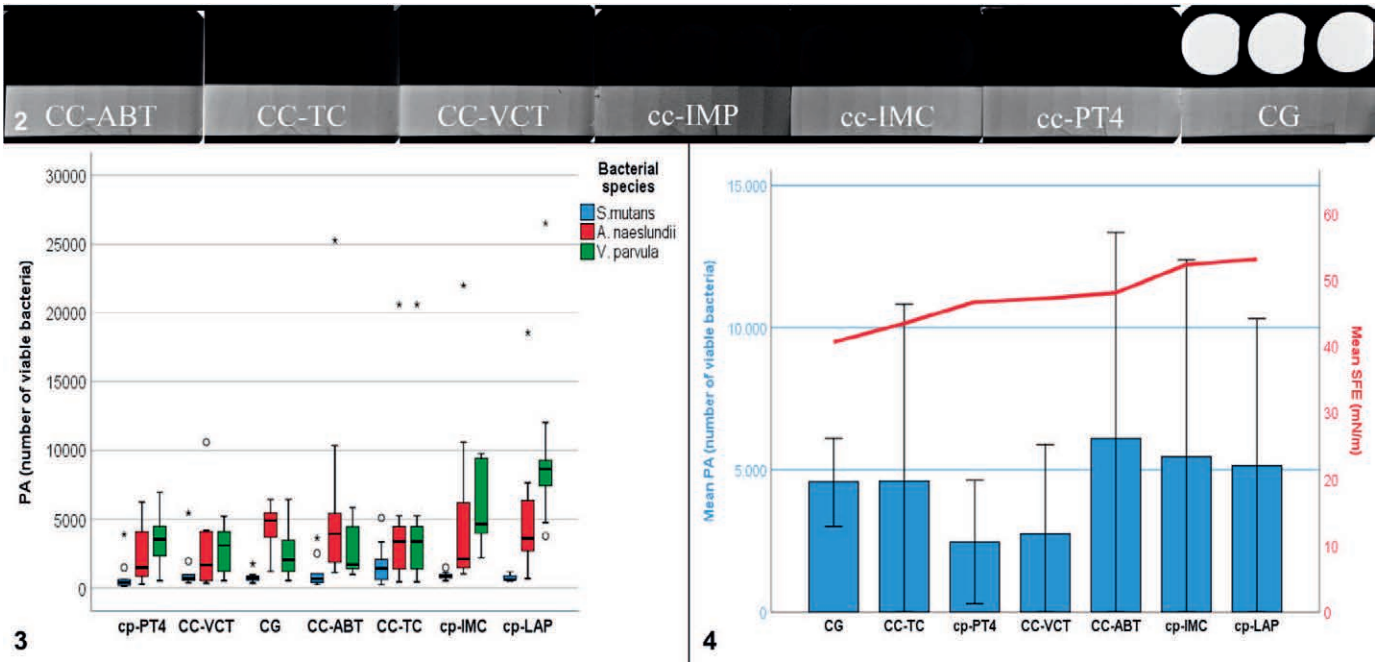

Figure 2. *Radiopacity (RO) of all tested materials.*

Figure 3. *PA (number of viable bacteria) according to the material and the bacterial species tested.*

Figure 4. *Correlation between PA (pooled data) and SFE values.*

were found ($p<0.001$), with higher bacterial colonization for cp-LAP compared to CG ($p=0.005$), and all CC temporary materials, namely CC-ABT ($p=0.009$), CC-VCT ($p=0.016$), and CC-TC ($p=0.033$).

A positive correlation was found between SFE values and pooled data of PA ($R=0.253$, $p=0.039$) (Figure 4).

DISCUSSION

This study aimed to evaluate different properties of polymer-based temporary restorative materials. Zirconia was chosen as a control because it is known for its excellent biocompatibility, surface properties, and high RO.[12,24] Regarding RO, the null hypothesis could be confirmed, as no temporary material showed any RO. The reason for this may be found in the composition of the materials, as they contain no components with high RO, such as zirconia or barium sulfate. These additives are used in other resins to improve their RO and could also be added to temporary materials. Although it is not necessary for temporary restorative materials to show similar RO values, a certain visibility in radiographs would be of interest to detect any excess restorative material. However, temporary materials not showing any RO do not have to be removed prior to presurgical cone beam computed tomography imaging, allowing practitioners to avoid interfering artifacts.

With regard to the SFE values, two cp materials (Luxatemp Automix Plus and Integrity Multi Cure) showed higher values than did the CC Telio CAD. Therefore, the stated hypothesis must be rejected. It seems that CAD/CAM processed materials generally show lower SFE values. This could be traced back to the industrial manufacturing process, particularly the polymerization process, realizing higher conversion rates and thus resulting in reduced quantities of functional groups on the polymer surface. The standardized manufacturing process could also be the reason for the obtained SR results, for which the cp materials also showed higher values compared to the CAD/CAM processed materials. In part, these results are confirmed by similar studies investigating different processed materials. Other groups reported tendential higher values.[9–11,25] This may be due to different polishing protocols, such as those involving different silicon polishers that are used chairside by the dentist. We chose a goat-hair brush with polishing paste, which is commonly used in dental laboratories.

During the microbiologic experiments, the specimens were cleaned mechanically by brushing, followed by 10 minutes of cleaning in an ultrasonic

Table 2: *Means and Standard Deviations for Surface Free Energy (SFE), Surface Roughness (SR), and Radiopacity (RO)*[a]

Type of Material	Product	SFE, mN/m	SR, μm			RO, mm/Al
			Initially	After Plaque Accumulation	*p*-Value Between SR Values	
Polymer-based temporary materials						
Conventionally processed	cp-IMC	52.4 ± 4.4 CD	0.054 ± 0.016 AB	0.074 ± 0.022 AB	0.051	8.01 ± 0.55 B
	cp-LAP	53.2 ± 3.3 D	0.099 ± 0.050* C	0.108 ± 0.017 B	0.139	8.45 ± 0.07 B
	cp-PT4	46.7 ± 1.4 BC	0.077 ± 0.012 BC	0.106 ± 0.033 B	0.011	0 A
CAD/CAM fabricated	CC-ABT	48.1 ± 5.0* BCD	0.041 ± 0.002 A	0.082 ± 0.006 AB	0.005	0 A
	CC-VCT	47.3 ± 4.1 BC	0.063 ± 0.014 AB	0.093 ± 0.020 AB	0.005	0 A
	CC-TC	43.2 ± 2.2 AB	0.051 ± 0.012 AB	0.071 ± 0.016 A	0.050	0 A
Zirconia (control group)	CG	40.7 ± 3.8 A	0.071 ± 0.009 ABC	0.090 ± 0.019* AB	0.009	226.7 ± 0.1 C

[a] *Different letters indicate significant differences among the tested materials.*
* *Indicates non–normally distributed groups.*

bath. This protocol may explain the increase in the SR values of all groups after microbiological testing. The applied cleaning process is similar to that used in the clinical situation, in which patients brush their teeth without paying particular attention to the temporary restorations. Thus, it is to be suspected that the surface properties of polymer-based temporary materials are changed after PA followed by a thorough cleaning. Another explanation for the observed differences in SR after testing the PA would be the alteration of the restoration surfaces by the bacterial colonization itself, which would lead to a "vicious cycle," compromising the quality of the surface over a longer time period.[26]

The SFE and the SR results influence the microbiological outcome. This is clearly demonstrated by the correlation between the SFE and the PA showing higher numbers of viable bacteria with increasing values of SFE. Higher bacterial counts of *Veillonella parvula* were found for the cp Luxatemp Automix Plus compared to all CC materials. Therefore, our hypothesis regarding PA must be rejected. *Veillonella parvula* plays an important role in the early stage of biofilm formation near the gingiva and opens the door for other periodontal pathogens, such as *Porphyromonas gingivalis* or *Fusobacterium nucleatum*.[27] A luminescence-based assay was used for the determination of the bacterial amount on the material surfaces. This assay measures the amount of ATP present in the tested cells, which is an indicator of the number of viable cells. Most previously published investigations use different kinds of staining or optical counting of cells.[28] Furthermore, a lot of different biofilm models are described in the literature.[29–31] This complicates the comparison of the present results to those of previous investigations. Other approaches in the literature tested temporary materials, with antimicrobial effects demonstrating promising results.[32]

For short-term restorative materials it is essential that no inflammation of the gingiva occurs during the temporary phase to avoid any bleeding during the restorative process. Regarding long-term temporary restorations, for which mainly CAD/CAM processed materials are used, this aspect must be widened for caries pathogens such as *Streptococcus mutans* and *Actinomyces naeslundii* because of the possibility of the formation of secondary caries on the restoration margin. Regarding *Streptococcus mutans* and *Actinomyces naeslundii*, no significant differences between the groups were found, which could be explained by the different adhesion mechanisms of these strains. Other reasons for differing PA rates are the chemical composition of the materials as well as further surface properties, such as SFE, SR, hydrophobicity, and surface-coating techniques.[33] Further investigations of the precise interaction of these parameters are necessary for a better understanding of biofilm formation on dental restorations.

In this study, we tested different materials with a biofilm model containing only a single bacterial species. Furthermore, three representative oral pathogens were chosen as test organisms, but the oral cavity is much more complex, so further *in vivo* investigations are necessary.

CONCLUSIONS

Considering all results, the following conclusions can be drawn:

- All of the tested temporary materials lack RO; therefore, it would be interesting to determine if there are any possibilities to alter the composition

of the materials to increase these values so that the practitioner can opt for a higher radiographic visibility if necessary.
- CC materials showed lower values for SR and SFE, thus providing better surface properties for the restorations.
- Conventionally processed temporary materials showed higher and more unsteady values for PA. Therefore, CC temporary materials may be superior to cp materials when used over longer periods of time.

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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 Computer-aided Design/Computer-aided Manufacturing Diamond Bur Wear on Marginal Misfit of Two Lithium Disilicate Ceramic Systems

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Clinical Relevance

Marginal misfit of monolithic lithium disilicate ceramic crowns obtained from a chairside computer-aided design/computer-aided manufacturing system is affected after successive millings using a single diamond bur set. This fact can be critical for the longevity of indirect restorations.

SUMMARY

Objectives: This laboratory study aimed to assess the effect of successive crown millings on the marginal misfit of monolithic full-ceramic restorations obtained from two lithium disilicate systems, with a single diamond bur set used for each material in a chairside computer-aided design/computer-aided manufacturing (CAD/CAM) unit.

Methods and Materials: Initially, 36 standardized composite resin dies were produced by additive manufacturing from a three-dimensional model of a right mandibular first molar with full-crown preparation generated in CAD software. Individual ceramic crowns were obtained in a chairside CAD/CAM unit (CEREC MC XL) for each composite resin die according to the ceramic system (IPS e.max CAD and Rosetta SM; n=18). Two new diamond burs were used as a set for obtaining the crowns in each experimental group (ceramic systems),

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and the milling periods were defined after three crown millings (T0-T6), when the diamond bur set of each system was removed for morphologic characterization using scanning electron microscopy (SEM). The marginal misfit of the crowns was assessed through coronal and sagittal micro-tomographic sectioning, in the vertical and horizontal directions of the ceramic crowns seated on their respective resin dies. The collected data were tabulated and subjected to one-way analysis of variance and Tukey's honestly significant difference test (α=0.05).

Results: SEM images showed changes in the superficial morphology of the diamond bur sets, with progressive loss of the diamond granules after the successive millings of crowns for both experimental groups. Significant differences were detected in the marginal misfit of the crowns from both ceramic systems at the different milling periods ($p<0.001$).

Conclusions: Diamond burs deteriorated after successive crown millings for both lithium disilicate ceramic systems. The marginal misfit of the crowns obtained increased with the successive use of the CAD/CAM diamond bur set employed for milling each ceramic material. In addition, new milling of full lithium disilicate ceramic crowns can be inappropriate after 11 successive millings for IPS e.max CAD and 12 for Rosetta SM, due to the increased marginal misfit observed under the parameters tested.

INTRODUCTION

Advances in the field of computer sciences have led to the development and improvement of computer-aided design/computer-aided manufacturing (CAD/CAM) technology. Dental CAD/CAM systems are used for the digital design and manufacture of indirect restorations in computerized milling units with prefabricated blocks or discs of different materials.[1,2] Chairside CAD/CAM systems allow indirect restorations to be manufactured in the office by the practitioner, without the need for prosthetic laboratory assistance. These systems have the advantage of not necessarily requiring conventional impressions and temporary restorations when all the steps for an indirect restoration can be performed in a single clinical session. Also, restorations with satisfactory esthetics and acceptable marginal accuracy can be obtained with chairside CAD/CAM systems.[1–3]

Most popular dental CAD/CAM milling units use diamond burs at high speed to mill distinct materials into indirect restorations.[4] The diamond burs from CAD/CAM systems are frequently used under water cooling, and the performance of these instruments is expected to decline when they are used successively.[5,6] Little is known about how CAD/CAM systems are capable of defining the number of milling cycles after which the diamond bur set needs to be replaced.[7–9] In other words, can the CAD/CAM milling units really have adequate control over the number of times the diamond burs can be securely used for milling the different restorative materials available without causing damage to the restorations?

Several types of dental ceramics are currently available, but the materials most commonly used in chairside CAD/CAM systems are blocks/discs from glass ceramics (feldspathic, leucite reinforced, and lithium disilicate reinforced).[10,11] Lithium disilicate–reinforced ceramic was first presented in 1998 as the IPS Empress 2 system (Ivoclar Vivadent, Schaan, Liechtenstein) for use in the heat-pressed technique.[12] Further, a new version of the material was released as IPS e.max Press in 2005, with improved mechanical and optical properties in relation to the first-generation system.[13] The lithium disilicate family of products was patent protected when introduced into the market, and after the patent's expiration, different manufacturers released alternative versions of this material,[11] some of which have different structural properties, sometimes showing behavior distinctly different from that of the original material.[14,15]

In 2006, IPS e.max CAD was introduced for use in CAD/CAM systems,[16] and recently, another lithium disilicate ceramic block became available (Rosetta SM, Hass, Gangneung, Gangwon, Korea).[11,17] The lithium disilicate ceramic blocks available on the market are partially crystallized and contain both lithium metasilicate (Li_2SiO_3) and lithium disilicate ($Li_2Si_2O_5$) crystal nuclei. In this state, the diamond burs of the CAD/CAM systems can be used for milling the blocks into restorations with reduced wear.[10,11]

The cervical adaptation of indirect ceramic restorations plays a key role in its clinical success,[18] since failure to provide good marginal adaptation can lead to plaque accumulation and periodontal destruction, recurrent caries, and, finally, failure of the restorations.[19] The marginal misfit size of an indirect restoration is dependent on the various steps involved in clinical and laboratory processing during the traditional manufacturing process.[14,20–25] Also,

while the marginal misfit of CAD/CAM restorations can be affected by several factors, the scanning quality and successive use of diamond burs in the milling unit for extended periods can be decisive in their adaptation.[1,2] It has been shown that the use of an opacifier powder before intraoral digital scanning can improve the marginal adaptation of full-ceramic crowns,[2] and because of severe use, CAD/CAM diamond burs must be used with caution, to reduce disturbances over the ceramic surface and cervical region, with consequent negative effects on final restoration strength.[15]

As has been seen, many factors can influence the quality of indirect restorations produced in chairside CAD/CAM systems. Thus, the purpose of this in vitro study was to assess the effects of successive crown millings on the marginal misfit of monolithic full-ceramic restorations obtained from two lithium disilicate systems with a single diamond bur set used for each material in a chairside CAD/CAM unit. Two null hypotheses were tested: 1) the monolithic full-ceramic crowns obtained would not show differences in marginal adaptation at the different milling periods for both ceramic systems and 2) the CAD/CAM diamond bur sets used for milling the monolithic full-ceramic crowns would not show differences in morphology at the different milling periods for both ceramic systems.

METHODS AND MATERIALS

Die Manufacturing

Thirty-six standardized composite resin dies were obtained and used for production of the ceramic crowns and evaluation of their adaptation. The die base drawing was obtained from a three-dimensional model of a right mandibular first molar with full-crown preparation with rounded axiogingival angles and shoulder termination,[20] which was created with CAD software (Rhinoceros 4.0, McNeel North America, Seattle, WA, USA) and nonuniform rational basis splines lines to generate the solid surfaces and volumes (Figure 1).[26,27]

Subsequently, the three-dimensional model was used for producing the standardized dies with additive manufacturing (Objet Connex350, Stratasys, Eden Prairie, MN, USA) with light-curing composite resin (Veroblack FullCure850, Objet Geometries, Rehovot, Central District, Israel). The dies obtained were checked for any defects and substituted when necessary. Then the dies were assigned to the experimental groups by simple randomization according to the ceramic system (n=18), IPS e.max CAD (LT A2, Ivoclar Vivadent), and Rosetta SM (LT A2, Hass) and subdivided according to the milling period (n=3).

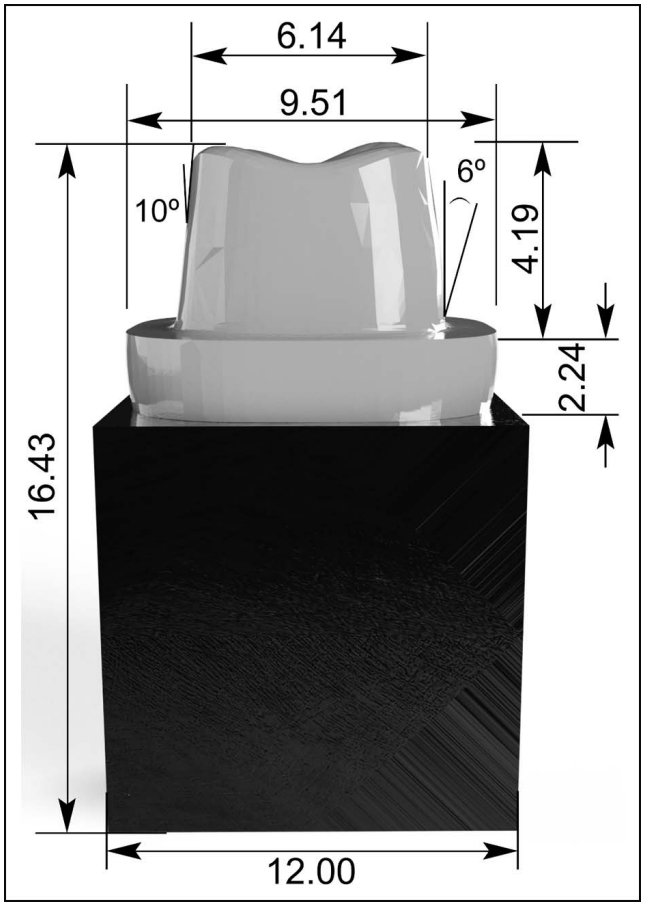
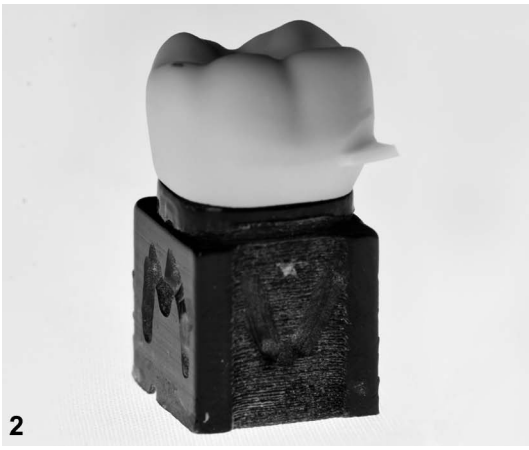


Figure 1. *Three-dimensional CAD model of a right mandibular first molar with full-crown preparation (mm).*

CAD/CAM Milling Process

To produce the ceramic crowns by means of the chairside CAD/CAM technique, all the composite resin dies were sprayed with a thin layer of an opacifier powder (CEREC Optispray; Dentsply Sirona, York, PA, USA), indicated for improving the vertical fit, being considered in this study as an option for reducing the need for internal surface adjustments of the crowns.[2] The die scanning was performed with a device formed by a custom stable base and a rotating platform developed for this study, to simplify image acquisition by the scanner. A digital camera (CEREC Omnicam; Dentsply Sirona) was positioned perpendicularly and as close as possible to the specimen, rather than at a 45° angle, and operated in dry conditions, and the scanning procedure was performed by a single experienced operator who had been previously



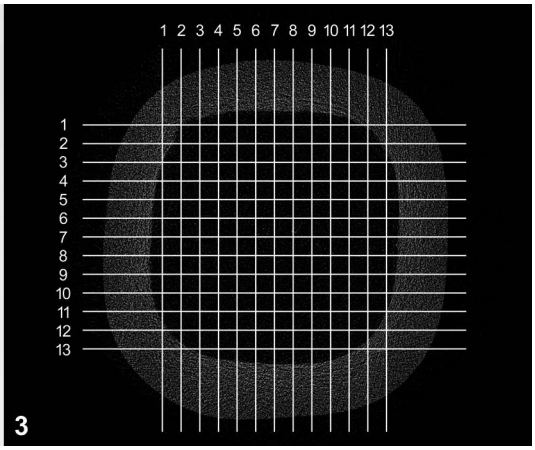


Figure 2. *Full monolithic lithium disilicate ceramic crown seated on its respective resin die.*

Figure 3. *Micro-CT sagittal and coronal cuts of the specimens.*

trained for this purpose.[28] After each resin die had been digitized, full-crown restorations were individually designed in the CAD software for the respective abutments, according to the standard occlusal anatomy of a mandibular first molar crown available in the software (CEREC v4.2.5; Dentsply Sirona). The luting space for adhesive cementation was set at 80 μm, according to the manufacturer's instructions, and the parameter for young teeth was disabled. A new set of CAD/CAM diamond burs (CEREC Step Bur 12 and Cylinder Pointed Bur 12S; Dentsply Sirona) was selected for each ceramic system before the first milling. Subsequently, the ceramic blocks were positioned in the CAM unit (CEREC inLab MC XL; Dentsply Sirona) to be milled according to the contours and dimensions previously defined on the CAD software in groups of three restorations per period. The milling periods for each ceramic material were defined after the completion of three crown millings per group (T0-T6), when the diamond bur set of each ceramic system was removed from the CAM unit for morphologic characterization. The accuracy of milling was ±25 μm, as described by the manufacturer. No polishing, glazing, or internal adjustments were made for any of the milled crowns before the marginal misfit measurements, to avoid additional interference.

Marginal Misfit Evaluation

The placement and adaptation of the ceramic crowns were individually checked with fluid vinylpolysiloxane material (Fit Checker, GC Dental Industrial Corp, Tokyo, Japan),[1,2] on the respective composite resin dies used for scanning and obtaining the restorations, to ensure passive adaptation was obtained. The vinylpolysiloxane material was also used for maintaining the crowns in position (cementation) for marginal misfit evaluation (Figure 2). The material was manipulated for 20 seconds according to the manufacturer's instructions before insertion inside the restorations and placement of the crowns on the resin dies. The excess material was removed from the cervical region using scalpel blades after the setting time was completed (two minutes). After the crowns were seated on their respective resin dies, the sets were first examined under stereomicroscopy at 40× magnification (MZ6, Leica Microsystems GmbH, Wetzlar, Germany) to ensure correct placement and avoidance of any cementation interference caused by the vinylpolysiloxane material. If any dislocation, unseating, or misadjustment was detected during this assessment, the ceramic crown and cementation material were removed from the die followed by new seating. No ceramic crowns were discarded at this time, as the intention was to check the marginal adaptation from the restorations as they were produced by the CAD/CAM system.

The assessment of the marginal misfit of the crowns was performed in a high-resolution micro–computed tomographic (micro-CT) scanner (1272, SkyScan, Bruker, Kontich, Belgium).[1] Micro-CT scanning was performed at 100 kV and 100 mA, with a pixel size of 12.0 μm, a filter Cu of 0.11 mm, and resolution of 1632 × 1092 pixels. The selected scanning was performed at 0.8° rotation steps to 180°. To reduce artifacts, an average of two frames was collected with 20-pixel random movements, resulting in a scanning time of 20 minutes per specimen for the tomographic sections to be obtained. The tomographic sections were combined in

three-dimensional images with specific software (NRecon v1.6.8.0; SkyScan, Bruker) with the following parameters: smoothing of 3%, ring artifact correction of 5%, and beam-hardening correction of 10%. After image reconstruction, specific software (DataViewer v1.5.0.2; SkyScan, Bruker) was used to obtain image data sets of the sagittal and coronal planes. Next, 13 images from the sagittal set and 13 images from the coronal set were selected to illustrate specimen extension in these two different planes, according to previous studies (Figure 3).[1,2,29] The vertical and horizontal marginal fits were measured in all aspects of the crowns (buccal, lingual, mesial, and distal) considering absolute values, using previously described criteria,[1,2] with the maximum acceptable misfit being defined as 120 μm.[19]

The marginal misfits were evaluated in the images obtained with the measuring software (CTAN v1.12.0.0; SkyScan, Bruker). For each selected image, measurements were made for vertical and horizontal marginal misfits on each aspect of the crown-die set at 200× magnification (Figure 4). The linear distance from the cavosurface angle of the preparation to the margin of the restoration was defined as the marginal misfit, in the vertical and horizontal directions, which represents the maximum measurement of discrepancy at the margin.[1,2,30] Fifty-two measurements were made per specimen (two planes × 13 slices × two aspects), and the combined mean of all four locations for each specimen was calculated.[1,2] All equipment was calibrated, and the software parameters were the same for all specimens; an experienced operator conducted all the evaluations.

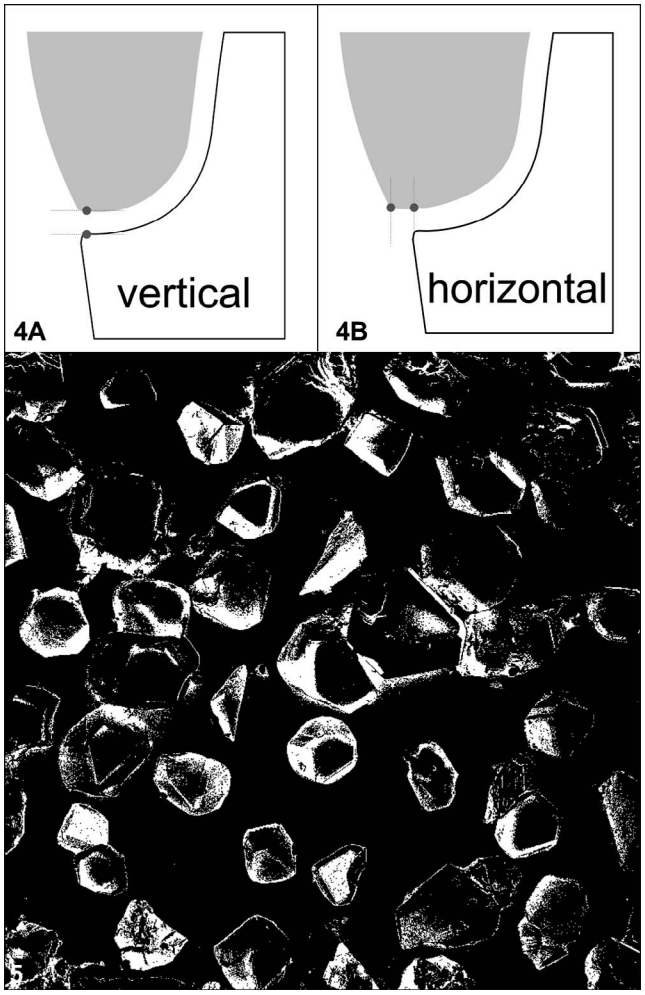


Figure 4. *Marginal misfit measurements. (A): Vertical misadjustment at the cervical region. (B): Horizontal misadjustment at the cervical region.*

Figure 5. *SEM threshold of diamond grains.*

Morphologic Characterization of Burs

Before the first milling and after the completion of every milling period (three crown millings), the diamond bur set corresponding to each ceramic system was removed from the CAM unit, and the burs were cleaned in an ultrasonic bath with distilled water for five minutes and then dried. At that point, scanning electron microscopy (SEM; VEGA 3, TESCAN Electron Microscopy, Brno, Czech Republic) images were taken from the surfaces of the diamond burs at 100× to 200× magnification and 10.0 KV accelerating voltage. For this qualitative analysis, the distribution, morphology, and sizes of the diamond granules were considered, as well as the presence of exposed metallic matrix on the surfaces of the burs. The total area of the diamond burs in each image was measured with image processing and analysis software (ImageJ 1.52a, National Institutes of Health, Bethesda, MD, USA). The threshold of granules was determined (Figure 5) to define the granule count per area (GC), in addition to the mean area (μm^2) and percentage (%) of diamond granule distribution in the image for quantitative morphologic characterization of the burs.[7]

Statistical Analysis

The marginal misfits (μm) verified in the coronal and sagittal sections selected for each specimen were tabulated, and the mean misfit values were calculated in the vertical and horizontal directions. Thus, one-way analysis of variance was used to show the differences between the marginal misfits verified isolated in the vertical direction and in the horizon-

Table 1: *Mean Marginal Misfit (μm) and Standard Deviation (±) for Lithium Disilicate Full-Ceramic Crowns According to the Ceramic Systems and Milling Periods (n=18)*[a]

Group/Milling Periods	IPS e.max CAD		Rosetta SM	
	Horizontal	Vertical	Horizontal	Vertical
T1 (crowns 1-3)				
1	78.3 ± 25.2 A	32.4 ± 12.3 A	41.7 ± 12.6 A	32.3 ± 5.3 A
2	86.4 ± 6.7 AB	35.1 ± 12.6 AB	51.9 ± 12.6 AB	35.0 ± 6.5 AB
3	100.0 ± 13.3 AB	37.4 ± 9.0 AB	57.4 ± 27.3 AB	36.2 ± 7.8 AB
T2 (crowns 4-6)				
4	102.5 ± 11.6 AB	39.8 ± 4.5 AB	73.2 ± 20.7 B	50.5 ± 3.0 B
5	106.6 ± 13 AB	41.6 ± 12.6 AB	86.2 ± 13.9 BC	67.9 ± 5.2 BC
6	123.2 ± 9.9 BC	45.1 ± 14.3 BC	100.6 ± 9.2 CD	72.1 ± 4.6 CD
T3 (crowns 7-9)				
7	135.7 ± 18.2 CD	80.2 ± 14.0 CD	113.2 ± 3.4 CDE	81.0 ± 2.9 CDE
8	156.1 ± 28.4 DE	88.7 ± 18.3 DE	117.5 ± 2.3 DEF	88.4 ± 3.4 DEF
9	173.9 ± 8.2 DEF	95.4 ± 17.2 DEF	124.9 ± 3.4 EF	92.4 ± 1.8 EF
T4 (crowns 10-12)				
10	181.4 ± 15.6 EFGH	107.5 ± 20.2 EFGH	129.4 ± 10.5 EFG	104.5 ± 6.3 EFG
11	192.3 ± 10.0 FGHI	121.0 ± 7.5 FGHI	138.7 ± 7.6 FGH	110.0 ± 5.5 FGH
12	197.6 ± 10.1 GHIJ	130.2 ± 6.7 GHIJ	148.3 ± 4.5 GH	120.8 ± 2.6 GH
T5 (crowns 13-15)				
13	203.5 ± 20.2 HIJK	135.2 ± 16.0 HIJK	156.5 ± 12.6 HI	130.2 ± 2.5 HI
14	209.6 ± 22.4 IJKL	143.5 ± 7.0 IJKL	171.6 ± 19.7 IJ	145.7 ± 5.2 IJ
15	212.9 ± 15.1 IJKL	147.8 ± 9.0 IJKL	185.6 ± 12.6 JK	146.9 ± 5.9 JK
T6 (crowns 16-18)				
16	222.9 ± 20.6 JKL	151.4 ± 19.9 JKL	189.2 ± 9.8 JK	152.5 ± 13.8 JK
17	232.4 ± 6.6 KL	153.3 ± 2.9 KL	192.1 ± 5.8 JK	155.7 ± 6.8 JK
18	239.7 ± 8.0 L	157.3 ± 3.9 L	194.8 ± 16.9 K	158.9 ± 2.4 K

[a] *Different letters compare milling periods vertically by Tukey's post hoc HSD test (*$p<0.05$*).*

tal direction for both ceramic systems in the different milling periods, followed by the Tukey honestly significant difference (HSD) test, with a confidence level of 0.05 to determine the main differences. The marginal misfit verified in the different directions (vertical and horizontal) and the ceramic systems (IPS e.max CAD and Rosetta SM) were not considered for multiple comparisons in the analysis. The analysis for the marginal misfit was performed only on the milling periods for the ceramic systems individually, in the vertical and horizontal directions separately. All tests were performed with a statistical software package (SigmaPlot 12.0 for Windows, Systat Software Inc., Chicago, IL, USA). The data for the morphologic characterization of the diamond burs were descriptively analyzed.

RESULTS

The data for the marginal misfit measurements were tabulated and subjected to the normality test (Shapiro-Wilk; $p=0.165$) and the equal variance test ($p=1.0$), presenting normal distribution. Significant differences were detected in the marginal misfit of the crowns at the different milling periods (T1-T6) for both ceramic systems, with a progressive increase in the marginal gap according to the successive millings of the ceramic crowns ($p<0.001$). The mean marginal misfit values are shown in Table 1.

The results of the morphologic characterization of the diamond burs by quantitative image analysis are shown in Table 2. Progressive loss of diamond granules from the surfaces of the burs was observed following the successive millings of the ceramic crowns, as shown by the reduction in granule count and the percentage of granules. The surface changes were observed on the morphology of the diamond burs used for milling the crowns of both ceramic systems (Figure 6A-D). Loss of the diamond granules and changes in the metallic matrix were also noted on the SEM images. Comparatively, the cylindrical bur showed more extensive morphologi-

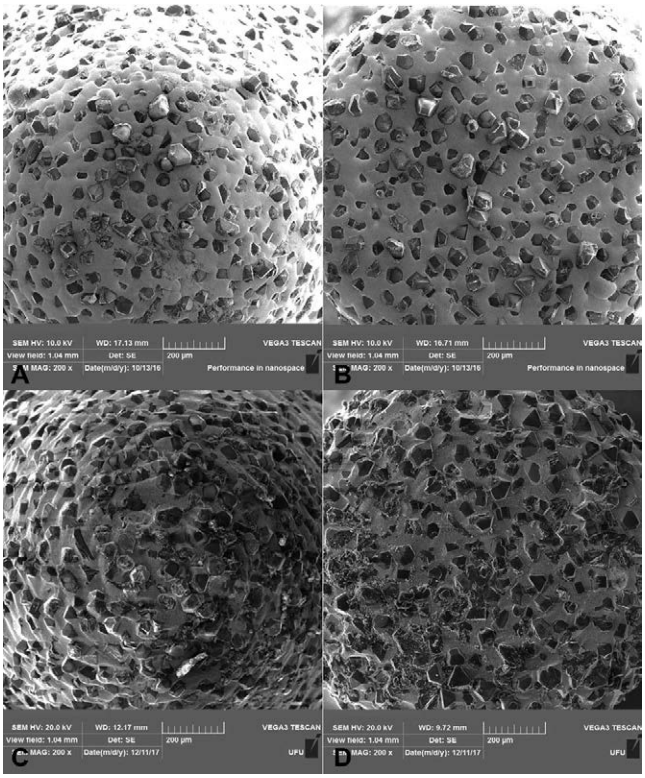


Figure 6. *(A, B): SEM images of the diamond burs before the first use—cylinder pointed bur and step bur, respectively.* (C, D): *SEM images of the diamond burs after successive millings—cylinder pointed bur and step bur, respectively.*

cal changes after successive use, such as loss/fracture of diamond grains, than the step bur (Figure 6C).

DISCUSSION

According to the results of the present in vitro study, the first null hypothesis was rejected, since significant differences were detected in the marginal fit of the monolithic ceramic crowns obtained in the different milling periods for both ceramic systems. The second null hypothesis was also rejected, since changes were observed in the surface morphology of the CAD/CAM diamond burs after successive millings of monolithic ceramic crowns for both ceramic systems, as observed by the decreased quantity of diamond granules. It seems that the marginal misfit of the ceramic crowns is directly related to the quality of diamond burs.

Since the tooth preparation design performed freehand may influence the marginal misfit of indirect restorations, standardized composite resin dies were used in the present study to reduce variables related to the operator and to avoid interference with the scanning procedure, besides allowing for the insertion of the crowns onto the dies to be performed accurately and reproducibly.[27] To avoid unadvertised wearing of a master resin die during the insertion and removal of the ceramic crowns, which could influence the marginal misfit assessments along the study, individual identical resin dies were produced by additive manufacturing instead of using a single die. The literature supports termination in shoulder with rounded axiogingival angles, since this design presents acceptable marginal misfit values.[26] The composite resin material used for obtaining the standardized dies presents distinct radiopacity properties when compared with those of dental ceramics, which are useful when measurements are performed by tomographic assessments, since it simplifies readings between/among the different structures. The occlusal and axial anatomy of the ceramic crowns produced was also standardized, but the software was allowed to calculate the best design for the cervical termination. The crowns tested in this study were not adhesively luted to the composite resin dies to avoid any possible influence in the marginal adaptation

Table 2: *Morphologic Characterization of the Diamond Burs Used for Obtaining the Lithium Disilicate Full-Ceramic Crowns According to the Ceramic System and Milling Periods: Mean of Diamond Granules per Area (μm), Percentage of Diamond Granules per Area (%), and Diamond Granules Count per Area (GC)*

Milling Period	IPS e.max CAD						Rosetta SM					
	Cylinder Bur			Step Bur			Cylinder Bur			Step Bur		
	Mean (μm)	Area %	GC	Mean (μm)	Area %	GC	Mean (μm)	Area %	GC	Mean (μm)	Area %	GC
T0	105.0	41.2	38	90.6	35.5	45	102.2	40.1	36	90.3	35.4	38
T1	92.0	36.1	32	71.7	28.1	36	98.9	38.8	33	87.3	34.2	37
T2	91.2	35.7	32	81.0	31.8	35	83.2	32.6	33	65.1	25.5	35
T3	75.5	29.6	30	84.9	33.3	31	82.3	32.25	31	64.6	25.3	32
T4	59.8	23.5	29	88.8	34.8	27	81.4	31.9	30	64.1	25.1	30
T5	66.0	25.9	22	71.2	27.9	26	71.9	28.2	27	60.8	23.9	30
T6	57.0	22.3	20	59.6	23.4	26	61.5	24.1	27	59.7	23.4	28

caused by the luting materials due to factors such as cement type, viscosity, composition, radiopacity, shrinkage stress, and fixation techniques.[14] Rather, the crowns were stabilized using only a thin layer of a vinylpolysiloxane material prior to marginal misfit assessments. As well, no polishing, glazing, or internal adjustments of the crowns were made for any of the specimens to avoid influence of those techniques.

The use of different CAD/CAM systems,[3] scanning cameras, and the application of opacifier powders may also influence the marginal misfit values, as demonstrated by previous investigations.[2,28] Therefore, in this study, all of these factors and even the operator were standardized to perform the scanning of all dies without further interference. During the pilot tests, the necessity for standardized scanning of the resin dies was noted, since this process was critical to be performed because of the die instability when it was held freehand. Thus, a custom device was developed by the authors for die stabilization as previously described, making the scanning procedure reproducible for all specimens. This change in the digitizing protocol resulted in increased accuracy of scanning with reduced marginal misfit values compared with the pilot specimens that were scanned freehand, showing that the cast scanning procedure must be performed rigorously.

The morphologic characterization of the diamond burs showed loss of the superficial diamond granules and changes in the metallic matrix following the successive millings of the ceramic crowns for both ceramic systems (Figure 6A-D). As seen, these findings may be related to previous results reporting progressive increases in the marginal misfit of ceramic crowns milled in CAD/CAM systems and the damage caused by the diamond burs on the ceramic surfaces,[6,15] which is also in accordance with the marginal misfit results found in the present investigation. Despite the fact that the previous study cited was performed with Y-TZP–based restorations showing that burs were similar after 30 successive millings, the main surface roughness values were significantly different after 27 millings for Y-TZP–based restorations with cyclic fatigue and after 24 millings for restorations without cycling fatigue.[6] Other studies using composite or glass ceramic CAD/CAM blocks also showed no differences in surface roughness of the materials after successive millings.[7–9]

The surface roughness of the ceramic crowns was not evaluated in the present study, but SEM images showed changes in the morphology of the diamond granules of the burs immediately after the first milling period (three crowns). Thus, the images of the diamond burs were quantitatively evaluated as a complementary analysis based on the total area of the images. The image analysis of the diamond burs showed progressive loss of the superficial diamond granules following successive millings of the ceramic crowns, as shown by the reduction in the percentage of granules (Table 2). The cylindrical bur showed increased morphological changes after the successive uses, such as loss/fracture of the diamond grains compared with those on the step bur (Figure 6C). This probably occurred because the diamond bur has a more severe regimen of use, since the occlusal anatomy of the crown is defined by this instrument, as described by the manufacturer.

The efficiency of the diamond burs was sequentially reduced following the successive millings of the lithium disilicate crowns, and a progressive increase in the marginal misfit of the full-ceramic crowns obtained was observed for both experimental groups. This behavior may have resulted from reductions in the cutting efficiency of the diamond instruments, as could be observed with the progressive loss of diamond granules on the surfaces of the burs. Reports in the literature support the separation of the diamond granules at the beginning of the cut, leaving fewer particles and a large number of diamond-shaped holes in the metal matrix on the surfaces of the CAD/CAM diamond burs.[5] Thus, the degradation of the diamond burs can be intimately associated with increased marginal misfit for the ceramic crowns milled, as observed in the present investigation. In addition, it can be assumed that the time required for milling the monolithic lithium disilicate ceramic crowns in CAD/CAM systems may increase with the sequential use and reduced efficiency of the diamond burs. Pilot measurements of the time taken for milling the crowns were performed in this study, and this assumption seems to be adequate, since increased milling time was observed for the crowns obtained in the last milling periods. Not only may this depend on the quantity of diamond granules remaining on the burs, but it also may be influenced by factors such as the cooling system and the performance of the electric motors of the CAM unit. Thus, further studies are needed.

Some studies have described that, despite the fact that micro-CT methodology was seldom used for the measurement of marginal adaptation, this is a reliable technique that allows two-dimensional and three-dimensional measurements to be made from any angle or position of the specimen.[1,2,18] A

previous investigation reported significant differences in the marginal adaptation of restorations produced by different CAD/CAM systems.[26] Therefore, in this study, a single CAD/CAM system and software were used to ensure that the marginal misfit values of ceramic crowns were obtained in equivalent form.

A marginal gap of less than 120 μm has been described as being clinically acceptable for indirect restorations.[19] Although this misfit value is supported by the literature, recent studies have indicated that the maximum level of vertical misadjustment should stand below the 75-μm limit,[1,2] which is a smaller misfit value than those found for the crowns of both experimental groups in the present study. Considering the marginal misfit values found in this study, the maximum number of subsequent millings per diamond bur set to produce indirect restorations with acceptable misfit in the vertical direction would be 11 and 12 for IPS e.max CAD and Rosetta SM ceramic systems, respectively. On the other hand, if the horizontal direction was considered, only six and eight millings would be performed for IPS e.max CAD and Rosetta SM, respectively. Any further milling with the same bur set resulted in increased marginal misfit values, higher than 120 μm. However, if 120 μm is considered the maximum acceptable misfit value for the marginal gap, after 12 lithium disilicate crown millings, the diamond bur set should no longer be used, since the next milling would result in increased misfit values that may jeopardize the success of the restorative procedure.

The present in vitro study has some intrinsic limitations, such as the absence of adjacent and antagonist teeth during the scanning procedures. Also, the influence of the crystallization process was not taken into consideration, and only a chairside CAD/CAM system was evaluated. Thus, further studies are needed to investigate additional variables. Regarding the increase in the milling irregularity demonstrated by the successive use of the diamond burs in the marginal misfit produced by these instruments, more studies about surface roughness and fracture resistance should be performed. Little information is available from the manufacturers about the number of milling cycles that can be performed before the diamond burs need to be replaced. The manufacturer of the CAD/CAM used in this study established that a new set of diamond burs is capable of milling up to 15 consecutive indirect restorations, regardless of the extension and restorative material; however, still no evidence is available to support this information.[8] As shown, the decision to change the diamond bur set is usually defined by the CAD/CAM unit, but ideally, this decision should also be made by the operator. More information is needed about these issues, and a better understanding of the criteria for replacing the diamond bur set is necessary to improve the quality of the ceramic restorations produced by chairside CAD/CAM systems.

CONCLUSIONS

Within the limitations of the present in vitro study, it was observed that the milling efficiency of the diamond bur set used in a chairside CAD/CAM system was progressively reduced with the sequential milling of lithium disilicate ceramic crowns for both ceramic systems evaluated. The reduced efficiency of the CAD/CAM diamond burs affected the cervical adaptation of the ceramic restorations, with a sequential increase in the mean marginal misfit. Inappropriate milling of new full lithium disilicate ceramic crowns occurred after 11 successive millings for IPS e.max CAD and 12 for Rosetta SM under the parameters tested.

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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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Effect of Various Surface Treatments on Ti-Base Coping Retention

K Kemarly • SC Arnason • A Parke • W Lien • KS Vandewalle

Clinical Relevance

Mechanical surface roughening of the titanium-abutment base is necessary to increase the pull-off bond strength of the lithium disilicate abutment material. Additional chemical surface treatment may further increase the bond strength, but the effects are product specific.

SUMMARY

Objective: The titanium-cement interface of a Ti-Base implant crown must be able to resist intraoral pull-off forces. The purpose of this study was to evaluate the effect of mechanical and chemical surface treatments of a titanium-abutment base (Ti-Base, Dentsply/Sirona) on the pull-off bond strength of a lithium disilicate abutment coping.

Methods and Materials: Ti-Bases were divided into nine groups of 10 copings each that varied in both mechanical surface treatment (none; Al_2O_3 air abrasion; CoJet silicoating, 3M ESPE) and chemical treatments (none; Monobond Plus, Ivoclar Vivadent; Alloy Primer, Kuraray). Lithium disilicate abutment copings (IPS e.max CAD, Ivoclar Vivadent) were designed and milled. After crystallization, the copings were cemented onto the Ti-Bases with a resin cement (MultiLink Hybrid-Abutment Cement, Ivoclar Vivadent) according to the manufacturer's recommendations. The copings were torqued to a mounted implant, and the access channel was sealed with composite. After 24-hour storage and 2000 thermal-cycles in distilled water, the copings were subjected to a removal force parallel to the long axis of the interface until fracture. Data were analyzed with multiple one-way analyses of variance and Tukey post hoc tests (α=0.05).

Results: Significant differences were found between groups based on type of surface treatment ($p<0.05$).

Conclusions: Chemical surface treatment with Monobond Plus and mechanical surface treatment with CoJet silicoating or Al_2O_3 air abrasion resulted in the greatest pull-off bond strength. Alloy Primer did not provide a statistically significant increased pull-off bond strength when the surfaces were mechanically treated with Al_2O_3 air abrasion or CoJet silicoating. The lack of any mechanical surface treatment resulted in the lowest pull-off bond

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strength regardless of the type of chemical surface treatment.

INTRODUCTION

The advent of computer-aided design/computer-aided manufacturing (CAD/CAM) dentistry has yielded several advantages over traditional dental workflows. Narrowing the focus on implant-supported prostheses, the Ti-Base and ScanPost system from Dentsply/Sirona (Charlotte, NC, USA) have made possible the fabrication of a ceramic implant abutment and prosthesis without the need for a traditional impression and cast.[1] Joda and Bragger[2] showed that this workflow decreased the cost to patients by 30% and halved the laboratory workload.

CAD/CAM technology is compatible with multiple restorative materials. For the posterior region, high occlusal forces have required materials with high fracture strengths, and zirconia has been traditionally shown as an effective material for abutment and restoration of implants in this critical region.[3-5] Zirconia also features high biocompatibility as well as more acceptable esthetics compared with metallic restorations.[3]

Recently, Ivoclar Vivadent has introduced a lithium disilicate CAD/CAM block marketed for use with the Ti-Base system. Lithium disilicate does not share a similar strength with zirconia; however, Elsayed and others[4] concluded that lithium disilicate abutments and hybrid-abutment crowns displayed sufficient strength during dynamic loading, lasting more than 1.2 million fatigue load cycles with "higher forces than physiological occlusal forces."[4] More esthetic restorations are possible with lithium disilicate in comparison with zirconia because of the higher degree of translucency. Also, lithium disilicate can be crystallized in the oven with a significantly shorter heating cycle, making it a very attractive restorative choice in terms of workflow.[6] Lithium disilicate also has the advantage of a more predictable adhesive bond than zirconia. The silica nature of the lithium disilicate allows for surface etching with hydrofluoric acid prior to silanization. The polycrystalline nature of zirconia may require other methods of surface treatment that may rely more on mechanical rather than chemical retention.[7]

Studies have investigated the effect of different mechanical and chemical surface treatments on titanium but not specifically the Ti-Base implant abutment base. The mechanical surface treatments commonly include air abrasion with Al_2O_3 and tribochemical silica coating. Air abrasion increases retention by roughening the titanium surface. Ebert and others[8] showed that air abrasion significantly increased bonding between zirconia copings and titanium compared with the control. Von Maltzahn and others[9] also investigated the mechanical surface treatment of titanium but included tribochemical coating. Tribochemical coating serves to embed silica particles into the surface of a material via high-speed impact from an air abrasion unit. In that study, it was found that the tribochemical surface treatment was less retentive than air abrasion with Al_2O_3.[9]

In addition to mechanical surface treatments, chemical surface treatments also exist for modifying titanium. According to the manufacturer, Monobond Plus (Ivoclar Vivadent, Amherst, NY, USA) and Alloy Primer (Kuraray, Houston, TX, USA) increase the bond strength to metals, with the use of Alloy Primer specifically mentioned for titanium. Both use functional monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) to promote chemical bonding between the metal and the cement.[10] Specifically, Monobond Plus contains ethanol, trimethylpropyl methacrylate (silane), methacrylated phosphoric acid ester (10-MDP), and disulfide acrylate. Alloy Primer contains methacrylated phosphoric acid ester (10-MDP) as well as 6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol (VBATDT) in acetone. Little research has been done studying the bond strength of Monobond Plus to titanium, but Veljee and others[10] showed that the addition of Alloy Primer increased the retention of a resin cement to pure titanium to a statistically significant level. They postulated that the Alloy Primer promotes wettability, thus increasing the adhesive bonding. Yanagida and others[11] also found that using only Alloy Primer combined with a resin cement showed significantly higher bond strength to pure titanium compared with air abrasion or tribochemical modification alone.

While research has shown that there is potential for differences in surface treatment in relation to the bond strength of titanium to resin cement, no research has evaluated the pull-off bond strength between the lithium disilicate abutment material and the titanium implant abutment base.[12] It is important to note that the Ti-Base is a medical grade 5 titanium aluminum alloy, which might behave differently than the pure titanium used in aforementioned studies. It is important to ensure that the restorations placed on the Ti-Bases are retentive and able to serve the patient in the long term. The purpose of this study was to evaluate several surface

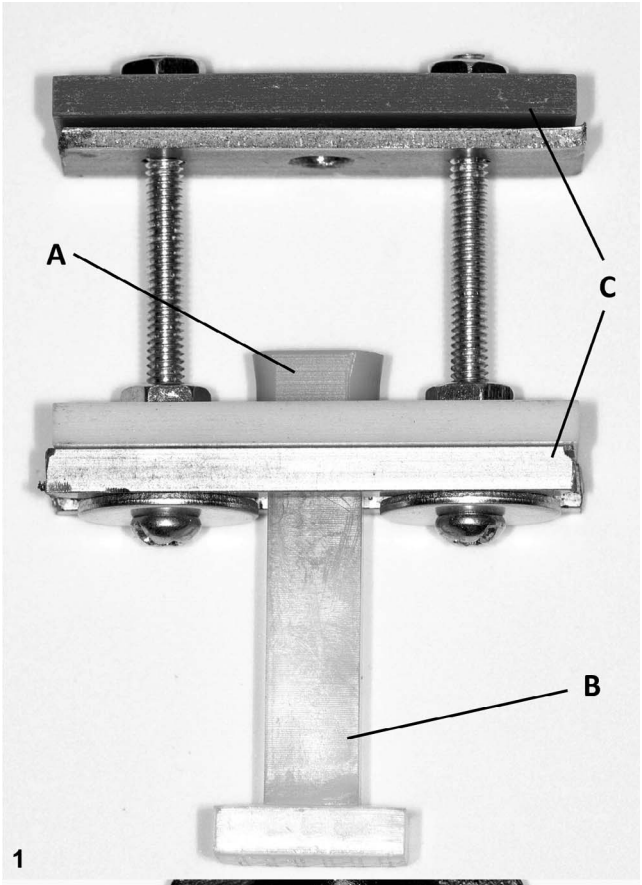
treatments in differing combinations and their effect on the pull-off bond strength of lithium disilicate to the Ti-Base implant abutment base. The null hypothesis tested was that there would be no difference in pull-off bond strengths of the lithium disilicate copings from the Ti-Base regardless of surface treatment modality.

METHODS AND MATERIALS

A custom coping was designed in SolidWorks CAD three-dimensional (3D) software (Dassault Systemes, Vélizy-Villacoublay, France). In addition, a custom cradle was designed to adapt an existing vice grip of the universal testing machine (model 5543, Instron, Norwood, MA, USA) to fit intimately with the coping to allow for even distribution of pull-off forces without possible fracturing due to compression of the lithium disilicate from the vice clamps. The cradles were 3D printed (Objet 260 Dental Selection, Stratasys Ltd, Eden Prarie, MN, USA). The coping was milled in lithium disilicate (IPS e.max CAD abutment, LT, shade A2, Ivoclar Vivadent) on a five-axis milling unit (CORiTEC 450i, imes-icore GmbH, Eiterfeld, Germany) and placed on an implant lab analog (Certain 4.1 mm, Biomet 3i, Palm Beach Gardens, FL, USA) and 3D scanned into the InLab software (v16.0, Dentsply/Sirona). Ninety copings were milled from the IPS e.max CAD abutment using a milling unit (MCXL, Dentsply/Sirona). The lithium disilicate copings were crystalized in a ceramic oven (Programat P500, Ivoclar Vivadent) following the manufacturer's instructions. To properly hold the implants, a custom base was designed in Solid Works with a channel. Holding towers were 3D printed (SLA Viper si2, 3D Systems, Rock Hill, SC, USA), and an implant (Certain 4.1 mm, Biomet 3i) was threaded into each. Each "implant tower" was analyzed to ensure that the implant was placed parallel to the long access of the tower to ensure pull-off forces would also be parallel.

In preparation for cementation, the titanium bases (Ti-Base, BC 4.1L, Dentsply/Sirona) were temporarily held in an implant lab analog (Certain 4.1 mm, Biomet 3i). Ninety Ti-Bases were divided into three groups of 30 each. Thirty of the Ti-Bases received no surface treatment. Thirty were air abraded (Basic Quattro IS, Renfert, Chicago, IL, USA) using 50 μm Al_2O_3 at 2.0 bar and then steam cleaned (i700B, Reliable, Toronto, Ontario, Canada). The remaining 30 Ti-Bases were treated with tribochemical silica coating (CoJet Sand, 3M ESPE, St Paul, MN, USA) at 2.0 bar for 15 seconds until the metal turned a uniformly dark color per the manufacturer's recommendation and steam cleaned. In each of the three groups of 30 Ti-Bases, 10 were primed with Monobond Plus primer applied to the Ti-Base bonding surface, allowed to react for 60 seconds, and gently blown dry with a three-way syringe. Ten were treated with Alloy Primer with a cotton pellet and left to dry per manufacturer recommendations. And the remaining 10 received no chemical treatment.

The intaglio surface of the custom lithium disilicate coping was etched for 20 seconds with hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent) and rinsed thoroughly with water from a three-way syringe. Monobond Plus primer was applied to the etched surfaces and allowed to react for 60 seconds and gently blown dry with a three-way syringe. The specimens were cemented to the Ti-Base using an autopolymerizing resin cement (MultiLink Hybrid Abutment Cement, Ivoclar Vivadent) according to the manufacturer's recommendations. Glycerin gel was applied to the cementation interface for seven minutes and then rinsed off with a three-way syringe. During setting of the cement, the specimens were set in a custom-made jig that allowed for placement of a 100-g weight onto the specimen to ensure standardized pressure during setting of the cement. After removal of the glycerin gel, the cement interface was polished to mimic actual clinical procedures. Next, the Ti-Base specimens were torqued to the implant in the experimental apparatus at 20 N/cm. Clearfil SE Bond (Kuraray) was applied to the screw channel and light cured (Bluephase G2, Ivoclar Vivadent). Irradiance was recorded with a power meter (Powermax, Coherent Inc, Santa Clara, CA, USA) and considered acceptable since it was greater than 1000 mW/cm^2. Filtek Z250 (3M ESPE) was placed incrementally and light cured. The cameo surface of the composite was polished with Enhance and Pogo polishing tips (Dentsply). The assembled specimens (Figure 1) were then placed in distilled water and stored in an incubator (model 20 GC, Quincy Labs, Chicago, IL, USA) for 24 hours at 37°C. Then, the specimens were thermal-cycled in distilled water for 2000 cycles at 5°C and 55°C with a dwell time of 30 seconds at each temperature (Sabri Dental Enterprise, Downers Grove, IL, USA). Each specimen was then loaded under tension in a universal testing machine (Instron) with a pair of customized vice jig assemblies holding the lithium disilicate restoration on one side and the 3D-printed resin tower in the other (see Figure 2). The universal testing machine subjected the lithium disilicate copings to a removal force parallel to the long axis of



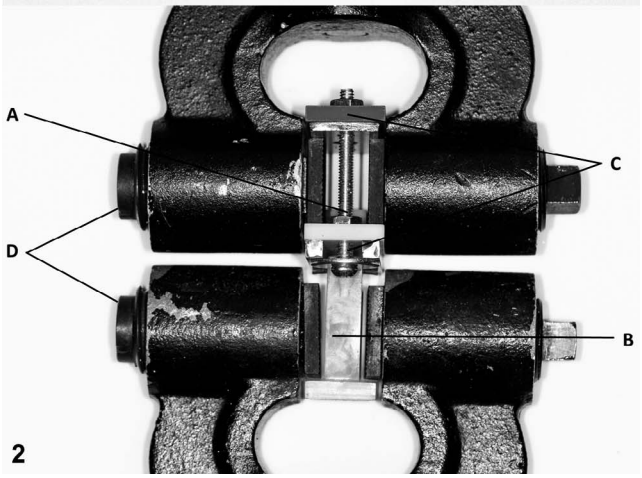


Figure 1. *Cemented specimen (A) loaded onto an implant threaded into a 3D-printed tower (B). The specimen is cradled by a custom adapter (C) designed to fit into the universal testing machine.*

Figure 2. *Cemented specimen (A) loaded onto an implant and threaded into a 3D-printed tower (B). The specimen is cradled by a custom adapter (C) fitted into the grips of the universal testing machine (D).*

the interface at a speed of 1 mm/min until the copings fractured or separated from the Ti-Bases. The maximum force between components was recorded in newtons.

Once the pull-off tests were complete, the fractured surfaces of all specimens were analyzed using a stereomicroscope at 10× magnification (SMZ-1B, Nikon, Melville, NY, USA). The fractured surfaces were evaluated and classified into the following failure modes: cement remaining on Ti-Base only, cement remaining on lithium disilicate only, cement remaining on both Ti-Base and lithium disilicate, fractured lithium disilicate with no cement remaining on Ti-Base, fractured lithium disilicate with some cement remaining on Ti-Base, and fractured lithium disilicate with a portion of lithium disilicate still bonded to the Ti-Base. The surface roughness of nine Ti-Bases was analyzed after mechanical modification (three per group). Surface roughness (Ra) was measured using a noncontact profilometer (3D Laser-Scanning Confocal Profilometer, Keyence, Itasca, IL, USA) and then analyzed using its proprietary software. The morphology of the Ti-Base surfaces was investigated by field-emission scanning electron microscopy (Sigma VP, Carl Zeiss, Oberkochen, Germany). The elemental composition of the Ti-Base surfaces was characterized by energy-dispersive spectroscopy (X-Max, Oxford Instruments, Abingdon, UK).

A mean removal force (N) and standard deviation were determined for each of the nine groups. Data were analyzed using a two-way analysis of variance (ANOVA) to evaluate the effect of mechanical (three levels) or chemical treatments (three levels) of the Ti-Base surface on the pull-off strength of the lithium disilicate specimens (α=0.05).

RESULTS

The results of the two-way ANOVA found significant differences between groups based on mechanical surface treatments ($p<0.001$) and chemical surface treatment, but also there were significant interactions ($p<0.001$). The data were further evaluated by multiple one-way ANOVAs per mechanical or chemical surface treatment (see Table 1).

Chemical surface treatment with Monobond Plus and mechanical surface treatment with Al_2O_3 air abrasion (896.0±173.1 N) or CoJet silicoating (1011.5±120.2 N) resulted in the greatest pull-off bond strengths, but they were not significantly different from each other. Both groups were significantly greater than Al_2O_3 air abrasion (650.3±54.7

Table 1: *Pull-off Bond Strength of Lithium Disilicate Abutment Copings After Various Mechanical and Chemical Surface Treatments of the Titanium-abutment Base* [a]

Mechanical Surface Treatment	Pull-off Bond Strength (SD), Newtons		
	Chemical Surface Treatment		
	Monobond Plus	Alloy Primer	None
Al_2O_3 air abrasion	896.0 (173.1) Aa	759.5 (127.1) ABa	650.3 (54.7) Ba
CoJet silicoating	1011.5 (120.2) Aa	491.1 (102.3) Bb	501.8 (49.0) Bb
None	340.9 (95.5) Ab	332.4 (85.4) Ac	393.1 (65.3) Ac

[a] *Groups with the same uppercase letter per row or lowercase letter per column are not significantly different ($p>0.05$).*

N) or CoJet silicoating (501.8±49.0 N) without any chemical treatment.

Chemical surface treatment with Alloy Primer and mechanical surface treatment with Al_2O_3 air abrasion (759.5±127.1 N) did not provide a significant increase in pull-off bond strength compared with no primer (650.3±54.7 N). Similarly, treatment with Alloy Primer and CoJet silicoating (491.1±102.3 N) did not provide a significant increase in pull-off bond strength compared with no primer (501.8±49.0 N). The lack of any mechanical surface treatment resulted in the lowest pull-off bond strength regardless of the type of chemical treatment. No mechanical treatment and Monobond Plus (340.9±95.5 N) was not significantly different from Alloy Primer (332.4±85.4 N) or no primer (393.1±65.3 N).

The most frequently observed failure mode for each group was as follows: Monobond Plus/Al_2O_3 abrasion, 100% had fractured lithium disilicate with a fragment of the lithium disilicate firmly bonded to the Ti-Base (see Figure 3); Monobond Plus/CoJet silicoating, 100% had fractured lithium disilicate with a fragment of the lithium disilicate firmly bonded to the Ti-Base; Monobond Plus/no mechanical, cement on both the lithium disilicate and the Ti-Base; Alloy Primer/Al_2O_3 abrasion, fractured lithium disilicate with a fragment of the lithium disilicate firmly bonded to the Ti-Base; Alloy Primer/CoJet silicoating, fractured lithium disilicate with a fragment of the lithium disilicate firmly bonded to the Ti-Base. Alloy Primer/no mechanical, fractured lithium disilicate with some cement left on the Ti-Base; no chemical/Al_2O_3 abrasion, cement on both the lithium disilicate and the Ti-Base; no chemical/CoJet silicoating, cement on both the lithium disilicate and the Ti-Base; no chemical/no mechanical, fractured lithium disilicate with no cement left on the Ti-Base. See Figure 4.

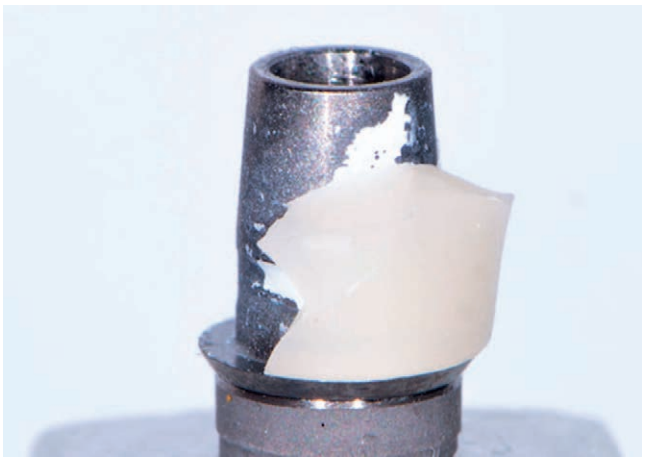


Figure 3. *Fragment of lithium disilicate bonded to the cervical area of the Ti-Base with cement remaining.*

In comparing surface roughness, Al_2O_3 abrasion gave an overall rougher surface (0.925±0.124 μm) compared with CoJet silicoating (0.555±0.000 μm) and control (0.297±0.040 μm). Evaluation of surface composition showed that the CoJet silicoating samples did in fact contain a higher surface composition of Si by weight (5.73%) compared with the Al_2O_3 abrasion (0.25%) or control (0.23%). Both the Al_2O_3 and CoJet silicoated Ti-Bases had less available Ti than the untreated Ti-Bases (35.53% and 27.29% compared with 70.9%). Scanning electron micrograph photos of the treated Ti-Base surfaces can be seen in Figures 5-7.

DISCUSSION

The purpose of this study was to evaluate several surface treatments in differing combinations and their effect on the pull-off bond strength of lithium disilicate to the Ti-Base implant abutment base. Within the limitations of this laboratory study, the null hypothesis was rejected because the results of the study found statistically significant differences in pull-off bond strengths of the lithium disilicate copings from the Ti-Base dependent on surface treatment modalities. Based on the results, it would appear that the most important factor in bonding to the Ti-Base is the use of some form of mechanical treatment. Without any mechanical surface treat-

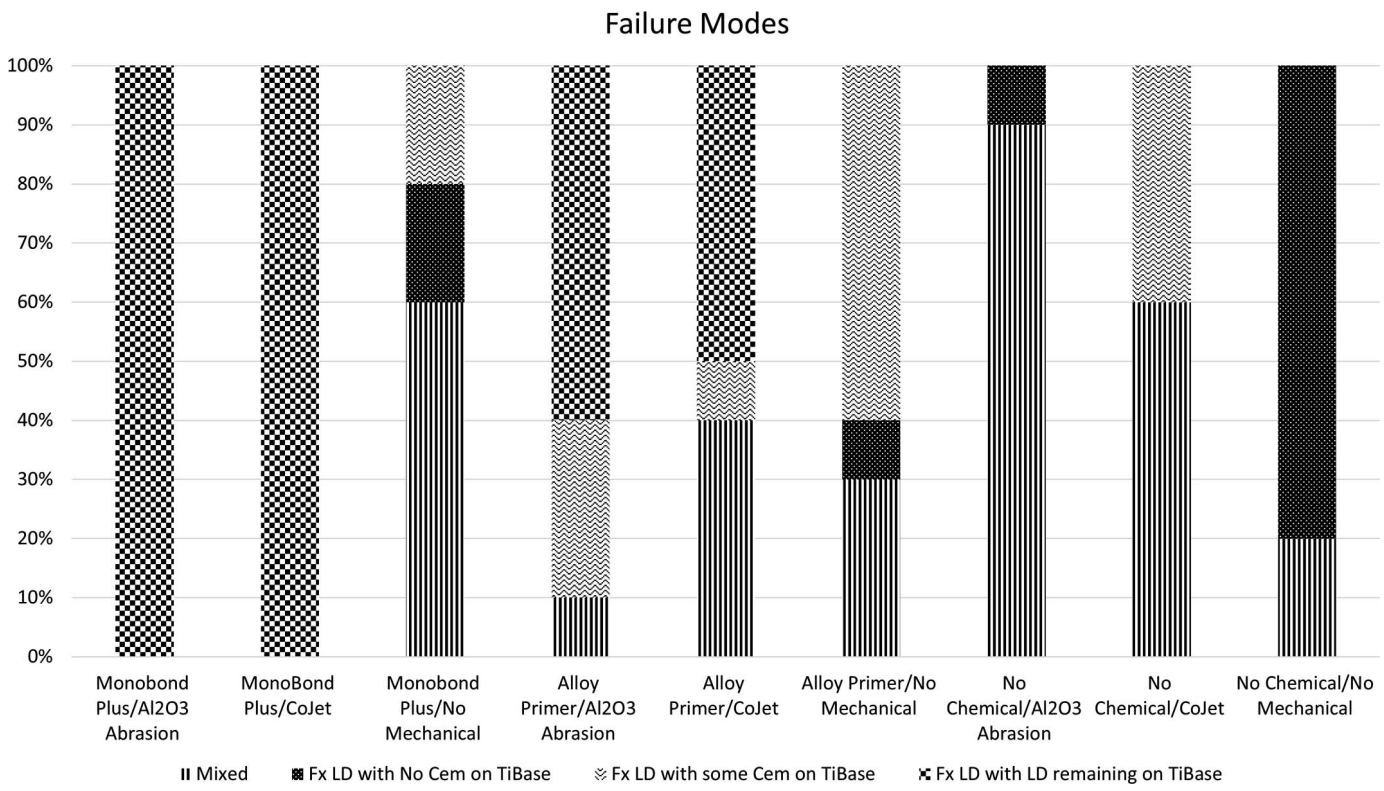
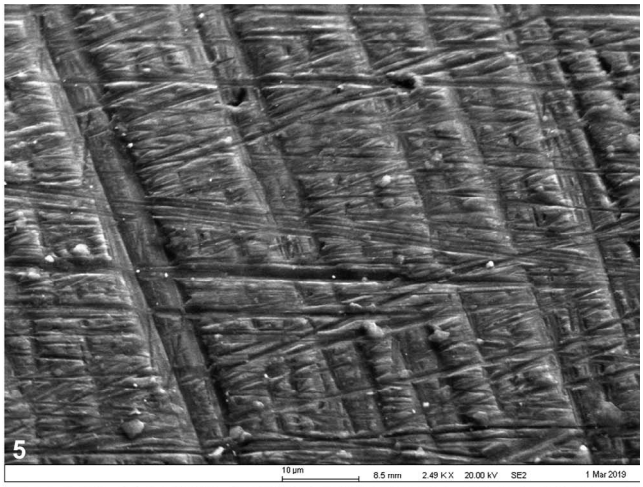


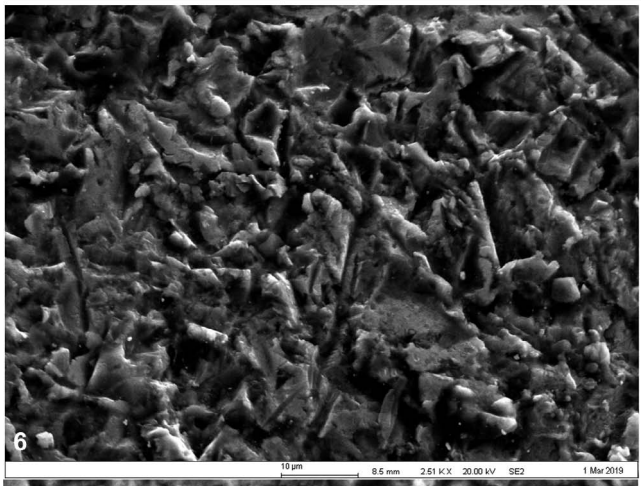
Figure 4. *Graphical representation of the various failure modes.*

ment, there was no statistically significant effect by any of the three chemical treatments. This is likely because of the increase in the bonding surface area due to increased surface roughness. The pull-off strengths correlated with the measured roughness of the Ti-Bases, with Al_2O_3 producing the roughest surface and the highest overall force with no chemical surface treatment. Papadopoulos and others[13] showed that the use of a large particle size increased the surface roughness and promoted increased mechanical retention when firing porcelain onto titanium. The effect of air abrasion on grade 5 alloy was shown to increase the shear bond strength to lithium disilicate.[14] However, a recent study by Linkevicius and others[15] showed that air abrasion of Ti-Bases with Al_2O_3 had a negative effect on retention of the zirconia coping. That study, however, used a different brand of titanium base (BioHorizons IPH Inc, Birmingham, AL, USA) that contains built-in retentive grooves. The air abrasion was shown to dull the retentive grooves, which could account for the discrepancy.[15]

The use of CoJet was overall less retentive than Al_2O_3 without chemical surface treatment. CoJet uses 30-μm particles, per the manufacturer. As described in this study, the smaller particle size yielded a smaller surface roughness according to the profilometer scan and is consistent with Fonseca and others,[16] who also showed particle size had a significant effect on bonding. Per the manufacturer's instructions, CoJet requires silane to be effective for bonding. When the silane containing Monobond Plus was added, the pull-off strength nearly doubled compared with the use of Alloy Primer, which does not contain silane. In this study's methodology, CoJet was applied at 2 bar. Per the manufacturer, this is the minimum accepted pressure that creates enough energy to embed the silica particles into the substrate. Were the maximum of 3 bar used, the bonding might have been significantly increased, but the authors felt it important to maintain consistency with the Al_2O_3 groups.

Monobond Plus was highly effective when combined with mechanical roughening of the Ti-Base with Al_2O_3 or CoJet silicoating. The effectiveness was likely due to a combination effect of each of its three functional components: trimethylpropyl methacrylate (silane), methacrylated phosphoric acid ester (10-MDP), and disulfide acrylate. As mentioned, silane in addition to CoJet allows for effective bonding. Air abrasion in addition to MDP and sulfur-containing compounds have also been shown to be





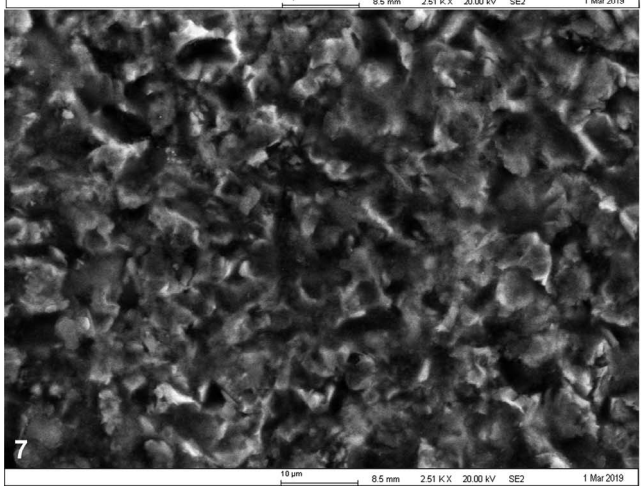


Figure 5. *Scanning electron micrograph photo of unmodified Ti-Base.*

Figure 6. *Scanning electron micrograph photo of Al_2O_3-treated Ti-Base.*

Figure 7. *Scanning electron micrograph photo of CoJet-treated Ti-Base.*

effective in bonding to titanium.[17-19] This study used Multilink Hybrid Abutment Cement, which is manufactured by Ivoclar Vivadent and is intended to be used with MonoBond Plus.

Unlike previous studies, the application of Alloy Primer did not appear to improve bonding between the Ti-Base and resin cement despite sharing similar components with Monobond Plus. One possible explanation is the potential differences between formulations. Ethanol is used as a solvent for Monobond Plus, while Alloy Primer uses acetone. The greater volatility of acetone may decrease the substantivity of the Alloy Primer. In their evaluation of the effect of organic solvents, Amaral and others[17] found that the type of solvent (ethanol or acetone) had no effect on the degree of conversion or resin-dentin bond stability; however, their study evaluated 4-methacryloyloxyethyl trimellitate anhydride adhesive (4-Meta Sun Medical Co, Kyoto, Japan) and not a primer, as investigated in this study.[17] In addition, thermal-cycling might have contributed to a decrease in the effects of Alloy Primer. Hiraba and others[18] looked at the effect of primers, including Alloy Primer and Monobond Plus, on the bond between tri-n-butylborane–initiated resin and a gold alloy. Part of the study design compared bond strengths before and after thermal-cycling. Their data showed that after thermal-cycling, the mean bond strength exhibited a significantly greater decrease in the groups using Alloy Primer compared with Monobond Plus.[18]

Groups with the greatest pull-off bond strengths combined mechanical modification and chemical surface treatments and shared the most common mode of failure: a fragment of lithium disilicate remaining firmly bonded to the Ti-Base with some cement on both the dislodged lithium disilicate coping and on the Ti-Base (see Figure 3). This failure mode indicates that while there was partial adhesive failure between the Ti-Base and coping interface, the bond between the remaining fragment and the Ti-Base was stronger than the tensile strength of that area of lithium disilicate coping. In all but two specimens, the remaining fragment was on the most cervical aspect of the Ti-Base and encased the tab used by the Ti-Base system for orientation of the crown on the abutment. Because of the taper of the Ti-Base, the cervical area has the largest diameter and thus the largest surface area for bonding. It is possible that the coronal portion of the Ti-Base with less surface area might have debonded first, creating greater tension between the coronal and apical segments. The failure mode of

the lithium disilicate copings in the remaining groups was more heterogeneous. As expected, the group with no modification of the Ti-Base featured the most cases of no cement remaining on the Ti-Base. The remaining groups all had some cement on both surfaces, indicating partial adhesive failure of both the Ti-Base cement interface and lithium disilicate cement interface.

The authors caution that this study used a single static test. While informative, static testing gives limited information on the effects of repeated forces on cement interfaces. In this study, the lowest tensile pull-off bond strength of 327.8 N was greater than the maximum jaw-opening strength of 142.86 N recorded in previous research.[20] In addition, more research is necessary using other types of surface primers and cements.

CONCLUSIONS

Based on the limitations of this study, when bonding lithium disilicate copings to Ti-Bases, mechanical roughening with either Al_2O_3 air abrasion or CoJet silicoating is recommended. Once mechanically modified, Monobond Plus appears to be the superior chemical primer of the materials tested for treating the Ti-Base when using MulitLink Hybrid Abutment Cement. Further studies are needed to compare additional combinations of materials for maximizing Ti-Base retention.

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 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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Dentin Staining Caused by Nano-silver Fluoride: A Comparative Study

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Clinical Relevance

The new formulations of nano-silver fluoride caused less dentin staining than the already available commercial agents 35% silver fluoride and silver diamine fluoride at 30% and 38%.

SUMMARY

The objective of this study was to evaluate the dentin staining potential of nano-silver fluoride (NSF 600 and 1500 ppm) compared with the following commercially available cariostatic agents: Advantage Arrest (Elevate Oral Care, West Palm Beach, FL, USA), Riva Star (SDI, Victoria, Australia), and Cariestop (Biodinâmica, Paraná, Brazil). Seventy-five extracted human molars were sectioned at the cementoenamel junction, and the occlusal enamel was removed for exposure of coronary dentin. The samples were divided among the five agents tested (n=15). The dentin staining (ΔE/ΔL) was analyzed with a digital spectrophotometer (VITA Easyshade, VITA Zahnfabrik, Bad Säckingen, Germany) at three different time points (before application, after two weeks, and after four weeks). Photographic images were also performed. The Kruskal-Wallis and Mann-Whitney tests compared the mean ΔE and ΔL values between groups. The NSF 600 and 1500 ppm resulted in the smallest color change (ΔE=1.02 and 1.53) and dentin staining after four weeks (ΔL=−0.76 and −1.2). The new formulations differed significantly from the commercial cariostatic agents ($p<0.001$). NSF might be an alternative to silver diamine fluoride since it does not compromise esthetics.

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INTRODUCTION

Dental caries is the most prevalent oral disease in the world, affecting all countries.[1,2] Oral hygiene is the most efficient method to prevent and control the disease.[3] However, several therapies have been proposed to reverse the carious process, including the topical application of fluoride, chlorhexidine, fluoride varnish, and silver diamine fluoride (SDF).[4-6] The last is a conservative, efficient, and less invasive alternative,[7-10] but its application can stain the tooth, compromising esthetics.[11-13]

The mechanism of action of SDF consists of the interaction of silver ions (Ag+) with the sulfhydryl groups of proteins and DNA of bacteria. This alters hydrogen bonding and thereby inhibits respiratory processes, cell wall synthesis, and cell division, which cause the death and eventual lysis of the cell.[14-16] However, remnant silver particles can precipitate and form a layer of silver phosphate on the carious dentin.[17,18]

The application of potassium iodide (KI) immediately after the use of SDF has been proposed to minimize this adverse effect.[11,17] In addition, new formulations based on silver nanoparticles also have been evaluated for their use in stain reduction.[19,20]

Smaller silver particles would be more efficient because of their greater surface area per volume, increasing the contact with microbial cells.[19,21-24] A new formulation, called nano-silver fluoride (NSF), contains silver nanoparticles, chitosan, and fluoride. This compound thus combines preventive and antimicrobial properties and promises not to cause tooth staining because of the size of its particles and because it does not undergo oxidation.[19,21,25,26] This new substance is safe to be used in humans, and its production costs are low.[25,27] In addition, it has shown good results in laboratory and clinical studies.[19,21,25-27] However, it remains to be established whether NSF causes less staining of permanent teeth.

Therefore, the objective of this study was to evaluate the dentin staining caused by NSF compared with other cariostatic agents (30% and 38% SDF and 35% silver fluoride). The null hypothesis was that there is no difference in the dentin staining between the cariostatic agents tested.

METHODS AND MATERIALS

Selection of the Sample

The sample calculation was based on the results of Sayed and others.[28] The mean ΔE and standard deviations at 14 days in the dark for SDF and SDF+KI were used for calculation. Sample calculation was performed using BioEstat 5.3 (Instituto de Desenvolvimento Sustentável Mamirauá, Manaus, Brazil). A 4.96 minimal difference was considered between mean ΔE of the treatment groups with a standard error of 0.55; six treatment groups were considered with an alpha type error of 0.05 and a beta power of 0.80. To note significant differences, the ideal sample size per group was four. A sample size of 15 was used to compensate for possible outlier values that might lead to sample loss.

Seventy-five permanent human molars extracted for therapeutic reasons were used. The teeth were examined under a stereomicroscope (40×). Carious teeth, teeth with restorations, and teeth with fractures, cracks, or fissures in the coronary portion were excluded.

Sample Preparation

The teeth were disinfected with 0.5% chloramine-T solution at 4°C for one week (ISO 11405:2003) and stored in distilled water (4°C) until the beginning of sample preparation. The teeth were cleaned with periodontal curettes, followed by prophylaxis with a pumice slurry and brush. The samples were cross-sectioned at the cementoenamal junction to access the pulp chamber. After cleaning the pulp chamber, the occlusal enamel was removed with a diamond disc in a cutting machine under refrigeration (Isomet, Buehler Ltd, Lake Bluff, IL, USA). The samples were embedded in PVC cylinders with acrylic resin with the occlusal surface facing upward.

Sample Treatment

The samples were randomly allocated into five groups according to the type of treatment (n=15). The products were applied as recommended by the manufacturers (Table 1). One drop of each agent was applied on the dentin surface followed by an active application with a microbrush for one minute. After treatment with the agents, the samples were placed in hermetically sealed containers and kept in artificial saliva (1% carboxymethylcellulose, 0.12% potassium chloride, 0.005% magnesium chloride, 0.18% methylparaben, and 100 mL of deionized water)[29-31] and changed every seven days. At each evaluation period, the samples were removed from artificial saliva, washed with 50 mL of distilled water, and dried with a gentle air jet.

Before the four-week assessment, samples were submitted to a brushing cycle. The brushing was performed in a simulated brushing machine (ElQuip, São Paulo, Brazil). Samples were fixed to the mechanical devices, and Colgate Classic Clean soft bristle toothbrushes (Colgate-Palmolive, São Paulo, Brazil) were used. Brushing was performed only with distilled water, and no abrasive agent was used. A load of 200g was applied with brushing speed of 250 cycles per minute for three minutes. This step aimed to analyze if brushing can reverse the staining caused by agents.

Table 1: *Materials, Composition, and Application Mode as Recommended by the Manufacturer*

Cariostatic Agent	Composition	Application
NSF 600: nano-silver fluoride, 600 ppm (CETENE, Pernambuco, Brazil	Silver nanoparticles, chitosan and sodium fluoride	Active application of one drop of the agent with a microbrush for 1 min
NSF 1500: nano-silver fluoride, 1500 ppm (CETENE, Pernambuco, Brazil)	Silver nanoparticles, chitosan, and sodium fluoride	Active application of one drop of the agent with a microbrush for 1 min
SDF 30: silver diamine fluoride, 30% (Cariestop, Biodinâmica, Paraná, Brazil)	Hydrofluoric acid, silver nitrate, ammonia hydroxide, and deionized water	Active application of one drop of the agent with a microbrush for 1 min
SDF 38: silver diamine fluoride, 38% (Advantage Arrest, Elevate Oral Care, Florida, USA)	Aqueous silver diamine fluoride, deionized water, and FD&C Blue 1	Active application of one drop of the agent with a microbrush for 1 min
SF: silver fluoride 35% (Riva Star, SDI, Victoria, Australia)	Silver particles, iodine, and fluoride + potassium iodide	Application of one drop of the content of the silver capsule with a specific applicator brush for 1 min; application of one drop of the content of the green capsule for 1 min

Color Measurement

Three color measurements were performed per sample: at baseline (0; before application of the cariostatic agent), after two weeks, and after four weeks (after a brushing cycle) from application. Dentin staining was determined based on the parameters of the CIELAB system (L*a*b*), in which L* corresponds to lightness and the mean value ranges from 0 (black) to 100 (white). The value of a* corresponds to the red-green axis and b* to the yellow-blue axis. Dentin staining was determined through ΔE ($\Delta E=[(L^*_2-L^*_1)^2+(a^*_2-a^*_1)^2+(b^*_2-b^*_1)^2]^{1/2}$) and ΔL ($\Delta L^*=L^*_2-L^*_1$). The degree of staining was evaluated within the following time points: ΔL, zero to two weeks and zero to four weeks.

Objective color measurement was performed with the VITA Easyshade device. Two-color measurements per block were obtained at each time point. The analyzed area was standardized using a custom thermo-formed tray with a 1-mm acetate plate. A hole was made at the central area of the tooth corresponding to the diameter of the spectrophotometer tip.

The mean ΔE and ΔL values were compared between groups using the Kruskal-Wallis test, followed by the Mann-Whitney test ($p=0.05$).

Photographic Images

To observe the dentin staining, the samples were photographed at the same time intervals (baseline, two weeks, and four weeks) using an EOS Rebel T6 digital camera (Canon, Tokyo, Japan), EF 100-mm f/2.8 Macro USM Lens (Canon), and MR-14EX II Macro Ring Lite Flash (Canon). Established parameters were used to standardize image acquisition: ISO 100, F22 aperture, and TTL flash mode at maximum power. The photographs were obtained at the same time of day (14:00 hours). A 40-cm distance between the lens and samples was established. The samples were placed on a white surface near the window with natural light, and the photographs were taken.

RESULTS

The staining of all experimental groups at different time points is shown in Table 2. Differences were observed for ΔE between Advantage Arrest and Cariestop groups at the intervals of zero to two weeks ($p=0.045$) and zero to four weeks ($p=0.041$). However, when ΔL was evaluated separately, differences were observed only between zero and four weeks ($p=0.031$).

Riva Star resulted in similar ΔE and ΔL as NSF 600 at the interval of zero to two weeks ($p=0.713$ and $p=0.724$, respectively). At the interval of zero to four weeks, more significant modifications were observed for Riva Star when compared with the NSF 1500 and NSF 600 groups. However, the intensity was still lower than the one observed for the Advantage Arrest and Cariestop groups.

Samples treated with NSF 1500 exhibited more significant staining than those treated with NSF 600 at the interval of zero to two weeks. At four weeks, the results obtained for the two groups were close to zero, indicating a small variation. No statistically significant differences were observed between these groups in the ΔE and ΔL evaluation ($p=0.081$ and $p=0.270$, respectively).

The photographic images can be seen in Figure 1. After two weeks, all samples exhibited some degree of staining when compared with the baseline assessment. However, after the brushing cycle and

Table 2: *Mean (Standard Deviation) of Dentin Staining ΔE and ΔL According to the Cariostatic Agent and Assessment Time (n=15)*[a]

Cariostatic Agent	$\Delta E_{0\text{-}2w}$	$\Delta L_{0\text{-}2w}$	$\Delta E_{0\text{-}4w}$	$\Delta L_{0\text{-}4w}$
Advantage Arrest	49.22 (8.62) A	−47.62 (8.67)A	52.95 (7.47) A	−51.69 (6.73) A
Cariestop	41.56 (9.32) B	−40.01 (9.87) A	47.15 (7.85) B	−45,11 (8.10) A
NSF 1500: nano-silver fluoride, 1500 ppm	17.97 (3.37) C	−18.95 (7.84) B	1.53 (0.89) C	−1.2 (1.08) C
NSF 600: nano-silver fluoride, 600 ppm	7.35 (3.23) D	−6.55 (3.36) C	1.02 (0.77) C	−0.76 (6.17) C
Riva Star	8.25 (4.49) D	−7.76 (6.17) C	30.27 (8.24) D	−29.93 (9.36) B

[a] *0-2w = 0 to 2 wk; 0-4 w = 0 to 4 wk. Negative values indicate staining. Different letters indicate significant differences between groups (columns) (Mann-Whitney test).*

color measurement after four weeks, the color was reestablished in the NSF 600 and NSF 1500 groups compared with baseline.

DISCUSSION

The hypothesis that no difference exists in the dentin staining between the cariostatic agents evaluated was rejected. Evaluation of ΔE and ΔL after the brushing cycle at four weeks showed that the nanoparticle formulations resulted in less staining of the teeth ($p<0.001$).

SDF is a cariostatic agent that has been investigated for years because of its proven clinical efficacy.[7,32] The good results obtained with this agent have encouraged studies aiming at the reduction of its adverse effects.[33] Parental acceptance was evaluated in New York City about the use of SDF for the treatment of caries in schoolchildren.[12] In that study, 32.5% of the participants classified the staining in posterior teeth as unacceptable, and 70.3% classified the esthetics of treated anterior teeth as unacceptable. These data suggest that, although effective, the use of SDF is limited and encounters resistance due to esthetic concerns.

The application of KI immediately after the use of SDF has been proposed to minimize the staining.[1,17] An *in vitro* study of extracted human molars evaluated tooth staining after treatment with SDF or SDF+KI by digital spectrophotometry and visual evaluation after four weeks of follow-up. The authors observed that the combined application of KI reduced or even prevented tooth staining, with the groups treated only with SDF being visibly darker (more significant variation in ΔL).[17] Once in contact with the silver particles, KI gives rise to silver iodide, which has a white-yellow color.[18] These results are consistent with the present study. Riva

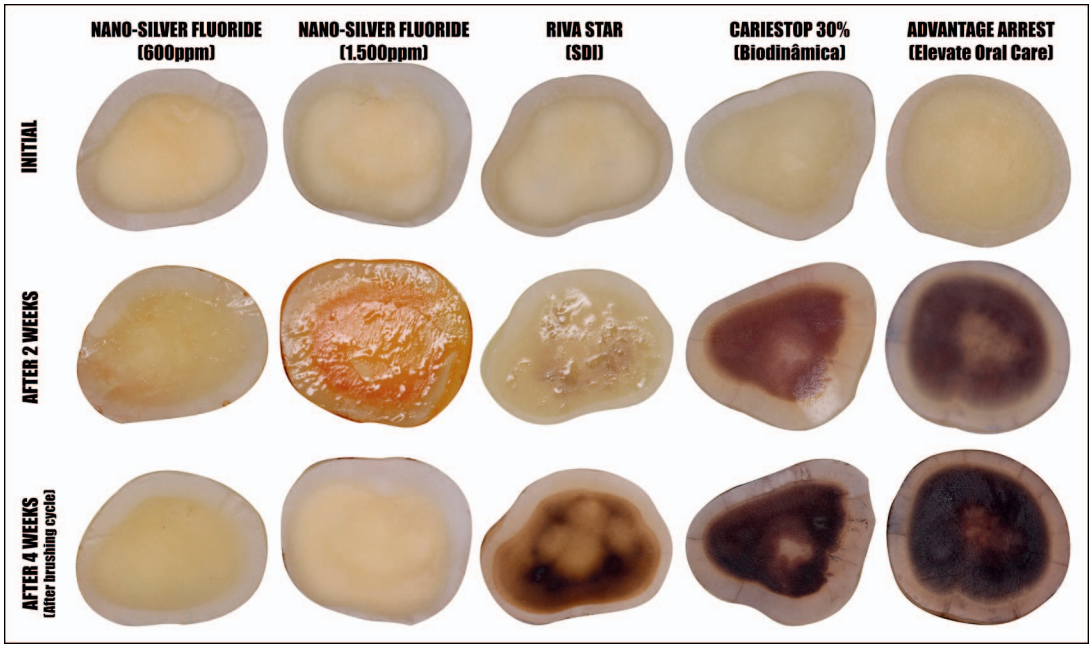

Figure 1. *Photographic images of the observed dentin staining*

Star (SDI) consists of two capsules: one containing silver fluoride and the other KI. This agent resulted in less staining of the teeth after four weeks when compared with Advantage Arrest (SDF 38%) and Cariestop (SDF 30%), which do not contain KI ($p<0.001$). However, a certain degree of dentin staining that could compromise esthetics could still be observed for Riva Star (Figure 1).

This staining is the result of the precipitation of silver particles through the formation of a layer of silver phosphate on the carious dentin followed by oxidation of the particles.[17,18] Patel and others,[18] who evaluated staining as a function of time, found that the black staining of carious dentin was clinically noticeable within two minutes after application of the agents. A relatively constant increase in the level of staining was observed after five minutes, and a deep black stain was detected on the dentin and enamel four to six hours after application. These results agree with the findings of the present study, in which time was the determinant factor for the extent of dentin staining in the case of the commercial agents. Regarding the experimental NSF formulations, the ΔE and ΔL data and photographic findings indicated minimal staining after four weeks. NSF does not form oxides on contact with the oxygen of the medium and therefore does not cause dentin staining.[25]

Evaluation after two weeks showed that NSF 600 and NSF 1500 caused a yellowish stain on the teeth, resulting in a decrease in lightness in these groups (−6.55 and −18.95, respectively). However, the brushing cycle seems to have removed the stained layer as a film, and the undesired staining effect and lightness returned to the baseline values (−0.76 and −1.2, respectively). On the other hand, all other groups reduced luminosity even after the brushing cycle. The presence of chitosan in the composition may have favored the formation of this film. Chitosan is a cationic biopolymer that is obtained by the deacetylation of chitin and is the second most abundant polymer found in nature.[35] In an aqueous medium, chitosan tends to agglomerate and adhere to the surface.[34] However, this film is easily removed, even with gauze. Samples were submitted to a short brushing cycle (three minutes) simulating clinical conditions to standardize the removal of the superficial pigments. When added to NSF, chitosan acts as a nanoparticle carrier. In addition, chitosan has a synergistic effect due to its antibacterial and remineralizing properties.[19,36]

Studies that evaluate tooth color variations, either bleaching or staining potential, have reported the results using ΔE and ΔL.[11,17,18,37,38] Both measures were used in this study, with approximate results. This finding may be related to the fact that the cariostatic agents cause dental darkening. Therefore, the luminosity (L*) was the color axis of the CIELAB system that mostly varied since it considers the variation between white and black.[37]

A digital spectrophotometer was used for the evaluation of dentin staining since the perception of colors by visual analysis is prone to subjective interpretation and varies among different observers. Visual color evaluation can be affected by factors such as lighting and human physiological variables (fatigue, age, and emotional state), while the spectrophotometer provides precise and accurate color measurements of teeth.[39,40] A previous clinical study compared three different methods for color evaluation (visual, spectrophotometer, and digital photography) of the right upper incisors of 50 patients. The authors observed a high agreement rate between digital methods and concluded that digital photography is a reliable method for color selection.[41] Hence, in the present study, dentin staining was objectively evaluated (spectrophotometer) and visually registered with photographs.

In clinical studies evaluating tooth staining caused by cariostatic agents, the researchers respond objectively only with yes/no (does cause or does not cause staining).[18,25] The photographic analysis allows the evaluation over time through visual monitoring as well as analysis of the gray scale.[18] Despite the illustrative nature of the photographic assessments in the present study, the findings were consistent with the quantitative approach to color measurement with a digital spectrophotometer.

CONCLUSIONS

The new formulations (NSF 600 and 1500 ppm) caused less dentin staining than the already available commercial agents 35% silver fluoride, 30% SDF, and 38% SDF. NSF might be an alternative to SDF since it does not compromise esthetics.

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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 Ethics Committee on Research

Involving Humans of the University of Pernambuco. The approval code issued for this study is 2.577.182.

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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A Comparative Study of Light Transmission by Various Dental Restorative Materials and the Tooth Structure

N Ilie • G Furtos

Clinical Relevance

Light transmission through dental materials and tooth structure has direct clinical implication on such factors as selecting an appropriate curing technique during a restorative process.

SUMMARY

Introduction: This study aims to quantify and compare the amount of light that passes through seven different types of direct and indirect restorative materials comprising light-cured resin based composites (regular and bulk-fill), computer-aided design/computer-aided manufacturing (CAD/CAM) restoratives such as resin based composites, poly(-methyl methacrylate) (PMMA) resin, leucite glass-ceramic, lithium silicate glass-ceramic, feldspar ceramic, and the natural tooth structure.

Methods and Materials: Individual sets (n=6) of plane-parallel test specimens (2 mm) of 32 restorative materials belonging to the aforementioned seven material types and the tooth structure were prepared. Within the analyzed materials, one leucite glass-ceramic and one lithium disilicate glass-ceramic were considered in two different translucencies. In addition, two light-cured resin composites, one CAD/CAM resin composite, and one lithium disilicate glass-ceramic were considered in two different shades. Optical properties (transmittance, *T*; absorbance, *A*; and opacity, *O*) of each material were calculated from the relationship between incident and transmitted irradiance [*I*(*d*)] using a violet-blue light-curing unit. Incident and transmitted irradiance were assessed in real time on a spectrophotometer. A multivariate analysis (general linear model) assessed the effects of various parameters on the optical properties.

Results: A very strong influence of the parameter *material* was identified on *I*(*d*) ($p<0.001$; partial eta squared, $\eta_P^2=0.953$), *T* ($p<0.001$; $\eta_P^2=0.951$), *A* ($p<0.001$; $\eta_P^2=0.925$), and *O* ($p<0.001$; $\eta_P^2=0.886$), while the effect of the parameter *material type* was not significant

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(p=0.079, p=0.05, p=0.05, and p=0.051, respectively). Light attenuation differed significantly by material within each shade category and by shade category within the analyzed material.

Conclusions: Attenuation of light through restorative materials and tooth structure is high (59.9% to 94.9%); thus, deficits in polymerization are difficult to compensate for by additional light exposure at the end of the restorative process.

INTRODUCTION

A prerequisite that esthetic dental restorative materials need to fulfill in order to accurately duplicate the tooth structure is the ability to be translucent. When the translucency of a restorative material misses an appropriate range, it is often the main factor responsible for failures in matching the shade of restorative materials to the natural teeth.[1,2] A similar effect was observed when repairing a resin composite restoration with a direct resin composite of the same shade[3,4] or when mixing different types of direct resin composites.[2] Equally important for esthetics is the impact of translucency on various material properties, such as the depth of cure in direct light-cured resin composite restoratives[5] and the sufficiency of curing in underlying, light-curing or dual-curing luting resin composites, when involving indirect restoratives.[6]

The light of a curing unit striking the surface of a dental restoration during polymerization may be reflected, absorbed, scattered, or transmitted. Based on these interactions with light, translucency is described as the relative amount of light transmission or the diffuse reflectance from a substrate surface through a turbid medium.[7] By contrast, opacity is a measure of impenetrability to light and is a consequence of extensive absorption and/or scattering of light in a specific medium. Attenuating the effects of light when passing through matter, such as scattering or reflection, are summarized in optics together with the absorption under the terms *extinction* or *absorbance*. Note that light attenuation increases exponentially with material thickness, as described by Lambert-Beer's law.[8]

Light absorption in dental restoratives occurs when the frequency of the light matches certain quantized frequencies within the constituent atoms and molecules of the material, such as resin components,[9] filler particles,[10,11] photoinitiator molecules,[12] dyes, pigments,[9,13] or structures surrounding the restoration. In contrast, light scattering is due to the deviation of light from its initial trajectory because of localized nonuniformities with a different index of refraction through which the light passes, such as reinforcing particles, porosity voids,[10] or surface roughness. In resin composites, for instance, absorption takes place within photoinitiators, color pigments, and the organic matrix,[9,14] whereas scattering is mainly attributed to filler particles.[10,11] For the tooth structure, a negligible absorption and a weak scattering (scattering coefficient ranging from 15 to 105 cm^{-1}) has been identified in enamel. In comparison, in dentin, scattering is strong (scattering coefficient 260 cm^{-1}), and the absorption low.[15]

It should be noted that the absorption of light on surfaces or when passing through matter depends on the material and the frequency (wavelength) of the incident light. If, for example, a yellow-appearing surface is irradiated with white light, the green light and red light are reflected/transmitted and the blue light is absorbed. When absorbing light, the absorbed energy is usually transformed into thermal energy, though other mechanisms are possible in both dental materials and natural teeth, such as the delayed release of light in the form of fluorescence.[16,17]

The aim of the present study was to compare and quantify the amount of light that would pass through 2-mm-thick increments of a large diversity of restorative materials: 1) regular light-cured resin-based composites (RBCs), 2) bulk-fill light-cured RBCs, 3) computer-aided design/computer-aided manufacturing (CAD/CAM) restoratives based on resin composites, 4) poly(methyl methacrylate) (PMMA), 5) lithium disilicate ceramic, 6) leucite glass-ceramic; and 7) feldspar ceramic, as well as the tooth structure, when irradiated with a contemporary violet-blue light-emitting diode (LED) light-curing unit (LCU). Moreover, optical properties calculated from the relationship between incident and transmitted irradiance, such as transmittance, opacity, and absorbance, are considered.

The results of the present study should allow clinicians to critically judge some customary routines in light polymerization during a restorative process, such as 1) pre-curing for a few seconds at the lowest increment when incrementally reconstructing a deep cavity with light curing RBCs, while assuming that sufficient light passes through the upper layers to complete the polymerization; 2) curing a light-cured or dual-cured luting material through various indirect restoratives (encompassing the novel high translucent CAD/CAM RBCs), assuming sufficient light transmittance through all indirect restoratives

to properly cure the luting material; and 3) curing through the tooth structure at the end of the restorative process to compensate for insufficient polymerization. Since all specimens were prepared and analyzed under identical conditions, the presented data allowed for a direct comparison among different material types.

The null hypotheses assume similar transmitted irradiance and optical properties through 1) all material types and tooth structure, 2) materials belonging to one material type, 3) materials belonging to one shade, and 4) materials belonging to one translucency.

METHODS AND MATERIALS

The light transmission through 32 restorative materials representing seven different material types (light-cured RBCs (regular and bulk-fill), CAD/CAM restoratives based on resin composites, PMMAs, lithium disilicate ceramics, leucite glass-ceramics, and feldspar ceramic) (Table 1) and in different shades and opacities was analyzed at a specimen thickness of 2 mm. Optical properties such as transmittance, absorbance, and opacity of each material, were calculated from the relationship between incident and transmitted irradiance using a violet-blue LED LCU (VALO, serial number VO 7710, Ultradent, South Jordan, UT, USA) in the standard exposure mode with an exposure time of 20 seconds. The light transmission through the tooth structure was also considered. For direct comparison of the optical properties among analyzed restorative materials and tooth structure, specimens of 2-mm thickness were considered. Various tooth structure specimen thicknesses (1.7 mm to 3 mm) have also been considered to simulate light transmittance through a tooth cavity wall, as this may occur in a clinical situation.

Specimen Preparation

Individual sets (n=6) of plane-parallel test specimens (2 mm) of 32 materials were prepared. Tooth specimens (n=50) were manufactured in a thickness range of 1.7 mm to 3 mm.

Light-cured RBC pastes were condensed in white cylindrical Teflon molds (6-mm diameter, 2-mm height) and applied on the sensor of a spectrophotometer device (MARC (Managing Accurate Resin Curing) System, Blue light Analytics Inc, Halifax, NS, Canada) while being separated from the sensor by a thin Mylar foil (Frasaco GmbH, Tettnang, Germany).

CAD/CAM blocks were cut using a low-speed diamond saw (Isomet low-speed saw, Buehler, Esslingen, Germany) under water-cooling, then ground with 4000-grit SiC paper and polished with a diamond suspension (mean grain size: 1 μm). The thickness of each specimen was determined at two points using a digital micrometer screw gauge (Mitutoyo, Kawasaki, Japan). Thickness tolerance was set at 0.05 mm.

Tooth specimens 1.7 mm to 3 mm thick were obtained from 25 caries-free human molars. For this purpose, one slice from the buccal and one from the lingual side of each tooth were cut perpendicular to the long axis of the tooth, thus providing specimens with one flat side, while leaving the tooth anatomy intact on the opposite side. This simulated the tooth wall in a Class II cavity through which a light-curing RBC restoration may be additionally irradiated at the end of a restorative procedure. The flat side of the tooth specimen was ground and polished similar to the CAD/CAM specimens and positioned in contact to the sensor of the spectrophotometer. The opposite, intact tooth side was irradiated with the LCU as described previously. Light transmittance was accordingly measured through a natural tooth wall of 1.7 to 3 mm thickness, thus simulating a clinically relevant polymerization condition.

Spectrophotometry: Measurement of the Incident and Transmitted Irradiance

Incident and transmitted irradiance through the described specimens were assessed on a laboratory-grade National Institute of Standards and Technology (NIST)–referenced USB4000 Spectrometer while using the aforementioned LCU. The incident irradiance (the irradiance reaching the specimen's surface) was determined on six occasions by applying the LCU directly to the sensor. The transmitted irradiance was measured by positioning the specimens between the LCU and the sensor. The Teflon molds containing the light-cured RBC, as well as the CAD/CAM and tooth structure specimens, were aligned and centered on the round detector of the spectrometer, which had a diameter of 3.9 mm. Consequently, the irradiance and radiant exposure reaching this area was considered. The miniature fiber optic USB4000 Spectrometer uses a 3648-element Toshiba linear Charge-coupled Device array detector and high-speed electronics (Ocean Optic, Largo, FL, USA). The spectrometer was calibrated using an Ocean Optics' NIST-traceable light source (300–1050 nm). The system uses a CC3-UV Cosine Corrector (Ocean Optic) to collect radiation over a

Table 1: *Materials, Manufacturer, Shade, and Lot Number for Light-cured RBCs and CAD/CAM Restoratives*

Type	Material	Manufacturer	Shade	Lot
Light-cured RBCs				
Regular (nanohybrid)	Filtek Supreme XTE	3M, Seefeld, Germany	A3 Dentin	N229448
	Filtek Supreme XTE flow	3M	A3	N236527
	Synergy D6 Dentin	Coltene, Altstätten, Switzerland	A3/D3	F88495
Regular (microhybrid)	G-eanial	GC, Leuven, Belgium	A3	140919A
			A2	160208A
	Gradia Direct	GC	A3 Anterior	140709A
			A2 Anterior	151221A
	Charisma	Heraeus, Hanau, Germany	A3	010700A
	XRV Herculite Enamel	Kerr, Orange, USA	A3	5223302
	Filtek Z250	3M	A2	N768186
Bulk-fill (low viscosity)	SDR	Dentsply Sirona, Charlotte, USA	Universal	1309183
	Venus	Heraeus	Universal	010100
Bulk-fill (high viscosity)	Admira Fusion X-tra	Voco, Cuxhaven, Germany	Universal	1537600
	Sonic Fill 2 Composite	Kerr	A3	5767358
	Filtek One	3M	A3	N782223
CAD/CAM restoratives				
RBC	VITA ENAMIC	VITA Zahnfabrik, Bad Säckingen, Germany	3M2	33000
	Luxacam Composite	DMG, Hamburg, Germany	A3	769515
	Grandio Blocs	Voco	A3 HT	1709591
	Lava Ultimate	3M	A3 HT	N933658
			A2 HT	N372985
	Brilliant Crios	Coltene	A3 HT	I35186
	Cerasmart	GC	A3 HT	1702011
	Tetric CAD	Ivoclar Vivadent, Schaan, Liechtenstein	A3 HT	W93631
PMMA	VITA CAD-Temp	VITA Zahnfabrik	2M2	CE 0124
	Telio CAD	Ivoclar Vivadent	A2 LT	N73354
Leucite glass-ceramic	IPS Empress CAD		A2 LT	R39335
			A2 HT	K24309
Lithium silicate glass-ceramic	IPS e.max CAD		A2 LT	R37085
			A3 LT	N73805
			A2 HT	T1888
	CELTRA Duo	Dentsply Sirona	A2 LT	18015733
Feldspar ceramic	VITA Mark II	VITA Zahnfabrik	A2	N502353

Abbreviations: CAD/CAM, computer-aided design/computer-aided manufacturing; PMMA, poly(methyl methacrylate; RBC, resin-based composite.
[a] A total of seven material types were considered: 1) regular light-cured RBCs, 2) bulk-fill light-cured RBCs, 3) CAD/CAM restoratives based on resin composites, 4) PMMA, 5) lithium silicate ceramics, 6) leucite glass-ceramics, and 7) feldspar ceramic.

180° field of view, thus mitigating the effects of optical interference associated with light collection sampling geometry. Irradiance at a wavelength range of 360–540 nm was individually collected at a rate of 16 records/second. The sensor was triggered at 20 mW.

Transmittance, Opacity and Absorbance

Transmittance (T) is defined as the ratio of transmitted irradiance (radiant power) to incident irradiance: $T = I(d)/I_0$, where $I(d)$ is the irradiance after the beam of light passes through the specimen of thickness d, and I_0 is the irradiance of the incident light. The inverse of transmittance is called *opacity* (O) ($T^{-1}=I_0/I(d)$). The negative decadic logarithm of the transmittance, that is, the decadic logarithm of the opacity, is the absorbance (A) ($-\log(T)=-\log(I(d)/I_o)$, where absorbance stands for the number of photons absorbed. Being defined as ratios of irradiance values, T, O, and A are dimensionless.

Statistical Analysis

A Shapiro-Wilk test verified the normal distribution of the data. A multivariate analysis (general linear

model) assessed the effects of various parameters as well as their interaction terms on the transmitted irradiance and optical parameters. The partial eta-squared statistic reports the practical significance of each term based on the ratio of the variation accounted for by the effect. Larger values of partial eta-squared indicate a greater amount of variation accounted for by the model effect, to a maximum of one. In all statistical tests, p-values < 0.05 were considered statistically significant when using SPSS software (Version 24.0, SPSSS Inc, Chicago, IL, USA).

RESULTS

The incident irradiance at the applied curing conditions amounted to 1174.1 (12.4) mW/cm^2. The transmitted irradiance through 2-mm-thick specimens varied between 471.0 ± 0.4 mW/cm^2 (flowable bulk-fill resin composite, Venus, universal) and 60.4 ± 0.1 mW/cm^2 (CAD/CAM resin composite, VITA Enamic). This denotes a drop in irradiance of 59.9% to 94.9% when light is passing through diverse restorative materials. In comparison, the attenuation of light through the tooth structure of the same thickness was 89.4% (transmitted irradiance = 124.6±36.7 mW/cm^2).

In the material range described earlier (Venus to VITA Enamic), the absorbance varied between 0.40 ± 0.02 and 1.29 ± 0.03, and the opacity between 2.52 ± 0.11 and 19.56 ± 1.13 (Table 2). The related optical properties of the tooth structure of the same thickness were 1.0 ± 0.03 and 10.5 ± 3.3, respectively.

A very strong influence of the parameter *material* was identified on the transmitted irradiance $I(d)$ ($p<0.001$; partial eta squared, $\eta_P^2=0.953$), T ($p<0.001$; $\eta_P^2=0.951$), A ($p<0.001$; $\eta_P^2=0.925$), and O ($p<0.001$; $\eta_P^2=0.886$), while the effect of the parameter *material type* was not significant ($p=0.079$, $p=0.05$, $p=0.05$, $p=$and 0.051, respectively).

Individual shades (A2, A3, 2M2, and U), irrespective of the chemical composition and structure of the analyzed materials, have been identified as having a highly significant influence on all determined optical parameters ($p<0.001$; $\eta_P^2=0.461$ for $I(d)$; 0.479 for T; 0.393 for A; and 0.361 for O).

The singular effect of shade (A2 and A3) on the transmitted irradiance for a given chemical composition and structure of a material can be directly observed in the light-cured RBCs G-eanial and Gradia Direct Anterior, the CAD/CAM RBC Lava Ultimate, and the lithium disilicate glass-ceramic IPS e.max CAD. Accordingly, light was attenuated significantly stronger in specimens of shade A3 compared with A2, in all aforementioned material types. Note, however, that difference in light attenuation owing to a difference in shade was higher in the glass-ceramic (relative change=29.9%) compared with the RBCs (15.6% Lava Ultimate, 13.2% G-eanial and 11.9% Gradia Direct Anterior).

The individual effect of translucency on the transmitted irradiance for a given shade (A2) was gathered in the leucite glass-ceramic IPS Empress CAD and the lithium disilicate glass-ceramic IPS e.max CAD (Table 2). The comparison indicates a strong attenuation of light in low translucent (LT) compared to the high translucent (HT) specimens. While the transmitted irradiance was higher through the leucite glass-ceramic, the difference in transmitted irradiance between HT and LT formulations was higher in the lithium disilicate glass-ceramic (49.72% vs 39.25% in the leucite glass-ceramic).

Figure 1 illustrates the exponential decrease of the transmitted irradiance through the tooth structure as a function of specimen thickness.

The direct comparison of 2-mm-thick tooth specimens with the analyzed restorative ceramics and glass-ceramics (Figure 2) emphasizes statistically similar transmitted irradiances through the tooth structure and the materials IPS e.max CAD, LT, A2 and CELTRA Duo, LT, A2. Compared with the tooth structure, significantly lower light transmittance was identified in IPS e.max CAD, LT, A3, while significantly higher light transmittance was found in all other materials. In the same material group presented in Figure 2, the opacity was significantly highest in the lithium disilicate glass-ceramic IPS e.max CAD, LT, A3, and the tooth structure.

Figure 3 illustrates graphically, in descending order, the transmitted irradiance through all analyzed RBC materials, both light-cured and CAD/CAM, in comparison to 2-mm-thick tooth structure specimens. A large range of RBC materials, comprising the material interval Charisma A3 (mean transmitted irradiance=92.33 mW/cm^2) to Telio CAD, A2 (170.6 mW/cm^2), showed a statistically similar transmitted irradiance to the tooth structure.

DISCUSSION

The present study quantifies the attenuation of light when traveling through different types of restorative materials of a predetermined thickness of 2 mm.

Table 2: *Transmittance, Absorbance, and Opacity as a Function of Material and Material Type (Mean and Standard Deviation)*[a]

Material	Type	Transmittance		Absorbance		Opacity	
FiltekTM Supreme XTE, A3	Resin composite (LC)	0.02 A	0.001	1.61	0.01	40.46	1.79
VITA Enamic, 3M2	Resin composite (CAD/CAM)	0.05 A	0.004	1.29	0.03	19.56	1.13
IPS e.max CAD, LT, A3	Lithium silicate glass-ceramic	0.08 AB	0.004	1.12	0.01	13.09	0.37
Charisma A3	Resin composite (LC)	0.08 AB	0.001	1.11	0.02	12.86	0.44
Sonic Fill 2 A3	Resin composite (LC)	0.08 AB	0.001	1.09	0.01	12.24	0.25
FiltekTM Supreme XTE flow	Resin composite (LC)	0.08 AB	0.024	1.10	0.01	12.48	0.39
XR V Herculite A3	Resin composite (LC)	0.08 AB	0.01	1.07	0.01	11.80	0.33
G-aenial A3	Resin composite (LC)	0.09 BC	0.001	1.06	0.01	11.52	0.20
Synergy D6 A3/D3	Resin composite (LC)	0.09 BC	0.001	1.04	0.01	10.92	0.21
Gradia Direct A3	Resin composite (LC)	0.09 BC	0.01	1.03	0.02	10.77	0.55
G-aenial A2	Resin composite (LC)	0.10 BC	0.001	1.00	0.01	10.00	0.17
Enamel+Dentin	Enamel+Dentin	0.10 BC	0.03	1.00	0.12	10.51	3.27
Gradia Direct A2	Resin composite (LC)	0.11 BC	0.01	0.98	0.02	9.48	0.38
Filtek Z250 A2	Resin composite (LC)	0.11 BC	0.01	0.96	0.02	9.25	0.29
IPS e.max CAD, LT, A2	Lithium silicate glass-ceramic	0.11 BCD	0.001	0.96	0.01	9.16	0.30
Luxacam Composite, HT, A3	Resin composite (CAD/CAM)	0.11 BCDE	0.001	0.95	0.01	8.92	0.18
CELTRA Duo, LT, A2	Lithium silicate glass-ceramic	0.12 CDEF	0.01	0.92	0.04	8.32	0.75
Grandio Blocs, LT, A3	Resin composite (CAD/CAM)	0.13 CDEF	0.01	0.90	0.04	7.95	0.71
VITA CAD-Temp, 2M2	Resin composite (CAD/CAM)	0.14 DEF	0.01	0.85	0.02	7.06	0.36
IPS Empress CAD, LT, A2	Leucite glass-ceramic	0.15 DEFG	0.01	0.84	0.03	6.89	0.51
Lava Ultimate, HT, A3	Resin composite (CAD/CAM)	0.15 DEFG	0.01	0.84	0.02	6.84	0.26
Telio CAD	Resin composite (CAD/CAM)	0.15 DEFGH	0.01	0.84	0.01	6.91	0.13
Filtek One A3	Resin composite (LC)	0.15 EFGH	0.01	0.83	0.02	6.83	0.35
Brilliant Crios, HT, A3	Resin composite (CAD/CAM)	0.16 FGH	0.01	0.81	0.01	6.42	0.12
Cerasmart, HT, A3	Resin composite (CAD/CAM)	0.18 GHI	0.01	0.75	0.02	5.62	0.24
Lava Ultimate, HT, A2	Resin composite (CAD/CAM)	0.18 HI	0.01	0.74	0.03	5.54	0.36
Vita Mark II, A2	Feldspar ceramic	0.20 I	0.01	0.71	0.02	5.18	0.21
Tetric CAD, HT, A3	Resin composite (CAD/CAM)	0.20 I	0.00	0.70	0.01	5.02	0.07
ADMIRA Fusion X-tra, U	Resin composite (LC)	0.2 IJ	0.01	0.68	0.02	4.76	0.13
IPS e.max CAD, HT A2	Lithium silicate glass-ceramic	0.21 IJ	0.01	0.67	0.01	4.66	0.09
IPS Empress CAD, HT, A2	Leucite glass-ceramic	0.24 J	0.001	0.63	0.01	4.21	0.04
SDR U	Resin composite (LC)	0.29 K	0.01	0.53	0.02	3.42	0.13
Venus U	Resin composite (LC)	0.40 L	0.02	0.40	0.02	2.52	0.11

Abbreviations: CAD/CAM, computer-aided design/computer-aided manufacturing; HT, high translucency; LT, low translucency; LC, light cured.
[a] *Material description followed in the sequence brand, followed by translucency (LT or HT, if applicable) and shade. Data are arranged in ascending order of transmittance, descending order of absorbance, and opacity. Letters indicate statistically homogeneous subgroups for transmittance (Tukey honestly significant difference test, $\alpha=0.05$).*

This thickness was chosen to allow for a direct comparison among a large variety of direct and indirect restoratives that might be involved in light curing during the restorative procedure and is due to the need for curing regular RBCs in increments that should not exceed 2 mm. The comparison of the optical properties among tooth and material specimens refers only to this thickness.

The light transmission through light-cured RBCs and the tooth structure has direct clinical implications, such as selecting an appropriate curing technique. While there is no experimental evidence, clinicians are often taught to only pre-cure the lowest increment when incrementally reconstructing a deep cavity, based on the estimate that the lowest increment will receive enough light when curing the upper increments or when curing through the tooth structure. The latter is recommended when completing the restoration.

The present study gives a comparison of the light transmittance through some representative regular micro- and nanohybrid light-cured RBCs (Table 2) in the clinically most commonly used shades (A2, A3), thus identifying light transmitted irradiances rang-

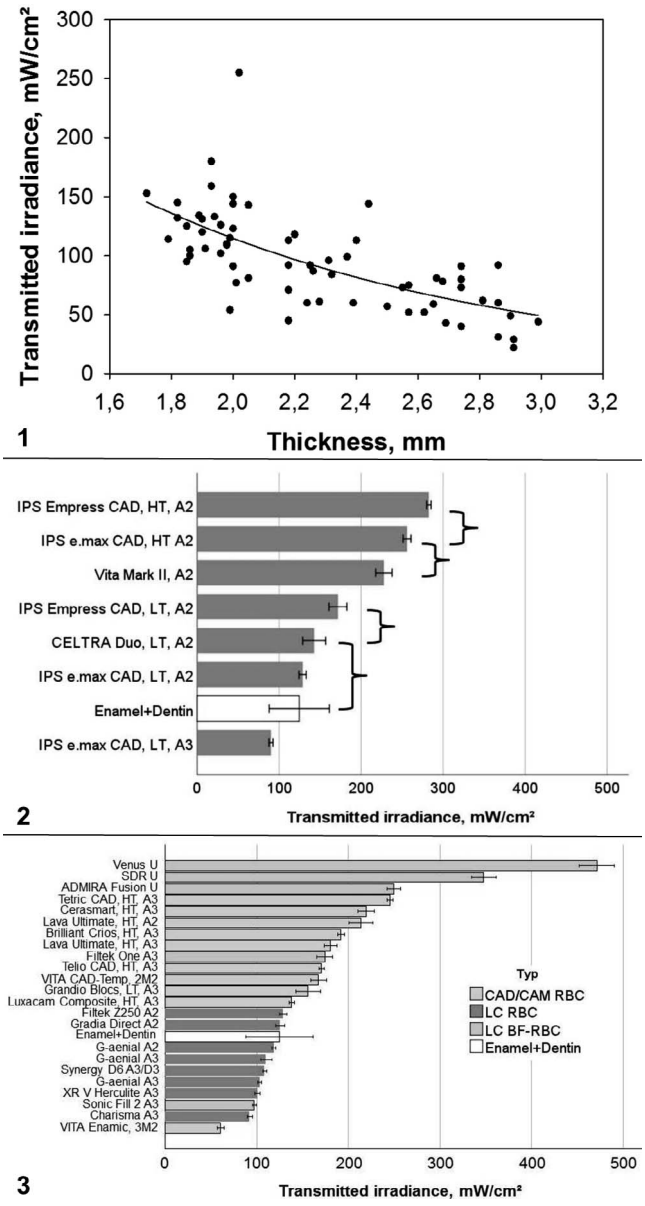

Figure 1. *Transmitted irradiance through the tooth structure (enamel + dentin) as a function of specimen thickness.*

Figure 2. *Transmitted irradiance through diverse ceramics and the tooth structure for 2-mm-thick specimens.*

Figure 3. *Transmitted irradiance through diverse resin composites (light-cured regular, light-cured bulk-fill and CAD/CAM) and tooth structure for 2-mm-thick specimens.*

ing from 92.32 ± 3.21 mW/cm^2 (Charisma, A3) to 128.33 ± 4.04 mW/cm^2 (Filtek Z250, A2). The indicated values represent the maximal irradiance recorded at the bottom of 2-mm-thick specimens during curing for 20 seconds. Converted to radiant exposure (incident irradiance × exposure time), the allocated values to underlying increments are 1.8 to 2.5 J/cm^2. Corroborated with the radiant exposure values indicated in the literature for sufficient polymerization in regular RBCs (21-24 J/cm^2),[18,19] the light transmitted through the upper increment may not be sufficient to compensate for a short precuring of the underlying increment. Thicker RBC layers, as may occasionally occur in a clinical situation during restoring a cavity, are even completely impermeable to light.[20] Note that the majority of RBCs,[20,21] including all light-cured materials analyzed in the present study, become progressively more translucent during polymerization, thus allowing more light to be transmitted through the 2-mm-thick specimens toward the end of the exposure time. Changes in translucency during polymerization are related in large part to the monomer reactivity and particularly to the filler/resin refractive index mismatch and their interface.[5,22,23] The measured transmitted irradiances through RBCs are similar to values measured through the tooth structure (enamel and dentin) of the same thickness (124.55±36.73 mW/cm^2). The high standard deviation is ultimately related to the diversity of the tooth structure as well as to the different fraction of enamel and dentin in the selected specimen, as it represents the real anatomy of the tooth. This enables quantification of light transmission as it will occur in a clinical situation. It is noteworthy that the transmitted irradiance through 3-mm-thick tooth specimens is lowered to insignificant values (25 mW/cm^2) while following an exponential decrease with the thickness. It should be emphasized that the incident irradiance within the curing conditions of the present study amounted to 1174.1 ± 12.4 mW/cm^2. The indicated transmitted irradiances will be even lower when using LCUs of lower irradiance, enhancing the exposure distance or changing the angulation of the LCU, as might occur in a clinical situation. All of these aspects raise questions about the benefit of additional polymerization through the tooth structure and limit it to a thermal effect.

To simulate clinical treatment, measurements were done on a dehydrated tooth structure. This procedure encompassed a 10-second exposure to the air stream followed by a period of 5 minutes in air, which was used to standardize the position of sample and LCU previous to measurement. Dehydration was identified to reduce the translucency of the tooth structure as a result of an increased difference in refractive indexes between the enamel prisms and the surrounding medium when water is replaced by air.[24] The reduction in translucency was quantified for enamel at 82% of the value measured in wet

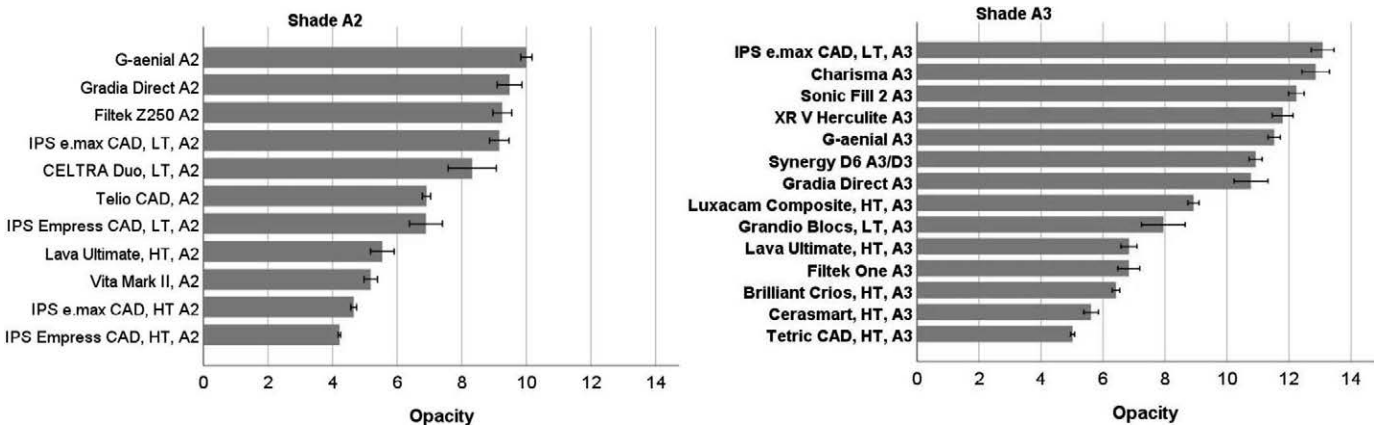

Figure 4. *Opacity of the analyzed materials grouped for the shades A2 (left) and A3 (right).*

enamel,[24] an aspect that needs to be considered when translucency is measured, other than in the present study, for esthetic purposes. Moreover, the chosen period of 5 minutes for drying the specimens before measurement was based on the fact that the assumptive water content change during drying of human enamel samples does not alter optical measurements above 5 minutes and up to 48 hours after specimens were taken out of the water.[25]

As for the analyzed bulk-fill RBCs, significantly higher light transmittance has been identified through 2-mm increments, except for Sonic Fill 2 A3, which performed similarly to regular RBCs and the tooth structure. It has already been shown that bulk-fill RBCs permit light transmission even in 4-mm-thick increments that may amount to up to 24% of the incident blue light,[26] which explains the good polymerization in depth of such materials. The reasons for enhanced light transmission in bulk-fill RBCs is mostly attributed to a reduced amount of pigments and enhanced filler sizes that lower the total interfacial area between the filler and matrix, reducing scatter.[5]

For comparative purposes, a series of modern CAD/CAM RBCs as well as two temporary, PMMA-based materials were studied. The transmitted irradiance through CAD/CAM RBCs and PMMA-based materials (p=0.90) as well as through each material category and the tooth structure (p=0.16 and p=0.149, respectively) were similar. Compared with light cured RBCs, transmitted irradiance was higher through CAD/CAM RBCs and PMMA-based materials (p<0.001, 0.024). Within the CAD/CAM RBCs material category, however, the variation in light transmission was high (60.4 to 245.2 mW/cm^2) and was due to differences in microstructure, pigments, chemical composition of the resin and filler, and filler morphology. It followed the sequence VITA Enamic < Luxacam Composite < Grandio Blocs < Lava Ultimate A3 and Brilliant Crios < Lava Ultimate A2 and Cerasmart < Tetric CAD. Note that all materials, except for VITA Enamic and Luxacam Composite, were analyzed in the HT version, thus explaining the ranking. The latter material category comprises a polymer infiltrated ceramic network material (VITA Enamic) with a three-dimensional interconnected dual network structure[27] that differs from the structure of the other analyzed CAD/CAM RBCs, in which only the polymer matrix phase is continuous, while fillers of different sizes and morphology are dispersed in the matrix.[28] The organic phase in all CAD/CAM RBCs is composed of dimethacrylates, which consist of either a mixture of urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA) in VITA Enamic[27] or variations of UDMA, TEGDMA, bisphenol a glycol dimethacrylate (Bis-GMA), and ethoxylated bisphenol a dimethacrylate (Bis-EMA) in the other materials. The inorganic phase amount is highest in VITA Enamic (86 wt% or 75 vol%) and Grandio Blocks (86 wt%), being followed by Lava Ultimate (86 wt%) while varying between 61 wt% to 71.1 wt% in the other materials. This variation is mostly reflected in the light transmittance ranking of the materials. Significant variations are also identified in the chemical composition of the fillers, which is specified as a feldspar ceramic (predominant element silicon, followed by aluminum, potassium, sodium, and inclusions of yttrium) in VITA Enamic,[27] silicon and zirconium oxide in Lava Ultimate, and silicon oxide and barium glass in the other materials. An increased scattering, and thus a lower light transmittance, must be attributed to fillers containing elements of a higher atomic number. In addition, radiopaque agents,[29-31]

which are introduced to improve the radiopacity of dental RBCs, also include elements with a high atomic number and may thus contribute to an increased scattering as well.

As for both analyzed temporary PMMA materials, their optical parameters were statistically similar (p=0.99) and lined up well in the sequence of the CAD/CAM RBCs. Similar light transmittances have been identified in VITA CAD-Temp and Grandio Blocs (p=0.161) as well as in Telio CAD and Lava Ultimate A3 (p=0.373).

In the analyzed CAD/CAM ceramics and glass-ceramics, the variation in translucency (HT or LT) showed a higher impact on light transmission than the shade (A2 or A3) or the ceramic systems (feldspar ceramic, leucite and lithium disilicate glass-ceramic). In ceramic systems, translucency in general decreases with increasing crystalline content.[32] Apart from the fact that the optical properties of each constituent of a system must be considered individually, it can be stated that the crystalline content in the analyzed ceramic systems increases in the sequence feldspar ceramic (30 vol%[32]), leucite glass–ceramics (35 vol%[32]), and lithium disilicate glass-ceramic (70 vol%[33]). At a given translucency (HT or LT) and shade, this sequence is also reflected in the range of transmitted irradiances (Figure 2). Within one ceramic system, the translucency (HT or LT) seems to be regulated by adjustments in the sintering program as well as by controlling crystal nucleation and growth in glass-ceramics, which allows intervention in the crystal type and size.[32] Interestingly, the present analysis identified larger differences in light transmittance between HT and LT versions in the lithium disilicate glass-ceramic compared with the leucite glass-ceramic.

A large portion of the light attenuation through a material is due to reflection at the incident surface.[34] The surface-reflection ratio (fraction of incident radiance that is reflected at the interface), as calculated by Watts and Cash[34] by considering the shift in absorbance-path length relation from its theoretical origin, amounts to 30% to 90%,[34] while recently, values of 11% to 27% were determined in CAD/CAM RBCs.[28] The reflection of light, when incident on the interface between air and material, varies with the refractive index of optical media (Fresnel equation); however, a large part of the reflection must be related to surface roughness. Since all the specimens in the present study were polished following an identical protocol, the surface-reflection ratio was also related to the material's ability to be polished. Considering that the angle of incidence was constant, since the LCU was placed in a standardized manner, perpendicular to the specimens' surface, by means of a mechanical arm, the transmitted light might be considered a material characteristic, determined not only by the optical properties of constituents and microstructure but also by the inherent surface roughness.

The results of the present study clearly indicate that the light passing through the upper layers of an incrementally restored cavity with light-cured RBCs is insufficient to complete the polymerization of an undercured lowest increment; consequently, each increment should be *ab initio* adequately cured. Moreover, the attenuation of light through various indirect restoratives and the restoration thickness need to be considered clinically when deciding the type of luting RBCs to be used. The outcome of an additional curing through the tooth structure at the end of the restorative process to compensate for insufficient polymerization may be very low and is dependent on the thickness of the tooth structure. In this context, it must be emphasized that transmitted irradiance decreases exponentially with tooth structure thickness and is lowered to insignificant values in 3-mm-thick tooth walls.

CONCLUSIONS

The attenuation of light when passing through diverse 2-mm-thick restorative materials is high and varies between 59.9% to 94.9%; thus, deficits in polymerization are difficult to compensate for by additional light exposure at the end of the restorative process. The values measured for the tooth structure are identified toward the upper end of this range (89.4%). Light attenuation differs significantly by material within each shade category and by shade category within each analyzed material. Light attenuation was determined by shade and translucency rather than by the type of restorative material. Therefore, all null hypotheses are rejected.

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 Operative Dentistry and Periodontology, University Hospital, Ludwig-Maximilians-Universität München. The approval code for this study is 19-118 KB.

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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Letter to the Editor

Dear Editor,

We appreciated the article published in your journal, *Operative Dentistry*, titled "Ferrule design does not affect the biomechanical behavior of anterior teeth under mechanical fatigue: an in vitro evaluation."[1] It was a quality study and threw light upon the factors that are necessary to consider for clinical application, while performing similar studies on ferrule. However, we would appreciate it if the authors could attend to the following concerns regarding the study.

1. There are differences in the properties of bovine and human teeth, and since this study evaluated biomechanical properties, the results may not be clinically relevant. Bovine enamel has been found to have significantly less microhardness value and higher wear as compared with human enamel.[2] There are also significant differences in the bond strength and shear bond strength of bovine and human teeth.[3-4]
2. The title of the study is suggestive of a conclusion rather than an insight to the study.
3. The conclusions of the cross reference quoted in the study have shown contrasting results, but the present study mentioned otherwise, stating "Ma and others reported a statistical difference between no-ferrule, 0.5-mm ferrule, and 1-mm ferrule designs, with an effect size that varied from 154,925 load fatigue cycles (mean difference between no-ferrule and 0.5-mm ferrule groups) to 262,659 cycles (mean difference between no-ferrule and 1-mm ferrule groups)"; however, in the original article by Ma and others, it was concluded that teeth with a 1.0-mm ferrule showed a significantly higher fatigue cycle count than the no-ferrule group (p=0.008) but were not statistically different from the 0.5-mm ferrule group (p=0.032).[5]
4. The masticatory forces acting on the anterior teeth ranges from 382N to 108N, respectively, for males and females.[6] However, in the present study the dynamic load testing applied a load of up to 550N, which may not be clinically relevant.

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Authors' Response

We appreciate your interest in our manuscript. Please find below the response to your queries.

1. Both wear and hardness are barely related to the biomechanical behavior of restorations. In fact, the biomechanical behavior of restored teeth is closely related to mechanical properties such as elastic modulus or the Poison coefficient. Otherwise, several studies have reported the similarity between the mechanical properties of bovine and human teeth:
   1. Novais VR, Soares PB, Guimarães CM, Schliebe LR, Braga SS, & Soares CJ (2016) Effect of gamma

radiation and endodontic treatment on mechanical properties of human and bovine root dentin *Brazilian Dental Journal* **27(6)** 670-674.
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Moreover, it has been demonstrated that bovine teeth are feasible substitutes for human teeth in either dentin or enamel bond testing. See: Reis AF, Giannini M, Kavaguchi A, Soares CJ, & Line SR (2004) Comparison of microtensile bond strength to enamel and dentin of human, bovine, and porcine teeth *Journal of Adhesive Dentistry* **6(2)** 117-121.

2. The title of the article is a personal choice of the authors. We chose this title since it quickly informs the readers about the content and conclusion of the study.
3. The author of the letter misunderstood the results of the quoted study. The "Results" section of study from Ma et al (2009) reported the following: "The Mann-Whitney U test with Bonferroni correction (α=0.017) was used for pairwise comparison and indicated that the no-ferrule group was significantly different from the 0.5-mm ferrule group (p=0.008) and 1.0-mm ferrule group (p=0.008), and there was no significant difference between the 0.5-mm and 1.0-mm groups (p=0.032)." Therefore, the absence of a statistical difference was between the specimens presenting a ferrule of 0.5 and those with 1.0 mm.
4. The author misunderstood the meaning of "accelerated fatigue testing protocol," which is presented in the second paragraph of the "Discussion" section. As discussed, a stepwise fatigue approach is used to accelerate the failure, since the specimens could not present failure if loaded below its endurance limit.

Best regards,

André L. Faria-e-Silva

Letter to the Editor

Dear Editor,

First of all we thank the members of the editorial team for forwarding our letter to authors for clarification and also considering our letter for publication. We appreciate the authors for their prompt reply and clarifying all the doubts with proper explanation.

With warm regards,

Dr Anwetakshmi Ray, Dr M Kundabala, and Dr Neeta Shetty

Online Only Articles

On occasion we receive manuscripts that we would like to publish, but do not have the page room to include in the print journal. For the full article, please go to https://meridian.allenpress.com/operative-dentistry or enter the provided address into your address bar.

External Marginal Gap Evaluation of Different Resin-filling Techniques for Class II Restorations—A Micro-CT and SEM Analysis

CS Sampaio • GA Garcés • N Kolakarnprasert • PJ Atria • M Giannini • R Hirata

Clinical Relevance: Secondary caries are the main reason for the failure of restorations, class II being the most affected. Techniques that promote less gap percentage are important. Flowable bulk fill composites used at such locations have been shown to decrease gap formation while being a faster procedure than an incremental technique.

https://doi.org/10.2341/19-053-L

Degradation of Computer-aided Design/Computer-aided Manufacturing Composites by Dietary Solvents: An Optical Three-dimensional Surface Analysis

SM Munusamy • AU Yap • HL Ching • NA Yahya

Clinical Relevance: Computer-aided design/computer-aided manufacturing (CAD/CAM) composite resins are susceptible to degradation by dietary solvents. Dietary counselling is prudent when placing such CAD/CAM restorations.

https://doi.org/10.2341/19-070-L

Effect of Different Surface Treatments of Resin Relined Fiber Posts Cemented With Self-adhesive Resin Cement on Push-out and Microtensile Bond Strength Tests

RV Machry • PE Fontana • TC Bohrer • LF Valandro • OB Kaizer

Clinical Relevance: When luting relined fiber posts with self-adhesive cement, the surface treatment of the posts influences the adhesion of the fiber posts to root dentin.

https://doi.org/10.2341/19-108-L

Effect of Tack Cure on Polymerization Shrinkage of Resin-based Luting Cements

Y-S Kim • S-H Choi • B-N Lee • Y-C Hwang • I-N Hwang • W-M Oh • JL Ferracane • H-S Chang

Clinical Relevance: Self-cure after tack cure could result in a lower polymerization shrinkage in some resin-based luting cements, which is closely related to lower degree of cure.

https://doi.org/10.2341/19-159-L

Influence of Bulk-fill Restoration on Polymerization Shrinkage Stress and Marginal Gap Formation in Class V Restorations

AMO Correia • MR Andrade • JPM Tribst • ALS Borges • TMF Caneppele

Clinical Relevance: Restoring Class V cavities with a regular bulk-fill composite presents a more favorable biomechanical behavior than restoring with a regular nano-filled composite.

https://doi.org/10.2341/19-062-L

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External Marginal Gap Evaluation of Different Resin-filling Techniques for Class II Restorations—A Micro-CT and SEM Analysis

CS Sampaio • GA Garcés • N Kolakarnprasert • PJ Atria • M Giannini • R Hirata

Clinical Relevance

Secondary caries are the main reason for the failure of restorations, class II being the most affected. Techniques that promote less gap percentage are important. Flowable bulk fill composites used at such locations have been shown to decrease gap formation while being a faster procedure than an incremental technique.

SUMMARY

Purpose: To evaluate gap formation of class II restorations, resin-filling techniques using microcomputed tomography (μCT) and scanning electronic microscopy (SEM) are discussed.

Methods and Materials: Class II cavities were prepared in 30 third molars and analyzed in distal and mesial views. Prime&Bond Universal adhesive was applied in all teeth and divided into five groups (n=6): G1, SS+HIT (Spectra Smart+Horizontal Incremental Technique); G2, SS+OBL (Oblique Incremental Technique); G3, SDR+BFT (Surefil SDR+Bulk Fill Technique); G4, SDR+SS (SDR placed on cervical floors from mesial and distal boxes (not light cured), followed by incremental layering with SS and light curing incrementally with the horizontal technique); and G5, BEZ+BFT (Bulk EZ+BFT). All light-curing procedures were performed with high-mode/1200 mW/cm², Bluephase Style 20i. Teeth were scanned twice (first scan, empty tooth; second scan, filled tooth after light curing) by μCT.

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https://doi.org/10.2341/19-053-L

Acquired μCT data were evaluated with software looking for gaps at the external mesial and distal margins and submitted to statistical analysis (one-way analysis of variance and least significant differences *post hoc* test). Validation of the μCT analysis was performed by SEM.

Results: G5 showed the lowest gap formation percentage, similar to G4 (p=0.20). G4 also showed statistical similarities to G1 and G3 (p>0.05). G2 showed the highest percentages, similar to G1 (p=0.10) but different from the rest of the groups (p<0.05). SEM validated the μCT technique by showing qualitative similar images regarding external marginal gap.

Conclusions: The dual-cure composite and the use of flowable nonpolymerized plus horizontal filling technique showed the best marginal adaptations. The μCT technique was validated for visualization of gap formation after being compared to the SEM technique.

INTRODUCTION

The current composition of dental resin composites allows their use in a variety of applications in the dental field, including but not limited to restorative procedures.[1] However, although the composition of composites has significantly evolved since their introduction, they still lack antibacterial properties and seem to favor the growth of cariogenic bacteria on their surfaces.[2] Thus, recurrent caries is one of the main reasons for resin composite restorations to fail or need replacement.[3,4]

Even though a perfect marginal seal is considered impossible to achieve, clinicians should aim to obtain adaptations that are as good as possible,[5] as it was observed that the bigger a gap is between the restoration and the tooth, the bigger it will become with time and the higher the microleakage at that specific surface will be.[6,7] The presence of gaps at the composite-tooth interface and subsequent microleakage of bacteria have long been considered to be the cause of recurrent caries wall lesions.[2,8]

Apart from marginal sealing and the characteristics of the material itself,[7] as recurrent caries is a multifactorial disease, other factors can also influence the survival rate of resin composite restorations, such as the skill of the clinician,[7] the caries risk of the patient,[9] the type of dental substrate on which the composite will be placed and bonded,[7,10] the cavity size,[11] the restorative technique used,[12] and the position of the tooth in the mouth,[2,11] among others. In this context, it is clearly stated in the literature that class II restorations are more prone to secondary caries and failures than class I restorations.[2] When performing class II restorations, however, it is obvious that the clinician cannot control all of the above-mentioned aspects. Still, what the clinician can control are the materials and techniques that will be used.

The incremental filling technique has been recommended for a various number of aspects, such as decreasing polymerization shrinkage stress[13] and promoting adequate light penetration[12] within the mass of the composite. However, disadvantages have also been mentioned and include the possibility of trapping voids within layers of composite during insertion and longer working times,[2,14] which can also increase the possibility of contamination when performing the restoration.[15] A newer class of material, the so-called bulk fill composites, surpasses those difficulties by modifying monomeric and loading composition.[12] Such composites present a higher translucency and incorporate a photoactive group in the methacrylate resin that allows for an adequate degree of cure within a 4-mm depth cavity and the lower shrinkage stress than conventional composites,[15] resulting in a quicker restorative procedure. Moreover, the composition of these materials allows for a modulated polymerization reaction by use of special stress-relieving monomers, the use of more reactive photoinitiators, and the incorporation of different types of fillers, such as prepolymer particles and fiberglass rod segments.[12]

Further improvements regarding such material relate to polymerization stress, which is considered one of the major drawbacks of direct composite restorations.[16] Shrinkage stress is influenced by cavity configuration and size as well as its compliance;[16] important properties influencing shrinkage stress are volumetric shrinkage and elastic modulus of the composites.[16] A recent literature review showed that depth of cure of such bulk fill composites is increased when compared to conventional composites, likely due to its increased translucency.[16] SDR, a flowable bulk fill resin composite, has been shown to present increased depth of cure[17] with decreased polymerization stress[16] and shrinkage[18] when compared to several other resin composites. Another class of resin composite material that also seems to present an advantage consists of the dual-curing resin composites; previous findings showed that chemical-cured core buildup composites lead to a significantly lower stress than light-cured ones.[19] Today, new dual-curing resin composites are avail-

able for the clinician, although they still lack scientific reports.

However, when it comes to marginal adaptation, results of studies seem to be controversial. On the one hand, most of the studies observe gaps in the internal margin of the restoration,[12,20–24] while it is markedly noted that the external proximal and cervical proximal margins in class II restorations are often considered to be their Achilles' heel,[25] as dentin bonding is often less predictable.[7,10,25] On the other hand, some authors state that the use of a flowable composite as a lining for the restoration promotes better composite adaptation,[20] while others show that thick flowable increments can promote higher degradation of the restoration due to the flowable resin's characteristics. It has also been shown that flowable bulk fill composites can demonstrate inferior internal adaptation in gingival walls compared to packable bulk fill composites in class II restorations, showing higher degradation.[21] Controversies also are related to the use of the incremental layering technique versus bulk filling, with some studies defending the use of different types of incremental layering (eg, oblique or horizontal),[26] while others defend the use of bulk filling.[12]

For evaluating marginal adaptation, different methodologies can be performed. Commonly, destructive methodologies are available, such as the use of dies and the sectioning of specimens to enable visualization of the margins through different types of microscopes or by replicas.[7,10,12,20,24,27] Currently, modern technologies allow for the evaluation of marginal adaptation without the need to destroy the specimens. Nondestructive technologies involve optical coherence tomography[22,23,28] and microcomputed tomography (μCT).[21,23,29] Although the first presents limited depth for visualization[22,23,28] because of a limitation of the technique, μCT allows for a 3D evaluation of the entire cavity, also enabling different types of parameters and tests to be analyzed for the restoration within the same scan.[21,29] Moreover, μCT has a series of different purposes, such as to quantify and evaluate polymerization shrinkage[18] and to evaluate voids within fiber-post cementations[30] and the film thickness of veneer cementations,[31] among others.

Thus, the aim of this study was to compare the external marginal adaptation at proximal boxes of different resin composites and filling techniques, using μCT scanning and validated by scanning electronic microscopy (SEM). The hypotheses tested were 1) that different resin filling techniques promote different percentages of gap formation on class II cavities and 2) that the μCT technique is effective for evaluating external gap formation when compared to the destructive technique of SEM that is evaluated qualitatively.

METHODS AND MATERIALS

The materials and their manufacturers, composition, and batch number are described in Table 1. Thirty human molars free of caries were obtained according to protocols approved by the ethical committee. Sample size was determined according to previous studies.[27–29] Teeth had their roots cut and their cusps flattened. After that, standardized class II cavities (2.5-mm occlusal depth×4 mm wide×4 mm mesial box depth and 1 mm beyond the cemento-enamel junction distal box depth) were prepared in each tooth (n=6 per group) and analyzed in distal and mesial views. In the mesial box, 4-mm-depth preparations were done in all teeth, while in the distal box, cemento-enamel junctions were visually detected and preparations done 1 mm beyond them to have a cementum margin. Cavities were prepared using two diamond burs (codes 845kr018 and 868A021, Brasseler, Savannah, GA, USA), which provided a standardized active head size to deliver consistent cavity preparation depth. Burs were changed after every five preparations. The final cavity dimensions were then checked with a digital caliper.

After that, teeth were divided into five groups: G1, SS+HIT (Spectra Smart, Dentsply Sirona+Horizontal Incremental Technique); G2, SS+OBL (Spectra Smart+Oblique Incremental Technique); G3, SDR+BFT (Surefil SDR Flow, Dentsply Sirona+Bulk Fill Technique); G4, SDR+SS (Surefil SDR Flow placed on the cervical floors from the mesial and distal boxes from the class II cavity and not light cured), followed by incremental layering of conventional composite Spectra Smart and light curing incrementally with the horizontal technique; and G5, BEZ+BFT (Bulk EZ, Danville Materials+Bulk Fill Technique). For all teeth, the adhesive protocol used was Prime&Bond Universal adhesive (Dentsply Sirona, York, PA, USA) applied according to the self-etch protocol of the manufacturers' instructions, as most of the adhesion was performed in dentin. All light-curing procedures were performed for 20 seconds at occlusal, mesial, and distal surfaces (high mode/1200 mW/cm^2, Bluephase Style 20i, Ivoclar Vivadent, Schaan, Liechtenstein); when the incremental technique was used, each increment was light cured separately for 20 seconds. The polymerization unit step-over distance was kept constant at approximately 2 mm.

Table 1: *Materials Used in This Study and Their Manufacturers, Composition, and Batch Number*

Material (Manufacturer)	Color	Batch	Composition
Spectra Smart (Dentsply Sirona, York, PA, USA)	A2	2758401	Glass, silica, colloidal hydrophobe, dimethacrylate, benzophenone III, EDAB, FluBlau concentrate, camphorquinone, BHT butylated hydroxytoluene, yellow iron oxide, red iron oxide, black iron oxide, titanium dioxide Filler percentage: 75-77 wt%
Surefil SDR Flow (Dentsply Sirona)	U	1609143	Barium-alumino-fluoro-borosilicate glass, strontium alumino-fluoro-silicate glass, modified urethane dimethacrylate resin, ethoxylated bisphenol A dimethacrylate, triethylene glycol dimethacrylate, camphorquinone, photo-accelerator, butylated hydroxyl toluene, UV stabilizer, titanium dioxide, iron oxide pigments, fluorescing agent Filler percentage: 68 wt%
Bulk EZ (Danville Materials, Anaheim, CA, USA)	A2	42728	Ethoxylated bisphenol A dimethacrylate esters, triethylene glycol dimethacrylate, bisphenol A glycidyl methacrylate, urethane dimethacrylate, fluoride compound, glass compound Filler percentage: proprietary
Universal adhesive Prime&Bond Universal (Dentsply Sirona)		1702004905	Phosphoric acid modified acrylate resin, multifunctional acrylate, bifunctional acrylate, acidic acrylate, isopropanol, water, initiator, stabilizer
Acid phosphoric gel 37% (Maquira, Maringá, Brazil)		015216	Phosphoric acid, chlorhexidine digluconate, thickener, purified water, dye
Bluephase Style 20i light-curing unit (Ivoclar Vivadent, Schaan, Liechtenstein)		1140000142	

Filling techniques are described as follows (Figure 1): G1, SS+HIT: Resin composite was placed in horizontal consecutive layers of 2 mm each, individually for mesial, distal, and occlusal walls. G2, SS+OBL: Resin composite was placed in oblique consecutive layers of 2 mm each, without adhering two opposite walls, individually for mesial, distal, and occlusal walls. G3, SDR+BFT: Flowable composite was placed at the gingival margin without light curing, and the first layer was applied horizontally in a 2-mm layer and light cured; after that, resin composite was placed using the horizontal technique previously described. G4, SDR+SS: Resin composite was placed in one single increment. G5, BEZ+BFT: Resin composite was placed in one single increment.

μCT Analysis

For all groups, teeth were individually scanned before and after resin composite application (first scan, empty tooth; second scan, filled tooth after light activation) by μCT (μCT40, Scanco Medical, AG, Basserdorf, Switzerland). The μCT apparatus was calibrated using a phantom standard at 70 kVp per beam hardening, 200 mgHA/ccm. The operating condition for the μCT device was an energy (70 kVp and 114 mA) with a medium resolution and a voxel size created of 16 μm per slice, using a specimen holder of 16.5 mm. The average of the total number of slices was approximately 250, with an average scan time of 30 minutes per tooth, as previously described.[18]

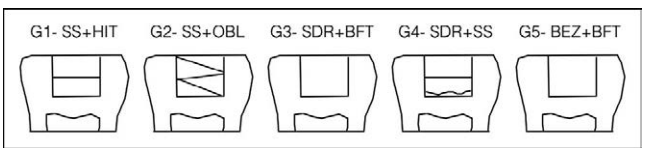


Figure 1. *Filling techniques used in this study. G1, horizontal technique; G2, oblique technique; G3, bulk fill technique; G4, flowable composite placed at the gingival margin without light curing, followed by horizontal technique; G5, bulk fill technique.*

Since enamel, dentin, and resin composite have similar densities, the first scan was performed after cavity preparation but before cavity filling (empty tooth) for all teeth. This procedure allows for posterior subtraction of both scans and acquiring a pure image of the restorations and also minimizes artifacts during the threshold segmentation.

For the second scan, a plastic laboratory sealing film (Parafilm M, SPI Supplies, West Chester, PA, USA) was used as a matrix and placed around each tooth to keep the composites in place for performing the restorations and during the scanning time. Since this matrix presents some level of elasticity, it allows for a good adaptation around the dental tissue, facilitating the placement of the restoration. Moreover, as the sealing film is transparent, light curing could be performed with the film in place. The instrument used for composite placement was a

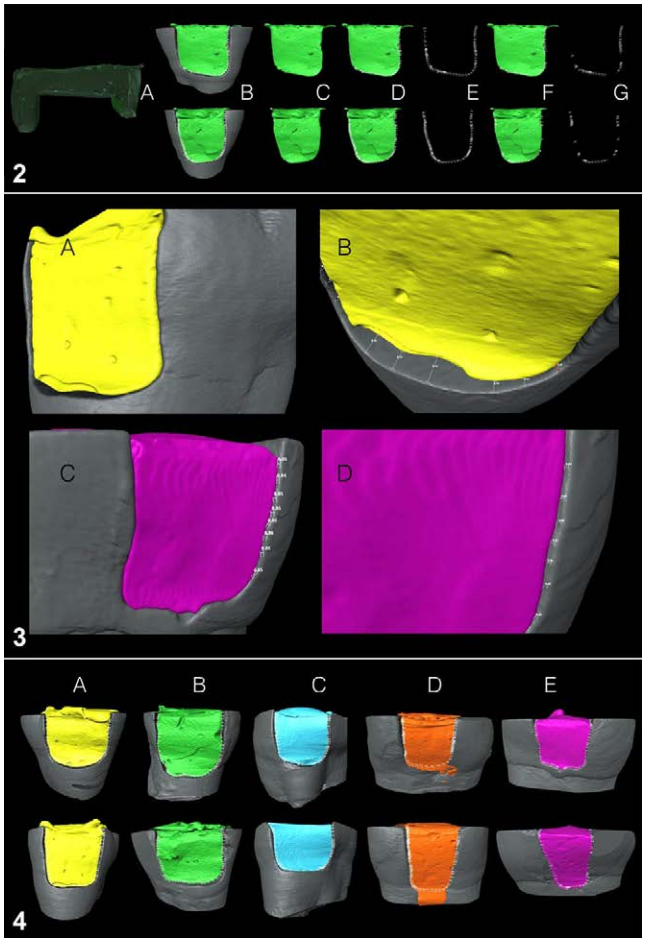


Figure 2. *Schematic depicting image analysis. (A): Restoration outside the tooth cavity. Observe the presence of voids within the composite mass. (B): Restoration observed from mesial and distal surfaces of the tooth. (C): Restoration outside the tooth at mesial and distal views. (D): Measurement of the length of the restoration to be used as total length for analysis of gap percentage. (E): Observation of the total length separated from the restoration and the cavity. (F): Measurement of the gap length within the restoration. (G): Observation of the gap length separated from the restoration and the cavity.*

Figure 3. *Gap measurements were performed from the outer surface of the cavity until its contact with the composite surface, as seen in the white lines. (A): Restoration showing a large gap and a bubble on the cervical wall. (B): Measurement of the maladjustment with the measurement tool. (C): Restoration showing adaptation in the proximal wall. (D): Maladjustment from this wall was checked to see if it was within the limit of 0.06 mm. Maladjustments below this limit were considered as gap-free margins.*

Figure 4. *Representative images of each group for μCT. (A): Group 1 (Spectra Smart, horizontal technique). (B): Group 2 (Spectra Smart, oblique technique). (C): Group 3 (SDR, bulk fill technique). (D): Group 4 (SDR placed on cervical floors, not yet light cured, followed by application of Spectra Smart with the horizontal technique). (E): Group 5 (Bulk EZ dual-curing composite, bulk fill technique).*

resin composite spatula with a burnisher at one end (1 Goldstein Flexi-Thin composite instrument, Ref CIGFT16, Hu-Friedy, Chicago, IL, USA). A single operator performed all restorative steps.

Acquired μCT data were imported into a workstation and evaluated with Amira software (version 5.5.2, VSG, Burlington, MA, USA). Both scans (empty and filled) were superimposed with the software ("superimposition" tool), and a subtraction was performed with the Boolean operation (filled tooth minus empty tooth), obtaining an image of the restoration only. Thresholds of the restorations were visually determined per specimen since the same threshold could not be used for all groups due to the different radiopacities and attenuation levels from the resin composites.

For analysis of gaps and maladjusted mesial and distal margins, the "3D measurement" operation tool was used. Quantification of gaps was performed throughout the entire length of the mesial and distal margins and quantified as percentages. A schematic depicting image analysis is shown in Figure 2. First, the entire length of the contour of the cavity was measured; after that, locations where gaps were identified and measured, and a percentage of the total was considered. Gaps were considered when maladjustments were bigger than 0.06 mm from the outer surface of the external wall toward the axial wall where resin composite was present, according to previous findings.[2,9] If the maladjustment was >0.06 mm from the external margin to the resin composite, it was considered a gap; if the maladjustment was <0.06 mm, it was not considered a gap. Measurements were performed from the outer surface of the cavity until its contact with the composite surface (Figure 3). Images from μCT scans for each group can be seen in Figure 4.

Data were checked for normality and analyzed by a one-way analysis of variance (ANOVA), and a least significant differences *post hoc* test was carried out using 95% confidence intervals. The software used was SPSS Statistics (IBM, Armonk, NY, USA).

SEM

SEM was performed for validation of the gaps and misfits observed in the μCT analysis. Each tooth was sectioned in the middle of its restoration (from mesial to distal) in its long axis in order to allow visualization of the mesial and distal margins of the restoration (Figure 5), which was also where μCT analyses were performed. SEM images were qualitatively compared with the μCT images in order to check if gaps were present or absent (Figure 6). Variable-pressure secondary electron (SEM) images were acquired with a Zeiss EVO 50 (Carl Zeiss Microscopy GmbH, Jena, Germany) in a vacuum of 20 Pa, an accelerating voltage of 15 kV, and a working distance of 13.5 mm at 5.716-mm field width.

Table 2: *Mean (%±SE) of Gap Formation (n=6, Two Sides) Determined for Each Class II Restoration Technique Observed Through μCT*[a]

Group	Gap Formation (%)
G1: SS+HIT	76.1 (5.8) AB
G2: SS+OBL	89.1 (2.9) A
G3: SDR+BFT	70.5 (1.9) B
G4: SS+SDRNP	64.0 (5.7) BC
G5: BEZ+BFT	53.7 (8.9) C

[a] *Means followed by different letters differ from each other in the same column (p<0.05).*

RESULTS

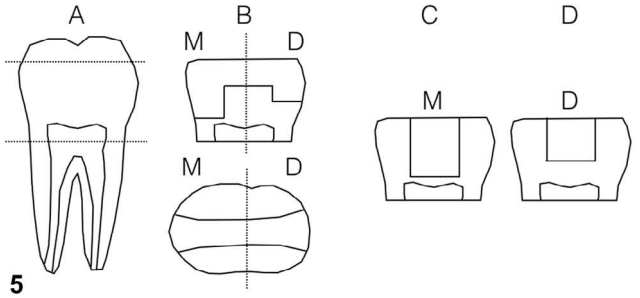
Table 2 shows the gap formation mean percentages determined for each class II restoration technique. One-way ANOVA revealed that there were significant differences between groups ($p<0.05$). Figure 4 shows a representative image obtained from a μCT scan for each group. Figure 6 shows representative images of each group for both μCT and SEM analysis.

Group 5, representing the bulk fill technique with the dual-curing composite, showed the lowest percentages of gap formation in the resin/dentin external marginal interface and did not differ from group 4, which represented the SDR resin composite unpolymerized with the addition of a conventional composite ($p>0.05$). Group 4 was also not statistically different than group 3, representing the SDR bulk fill technique ($p>0.05$). Group 2, the oblique incremental technique, was shown to be the technique with the highest amount of gap formation percentages, not statistically different than the horizontal incremental technique (group 1) ($p>0.05$), both incremental techniques being performed with a conventional composite.

Qualitatively, it was observed that most of the gaps were observed on the cervical floor rather than in the proximal walls. Such results were validated by the images acquired by SEM, as seen in Figure 6. Gap observation in the SEM images corresponded to the same location where gaps were observed in the μCT images.

DISCUSSION

Gap formation around composite restorations has been previously described in the literature; however, controversies still exist regarding the ideal technique for decreasing its formation. The present study focused on characterizing different filling techniques for class II restorations in order to observe how gap



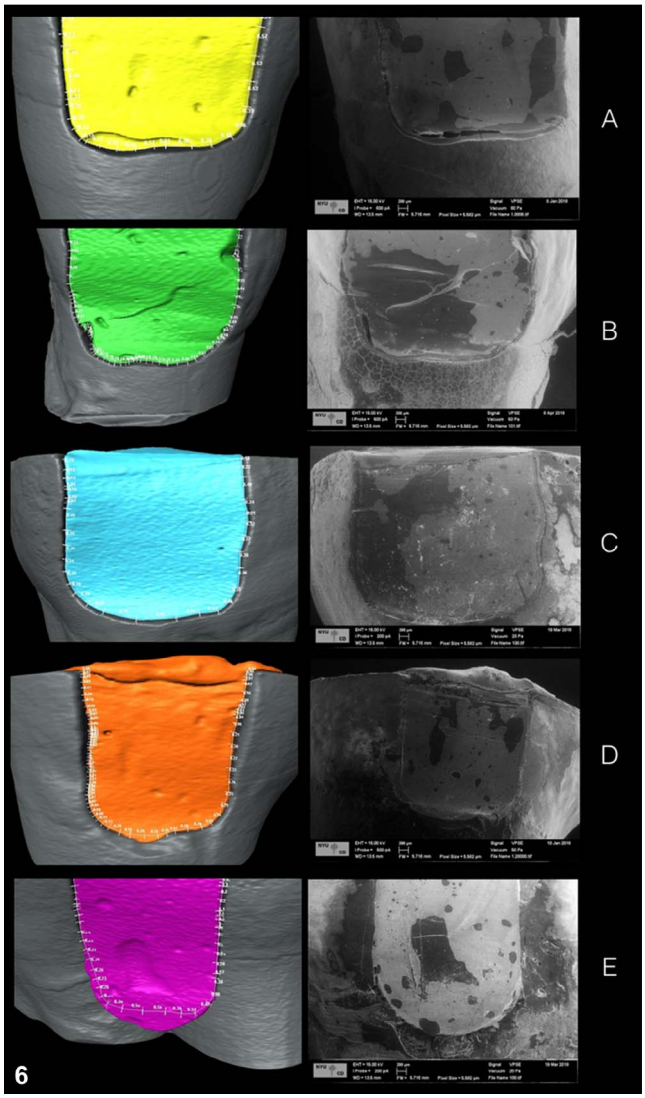


Figure 5. *Schematic depicting image analysis scanning electron microscopic specimen preparation. (A): Teeth had their roots cut and their cusps flattened. (B): After microcomputed tomographic scans, each tooth was sectioned in the middle of its restoration (from mesial to distal) in its long axis. (C): Visualization of the mesial margin of the restoration. (D): Visualization of the distal margin of the restoration.*

Figure 6. *Representative images correlation within μCT and SEM. Qualitatively, it was observed that most of the gaps were observed at the cervical wall rather than in the buccal/lingual walls. One can observe specimens that showed spreading of the material beyond the margin. A correlation was observed within the images from μCT and SEM. μCT, microcomputed tomography; SEM, scanning electron microscopy.*

formation can be decreased in the external interproximal margins of these procedures. Such margins (external interproximal mesial and distal) of class II restorations were chosen since it was observed that they are the type of restorations with the highest incidence of secondary caries formation,[2] usually located gingivally (or at cervical margins).[32] Moreover, it was observed that after aging, marginal adaptation in dentin is decreased.[7]

Since there is no accepted specific threshold for determining the correlation between gap size and wall lesion, the one used in the present study was 0.06 mm, any maladjustment bigger than this being considered a gap. This threshold was chosen because a recent study observed that a gap of about 60 μm may lead to the development of a wall lesion and predispose interfacial demineralization,[2,9] which is much less than what was previously thought, explaining the much higher values of gap percentages in this study, reaching from 53.7% to 89.1% of external proximal marginal gap when compared to other studies that showed much less a percentage of internal and external gap formation.[6,27,29] Results from a recent study showed that no matter which insertion technique was used, all resin composites present polymerization shrinkage, voids, and gaps[6] in accordance with our study. Findings from the current research indicate that different filling techniques do affect gap formation on the external interproximal margins of class II restorations, thus accepting the first hypothesis.

In this study, it was observed that the use of a flowable composite, being either the dual-curing bulk fill composite (Bulk EZ), the flowable bulk fill composite (SDR), or a combination of the nonpolymerized bulk fill flowable with incremental filling, resulted in lower percentages of gap formation than when the incremental filling technique was used purely with a conventional composite. The placement of low-viscosity materials provided better adaptation to the cavity walls. For groups 4 and 5, an excess of resin composite in the cervical floor for all of the specimens and for half of the specimens, respectively, caused by the spreading of the material in this region was observed, decreasing thus the gap formation. For group 3, this also occurred but was limited to one specimen only, while for the other specimens, the material flowed until the margin or a bit before the margin, thus causing a gap formation. For groups 1 and 2, spreading of the material was not observed beyond the cervical margins of the teeth for any specimen. The presence and absence of spreading of material beyond the margins of the teeth explain the statistical differences within groups. The gingival margin of class II restorations is the most susceptible for recurrent caries and also the place where more misfits, maladjustments, and gaps occur.[25] When such regions were covered with an excess of resin composite, such gaps were prevented from forming, thus decreasing the gap percentage of the restorations. Groups 1 and 2 showed, for all specimens, gaps in the cervical floor, explained by the lack of spreading at this region. It is believed that, for group 4, excess of material occurred for all specimens since this technique requires slight pressure in the conventional resin layer after the flowable material is applied and before it is light cured.

Obviously, flash of material is also of concern since it can lead to overhangs in the restoration, which were documented as promoting the trapping of food, periodontal problems, and subsequent recurrent caries.[33,34] Thus, mostly when flowable materials are used, it is important to observe using dental floss if this excess of material is leading to an overhang at the interproximal margins of the restoration. According to the authors' visualization in such restorations, the excess of material was not sufficient to promote overhangs that could lead to such problems for any group, although it could be a subject for future studies.

Another topic that can be related to the findings from this study is that, in a previous study, it was observed that in the postgel period, shrinkage is counteracted by adherence and by plastic flow of the resin composite;[27] the authors mentioned that the higher the plastic flow, the longer the resin composite can withstand gap formation and the smaller the resulting gap.[27] This observation can explain our results, which showed better marginal adaptation and lower gap formations when flowable composites were used. The flowability of resin composites is a determinant of gap formation, mostly in dentin cavities,[27] since they are less viscous materials that can flow easily and adapt well to the tooth surface.[20] They also serve as a flexible intermediate layer that absorbs stress during polymerization shrinkage stress of composite resin.[20]

However, the use of thick layers of flowable resin composite can also cause higher polymerization shrinkage because of the lower filler content in relation to conventional resin composites, although it depends on the type of flowable material.[35] Nevertheless, this is not the case in our study since the flowable composite used was a bulk fill flowable composite type, which has been proven to present less polymerization shrinkage than some conven-

tional packable and flowable composites[12,18] and similar depth of cure in the entire 4-mm extension of cavities.[12] Polymerization shrinkage and depth of cure of the studied dual-curing composite has not been studied to date to the authors' knowledge.

It was observed that the μCT technique was an effective tool for the evaluation of external interproximal gap formation visualization, validated against SEM, a destructive technique, thus accepting the second hypothesis. The microcomputed analysis has shown to be efficient at a series of different uses,[6,18,21,31] and its nondestructive aspect undoubtedly presents an advantage for *in vitro* studies.

Limitations of this study include the fact that the authors used a self-etch approach with a universal adhesive; possibly, by using a selective enamel etching technique, gap percentages could have been different where enamel is present. Moreover, gaps on class II cavities also depend on the anatomy of the teeth. To reduce the variation of tooth anatomy, teeth were chosen according to the most adequate anatomy to adapt in the matrix used (ie, Parafilm, which presents good adaptation in the cavity walls), and after that, teeth were assigned randomly within the groups. It was observed that marginal deterioration is a sensitive predictor of failure of a posterior composite;[36] thus, future studies should evaluate the size of gap related to wall lesion after demineralization through μCT since it was observed in this study that the size of gap and its extension are easily measurable through this method.

CONCLUSIONS

The results from this study suggested the following:

1) The use of flowable bulk fill composites at the gingival wall of class II restorations, being used either in a dual-curing technique, a conventional bulk filling technique, or a flowable bulk fill resin composite in combination with conventional incremental resin composite, was shown to be a good alternative for decreasing the percentage of gap formation besides being a faster procedure when compared to incremental technique.
2) The bulk fill techniques, along with the reported technique of filling the cervical floor with the flowable composite without polymerization and posterior filling with the incremental technique with a conventional composite, performed better than incremental resin composite filling techniques when gap percentages in the external class II proximal areas were analyzed. An excess of material was observed in all flowable composite groups, although it was shown not to be a problem involving overhang after being observed with dental floss.
3) The nondestructive μCT technique was shown to be an effective alternative tool for external gap formation evaluation when compared to SEM, a destructive technique, which was evaluated qualitatively.

Acknowledgement

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of University of the Andes, Chile.

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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Degradation of Computer-aided Design/Computer-aided Manufacturing Composites by Dietary Solvents: An Optical Three-dimensional Surface Analysis

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Clinical Relevance

Computer-aided design/computer-aided manufacturing (CAD/CAM) composite resins are susceptible to degradation by dietary solvents. Dietary counselling is prudent when placing such CAD/CAM restorations.

SUMMARY

This study determined the effect of dietary solvents on the surface roughness (Ra) of direct, indirect, and computer-aided design/computer-aided manufacturing (CAD/CAM) dental composites. The materials evaluated were a direct composite (Filtek Z350 XT [FZ]), an indirect composite (Shofu Ceramage [CM]), and four CAD/CAM composites (Lava Ultimate [LU], Shofu Block HC [HC], Cerasmart [CS], and Vita Enamic [VE]). Specimens (12×14×1.5 mm) of each material were prepared, measured for baseline Ra, ranked, divided into six groups (n=12), and conditioned in the following media for 1 week at 37°C: air (control), distilled water, 0.02 N citric acid, 0.02 N lactic acid, heptane, and 50% ethanol-water solution. The composite specimens were then subjected to postconditioning Ra testing using an optical three-dimensional surface analyzer (G4e, Alicona Imaging GmbH, Raaba, Austria). Inter-medium and inter-material comparisons were performed with one-way analysis of variance and *post hoc* Bonferroni test at a significance level of α=0.05. Mean Ra values ranged from 0.086 ± 0.004 μm to 0.153 ± 0.005 μm for

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the various material/medium combinations. For all materials, conditioning in air (control) and distilled water generally resulted in significantly lower mean Ra than exposure to other dietary solvents. Conditioning in citric acid presented the roughest surfaces for FZ, CM, and CS. For LU, HC, and VE, exposure to lactic acid, heptane, and ethanol solution resulted in the highest mean Ra. Regardless of conditioning media, FZ had the highest and VE the lowest mean Ra compared with other composites. The CAD/CAM composites remained susceptible to surface degradation by dietary solvents despite their industrial polymerization.

INTRODUCTION

Over the past decade, dental composites have evolved to meet increasing demands for esthetics, durability, and efficiency. Industrial polymerization, filler innovations, and digital dentistry are key drivers of dental composite development. Contemporary dental composites can be broadly categorized into direct, indirect, and computer-aided design/computer-aided manufacturing (CAD/CAM) materials.[1] Although direct composites are placed intraorally, indirect composites are fabricated extraorally in the laboratory before being cemented onto teeth. CAD/CAM dentistry, which was introduced in the 1970s,[2] involves the use of digitized optical scanners and chair-side or networked milling machines that fabricate single-unit restorations as well as multiple-unit bridges and frameworks.[3] The recent transition to open-access CAD/CAM systems allowed for greater data acquisition flexibility and increased choice of materials, design software, and manufacturing techniques. CAD/CAM dentistry has improved quality control, saves time, and is more cost effective than conventional dental laboratory processes.[4,5]

Ceramics and composites are the two main materials used for esthetic CAD/CAM restorations. CAD/CAM ceramics have traditionally been used because of their good mechanical and superior optical properties.[6] However, the advantages of CAD/CAM composites, such as ease in milling, better accuracy, reduced cost, simple polishing procedure, and intraoral repairability, have promoted their use over CAD/CAM ceramics.[7] Moreover, several studies found that CAD/CAM composites displayed superior fatigue or fracture resistance than glass ceramics and conventional composites.[8,9] Based on microstructure, CAD/CAM composite blocks can be divided into two subclasses: dispersed fillers and polymer-infiltrated ceramic network.[10] The first photopolymerized composite block introduced (Paradigm MZ100, 3M ESPE, St Paul, MN, USA) was composed of 85% weight-dispersed zirconia-silica fillers in a bisphenol A–glycidyl methacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) matrix. Newer CAD/CAM composites in the dispersed fillers group, including Lava Ultimate (3M ESPE), Cerasmart (GC Corporation, Tokyo, Japan), and Shofu Block HC (Shofu Inc, Kyoto, Japan), use urethane dimethacrylate (UDMA), a highly viscous monomer that requires high-temperature polymerization, in their resin matrix.[10] Vita Enamic (VITA Zahnfabrik, Bad Säckingen, Germany), the only material in the polymer-infiltrated ceramic network group, is composed of a pre-sintered glass-ceramic scaffold that is infiltrated with monomer and manufactured using high-temperature/high-pressure polymerization.

Surface roughness (Ra) of restorations can influence biofilm formation, which contributes to periodontal disease and/or recurrent caries.[11,12] The arithmetic mean of Ra is represented by a threshold Ra of 0.2 μm, above which bacterial retention may occur, proposed for restorative materials.[13] In addition, rough restoration surfaces may promote antagonist tooth wear[14,15] and are more susceptible to staining.[16] Restorations must be finished and polished properly to attain smooth surfaces,[17,18] and both laboratory and chair-side polishing of CAD/CAM composites are able to achieve Ra values below the critical threshold of 0.2 μm.[19]

Composite restorations are intermittently or constantly exposed to chemical agents in food and beverages.[20,21] The latter can occur when chemical agents are trapped around poorly finished/polished restorations, absorbed by adherent debris (calculus or food particles) at restoration margins, or produced by bacterial degradation of debris.[22,23] In research, dietary solvents or food-simulating liquids are used to simulate the diversity of ingredients in food and beverages.[24] They usually include heptane, citric acid, lactic acid, ethanol solution, and distilled water based on US Food and Drug Administration guidelines.[25]

Chemical degradation of dental composites is a clinical problem in the oral environment.[22,24] It is initiated by water absorption, which softens the resin matrix and causes hydrolytic degradation of silane couplers and fillers.[26] In addition, leaching of residual monomers, organic substances, filler particles, and ions also occurs.[27] The effects of chemical degradation on dental composites include decreased

Table 1: *Technical Profile and Manufacturers of Materials Investigated*

Material (Abbreviation)	Manufacturer	Manufacturing Process	Monomer Composition	Filler Composition	Filler % by Weight	Lot Number
Filtek Z350 XT (FZ)	3M ESPE, St Paul, MN, USA	Direct	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA	Silica nanoparticles and zirconia nanoparticles	78.5	N771467
Shofu Ceramage (CM)	Shofu, Kyoto, Japan	Indirect	UDMA (+ HEMA in opaque paste)	Silica-based glass	74	011605
Lava Ultimate (LU)	3M ESPE, St Paul, MN, USA	CAD/CAM	UDMA	SiO_2 (20 nm), ZrO_2 (4-11 nm), ZrO_2/SiO_2 clusters	79	N554839
Shofu Block HC (HC)	Shofu, Kyoto, Japan		UDMA + TEGDMA	SiO_2, Zirconium silicate	61	091501
Cerasmart (CS)	GC, Tokyo, Japan		UDMA + other DMA	SiO_2 (20 nm), barium glass (300 nm)	71	1410271
Vita Enamic (VE)	VITA Zahnfabrik, Bad Säckingen, Germany		UDMA + TEGDMA	86% glass-ceramic sintered network	86	20160422

Abbreviations: Bis-EMA, ethoxylated bisphenol-A-glycidyl methacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; CAD/CAM, computer-aided design/computer-aided manufacturing; DMA, dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; SiO_2, silicone dioxide; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; ZrO_2, zirconium dioxide.

surface hardness and change in surface topography, resulting in an increase in roughness values.[26,28] The aforementioned effects can be detected in about a month *in vivo*.[28] However, exposure to dietary solvents allow chemical degradation of dental composites to be observed within a week *in vitro*[20,22] while taking into account processes like chemical affinity, elution, and/or bonding of filler-silane.[23]

Although the effect of dietary solvents on the surface degradation of direct and indirect composites has been previously reported,[20,21,29,30] their effects on CAD/CAM composites are still not widely researched. CAD/CAM composites are anticipated to be more resistant to the effects of dietary solvents in view of their industrial high-heat and high-pressure polymerization. The objective of this study was to determine the effect of dietary solvents on the Ra of direct, indirect, and CAD/CAM composites. The null hypotheses were as follows: 1) the Ra of CAD/CAM composites is not significantly affected by dietary solvents, and 2) there is no significant difference in Ra between composite types after exposure to the various dietary solvents.

METHODS AND MATERIALS

Specimen Preparation and Conditioning

The materials investigated and their technical profiles are shown in Table 1. The materials consisted of one direct (Filtek Z350 XT [FZ]), one indirect (Shofu Ceramage [CM]), and four CAD/CAM dental composites (Lava Ultimate [LU], Shofu Block HC [HC], Cerasmart [CS], and Vita Enamic [VE]). All materials were shade A2. Sample size was calculated using the G*Power Software version 3.1.9.3[31] based on an analysis of variance (ANOVA) test with effect size of 0.5[20], alpha error of 0.05, and power of 80% for six materials. In total, 72 specimens were prepared for each material with provision for a 20% specimen rejection. The FZ and CM specimens were fabricated by using a customized stainless steel mold with a recess of 12×14×1.5 mm. The direct and indirect composites were placed in one increment, covered with a transparent cellulose acetate strip, and compressed between two glass slides. Excess materials were extruded, and a 1000 g load was applied for 15 seconds through the top glass slide. The FZ specimens were then cured with four overlapping irradiations of 20 seconds each using a light-emitting diode (LED) curing unit (Demi Plus, Kerr, Orange, CA, USA). The LED curing unit had an 8-mm-diameter light guide, output irradiance of 1330 mW/cm^2, and a wavelength range of 450-470 nm. Irradiance was measured using a LED radiometer (Demetron LED Radiometer, Kerr) to ensure consistency of light output for full depth cure. The CM specimens were irradiated using a calibrated Solidilite V (Shofu Dental, Kyoto, Japan) laboratory curing unit that had 4 halogen lamps with a total power of 600 W and wavelength range of 400-550 nm. After an initial cure for 1 minute on the turntable, CM specimens were removed from their molds and further cured for another 3 minutes. Both FZ and CM specimens were left in an incubator at 37°C for 24 hours to allow post-cure after photopolymerization. For LU, HC, CS, and VE, CAD/CAM blocks of 12×14 mm were sectioned into 1.5-mm-thick specimens using a high-speed diamond saw under water coolant (Micracut 176, Metkon, Bursa, Turkey).

The direct, indirect, and CAD/CAM composite specimens were examined for defects and replaced where needed. The specimens were then polished sequentially on both sides using a twin variable-speed grinding and polisher machine (Buehler, Lake Bluff, IL, USA) with a series of silicon carbide abrasive paper discs from P600 to P1200 at 250 rpm for 30 seconds.[32,33] They were subsequently measured with a digital micrometer (Mitutoyo Corporation, Kawasaki, Japan) to ensure dimensional uniformity (±0.15 mm) after polishing. The polished specimens were ultrasonically cleaned in distilled water and washed with isopropanol to remove any impurities. Baseline Ra of the 72 specimens of each composite were measured and ranked from smallest to largest before being divided into six groups (n=12) to ensure equal distribution of specimens with varying Ra and to minimize sampling bias. The six groups were conditioned for 1 week at 37°C in the following media: air (control), distilled water, 0.02 N citric acid (pH 2.6), 0.02 N lactic acid (pH 2.6), heptane, and 50% ethanol-water solution. Ten milliliters of each dietary solvent was used for each group. At the end of the conditioning period, the specimens were rinsed with distilled water, gently blotted dry with absorbent paper, and subjected to postconditioning Ra measurement.

Ra Testing and Scanning Electron Microscopy Observation

Ra was measured using the Infinite Focus Optical 3D Measurement G4e machine (Alicona Imaging GmbH, Raaba, Austria). The magnification of the three-dimensional (3D) surface analyzer was set at 20× with vertical and lateral resolutions of 325.66 nm and 2.93 μm, respectively. Images from five standardized areas of each specimen were captured, and the average Ra was determined using IFM version 3.5 software (Alicona Imaging GmbH, Raaba, Austria). The measurements were subsequently tabulated, and mean values for each material/medium combination were computed. Representative control and postconditioning samples with the highest mean Ra value were examined with scanning electron microscopy (SEM) (Quanta FEG 250, Thermo Fisher Scientific, Brno, Czech Republic), at 5000× magnification and 10 kV to ascertain microstructural changes.

Statistical Analysis

Statistical analysis was performed using the SPSS Statistic 24.0 software (IBM, Chicago, IL, USA). Parametric analysis was used as data was found to be normally distributed with the Kolmogorov-Smirnov test. Two-way ANOVA was performed to determine interactions between materials and conditioning media. Inter-medium and inter-material comparisons were performed with one-way ANOVA and *post hoc* Bonferroni test**.** Statistical analyses were conducted at a significance level of $\alpha = 0.05$.

RESULTS

Mean Ra values of the composite materials after conditioning in the various media are shown in Table 2. Mean Ra ranged from 0.086 ± 0.004 μm to 0.153 ± 0.005 μm for VE exposed to air or distilled water and for FZ conditioned in citric acid, respectively. Tables 3 and 4 showed inter-medium comparisons for the different materials and inter-material comparisons after exposure to the various conditioning media. Two-way ANOVA revealed significant interactions between materials and conditioning media ($p<0.05$).

For all composites, the smoothest surfaces were observed after exposure to air or distilled water, and mean Ra values were mostly significantly lower than the other dietary solvents (Table 3). With the exception of CS and VE, conditioning in citric acid, lactic acid, heptane, and ethanol solution resulted in significantly rougher surfaces than the control. No significant difference in mean Ra was observed between lactic/citric acid and the control for CS/VE, respectively. Conditioning in citric acid presented the roughest surfaces for FZ, CM, and CS. Significant differences in mean Ra values were observed between exposure to citric acid and heptane for FZ. Conditioning in lactic acid presented the roughest surface for LU. Significant differences in mean Ra values were observed between exposure to the two acids and heptane as well as ethanol solution. For HC and VE, conditioning in heptane and ethanol solution resulted in the roughest surfaces, respectively. VE, however, appeared to be less susceptible to degradation by citric acid.

For all conditioning media, mean Ra of FZ was the highest while that of VE was the lowest compared with other composites investigated (Table 4). When exposed to air (control) and distilled water, mean Ra ranking and significant differences between materials were alike. Conditioning in citric acid presented similar results, with the exception of the lack of significant inter-material difference between CM and CS. No significant differences in mean Ra values were observed between FZ, LU, and CM after conditioning in lactic acid. They were, however, still significantly rougher than CS, HC, and VE. For

Table 2: *Mean (Standard Deviation) Ra Values for the Various Materials*

Dietary Solvents	Mean Ra values (μm)					
	FZ	CM	LU	HC	CS	VE
Control (air)	0.136 (0.006)	0.125 (0.006)	0.120 (0.005)	0.105 (0.006)	0.116 (0.008)	0.086 (0.004)
Distilled water	0.137 (0.005)	0.125 (0.006)	0.120 (0.006)	0.106 (0.006)	0.117 (0.008)	0.086 (0.004)
0.02 N citric acid	0.153 (0.005)	0.140 (0.008)	0.140 (0.008)	0.122 (0.006)	0.134 (0.006)	0.087 (0.005)
0.02 N lactic acid	0.147 (0.008)	0.138 (0.007)	0.142 (0.008)	0.121 (0.004)	0.124 (0.009)	0.096 (0.004)
Heptane	0.144 (0.006)	0.137 (0.006)	0.132 (0.005)	0.125 (0.006)	0.131 (0.008)	0.103 (0.007)
50% ethanol-water	0.146 (0.006)	0.137 (0.009)	0.131 (0.004)	0.123 (0.006)	0.131 (0.009)	0.107 (0.007)

Abbreviations: CM, Shofu Ceramage; CS, Cerasmart; FZ, Filtek Z350 XT; HC, Shofu Block HC; LU, Lava Ultimate; Ra, surface roughness; VE, Vita Enamic.

heptane, FZ was only significantly rougher than the CAD/CAM composites. In addition, CM was also considerably rougher than HC and VE. The mean Ra of FZ was significantly higher than that of the other composites after conditioning in ethanol solution. CM was, again, notably rougher than HC and VE.

Representative postconditioning samples with the highest mean Ra values were subjected to SEM examination and compared with the control group. SEM micrograph of FZ after conditioning in 0.02 N citric acid showed formation of surface voids and exposure of filler particles (Figure 1). Erosion of resin matrix with appearance of filler particles was also seen in CM after conditioning in 0.02 N citric acid (Figure 2). LU displayed accompanying filler/matrix interfacial failure after conditioning in 0.02 N lactic acid (Figure 3). HC showed sizeable areas of matrix erosion and localized porosity with some protrusion of fillers after exposure to heptane (Figure 4). SEM micrograph of CS showed nano-sized filler particles. Matrix erosion occurred with exposure to 0.02 N citric acid, but surface damage was not as apparent (Figure 5). For VE, considerable matrix erosion occurred after conditioning in 50% ethanol-water solution, revealing the glass-ceramic network (Figure 6).

Table 3: *Comparison of Mean Ra Values Between Dietary Solvents Based on Materials*[a]

Materials	Differences Between Dietary Solvents
FZ	Citric acid, lactic acid, 50% ethanol-water, heptane > Control (air); Citric acid, lactic acid, 50% ethanol-water > Distilled water; Citric acid > Heptane
CM	Citric acid, lactic acid, heptane, 50% ethanol-water > Control (air) and distilled water
LU	Lactic acid, citric acid, heptane, 50% ethanol-water > Control (air) and distilled water; Lactic acid, citric acid > Heptane and 50% ethanol-water
HC	Heptane, 50% ethanol-water, citric acid, lactic acid > Distilled water and control (air)
CS	Citric acid, heptane, 50% ethanol-water > Control (air) and distilled water
VE	50% ethanol-water, heptane, lactic acid > Control (air); 50% ethanol-water, heptane > Lactic acid, citric acid, and distilled water; Lactic acid > Citric acid and distilled water

Abbreviations: CM, Shofu Ceramage; CS, Cerasmart; FZ, Filtek Z350 XT; HC, Shofu Block HC; LU, Lava Ultimate; Ra, surface roughness; VE, Vita Enamic

[a] *Results of one-way analysis of variance and post hoc test ($p < 0.05$); > indicates statistically significant differences in mean Ra values between conditioning in different dietary solvents for each material.*

DISCUSSION

The effect of dietary solvents on the Ra of direct, indirect, and CAD/CAM composites was examined in this study. As the Ra of CAD/CAM composites was affected and significant differences existed between composite materials after conditioning in the various dietary solvents, both null hypotheses were rejected. Significant two-way interactions (ANOVA) between materials and conditioning media were observed, indicating that the effect of dietary solvents on Ra of the composites was material dependent. This finding corroborated those of earlier literature.[27]

For all composite materials evaluated, no significant difference in mean Ra was observed between conditioning in air (control) and distilled water. The polymer network in dental composites may contain porosity and intermolecular spaces that allow water or solvents to diffuse.[34] Two important factors that influence water sorption in dental composites are degree of conversion and amount of residual monomers remaining in the polymer network.[35] When matrix polymer is subjected to high temperature or a combination of high temperature and high pressure polymerization, the degree of conversion is increased. The higher cross-linked network reduces the diffusion of water/solvents and amount of leachable unreacted monomers.[36,37] The solubility parameter describes a solvent's ability to dissolve a

Table 4: *Comparison of Mean Ra Values Between Materials Based on Dietary Solvents*[a]

Dietary Solvents	Differences Between Materials
Control (air)	FZ > CM, LU, CS > HC > VE; CM > CS
Distilled water	FZ > CM, LU, CS > HC > VE; CM > CS
Citric acid	FZ > CM, LU, CS > HC > VE
Lactic acid	FZ, LU, CM > CS, HC >VE
Heptane	FZ > LU, CS, HC > VE; CM > HC, VE
50% ethanol-water	FZ > CM, LU, CS, HC > VE; CM > HC, VE

Abbreviations: CM, Shofu Ceramage; CS, Cerasmart; FZ, Filtek Z350 XT; HC, Shofu Block HC; LU, Lava Ultimate; Ra, surface roughness; VE, Vita Enamic

[a] *Results of one-way analysis of variance and post hoc test ($p < 0.05$); > indicates statistically significant differences in mean Ra values between different materials after conditioning in each dietary solvent.*

substance.[30] Together with polymer network density, the solubility parameter affects the extent and rate of solvent uptake.[30,35] Degradation of dental composites is greater when the solubility parameter mismatch between polymers and solvent is small.[35] The type of polymer, filler load and composition, as well as surface treatment of filler particles also influence hydrolytic degradation.[38] Notwithstanding the higher degree of conversion of indirect and CAD/CAM composites, the direct composite FZ also displayed no significant difference in Ra between air (control) and distilled water. The resistance of FZ to hydrolytic degradation may be attributed in part to the use of spherical shaped nanoparticle fillers. These nanoparticles form nanoclusters that improve packing and reduce stresses that commonly occur on irregularities of filler/matrix interfaces.[39,40] Furthermore, water uptake is a diffusion-controlled process and may take a minimum of one week to show significant effect on surface properties of composites.[41,42]

Dietary solvents used in this study included heptane, which simulates butter, fatty meats, and vegetable oils as well as citric acid, lactic acid, and ethanol, which mimic certain alcohols, vegetables, fruits, candy, and syrup.[22] Distilled water simulates nonacidic foods with pH more than 5 and models the wet oral environment.[21] Alcohol and acidic/basic solvents exhibit hygroscopic and hydrolytic effects on dental composites, and the degree of degradation is influenced by the chemistry of the solvents.[30,43] This was in accordance with our findings, where conditioning in citric acid, lactic acid, heptane, and 50% ethanol-water resulted in significantly greater postconditioning mean Ra values than with air (control) and distilled water. Acids such as citric and lactic acids are known to have softening effects on polymers through chemical degradation of the resin matrix and silane agent as well as hydrolytic breakdown of the filler particles.[44,45] The aforementioned effects are supported by SEM micrographs of FZ, CM, CS in citric acid, and LU in lactic acid, respectively. VE was not significantly degraded by citric acid, and this may be ascribed to the filler composition (interpenetrated resin matrix) that differed from the other composites investigated.

Previous studies have reported superior and stable surface characteristics when dental composites are stored in heptane as it can prevent leaching of silica and combined metal from filler particles.[20,38] Interestingly, conditioning in heptane presented the roughest surface for HC in our study. This may be attributed partly to the relatively low filler loading of HC (61%) compared with the other materials (71% to 86%) and higher monomer composition accordingly. Moreover, TEGDMA in HC has been reported to cause a plasticizing effect and solvent susceptibility.[46] The latter effect is supported by changes in surface morphology, including considerable areas of resin matrix erosion observed under SEM. Degradation of the polymer matrix and filler-silane bond by ethanol-water solution is influenced by the composition and chemical structure of the polymer networks.[23,47] In this study, 50% ethanol-water solution was selected because it mimicked food and beverages consumed by people and allowed composite degradation to be observed.[20,21,44,48] The solubility parameter of pure ethanol approximated that of Bis-GMA resin, whereas TEGDMA resin absorbed higher amounts of 50% ethanol-water solution than water or pure ethanol.[49] Since FZ contains both Bis-GMA and TEGDMA, the aforementioned may explain the significantly roughened surfaces of FZ after conditioning in 50% ethanol-water solution. In this study, it was observed that for VE, conditioning in 50% ethanol-water solution presented the highest Ra. The SEM micrograph of VE showed irregular preinfiltrated porous ceramic[50] for the control sample and obvious volume defects after conditioning in 50% ethanol-water solution. The microstructural changes may be attributed to the degradation and loss of the infiltrated polymer, leading to exposure of the porous ceramic network.

The chemical degradation of dental composites can be assessed by changes in Ra. Moreover, Ra of restorations is found to affect clinical performance as adhesion of plaque on rough surfaces could promote caries and periodontitis.[51] The baseline Ra values for

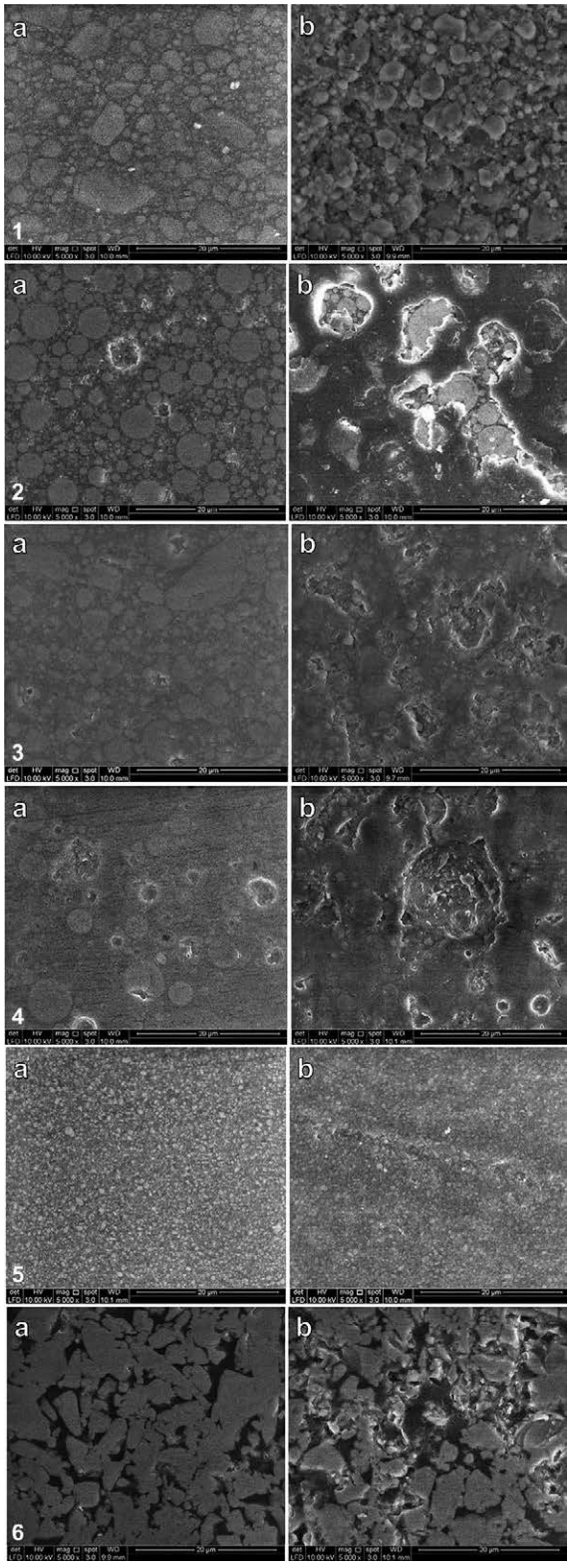

Figure 1. *Scanning electron micrograph of Filtek Z350 XT (a): air (control) and (b): after conditioning in 0.02 N citric acid.*

Figure 2. Scanning electron micrograph of Shofu Ceramage (a): air (control) and (b): after conditioning in 0.02 N citric acid.

CAD/CAM composite blocks were significantly lower than those of the direct composite FZ and consistent with findings of a previous study where laboratory-based polishing of CAD/CAM composites presented Ra values between 0.06 μm and 0.16 μm, while chair-side polishing achieved values between 0.11 μm and 0.13 μm.[19] After conditioning in dietary solvents, the mean Ra of all composites tested were below the threshold Ra of 0.2 μm. The performance of the composites was thus acceptable for the duration of dietary solvent exposure in this study. Chemical degradation observed with one week of conditioning may actually take a few months or years to occur intraorally, depending on exposure time and the patient's oral condition.[21] A recent study found that elution of unreacted components from conventional resin-based materials can occur up to one year and is influenced by composition of composite, degree of conversion, solvent type, size, and the chemical nature of released components.[52] This warrants further investigation via chemical analysis, and longer storage periods are needed to assess progressive chemical degradation of the CAD/CAM dental composites.

CONCLUSIONS

Within limits of this study, the following can be concluded:

1. Ra of dental composites, including CAD/CAM materials, was significantly affected by dietary solvents, with the exception of distilled water.
2. For CAD/CAM composites, conditioning in citric acid, lactic acid, heptane, and 50% ethanol-water solution presented the roughest surface for CS, LU, HC, and VE, respectively.
3. Differences in Ra between composite types were both material and solvent dependent.

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Figure 3. Scanning electron micrograph of Lava Ultimate (a): air (control) and (b): after conditioning in 0.02 N lactic acid.

Figure 4. Scanning electron micrograph of Shofu Block HC (a): air (control) and (b): after conditioning in heptane.

Figure 5. Scanning electron micrograph of Cerasmart (a): air (control) and (b): after conditioning in 0.02 N citric acid.

Figure 6. Scanning electron micrograph of Vita Enamic (a): air (control) and (b): after conditioning in 50% ethanol-water solution.

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Conflict of Interest

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

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Effect of Different Surface Treatments of Resin Relined Fiber Posts Cemented With Self-adhesive Resin Cement on Push-out and Microtensile Bond Strength Tests

RV Machry • PE Fontana • TC Bohrer • LF Valandro • OB Kaizer

Clinical Relevance

When luting relined fiber posts with self-adhesive cement, the surface treatment of the posts influences the adhesion of the fiber posts to root dentin.

SUMMARY

This study evaluated the effect of surface treatment and silanization of resin composite on the bond strength of relined fiber posts cemented with self-adhesive resin cement. Push-out and microtensile bond strength (MTBS) tests were performed in this study. The endodontic treatment of 80 single-rooted bovine teeth was first performed in the push-out test segment, followed by weakening the intracanal walls by diamond bur. Then, the glass fiber posts were adapted with resin composite to fill the root canals, followed by photoactivation and resin surface conditioning according to four different experimental conditions: no conditioning as control, 10% hydrofluoric acid, 35% hydrogen peroxide, or air abrasion with alumina particle (all groups were subdivided into "with silanization" or "without silanization," thus totaling eight experimental groups). Self-adhesive resin cement was used for the post cementation. Four slices per tooth were obtained for the push-out tests. Next, 160 blocks of resin composite were first produced for the MTBS tests; their bonding surfaces were conditioned (as mentioned,

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ie, eight treatments), and they were cemented to each other. The 80 sets (n=10/treatment) were then cut into microbars (16/set): eight were immediately tested, while the other eight were thermocycled (12,000×) and stored (120 days) before MTBS. Failure modes and topographic analyses were performed after treatments. There was no statistically significant difference for the push-out results. In MTBS, surface treatment and silanization had a significant effect ($p<0.001$). Aging decreased bond strength for all groups. Considering the aged groups, air abrasion promoted the highest values and silanization improved bond strength for all treatments except air abrasion. The alumina particle air abrasion of the relining resin composite promoted the highest bond strengths when luting with self-adhesive resin cement.

INTRODUCTION

Teeth with extensive coronary destruction usually require the use of retainers, which provide core retention and, consequently support for prosthesis or coronary restoration.[1,2] Although cast post-and-cores and fiber posts present equally favorable clinical results regarding longevity in teeth with regular root canals,[3] the high modulus of elasticity of the cast post-and-cores contraindicates their use in teeth with an excessively wide root canal.[4] Meanwhile, fiber posts seem more indicated because they have similar modulus of elasticity to that of the dentin, promoting better distribution of the stresses in the root and less probability of catastrophic root fractures.[5,6]

The problem intensifies when the root canal presents oval anatomy or is greatly enlarged by endodontic treatments, internal resorption, and caries, thus leading to critical situations such as displacement of the posts resulting from poor adaptation to the root canal, problems caused by excessive cement thickness (voids and irregular contraction of polymerization), and risk of catastrophic fractures due to thin root walls.[7–9] Thus, relined posts (fiberglass posts relined in the root canal with resin composite) have been proposed to enable better adaptation of prefabricated fiber posts in nonuniform anatomy or flared or oval-shaped canals.[10] This technique considerably reduces the resin cement thickness required to fill the space between the post and the root canal walls and reduces the occurrence of voids and failure by polymerization contraction of the resin cement, consequently improving the adhesive bond strength of the post to the root canal.[8,11–13]

Self-adhesive resin cements present a less sensitive technique because they eliminate the precementation steps of the resin cement inside the root canal.[14] Moreover, these cements promote better bond strength results than conventional resin cements when tested with the relined posts.[15,16] However, until now no study has evaluated surface conditioning of resin composite specifically applied for relining fiber posts to improve the bond strength of resin cements to the root canals.

Some techniques for surface treatment of resin composite blocks, such as aluminum oxide particle air abrasion and hydrofluoric acid etching have been previously tested to cement these blocks with self-adhesive resin cement for bond strength improvements.[17] These treatments increased the surface roughness (adhesive area), increased the surface free energy, and exposed resin composite filler components.[5,18–21] Similarly, hydrogen peroxide was found to increase the roughness of resin composite restoration when it was clinically applied for bleaching.[22] Therefore, bond strength improvements might also be expected when resin relined fiber posts are subjected to this kind of surface treatment before cementation.

Also, 3-(methacryloxy)propyl-trimethoxysilane-based primers are also indicated as coupling agents (chemical treatment) as part of a surface treatment, with or without topographic changes via physical treatments in order to promote bonding between organic and inorganic compounds from ceramic/resin composite and resin cements.[23] Its application is recommended in repairing resin composite restorations, especially after surface treatments such as air abrasion with aluminum oxide, as it interacts with the filler particles, as well as in conditioning of glass-ceramic and silica-coated zirconia polycrystal.[4,24–26] However, its application in resin composite is still questioned.[21]

Therefore, the aim of this present study was to evaluate the effect of different surface treatments and silane application on the bond strength durability of relined posts cemented with self-adhesive resin cement to the root canals. This question deserves to be evaluated, taking into account that adhesive failures of the relined post occur and have been investigated.[15,16] Therefore, in view of the previous considerations, the null hypothesis of our study was that there is no difference in bond strength between resin composite and resin cement after the different

Table 1: *Experimental Groups Regarding the Surface Treatment Used, Application or Not of the Silane Agent and Thermocycling Plus Storage (Only for Microtensile Bond Strength Test)*

Surface Treatment	Name/Brand Protocol Application	Silane Application[a]	Push-out Groups	Thermocycling and Storage[b]	MTBS Groups
Control	No surface treatment	No	CTRL	No (baseline)	CTRL
				Yes	CTRLtherm
		Yes	CTRL+S	No (baseline)	CTRL+S
				Yes	CTRL+Stherm
10% hydrofluoric acid	(Dentsply, Petrópolis, Brazil) Acid application for 60 seconds.[39]	No	HF	No (baseline)	HF
				Yes	HFtherm
		Yes	HF+S	No (baseline)	HF+S
				Yes	HF+Stherm
Hydrogen peroxide 35%	(Whiteness HP; FGM, Joinville, Brazil) Gel application for 60 seconds.	No	HPerox	No (baseline)	HPerox
				Yes	HPeroxtherm
		Yes	HPerox+S	No (baseline)	HPerox+S
				Yes	HPerox+Stherm
Air abrasion with 45 μm aluminum oxide	(Polidental, Cotia, Brazil) 10 seconds at distance of 5mm and pressure of 2.8 bar.[39]	No	AAbr	No (baseline)	AAbr
				Yes	AAbrtherm
		Yes	AAbr+S	No (baseline)	AAbr
				Yes	AAbrtherm

Abbreviations: CTRL, control; S, silane; HF, Hydrofluoric Acid; HPerox, Hydrogen Peroxide; AAbr, air abrasion; Therm, thermocycling.
[a] *Rubbing for 5 seconds and waiting for 5 minutes for solvent evaporation.*
[b] *Only the specimens of the microtensile test were divided for thermocycling and storage.*

surface conditioning protocols or silane application. In addition, aging does not reduce the microtensile bond strength values.

METHODS AND MATERIALS

Study Design

Two mechanical tests were applied in this study: push-out and microtensile tests.

Push-out Test

The sample size was calculated using the OpenEpi 3.01 program implementing parameters that were based on a previous pilot study considering a power of 80% and a significance level of 0.05, requiring eight bovine teeth per group. However, 10 teeth per group were used in this study because of the variability of the root anatomy of bovine teeth (n=10). The specimens were randomly allocated into eight groups considering the surface treatment with or without silane application (Table 1).

Tooth Selection and Preparation—Bovine incisors were obtained and sectioned to get roots with standard lengths of 16 mm. The roots were then selected according to the diameter of a size 80 K-file (Dentsply Maillefer, Ballaigues, Switzerland) to reduce the size variation between root canals. Apical root portions were included in a chemically cured acrylic resin (VIPI, Pirassununga, Brazil) block. The specimens were attached on a dental surveyor with the long axes of the teeth and the resin block parallel to each other and perpendicular to the ground. All tooth preparation protocols were performed by one operator (RVM).

Endodontic Procedures—Canal patency was established with a size 15 K-file (Dentsply Maillefer). The working length was set at 1 mm from the apex. Root canals were prepared by using endodontic files (Dentsply Maillefer). The coronal portion of the roots was initially prepared by using Gates-Glidden drills (Dentsply Maillefer). The step-back technique was subsequently applied. Each canal was irrigated with 2 mL of a 2.5% sodium hypochlorite (Novaderme, Santa Maria, Brazil) between each instrument change. Specimens were irrigated with 5 mL of 17% ethylenediaminetetraacetic acid (EDTA, Novaderme) for 3 minutes and subsequently rinsed with 2 mL of distilled water. Next, they were dried using size 80 paper points (Dentsply Maillefer).

AH Plus (Dentsply Maillefer) was mixed according to the manufacturer's instructions and placed to working length using a lentulo spiral (Dentsply Maillefer). Gutta-percha cones (Dentsply Maillefer) compatible with the diameter of the last instrument used for the apical third of the root canal were used. The compression technique was cold lateral condensation with R8 accessory cones (Tanari, Manaca-

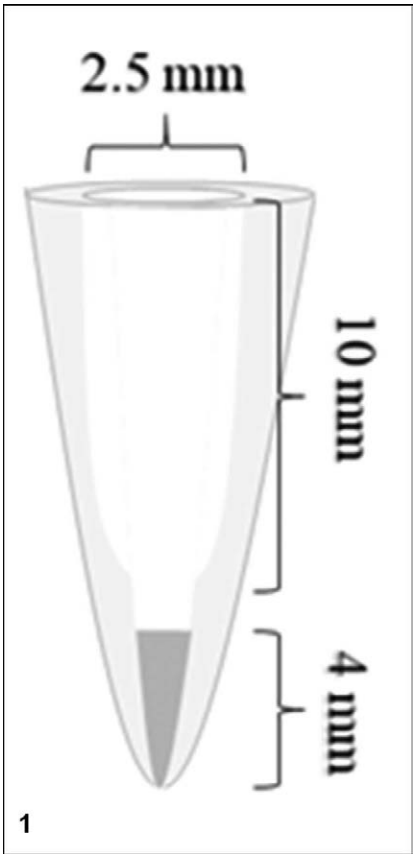

Figure 1. *Representative image of tooth enlarging preparation for push-out test.*

puru, Brazil). Excess gutta-percha in the coronal portion was removed with a hot instrument. Roots were stored for 72 hours at 37°C and 100% humidity to allow the sealers to set.

Post Space Preparation—Root canal filling was partially removed using a hot instrument and a Whitepost DC N2 (FGM, Joinville, Brazil) bur at 12 mm. Then, the root canals were enlarged by one operator (RVM) who ground the intracanal walls with No. 4137 diamond burs (KG Sorensen, Cotia, Brazil) in high rotation (Extra Torque 605C; Kavo, Joinville, Brazil) under distilled refrigerated water (Figure 1). This enlarging was performed in the most coronal portion of the root up to 10 mm deep, standardizing the canal diameter opening through the total diameter of the bur (2.5 mm).

Luting Procedures—To obtain relined fiberglass posts, Whitepost DC N2 (FGM) posts were cleaned with 70% alcohol, and a silane (RelyX Ceramic Primer; 3M ESPE, Seefeld, Germany) was applied according to the manufacturer's instructions. The resin composite (Filtek Z250; 3M ESPE) was inserted inside the previously lubricated root canal (K-Y gel; Johnson & Johnson, São José dos Campos, Brazil), the post was positioned, and the resin was light cured for 5 seconds using a 1200 mW/cm^2 light-emitting diode light-curing unit (Radii Cal; SDI, Melbourne, Australia) from the occlusal surface. The relined posts were removed from the canal, light-cured for 40 seconds, and reinserted to verify the adaptation.[9] The 80 relined posts were divided into surface treatment and silane application groups (Table 1). All specimens were subjected to the same washing and drying process (washing with distilled water for 10 seconds using a dental syringe and air-jet drying for 30 seconds) after each treatment procedure.

The self-adhesive resin cement (RelyX U200; 3M ESPE) was handled properly and inserted into the canal, followed by insertion of the relined fiber post. The cement was light-cured inside the root for 40 seconds (Radii Cal; SDI), 10 seconds on each face. The specimens were stored for 24 hours at 37°C.

Push-out Test—The teeth were fixed on a metal base in the cutting machine (Isomet 1000 Precision Saw; Buehler, Warwick, UK) and then sectioned perpendicular to the long axis of the root. The first coronal slice (approximately 1 mm thick) was discarded, and four other slices per specimen (thickness: 1.5±0.3 mm) were obtained (40 per group). Each slice was positioned on a metallic device with a central opening (Ø=3 mm) larger than the canal diameter. The most coronal portion of the specimen was placed downward.

The push-out test was performed in a universal testing machine (Emic DL-2000; Emic, São José dos Pinhais, Brazil) at a speed of 1 mm/min. A metallic cylinder (Ø extremity= 0.8 mm) induced a load on the post in an apical to coronal direction without applying any pressure on the resin composite, cement, or dentin.[16]

Push-out bond strength values (α) in MPa were obtained with the formula $\alpha = F/A$, where F = load for specimen rupture (N) and A = bonded area (mm^2). The formula to calculate the lateral area of a circular straight cone with parallel bases was used to determine the area. The formula used was $A = \pi g(R_1 + R_2)$, where $\pi = 3.14$, g = slant height, R_1 = smaller base radius, and R_2 = larger base radius. The following formula was used to determine the slant height: $g = (h^2 + [R_2 - R_1]^2)^{1/2}$, where h = section height; R_1 and R_2 were obtained by measuring the internal diameters of the smaller and larger base, respectively, which corresponded to the internal diameter between the root canal walls. The diame-

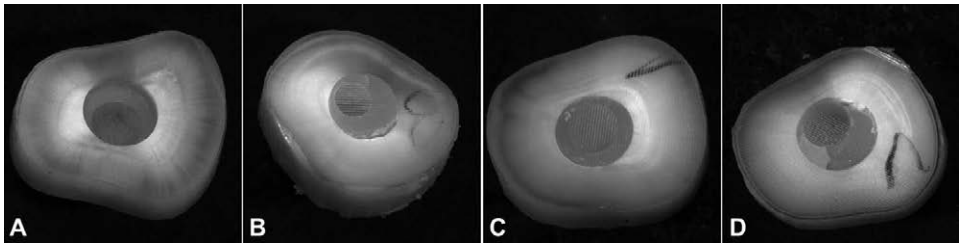

Figure 2. *Representative failure images of push-out test at 10x magnification. (A) adhesive at cement/dentin interface, (B) adhesive at resin composite/cement interface, (C) adhesive at resin composite/fiber post interface, (D) cohesive in some material or dentin.*

ters and section height were measured using a digital caliper (Starret 727, Starrett, Itu, São Paulo, Brazil).[16,27–29]

Failure Analysis—Specimens were analyzed at 10× magnification with a stereomicroscope (Zeiss Stemi SV6; Carl Zeiss, Jena, Germany) after the push-out test. Failure modes were categorized as follows: Ac/d = mainly adhesive at cement/dentin interface, Ac/cr = mainly adhesive at resin composite/cement interface, Cr/p = mainly adhesive at resin composite/fiber post interface, COE = mainly cohesive in some material or dentin (Figure 2). The color of the composites was carefully selected in order to clarify the evaluated interfaces. Only the Ac/cr failures were considered for statistical analysis for the push-out test because this was the interface of interest.

Microtensile Test

One hundred and sixty (160) microbars for each surface treatment (as designed for push-out tests) were obtained. For this, microhybrid resin composite blocks were cemented together both to the same experimental group according to the surface treatment with or without silane application.

In addition, the 160 specimens from each group were divided equally (n=80) according to the aging condition. Aging and storage for MTBS were evaluated to test the bond strength durability (immediate test or aging condition) (Figure 3).

Specimen Production for MTBS—One hundred and sixty (160) resin composite blocks (Filtek Z250; 3M ESPE, eighty shade A1 and eighty shade D3) were prepared using a silicon template (4 mm high and 8 mm sides) placed on a glass plate covered by a polyester strip. Each increment (±2 mm) was inserted using a No. 1 spatula (Golgran, São Caetano do Sul, Brazil) and photoactivated for 40 seconds (Radii Cal; SDI, 1200 mW/cm^2). The last layer was covered with a polyester strip and compressed using a glass slide to obtain a flat surface. The sample was photoactivated through the glass plate with the polyester strip in contact with the resin composite surface. The obtained blocks were divided by shade into surface treatment and silane application groups (Table 1). One operator (RVM) performed all specimen production procedures.

Cementation Procedures—The blocks were washed for 10 seconds with distilled water spray and dried with water and oil free spray before surface treatment and cementation. The bonding surface of each block received the surface treatment, as described in Table 1. The surfaces of those included in the silanization groups were cleaned with 70% alcohol and then the silane agent was applied with a disposable microbrush (Cavibrush; FGM) and rubbed for 5 seconds with evaporation of solvent for 5 minutes.

The self-adhesive resin cement (RelyX U200; 3M ESPE) was mixed properly and applied on the conditioned surface of one of the blocks and another block from the same group was positioned/cemented, followed by static load application of 2.5 N onto the assembly and then cement excess was removed with a microbrush, waiting for 3 minutes before proceeding with photoactivation for 25 seconds on the interface on one side of the blocks. The assembly then received additional photoactivation (80 seconds, ie, 20 seconds each side) after load removal.

Microtensile Bond Strength Test—Each block was stored in 37°C distilled water for 24 hours and then sectioned into microbars with an interface area of about 1 mm × 1 mm, using a diamond disk at low speed under water cooling (Isomet, Buehler), producing a total of approximately 16 microbars 8 mm long. Half of the samples (8 microbars) were immediately subjected to the microtensile test (baseline), and the other half were aged for 12,000 cycles between 5°C and 55°C with a dwell time of 30 seconds and a transfer time of 2 seconds (Nova Etica, São Paulo, Brazil), then stored in 37°C distilled water for 120 days.[20,30]

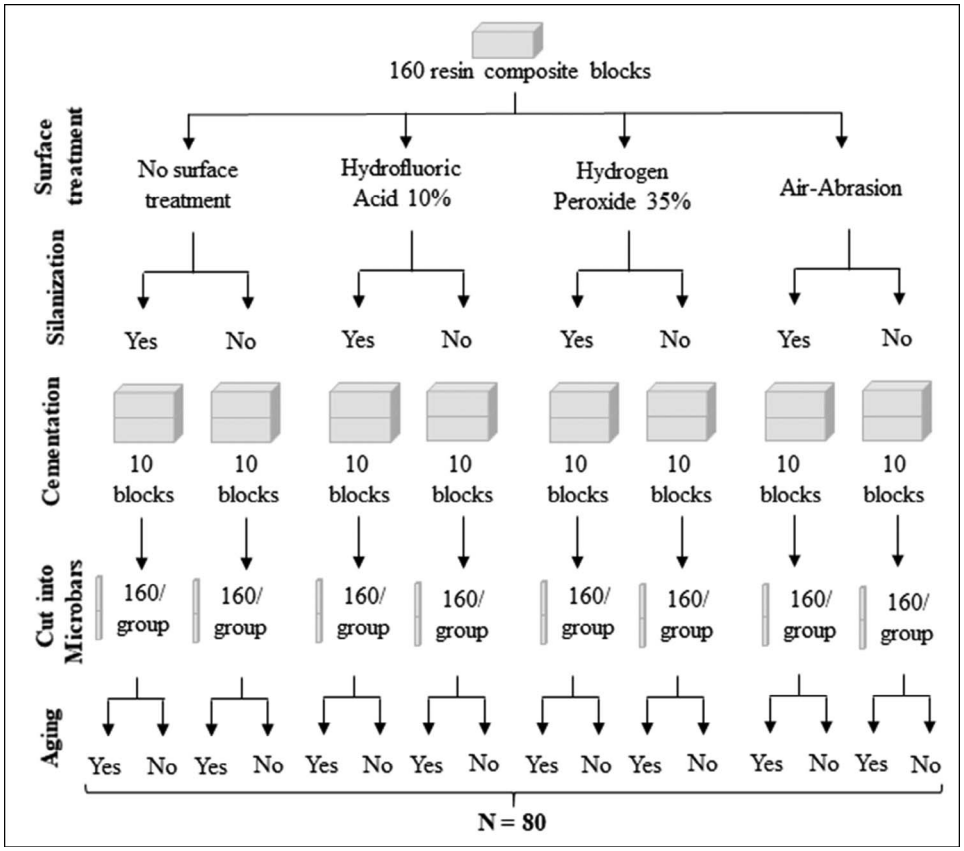

Figure 3. *Representative image diagram of the MTBS experimental groups.*

Each sample was measured using a digital caliper (Starrett 727; Starrett, Itu, Brazil) and positioned in Geraldeli devices with cyanoacrylate glue (Three Bond Gel; Three Bond, Diadema, Brazil). The MTBS was determined in a universal testing machine (EMIC DL-2000, São José dos Pinhais, Brazil) with a load cell of 50 kN (force limit = 500 N) at a speed of 0.5 mm/min. The bond strength (α) in MPa was calculated by $\alpha = f/a$, where f = the force required to induce failure (in newtons) and a = the area of the bonded interface (mm^2; thickness 1 × thickness 2, measured at the adhesive zone).[20,31]

Failure Analysis—All specimens submitted to the microtensile test were analyzed under a stereomicroscope (Zeiss Stemi SV6; Carl Zeiss, Jena, Ger-

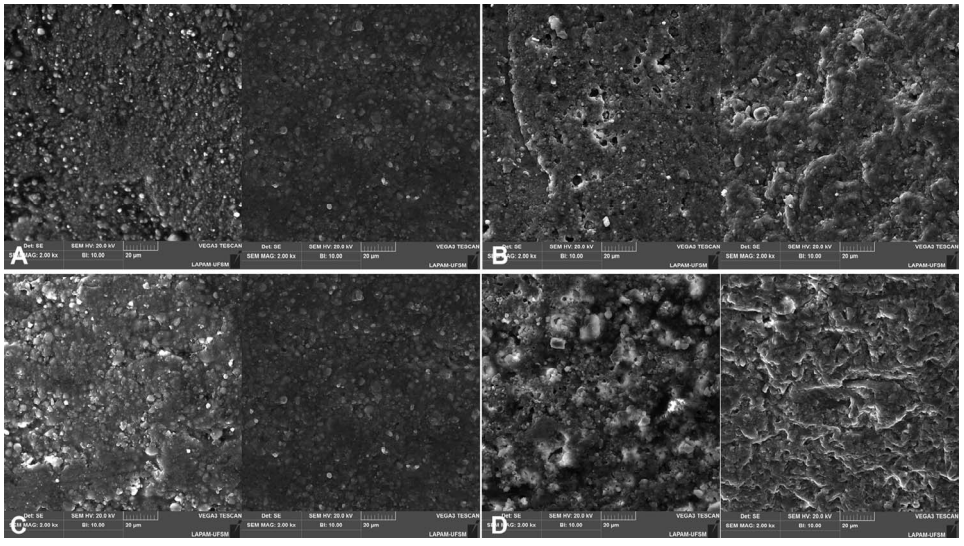

Figure 4. *Representative SEM images at 2000x magnification of relined fiber post surface non-silanized and silanized: (A) non-surface treatment; (B) 10% Hydrofluoric Acid treatment; (C) 35% Hydrogen Peroxide treatment; (D) air-abrasion treatment.*

Table 2: *Results of Push-out Bond Strength Tests and Failure Modes Distribution*

Groups	Bond strength	Failures				
		Ac/d	Ac/cr	Cr/p	COE	Total
Control	2.7 ± 1.8	27 (67.5%)	5 (12.5%)	7 (17.5%)	1 (2.5%)	40
Control + silanization	2.0 ± 1.4	32 (80%)	3 (7.5%)	5 (1.25%)	0	40
Hydrofluoric acid	2.5 ± 1.4	27 (67.5%)	5 (12.5%)	8 (20%)	0	40
Hydrofluoric acid + silanization	2.2 ± 1.0	21 (52.5%)	7 (17.5%)	12 (30%)	0	40
Hydrogen peroxide	2.6 ± 2.7	10 (25%)	15 (37.5%)	15 (37.5%)	0	40
Hydrogen peroxide + silanization	1.6 ± 0.3	27 (67.5%)	8 (20%)	5 (12.5%)	0	40
Air abrasion	2.2[a]	22 (55%)	2 (5%)	16 (40%)	0	40
Air abrasion + silanization	2.0 ± 0.5	26 (65%)	6 (15%)	8 (20%)	0	40
	Total	192 (60%)	51 (15.94%)	76 (23.75%)	1 (0.31%)	320

Abbreviations: Ac/d, cement/dentin; Ac/cr, composite resin/cement; Cr/p, composite resin/fiber post; COE, cohesive failure in dentin.
[a] *Insufficient values were obtained to calculate the standard deviation.*

many). The failure modes were categorized as mainly adhesive (at resin composite/cement interface failure) or cohesive (predominant in the resin composite or cement).

Scanning Electron Microscopy (SEM)

One sample of relined fiber post from each group was prepared for surface analysis by scanning electron microscopy (VEGA3; TESCAN, Brno, Kohoutovice, Czech Republic) at 2000× magnification to assess changes in surface topography.

Data Analysis

Normality and homogeneity analyses were performed, confirming normal distribution of the data from the two tests.

Two-way analysis of variance (ANOVA) (IBM SPSS Software; IBM, Armonk, NY, USA) was used for statistical analysis in the push-out test, considering the surface treatment in the same silane application condition. The silane application was analyzed with the Student *t*-test in the same surface treatment. The significance level was set at 5%.

One-way ANOVA was used for statistical analysis for MTBS to investigate the difference between the groups regarding the surface treatment within the same condition of silanization and/or aging. A Tukey test was applied to compare the same surface treatment, varying the silanization and aging.

RESULTS

Push-out Test

Two-way ANOVA showed that the surface treatment and silanization had no significant effect on push-out bond strength; thus, the groups had similar statistical results (Table 2). The main failure type was between cement and root dentin, while posts treated with hydrogen peroxide without silane application presented 37.5% of failures between cement and resin composite (Table 2).

Microtensile Bond Strength Test

At baseline condition, the following was observed (Table 3): 1) hydrofluoric acid etching had the lowest bond strength when the silane was not applied, and the other groups had higher and similar bond strength; 2) air abrasion had the highest values with silanization; and 3) the silane application improved the bond strength of the hydrofluoric acid group, reduced the bond strength of the hydrogen peroxide group, and had no effect for control and air-abrasion groups.

In the aging condition, the following was noted (Table 3): 1) air abrasion promoted the highest bond strength results for silane application and no silane application; and 2) silanization had no effect on the bond strength results of the air abrasion groups.

It was noted that the bond strength of all of the groups was lower after aging when comparing baseline vs aging and keeping the same surface treatment and silane condition (evaluation of the bond strength durability) (Table 3).

Regarding the failure mode, there were more adhesive failures after aging compared with the immediately tested specimens (Table 4); the exception was the hydrofluoric acid group without silane, which had less adhesive failure after thermal aging. The air-abrasion and untreated groups generally had fewer adhesive failures than the other two.

Table 3: *Results of the Microtensile Bond Strength Test in MPa* [a]

Treatment	Baseline		Aging	
	No Silanization	Silanization	No Silanization	Silanization
No surface treatment	63.6 ± 18.9 Aa*	66.7 ± 11.8 Ba*	42.4 ± 13.9 Bb°	52.3 ± 11.1 Ba°
Hydrofluoric acid 10%	55.5 ± 11.0 Bb*	68.5 ± 11.7 Ba*	45.9 ± 10.8 Bb°	51.8 ± 10.9 Ba°
Hydrogen peroxide 35%	65.2 ± 12.4 Aa*	48.7 ± 11.3 Cb*	34.7 ± 9.1 Ca°	35.3 ± 8.3 Ca°
Air abrasion	71.8 ± 16.3 Ab*	81.0 ± 11.4 Aa*	60.6 ± 12.7 Aa°	57.7 ± 12.7 Aa°
p-value	0.000	0.000	0.000	0.000

[a] *Different lowercase letters indicate statistical difference between silane application within the same treatment and aging condition. Different upper-case letters indicate statistical difference between treatments on same silanization and aging condition. Different symbols (*/°) indicate statistical difference between the means of the specimens tested immediately and those submitted to thermocycling on same silanization condition.*

Pretest failures (breaking the specimen during cutting or during the aging process) were not included; therefore, not all groups had a total of 80 specimens. The groups that had pretest failure were control with silane (2 losses), hydrofluoric acid 10% (6 losses, in which 4 were in aging specimens), and air abrasion with silane at the baseline (1 loss).

Topographic Analysis

Hydrofluoric acid etching and air abrasion changed the surface topography of the resin composite, removing its matrix with exposure of fillers, opening the spaces in nanoscale, resulting in a somewhat rougher surface (Figure 4). Voids and possible undercuts appeared in the surface etched by hydrofluoric acid owing to selective corrosion process, while air abrasion blasted and scratched the surface by partial material removal and alumina particle deposition (particle incrustation via kinetic energy from the particles air-induction).

DISCUSSION

This study showed significant differences in bond strength for the microtensile test (MTBS) between self-adhesive resin cement and resin composite when different surface treatments were applied, despite the push-out test not presenting any difference between surface treatments. Therefore, the null hypothesis was partially accepted.

The push-out test presents somewhat similar characteristics in terms of effect from forces when under clinical service on the fiber post, interfaces, and root dentin, that is, vectors of shear stress inducing pull-out of fiber post. Clinically, mainly adhesive failures at the interface have been observed between resin cement and dentin making this the most critical area.[12,13] These failures were identified as predominant in the push-out tests we performed. Thus, we suggest that it has been the reason for insignificant statistical differences in terms of the adhesion of the fiber posts treated with the surface treatments tested in the push-out test. These findings suggest that the priority is to improve adhesion of resin cement to dentin considering the context evaluated in our study.

In contrast, our findings show statistical differences in adhesion for distinct treatments when evaluated under the MTBS test, indicating that

Table 4: *Failure Modes in Each Group for Microtensile Bond Strength*[a]

	Adhesive		Cohesive		Glue Failure		Total	
	Baseline	Aging	Baseline	Aging	Baseline	Aging	Baseline	Aging
CTRL	29 (36.2)	39 (48.8)	46 (57.5)	41 (51.3)	5 (6.2)	0	80	80
CTRL+S	29 (36.7)	56 (70.9)	47 (59.5)	23 (29.1)	3 (3.8)	0	79	79
HF	55 (70.5)	50 (65.8)	22 (28.2)	26 (34.2)	1 (1.3)	0	78	76
HF+S	54 (67.5)	56 (70.0)	26 (32.5)	24 (30.0)	0	0	80	80
HPerox	52 (65.0)	71 (88.8)	28 (35.0)	9 (11.3)	0	0	80	80
HPerox+S	66 (82.5)	69 (86.3)	14 (17.5)	11 (13.8)	0	0	80	80
AAbr	22 (27.5)	46 (57.5)	57 (71.2)	34 (42.5)	1 (1.2)	0	80	80
AAbr+S	21 (26.6)	46 (57.5)	55 (69.2)	34 (42.5)	3 (3.8)	0	79	80
Total	328 (51.6)	433 (68.2)	295 (46.4)	202 (31.8)	13 (2.0)	0	636	635

[a]Absolute values and percentage of failures before aging (baseline) and after thermocycling (aging).
Abbreviation: CTRL, control; S, silane; HF, Hydrofluoric Acid; HPerox, Hydrogen Peroxide; AAbr, air abrasion.

treatments influence adhesion between the resin composite and the resin cement, especially after treatments that generate greater surface roughness. This methodologic approach isolates the interface of interest for this study, thereby generating a more homogeneous stress distribution at the interface than other mechanical tests.[32,33] Therefore, these outcomes present more specific interpretations of the adhesive resistance between resin cement and resin composite. In addition, the obtained results may even be considered for other interpretations and other applications of resin cements on resin composite restorations.

The cohesive failures of MTBS occur because the resin composite has a tensile strength around 66 MPa (28.1 - 102.1 MPa).[31] The adhesive failure rates obtained in the present study are within this range; therefore, the groups that presented the most cohesive failure had this outcome due to the high adhesive resistance between the self-adhesive resin cement and the resin composite, which was similar to the fracture strength of the resin composite. Thus, Palasuk and others[34] reported that the MTBS produced after air abrasion with aluminum oxide on resin composites was not different from the cohesive strength of this material. The same can be seen in our findings, since similar bond strength values between resin composite and resin cement for the cohesive strength of the resin composite occurred.[31]

The air-abrasion group in this study generally had the highest bond strength values compared with the other treatments; this is in accordance with previous studies that suggest that the bond strength of a composite improves with a new resinous material due to the increased roughness of the treated surface.[19,35–37] SEM evidenced the significant increase of surface roughness in specimens sandblasted with 45-μm aluminum oxide particles in the same way as previous studies.[36,38]

This increase in roughness seemed to be the most important reason for the bond strength improvement between the two composites among the factors that influenced adhesion of the self-adhesive resin cement to the prepared substrate. This may have been caused by increased mechanical interlocking and exposure of the silica particles, as well as the similar chemical nature between the old and new resins, inherently potentiating the chemical bonds.[38]

In the same way as air abrasion, hydrofluoric acid promotes changes in the topography of the composite when applied on a resin composite surface. However, there is water penetration and hydrolytic degradation during the conditioning process, which means breaking the silane bond between the matrix and filler and a consequent weakening of the composite.[39,40] Thus, the resin composite surface becomes rough, but the structure becomes very weak and prone to microcracks. The SEM image evidences areas of structure loss, which corroborate this behavior. The use of silane on resin composite under this treatment improved the bond strength results. However, it is possible to deduce that the reapplication of this element prevents this weakening since hydrofluoric acid acts by breaking the silane bonds present in the composites.

The union between silane and resin composite deteriorates over time due to hydrolysis since the resins are permeable.[41] It is believed this deterioration will be lower if the surface preparation is adequate, providing micromechanical retention performed before silane treatment.[41] However, contrary to what can be observed for dental ceramics,[25] our study demonstrated that this effect did not occur on the resin composite surfaces. When considering the no-treatment group, silane application after aging promoted higher bond strength than the no-silanization condition, meaning it appears to produce a relevant effect when no surface topographic changes occur; on the other hand, silanization had no effect for the air-abrasion group, thereby corroborating a study that showed no gain when associating alumina particle air abrasion with silane application.[42]

The thermal cycle aging process might lead to hydrolytic degradation in the resin matrix owing to the effect of contraction and expansion of the composite and distinct substrates.[20,30] Our findings show a significant reduction of MTBS after aging; all the treatments and their combinations had lower adhesion after aging compared with their counterpart at baseline, meaning distinct outcomes might be obtained before or after aging. This demonstrates that bond strength durability evaluation by means of aging procedure (long-term storage, thermal cycling) is crucial to better interpret and understand the adhesion promotion potential of surface conditionings.

Regarding study limitations, the cohesive failures in the microtensile test could be mentioned, even though only data from samples with adhesive failure were considered for statistical analysis. Also, no aging condition (thermal or mechanical cycling) were performed on the push-out specimens. Another aspect is that bovine teeth were used as substitutes

for human teeth for the push-out evaluation; however, the use of bovine teeth in adhesion tests has been well accepted due to the similarities to human teeth.[43]

CONCLUSIONS

- The surface treatments had no effect on the push-out bond strength results.
- The alumina particle air abrasion promoted the highest MTBS.
- In MTBS, aging reduced bond strength for all surface treatments.
- Silanization promoted better bond strength results for the MTBS specimens etched by 10% hydrofluoric acid and air abrasion when immediately tested.
- For the aging MTBS specimens, silanization improved bond strength for the control and 10% hydrofluoric acid groups.

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Conflict of Interest

The authors state that they do not present any conflicts of interest.

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Effect of Tack Cure on Polymerization Shrinkage of Resin-based Luting Cements

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Clinical Relevance

Self-cure after tack cure could result in a lower polymerization shrinkage in some resin-based luting cements, which is closely related to lower degree of cure.

SUMMARY

Objectives: To evaluate the effect of tack cure on polymerization shrinkage (PS) of resin-based luting cements.

Methods and Materials: One composite resin cement, Duo-Link (Duolink); two self-adhesive resin cements, RelyX U200 (U200) and G-CEM LinkAce (GCem); and one resin-modified glass ionomer cement, RelyX Luting Plus (Luting+), were used for measuring PS in light-cure (LC group), self-cure (SC group), and two tack-cure modes that were light cured (TC-LC group) or self-cured (TC-SC group) after tack cure. PS was measured by a modified bonded disc method for 1600 seconds and analyzed with two-way analysis of variance and Tukey honestly significant difference test. To investigate the effect of tack cure on light cure or self-cure, data were analyzed with an independent-samples *t*-test with tack cure as a variable. The significance level was 5%.

Results: Regarding cure mode, Duolink showed a significantly lower PS in the TC-SC group compared with the other groups. Luting+ showed a significantly lower PS in the TC-SC group than in the SC group. U200 showed a significantly lower PS in the self-cure groups compared with that in the light-cure groups. The PS of GCem was not affected by cure mode.

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Regarding cements, Luting+ showed the highest PS, followed by GCem, Duolink, and U200 ($p<0.05$). Self-cure of Duolink and Luting+ was negatively affected by tack cure, while light cure was not affected. U200 and GCem were not affected by tack cure either in the self-cure or light-cure groups.

Conclusion: For the tested cements, tack cure decreased the PS of Duolink and Luting+ when they were self-cured after tack cure. When the cements were light cured after tack cure, PS was not affected by tack cure in any cement.

INTRODUCTION

The tack cure procedure is generally used to remove excess cement from the margins of indirect restorations when using resin-based luting cements with the tack-cure option.[1] Briefly, a light-cure tip is placed over the margins and light cured for a short time to polymerize cements to a semi-gel state, which makes the removal of excess cement easier while fixing the restoration in place. Then, the cements can be light cured or self-cured for final cure according to the manufacturer's instructions.[2,3]

As dual-cure cements incorporate both light-cure and self-cure components, the polymerization initiates independently but overlaps with the other cure mode during the cure reaction, as both cure modes initiate free-radical formation and monomer conversion.[4] The exact mechanism for the interaction between light cure and self-cure is not yet known. However, studies have shown that both cure modes could not guarantee a uniform maximum cure.[2,5,6] The self-cure reaction is generally slower and shows less effective monomer conversion than when light cure is used as a supplement,[4,7] as there is a limit to the amount of self-cure promoters that can be added to dual-cure cements in order not to impair their working time. Therefore, the self-cure reaction is generally restricted to some extent.[7]

One issue encountered with the tack cure procedure is that immediate light cure of some dual-cure cements appears to interfere with the following self-cure reaction and restricts the cement from achieving its maximum mechanical properties.[7-9] Immediate light cure induces rapid formation of polymer chain cross-linking, which results in entrapment of initiators and unreacted monomers because of increased steric hindrance. Thus, segmental mobility of methacrylate groups is decreased along with the reactivity of the self-cure reaction.[10,11] In addition, studies showed that immediate low-dose light cure, which is more closely related to tack cure, could lower the degree of cure (DC) of dual-cure cements.[7,9]

Another issue is encountered when the cement is light cured after tack cure to reduce chair time. This is because the self-cure reaction is in progress during excess cement removal. The resulting interruption of the light-cure procedure could also result in the formation of relatively linear polymer structures with lower cross-link density, which makes polymers more susceptible to softening, as shown in pulse-delay cure of light-cure composite resins.[12,13]

Therefore, the incorporation of a tack-cure procedure offers a complex cure scenario consisting of light cure and self-cure in dual-cure cements. Previous studies showed that delaying or omitting light cure might modify the polymeric structure[14] and the extent of polymerization of dual-cure cements.[9] The moment of light cure, light intensity, duration of interruption, and frequency of interruption determine the way structure networks will be formed,[15,16] thus determining the structural integrity of the materials.[9] However, there are only limited studies on the evaluation of clinically relevant tack-cure scenarios for polymerization of dual-cure cements.[2,3,17] Therefore, the aim of this study was to evaluate the effect of tack cure on the polymerization shrinkage (PS) of resin-based luting cements. The null hypothesis was that tack cure would not affect the PS of resin-based luting cements.

METHODS AND MATERIALS

One conventional composite resin cement, Duo-Link (Duolink); two self-adhesive resin cements (SARCs), RelyX U200 (U200) and G-CEM LinkAce (GCem); and one resin-modified glass ionomer cement (RMGIC), RelyX Luting Plus (Luting+) were used for measuring the PS in light-cure mode (LC group), self-cure mode (SC group), and two tack-cure modes that were light cured (TC-LC group) or self-cured (TC-SC group) after tack cure (Table 1). Duolink and GCem were of the automix type, whereas U200 and Luting+ were of the clicker type. For the clicker type, the cement was dispensed on a mixing pad and hand mixed with a plastic spatula. Mixed cement was delivered into the C-R syringe (Needle Tip, Seil Global, Busan, Korea) and plugged and loaded into a gun. A volume of 14 mm^3 of each cement was measured with a flat 3-mm thick stainless-steel mold with a hole of 2.44 mm diameter, which was placed on a glass slide. After filling the mold to the upper limit, it was carefully lifted from the glass slide,

Table 1: *Materials Used in This Study and Tack-Cure Time for Each Material*

Group	Cement Type	Product (Lot No.)	Composition	Tack Cure Time, s
Duolink	Composite resin cement	Duo-Link Trans, Bisco, Schaumberg, IL, USA (1600006736)	Base: Bis-GMA, TEGDMA, glass filler Catalyst: Bis-GMA, TEGDMA, glass filler Filler load: 38 vol%, 61 wt%	2
U200	Self-adhesive resin cement	RelyX U200 TR, 3M, Neuss, Germany (630798)	Base paste: fiberglass, phosphoric acid esters methacrylate, TEGDMA, silica treated silane, sodium persulfate Catalyst paste: fiberglass, substitute dimethacrylate, silane-treated silica, p-toluenesulfonate sodium, and calcium hydroxide 70 wt%, 50 vol%	1
GCem	Self-adhesive resin cement	G-CEM LinkAce A2, GC, Tokyo, Japan (1610111)	Paste A: Fluoro-alumino-silicate glass, UDMA, dimethacrylate, silicon dioxide, initiator, inhibitor Paste B: Silicon dioxide, UDMA, dimethacrylate, initiator, inhibitor 55.3 wt%	1
Luting+	Resin-modified glass ionomer cement	RelyX Luting Plus, 3M, St Paul, MN, USA (N820543)	Paste A: Silane-treated filler, water, 2-hydroxyethyl methacrylate, silane-treated silica, 4-(dimethylamino)-benzene-ethanol, titanium dioxide Paste B: Silane-treated ceramic, copolymer of acrylic and itaconic acids, 2-hydroxyethyl methacrylate, water, glycerol 1,3 dimethacrylate 1-5%, potassium persulfate, potassium diphosphate, glyceryl methacrylate, 2,6 di-tert-butyl-P-cresol (BHT)	5

Abbreviations: Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

leaving the cement on the glass slide due to its flow. When there was a cement remnant inside the mold, a 0.5-mm-thick metal wire was used to remove and incorporate the remnant to the specimen. Then, the cement was pressed between the glass slide and a flexible cover glass (Marienfeld, Lauda-Koenigshofen, Germany) with the help of a 0.5-mm-diameter metal orthodontic wire, resulting in a specimen size of 0.5 mm in thickness and 6 mm in diameter (n=7). The glass slide was positioned on the metal shelf of a PS-measuring device (u-BioMechanics, IB Systems, Seoul, Korea), so that the specimen was located in the center of a blue light-emitting diode (LED) light (9 mm in diameter, 500 mW/cm^2 total irradiance) mounted in the middle of the metal shelf. The distance from the LED light to the specimen was 3 mm (glass slide of 1 mm + distance from the LED light source to the upper surface of 2 mm). Then, the linear variable differential transformer (LVDT) probe was set to touch the cover glass and adjusted to zero. On curing of the cement, the LVDT probe measured the real-time axial PS for 1600 seconds at a rate of 10 data points/s.[18] Baseline data were obtained for 20 seconds before tack cure or light cure to check the consistency of data collection before PS measurement. Also, 20 seconds could correspond to the time for a clinician to apply the cement to inlays. In the LC group, the specimen was light cured for 20 seconds. In the tack-cure groups, the specimen was tack cured for either one second, two seconds, or five seconds, respectively, according to the manufacturer's instructions. The cement was then light cured for 20 seconds in the TC-LC group after a two-minute delay, which simulated the time to remove the excess cement in the clinical situation. In the TC-SC group, the cement was left to self-cure after tack cure. In the SC group, the specimen was left to self-cure without tack cure or light cure.

Data acquired at 1600 seconds were analyzed with two-way analysis of variance (ANOVA; SPSS 23, SPSS, Chicago, IL, USA) to examine two factors, cure mode and cement, followed by post hoc comparisons with Tukey honestly significant difference (HSD) test. To investigate the effect of tack cure on light cure or self-cure, data were analyzed with an independent-samples *t*-test with tack cure as the variable. In addition, the PS data of each cement were analyzed with one-way ANOVA to examine either the cure mode factor or the time factor followed by Tukey HSD. The significance level was 5%.

RESULTS

Two-way ANOVA showed significant effects for cure mode factor, cement factor, and their interaction ($p<0.001$; Tables 2 and 3) on PS data at 1600 seconds. Independent-samples *t*-test showed that tack cure had a negative effect on self-cure in Duolink and Luting+ ($p<0.05$; Table 4). One-way ANOVA showed significant effects for cure mode factor and time factor on the PS data of each cement ($p<0.05$; Tables 5-8).

Table 2: *Summary of Two-Way ANOVA for Main Factors (Cement and Cure Mode) and Their Interactions for Polymerization Shrinkage of Resin-Based Luting Cements*

Source of Variation	Sum of Squares	*df*	Mean Square	*F*	Significance
Cement	23.328	3	7.776	402.147	<0.001
Cure mode	2.702	3	0.901	46.586	<0.001
Cement × cure mode	4.376	9	0.486	25.149	<0.001
Error	1.856	96	0.019		
Total	1961.349	112			

Regarding cure mode, PS of the LC or TC-LC group was higher than that of the SC group, which was higher than that of the TC-SC group ($p<0.05$). For Duolink, PS in the SC group was significantly lower than that in the other groups at 200 seconds. However, at 1600 seconds, PS was significantly lower in the TC-SC group, while there was no significant difference in PS among the LC, TC-LC, and SC groups (Figure 1a). Accounting for this, the PS rate in the SC group was higher between 200 seconds and 400 seconds than between 0 and 200 seconds only for Duolink (Figure 2a). For U200, PS was the highest in the LC group, followed by that in the TC-LC, TC-SC, and SC groups at 200 seconds ($p<0.05$). However, at 1000 seconds and thereafter, U200 showed a significantly higher PS in the LC or TC-LC groups compared with that in the SC or TC-SC groups, without significant difference between light-cure groups or between self-cure groups (Figure 1b). The PS or PS rate of GCem was not significantly affected by tack cure or cure mode (Figures 1c and 2c). For Luting+, PS was the highest in the TC-LC group, followed by that in the LC, TC-SC, and SC groups at 200 seconds ($p<0.05$). At 800 seconds and thereafter, however, PS was higher in the SC group than that in the TC-SC group ($p<0.05$). The SC, TC-LC, and LC groups showed no significant difference in PS (Figure 1d). Regarding cements, Luting+ showed the highest PS, followed by GCem, Duolink, and U200, respectively, with significant differences (Table 3).

DISCUSSION

The PS of resin-based luting cements is frequently identified as an important cause of the failure of indirect restorations.[19] Also, as a strong positive correlation has been observed between PS and DC,[20,21] PS is considered a feasible alternative to the conventional infrared method of measuring DC.[22] Therefore, the extent of PS in this study could be used to determine DC indirectly within the same material. In other words, high or low PS could be regarded as high or low DC within a resin-based luting cement.

As the independent-samples *t*-test showed that tack cure had a negative effect on self-cure in Duolink and Luting+, the null hypothesis was partially rejected. The short tack-cure procedure with a low radiant exposure increased the PS abruptly at the beginning of polymerization in all cements. It could be speculated that tack cure induced polymer growth centers, although such induction was relatively less than that induced by a final light cure.[12] Accordingly, the increased viscosity of cements entrapped activated free radicals and unreacted monomers as well as self-polymerization promoters needed for self-cure, which is known to usually have a lower polymerization rate than light cure.[2,23] Therefore, the PS of Duolink and Luting+ was lower in the TC-SC groups compared with that in the SC groups. However, GCem and U200 did not show any significant difference in PS between the TC-SC and SC groups.

Table 3: *Mean Polymerization Shrinkage (%± SD) of Resin-Based Luting Cements When Light Cured or Self-Cured With or Without Tack Cure at 1600 Seconds*[a]

	LC	TC-LC	TC-SC	SC	Pooled Average
Duolink	4.29 ± 0.11 Ab	4.23 ± 0.20 Ab	3.89 ± 0.05 Bc	4.14 ± 0.11 Ac	4.14 ± 0.20 c
U200	3.95 ± 0.07 Ac	3.89 ± 0.08 Ac	3.04 ± 0.10 Bd	2.96 ± 0.15 Bd	3.46 ± 0.48 d
GCem	4.28 ± 0.08 Ab	4.23 ± 0.15 Ab	4.19 ± 0.15 Ab	4.35 ± 0.13 Ab	4.26 ± 0.14 b
Luting+	4.72 ± 0.20 ABa	4.76 ± 0.14 ABa	4.57 ± 0.22 Ba	4.90 ± 0.15 Aa	4.74 ± 0.21 a
Pooled average	4.31 ± 0.30 A	4.28 ± 0.35 A	3.92 ± 0.59 C	4.09 ± 0.73 B	

Abbreviations: Duolink, Duo-Link; GCem, G-CEM LinkAce; LC, light cure; Luting+, RelyX Luting Plus; SC, self-cure; TC, tack cure; U200, RelyX U200.
[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD (p<0.05).*

Table 4: *Mean Polymerization Shrinkage (%± SD) of Resin-Based Luting Cements When Light Cured or Self-Cured at 1600 Seconds With Tack Cure as the Variable*[a]

Cement	Cure Mode	With Tack Cure	Without Tack Cure
Duolink	Light cure	4.23 ± 0.20 A	4.29 ± 0.11 A
	Self-cure	3.89 ± 0.05 B	4.14 ± 0.11 A
U200	Light cure	3.89 ± 0.08 A	3.95 ± 0.07 A
	Self-cure	3.04 ± 0.10 A	2.96 ± 0.15 A
GCem	Light cure	4.23 ± 0.15 A	4.28 ± 0.08 A
	Self-cure	4.19 ± 0.15 A	4.35 ± 0.13 A
Luting+	Light cure	4.76 ± 0.14 A	4.72 ± 0.20 A
	Self-cure	4.57 ± 0.22 B	4.90 ± 0.15 A

Abbreviations: Duolink, Duo-Link; GCem, G-CEM LinkAce; RelyX Luting Plus; U200, RelyX U200; Luting+.
[a] *Mean values followed by different uppercase letters (row) are significantly different by independent-samples* t*-test (*$p<0.05$*) within a cure mode.*

Differences in compositions of cements, including the initiator and catalyst system, might have caused the difference in the PS of these dual-cure cements.[10,17]

On the other hand, tack cure had no significant effect on light cure in all resin-based luting cements. This result was consistent with a previous study showing that light cure after tack cure did not negatively affect the microhardness of dual-cure cements.[17] In addition, a study on dual-cure cements reported that there was no significant difference in DC between the immediate light-cure group and two-minute delayed light-cure group, which had the same delay time as in the current study, except for the tack-cure procedure.[7] Therefore, it could be speculated that radiant exposure of final light cure in the TC-LC group might have induced enough growth centers to result in a PS similar to the LC group[12] despite the tack cure procedure and a two-minute delay time.

Duolink, a conventional composite resin cement, showed higher PS in the TC-SC group than in the SC group at the beginning of polymerization because a short light-cure reaction due to tack cure was added to the self-cure reaction. However, the PS rate in the SC mode increased thereafter, resulting in higher PS in the SC group than in the TC-SC group. Thus, it could be speculated that tack cure induced premature polymer growth centers before the self-cure PS rate was dominant and resulted in the interference of the self-cure reaction. In addition, the time to the plateau of PS decreased in the TC-SC group. This could also be a sign that the tack cure interfered with the self-cure reaction, resulting in premature termination of polymerization. For the light-cure groups, Figure 1a shows that the PS curves of LC and TC-LC groups were lined up exactly if the PS curve of the LC group was shifted 122 seconds (two seconds for tack cure and two minutes for delay time) on the *x*-axis. PS after 20-second light cure was 3.32% ± 0.12% at 40 seconds in the LC group and 3.35% ± 0.14% at 162 seconds in the TC-LC group. The final comparable PS values for both groups were 4.28% ± 0.11% at 1478 seconds in the LC group and 4.23% ± 0.20% at 1600 seconds in the TC-LC group. Data at those time points showed no significant difference between the LC and TC-LC groups ($p>0.05$, independent-samples *t*-test). Therefore, the tack cure and the self-cure reaction during the two-minute delay time seemed to have no significant effect on the light-cure reaction. Similar to this study, Pereira and others[9] reported that there was no significant difference in the extent of polymerization among light-cure, self-cure, and five-minute delayed light-cure modes with Duolink. The extent of polymerization in the five-minute delayed light-cure mode could be explained with the result of this study. As the PS rate of the SC group was the highest between 200 seconds and 400 seconds, the five-min delayed light cure might not have interfered with the self-cure reaction because the cement was light cured when the self-cure PS rate was high.

U200, an SARC, showed significantly higher PS in the light-cure groups than in the self-cure groups,[2,6,24] regardless of tack cure. In addition, the PS of U200 was the lowest among the dual-cure cements tested. Previous studies reported that water produced during neutralization of acidic monomers in SARCs could interfere with the polymerization reaction.[25] In addition, free radicals formed by acidic monomers are less reactive than free radicals formed by unmodified dimethacrylate monomers. This might be a reason for the lower PS of this cement especially in the self-cure mode when the PS rate is low.[2,24,26] Regarding the self-cure groups, although tack cure increased PS initially, the difference in PS between the TC-SC and SC groups was not significant after 1000 seconds. As the self-cure PS rate was the highest when the cement was tack cured, namely, between 0 and 200 seconds, it seemed that tack cure did not affect the self-cure reaction (Figure 2b). In addition, tack cure extended the time to reach the plateau of PS from 1200 seconds in the SC group to 1400 seconds in TC-SC group. Since the self-cure reaction had low PS and PS rate, it could be assumed that the light-cure reaction by the tack-cure procedure was added to the self-cure reaction to extend the time for polymerization. However, radiant exposure of the tack-cure procedure was not high

Table 5: *Polymerization Shrinkage (%±SD) of Duolink for 1600 Seconds at 200-Seconds Intervals*[a]

Group	Time, s					
	200	400	600	800	1000	1200
LC	3.89 ± 0.12 Ca	4.04 ± 0.12 BCa	4.13 ± 0.11 ABa	4.19 ± 0.11 ABa	4.23 ± 0.12 ABa	4.26 ± 0.12 Aa
TC-LC	3.63 ± 0.16 Ca	3.91 ± 0.16 BCa	4.01 ± 0.17 ABa	4.08 ± 0.17 ABa	4.13 ± 0.18 ABa	4.17 ± 0.19 ABab
TC-SC	2.41 ± 0.36 Eb	2.98 ± 0.25 Db	3.34 ± 0.16 Cb	3.54 ± 0.11 BCc	3.68 ± 0.08 ABc	3.77 ± 0.07 ABc
SC	0.97 ± 0.16 Fc	2.89 ± 0.11 Eb	3.51 ± 0.10 Db	3.78 ± 0.10 Cb	3.92 ± 0.10 BCb	4.02 ± 0.10 ABb

Abbreviations: Duolink, Duo-Link; LC, light cure; SC, self-cure; TC, tack cure.
[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD ($p<0.05$).*

Table 6: *Polymerization Shrinkage (%±SD) of U200 for 1600 Seconds at 200-Second Intervals*[a]

Group	Time, s					
	200	400	600	800	1000	1200
LC	3.48 ± 0.07 Fa	3.66 ± 0.07 Da	3.75 ± 0.06 CDa	3.81 ± 0.07 BCa	3.86 ± 0.07 ABCa	3.90 ± 0.07 ABa
TC-LC	3.17 ± 0.08 Db	3.55 ± 0.07 Ca	3.69 ± 0.08 Ba	3.77 ± 0.07 ABa	3.82 ± 0.08 Aa	3.85 ± 0.08 Aa
TC-SC	1.55 ± 0.07 Gc	2.03 ± 0.08 Fb	2.33 ± 0.09 Eb	2.55 ± 0.09 Db	2.71 ± 0.10 Cb	2.85 ± 0.10 BCb
SC	0.90 ± 0.18 Fd	1.67 ± 0.16 Ec	2.10 ± 0.15 Dc	2.38 ± 0.15 Cc	2.58 ± 0.15 BCb	2.74 ± 0.15 ABb

Abbreviations: LC, light cure; SC, self-cure; TC, tack cure; U200, RelyX U200.
[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD ($p<0.05$).*

Table 7: *Polymerization Shrinkage (%±SD) of GCem for 1600 Seconds at 200-Second Intervals*[a]

Group	Time, s					
	200	400	600	800	1000	1200
LC	3.28 ± 0.04 Fa	3.64 ± 0.05 Ea	3.84 ± 0.07 Da	3.98 ± 0.08 Ca	4.09 ± 0.08 BCa	4.17 ± 0.08 ABa
TC-LC	3.26 ± 0.13 Ea	3.66 ± 0.14 Da	3.85 ± 0.15 CDa	3.98 ± 0.15 BCa	4.06 ± 0.15 ABCa	4.13 ± 0.15 ABa
TC-SC	2.96 ± 0.15 Eb	3.47 ± 0.15 Da	3.73 ± 0.15 Ca	3.89 ± 0.14 BCa	4.01 ± 0.14 ABa	4.08 ± 0.15 ABa
SC	2.93 ± 0.20 Eb	3.55 ± 0.16 Da	3.83 ± 0.14 Ca	4.01 ± 0.14 BCa	4.13 ± 0.13 ABa	4.22 ± 0.13 ABa

Abbreviations: GCem, G-CEM LinkAce; LC, light cure; SC, self-cure; TC, tack cure.
[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD ($p<0.05$).*

Table 8: *Polymerization Shrinkage (%±SD) of Luting+ for 1600 Seconds at 200-Second Intervals*[a]

Group	Time, s					
	200	400	600	800	1000	1200
LC	3.89 ± 0.19 Cb	4.36 ± 0.18 Ba	4.52 ± 0.20 ABab	4.60 ± 0.21 ABab	4.64 ± 0.20 ABab	4.66 ± 0.20 ABab
TC-LC	4.36 ± 0.13 Ba	4.57 ± 0.14 ABa	4.64 ± 0.15 Aa	4.68 ± 0.15 Aab	4.71 ± 0.15 Aab	4.73 ± 0.15 Aab
TC-SC	3.03 ± 0.09 Cc	4.02 ± 0.16 Bb	4.30 ± 0.18 ABb	4.43 ± 0.20 Ab	4.49 ± 0.20 Ab	4.52 ± 0.21 Ab
SC	2.52 ± 0.30 Dd	4.07 ± 0.15 Cb	4.52 ± 0.12 Bab	4.69 ± 0.13 ABa	4.78 ± 0.14 ABa	4.83 ± 0.14 Aa

Abbreviations: LC, light cure; Luting+, RelyX Luting Plus; SC, self-cure; TC, tack cure.
[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD ($p<0.05$).*

enough to affect the final PS at 1600 seconds.[27] Regarding the light-cure groups, the relationship between the LC and TC-LC groups seemed to be just like that of Duolink: exactly the same PS curves with a 121-second (one second for tack cure and two minutes for delay time) time difference (Figure 1b). After 20 seconds of light cure, the PS was 2.66% ± 0.10% in the LC group at 40 seconds and 2.74% ± 0.08% in the TC-LC group at 161 seconds. The final comparable PS values for both groups were 3.94% ±

Table 5: *Extended.*

Group	Time, s	
	1400	1600
LC	4.28 ± 0.11 Aa	4.29 ± 0.11 Aa
TC-LC	4.20 ± 0.20 ABab	4.23 ± 0.20 Aa
TC-SC	3.83 ± 0.05 ABc	3.89 ± 0.05 Ab
SC	4.08 ± 0.11 ABb	4.14 ± 0.11 Aa

Table 6: *Extended.*

Group	Time, s	
	1400	1600
LC	3.93 ± 0.07 Aa	3.95 ± 0.07 Aa
TC-LC	3.87 ± 0.08 Aa	3.89 ± 0.08 Aa
TC-SC	2.95 ± 0.11 ABb	3.04 ± 0.10 Ab
SC	2.86 ± 0.15 Ab	2.96 ± 0.15 Ab

Table 7: *Extended.*

Group	Time, s	
	1400	1600
LC	4.23 ± 0.08 Aa	4.28 ± 0.08 Aa
TC-LC	4.18 ± 0.15 ABa	4.23 ± 0.15 Aa
TC-SC	4.14 ± 0.15 Aa	4.19 ± 0.15 Aa
SC	4.30 ± 0.12 Aa	4.35 ± 0.13 Aa

Table 8: *Extended.*

Group	Time, s	
	1400	1600
LC	4.70 ± 0.20 Aab	4.72 ± 0.20 Aab
TC-LC	4.75 ± 0.15 Aab	4.76 ± 0.14 Aab
TC-SC	4.55 ± 0.22 Ab	4.57 ± 0.22 Ab
SC	4.87 ± 0.15 Aa	4.90 ± 0.15 Aa

0.07% in the LC group at 1479 seconds and 3.89% ± 0.08% in the TC-LC group at 1600 seconds. At those time points, data showed no significant difference in PS between the LC and TC-LC groups ($p>0.05$, independent-samples t-test). Thus, it could be stated that the tack cure and the self-cure reaction during the two-minute delay time had no significant effect on the final light-cure reaction of this cement. The time to reach the plateau of PS was reduced to 800 seconds in the TC-LC group from 1000 seconds in the LC group. The increase in total light-cure time due to additional light exposure of the tack-cure procedure might be one reason for such a result.[3]

GCem, another SARC, showed no significant difference in PS regardless of tack cure or cure mode. Figures 1c and 2c showed that PS and PS rate in the SC group were as high as those in the LC group, unlike U200. As the self-cure PS rate was the highest when the cement was tack cured, namely, between 0 and 200 seconds, it seemed that tack cure did not affect the self-cure process. Therefore, it could be assumed that the self-cure reaction was not interfered by tack cure because of the high initial PS rate of the SC group. The relationship between the LC and TC-LC groups was different from that of Duolink or U200. High self-cure PS rate at the time of tack cure and during the two-minute delay time seemed to be one reason for the difference. Nevertheless, there was no significant difference in PS between the LC and TC-LC groups. The time to reach the plateau of PS was decreased in the TC-LC group compared with the LC group, probably because of the additional light exposure of the tack-cure procedure. One study reported that the effect of light cure was compensated by self-cure within five minutes, showing the high self-cure capacity of this cement.[28] The manufacturer has claimed that they incorporated a metal ion accelerator to increase the free radical in the self-cure reaction and called it the "high-efficiency triple initiator self-cure technology."[29]

Duolink showed a PS difference between the TC-SC and SC groups, while GCem or U200 showed no significant PS difference between these groups. The PS rates of Duolink between 0 and 200 seconds and between 200 seconds and 400 seconds in the TC-SC group were 2.41% and 0.57%, respectively, compared with PS rates in the SC group, which were 0.97% and 1.92%, respectively. Thus, tack cure increased the PS rate between 0 and 200 seconds but decreased the PS rate between 200 and 400 seconds. This reduction in self-cure PS rate due to tack cure contributed to lower PS in the TC-SC group. On the other hand, PS rates of U200 between 0 and 200 seconds and between 200 and 400 seconds in the TC-SC group were 1.55% and 0.48%, respectively, compared with PS rates in the SC group, which were 0.90% and 0.77%, respectively. Although tack cure increased the PS rate between 0 and 200 seconds and decreased the PS rate between 200 and 400 seconds, the PS rate at those time periods

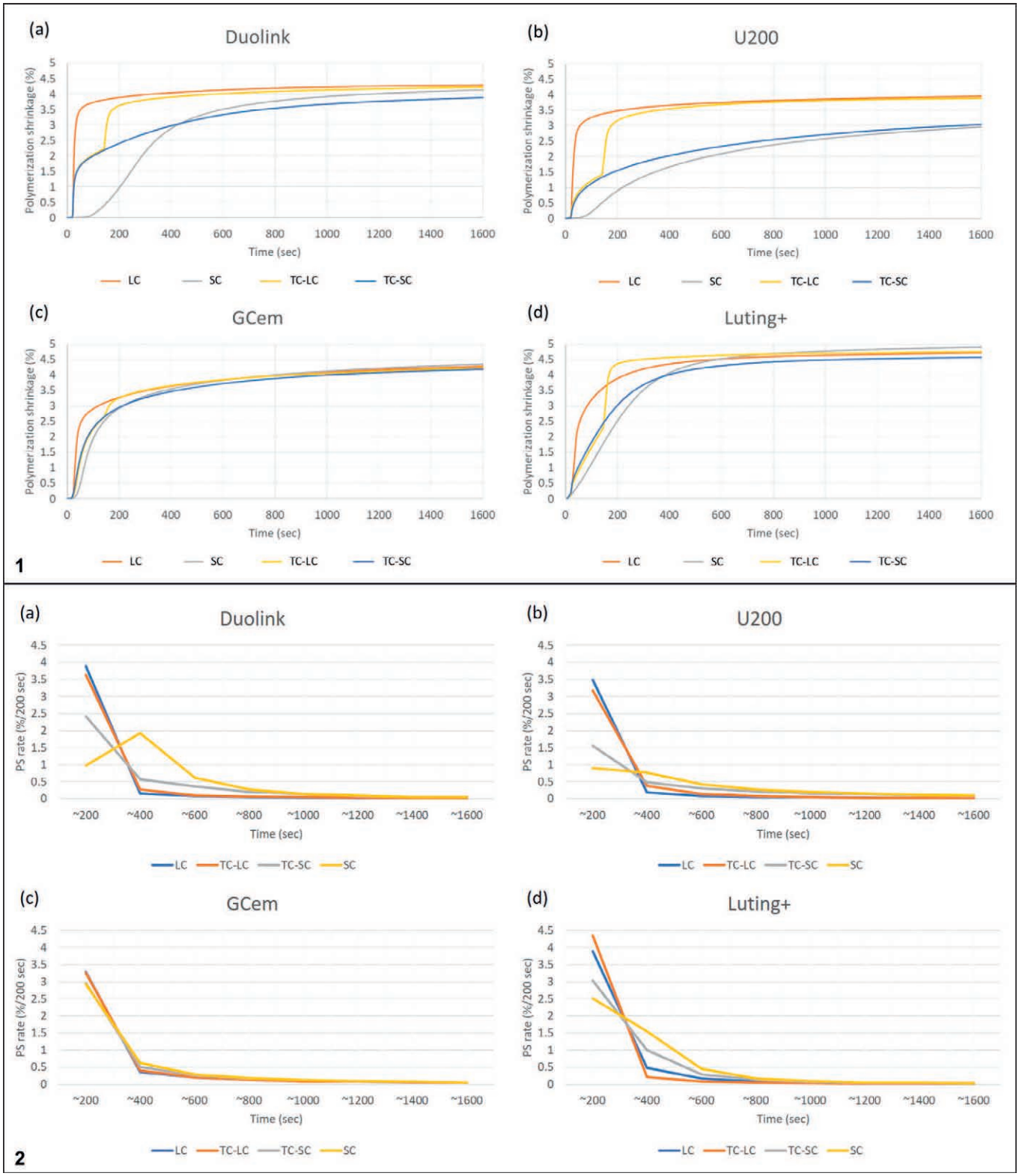

Figure 1. *Polymerization shrinkage of resin-based luting cements according to cure mode. (a): Duo-Link. (b): RelyX U200. (c): G-CEM LinkAce. (d): RelyX Luting Plus. LC, light cure; SC, self-cure; TC, tack cure.*

Figure 2. *Polymerization shrinkage rate (%/200 seconds) of resin-based luting cements. (a): Duo-Link. (b): RelyX U200. (c): G-CEM LinkAce. (d): RelyX Luting Plus. LC, light cure; SC, self-cure; TC, tack cure.*

was lower than those of Duolink, resulting in no significant difference between those groups and lower final PS for both self-cure groups. The PS rates of GCem between 0 and 200 seconds and between 200 and 400 seconds in the TC-SC group were 2.96% and 0.51%, respectively, similar to PS rates in the SC group, which were 2.93% and 0.62%, respectively. Therefore, it seemed that tack cure did not affect the self-cure reaction, as both cure modes exhibited a high PS rate and high final PS. Thus, the extent of decreased self-cure PS rate due to tack cure seemed to be one reason for the decreased PS of the TC-SC groups for the composite resin cements. In addition, if the dual-cure cements were tack cured when the self-cure PS rate was high, as in case of GCem and U200, the tack cure did not interfere with the self-cure reaction. However, if the dual-cure cements were tack cured before the self-cure PS rate reached its maximum, like in case of Duolink, the premature polymerization due to tack cure seemed to interfere with the self-cure reaction. Further study is needed to verify the relation between the timing of tack cure and the self-cure PS rate in detail.

Luting+, an RMGIC, showed higher PS than composite resin cements, similar to other studies.[30,31] The main setting of an RMGIC is achieved by an acid-base reaction, which has been reported to have higher setting shrinkage than composite resin.[31] In addition, HEMA contained in the RMGIC is a hydrophilic resin that also exhibits high PS.[32] Therefore, PS should be referred to as setting shrinkage in RMGIC. However, the term *polymerization shrinkage* was used throughout the text as in previous studies.[31,33] This is probably because PS is part of the setting shrinkage of RMGIC, making it difficult to distinguish between PS and shrinkage from an acid-base reaction. The highest PS was observed in the SC group, and there was a significant difference in PS between the SC and TC-SC groups. However, there was no significant difference in PS among the LC, TC-LC, and SC groups, although the manufacturer has claimed that this cement is basically a self-cure material with a five-second tack-cure option. In the self-cure groups, the PS rates of Luting+ between 0 and 200 seconds and between 200 and 400 seconds in the TC-SC group were 3.03% and 0.99%, respectively, compared with the PS rates in the SC group, which were 2.52% and 1.55%, respectively. Thus, tack cure seemed to interfere with the self-cure reaction of resin components as well as the acid-base reaction of glass ionomer components between 200 and 400 seconds, since the light-cure reaction and acid-base reaction could compete with each other and inhibit each other during the early RMGIC setting.[34] To support this, the acid-base reaction of an RMGIC in the SC mode was reported to show an exotherm peak at 4.2 minutes after mixing,[34] which is in the range of 200 seconds and 400 seconds. The time to reach the plateau of PS was reduced in the TC-SC group compared with that in the SC group, resulting in premature termination of polymerization. For the light-cure groups, tack cure did not affect the final PS, although the PS curves were different from those of resin cements. At 200 seconds, the PS in the TC-LC group was significantly higher than that in the LC group only for Luting+ (Figure 1d). One reason could be that the total light-cure time in the TC-LC group was 25 seconds, which was the longest light-cure time among the test groups. The longer tack-cure time might have resulted in a more crosslinked polymer network with higher exothermic heat, which accelerated the acid-base reaction and self-cure reaction to even earlier times. Nevertheless, it seemed that the light-cure time of 20 seconds in the LC group was enough to light cure the RMGIC, as the final PS showed no significant difference between the LC and TC-LC groups. As PS was not significantly different among the LC, TC-LC, and SC groups, Luting+ could be referred to as a dual-cure cement in terms of PS.

CONCLUSION

For the tested cements, tack cure decreased the PS of Duolink and Luting+ when they were self-cured after tack cure. However, light cure after tack cure showed no significant difference in PS compared with light cure alone. Duolink showed lower PS in the TC-SC group compared with the other cure groups. Luting+ showed lower PS in the TC-SC group than in the SC group. On the other hand, U200 showed lower PS in the self-cure groups compared with the light-cure groups regardless of tack cure. GCem was not affected by tack cure or cure mode.

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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 Bulk-fill Restoration on Polymerization Shrinkage Stress and Marginal Gap Formation in Class V Restorations

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Clinical Relevance

Restoring Class V cavities with a regular bulk-fill composite presents a more favorable biomechanical behavior than restoring with a regular nano-filled composite.

SUMMARY

Purpose: This study evaluated the influence of Class V cavity extension and restorative material on the marginal gap formation, before and after aging, and the theoretical polymerization shrinkage stress distribution in a tooth restoration.

Methods and Materials: Class V cavities with the depth of 2 mm, cervical/incisal distance of 4 mm, and margins located in the enamel 1 mm above the cementoenamel junction were prepared in 60 bovine incisors in two mesiodistal dimensions (n=30): 2.9-mm large extension cavities (LE) or 1.4-mm small extension cavities (SE). The cavities' depths were validated using a periodontal probe, while the mesiodistal and cervical/incisal distances were measured using a stereomicroscope. After adhesive application (Clearfil SE Bond), each group was randomly divided into two groups (n=15) according to the restorative material: Filtek Z350 XT (N) or Filtek Bulk Fill Posterior (BF). The marginal gap formation between the tooth structure and the restorative material was evaluated using a stereomicroscope before and after thermocycling for 15,000 cycles (5°C

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and 55°C). Data were analyzed using repeated-measures analysis of variance (ANOVA) and Tukey test for multiple comparisons (α=0.05). A three-dimensional geometric model with the same dimensions as the experimental test was created for each cavity, and the restorations were modeled for each restorative material. In the analysis software, the finite element mesh was created with tetrahedral quadratic elements, and the polymerization shrinkage was simulated by thermal analogy. The maximum principal stress was used to express the tensile stress in the adhesive interface through colorimetric graphs.

Results: For the marginal gap, the repeated-measures ANOVA revealed a significant effect only for the factors composite resin (df=1, F=4.09, p=0.04) and thermal aging (df=1, F=44.35, p<0.001). For all numerical simulations, higher stress concentration occurred at the enamel margin, and the stress peak decreased in the following sequence: LE-N (17.0 MPa) > SE-N (15.0 MPa) > LE-BF (9.1 MPa) > SE-BF (8.2 MPa).

Conclusion: Marginal gaps in the specimens fell between approximately 12 and 17 μm; however, the regular bulk-fill composite showed less gap formation and better stress distribution around the cavity margin than the regular nano-filled composite, regardless of the cavity extension.

INTRODUCTION

The complete loss of the restoration is one of the most common causes of failures in noncarious cervical lesion (NCCL) resin restorations, along with marginal gap and marginal staining.[1-4] This loss can be associated with the polymerization shrinkage of the composite resin, still present in low-shrinkage restorative materials, which is responsible for the development of stress at the adhesive interface.[5-7] In addition, the marginal integrity of resin restorations depends on other factors, such as cavity configuration (size, geometry, C-factor, and substrate compliance),[5,8-12] composite resin properties (composition, filler content, resin flow, and elastic modulus [E]),[7,13-15] adhesive systems,[2,16,17] and aspects related to the restorative technique (placement and photopolymerization of the composite).[7,9,18-20]

The size and geometry of a Class V cavity can be one of the key factors,[8,10,12] since for a deeper and larger restoration, higher shrinkage stress and microleakage could be observed,[8] and some geometries may favor the stress concentration at the restoration interface more than others.[10,12,21] In this situation, the incremental filling techniques (horizontal and oblique) have been used to minimize the adverse effects of shrinkage stress and thereby reduce marginal gap formation.[11,19,22] These procedures can still improve the degree of material conversion[23] because they allow the placement of the composite in increments of 2 mm or less. However, the chair time increases and there is the possibility of incorporating voids between the resin layers.[24]

For these reasons, changes in placement techniques and continual improvements in restorative materials aimed at improving the clinical longevity of resin restorations have been made.[25,26] Recently developed bulk-fill composites exhibit reduced volumetric shrinkage and lower polymerization shrinkage stress, which may be attributed to the incorporation of polymerization modulators and monomers able to relieve stress.[27-30] The use of single increments up to 6-mm thick, depending on the manufacturer, is attributed to increased translucency and inclusion of new photoinitiators, for example, acyl phosphine oxide; Ivocerin (bis[4 methoxybenzoyl]-diethylgermane), and phenyl propanedione. These allow for an increased cure depth because of their significantly more intense absorption of visible light irradiation as compared with conventional photoinitiator systems.[31-33] In addition, the use of this approach prevents voids throughout the composite, simplifies material placement, and reduces clinical time.[29,34] It should also be noted that, to improve the performance of the restoration, advances were made in relation to ultrasonic energy application during the insertion of these materials and including reinforcing fibers.[35-37]

Recent studies reported positive results for these materials in NCCLs.[20,38] One 1-year clinical evaluation showed acceptable clinical performances for the restorations of NCCLs with bulk-fill flowable composites compared with regular nano-filled composites. In a finite element analysis (FEA), the model restored with a bulk-fill composite showed reduced development of interfacial stresses. These investigations have not focused on how the cavity extension influences the stress distribution and marginal gap formation, which may differ depending on the volume and compliance of the cavity walls,[39] justifying further studies.

Table 1: *Technical Information About the Materials Used in the Study*

Material	Manufacturer	Composition
Clearfil SE Bond	Kuraray America Inc, New York, NY, USA	Primer: 10-MDP, HEMA, DMA, catalyst, water
		Bond: 10-MDP, HEMA, DMA, Bis-GMA, filler, catalyst
Filtek Z350 XT	3M, St Paul, MN, USA	Filler: 78.5 wt% (59.5 vol%) silica, zirconia, aggregated zirconia/silica
		Matrix: Bis-GMA, UDMA, TEGDMA, dimethacrylate
Filtek Bulk Fill Posterior	3M, St Paul, MN, USA	Filler: 76.5 wt% (58.4 vol%) silica, zirconia, ytterbium trifluoride, aggregated zirconia/silica
		Matrix: AUDMA, AFM, UDMA, DDDMA, EDMAB

Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; AFM, addition-fragmentation monomer; AUDMA, aromatic urethane dimethacrylate; Bis-GMA, bisphenol-glycidyl methacrylate; DDDMA, 1, 12-dodecanediol dimethacrylate; DMA, dimethacrylate; EDMAB, ethyl 4-dimethyl aminobenzoate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

The purpose of this study was to evaluate the influence of cavity extension and restorative material on 1) the marginal gap formation, before and after aging, and 2) the stress distribution by FEA in a tooth/restoration. FEA was used to ratify the results obtained experimentally for the marginal gap of resin composite restorations placed in simulated NCCLs.

METHODS AND MATERIALS

Tooth Preparation

Sixty undamaged and fresh bovine incisors were collected and stored in 0.1% thymol solution under refrigeration at 4°C until they were used in this laboratory study. The incisal portion was removed to 10 mm above the cementoenamel junction (CEJ), and the root portion was removed to 4 mm below the CEJ, both using a diamond disc coupled to a low-speed hand piece. The teeth were positioned with the enamel surface facing down and embedded in acrylic resin (Jet-Clássico, São Paulo, SP, Brazil). The enamel surface of each tooth was flattened by grinding using 600-grit silicon carbide paper (Extec Corp, Enfield, CT, USA) in a polishing device (Pantec Polipan 2, São Paulo, SP, Brazil) under water irrigation for 30 seconds. This procedure created a flat enamel surface for cavity preparation.

Cavity Preparation

The teeth were randomly divided into two groups (n=30): the large extension cavity group (LE) and the small extension cavity group (SE). In each tooth, a Class V cavity was prepared under abundant air-water coolant with a spherical diamond bur corresponding to the cervical/incisal distance (No. 3018 for the LE and No. 1014 for the SE; KG Sorensen, Barueri, SP, Brazil) and adapted to a cutting machine (spindle speed 2000 rpm, frequency 50-60 Hz) to produce standardized cavity preparation dimensions. The cavity margins were located in the enamel, 1 mm above the CEJ. New burs were used after every five cavity preparations. The depth and the mesiodistal distance of the preparations were controlled to keep 2 mm and 4 mm, respectively, independent of the cavity configurations. For LE cavities, the cervical/incisal dimension was 2.9 mm, and for SE cavities, it was 1.4 mm. The cavities' depths were validated using a periodontal probe, while the mesiodistal and cervical/incisal distances were measured using a stereomicroscope (Stemi-2000C, Zeiss, Gottingen, Germany).

Restorative Procedures

All prepared cavities were bonded using the same two-step self-etch adhesive (Clearfil SE Bond, Kuraray America, New York, NY, USA; Table 1), according to the manufacturer's instructions. One coat of primer was gently scrubbed on the entire lesion surface for 20 seconds, followed by a gentle air stream to evaporate the solvent. Then, the bonding agent was applied and light cured for 10 seconds at 1200 mW/cm^2 with a polywave light-emitting diode (LED) device (Bluephase N, Ivoclar Vivadent, São Paulo, SP, Brazil). The irradiance was assessed with a radiometer (L.E.D, Demetron, Kerr Corp, Orange, CA, USA).

Composite filling materials included one nano-filled composite (Filtek Z350 XT, 3M, St Paul, MN, USA) and one bulk-fill composite (Filtek Bulk Fill Posterior, 3M; Table 1). The LE group was divided into two subgroups (n=15): group LE-N, which was filled with Filtek Z350 XT, and group LE-BF, which was filled with Filtek Bulk Fill Posterior. The SE group was also divided into two subgroups using the same composites (n=15): group SE-N and group SE-BF. The experimental design of this study is shown in Figure 1.

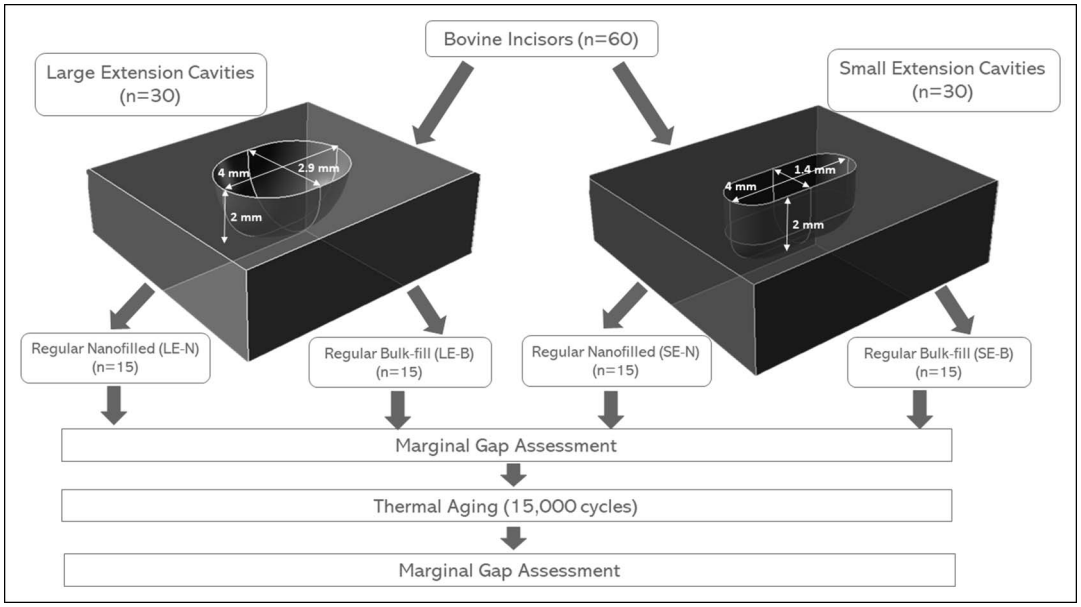

Figure 1. *Experimental design of the study.*

To ensure a smooth surface, the cavities were filled either in bulk placement (LE-BF, SE-N, and SE-BF) or with two oblique increments of 2 mm (LE-N) and covered with a transparent polyester strip until the end of the polymerization. For all groups, light-curing was performed for 40 seconds with the Bluephase N (Ivoclar Vivadent). The light tip was placed perpendicularly over the polyester strip at a 0-mm distance. The specimens were stored in ultrapure water at 37°C for at least 48 hours to ensure complete material polymerization.

The specimens' surfaces were ground flat and polished using a polishing device (Pantec Polipan 2, São Paulo, SP, Brazil) with 1200-, 2400-, and 4000-grit silicon carbide paper (Extec Corp) under continuous water cooling. All specimens were ultrasonically cleaned in ultrapure water (Type II, Adamo, Piracicaba, São Paulo, SP, Brazil) for 10 minutes. After cleaning, they were immersed in ultrapure water at 37°C until their next use.

Marginal Gap Assessment

The marginal gap between the tooth structure and the restorative material was assessed along the margin of the restoration at a magnification of 50× using a stereomicroscope (Discovery V20, Zeiss) with a bilateral LED light and corresponding digital camera. Immediately before the marginal gap assessment, each specimen was dried with absorbent paper. In each specimen, digital images of the three largest gaps were taken (16-bit resolution). AxioVision Software (Zeiss) was used to measure the width of each gap. A mean of these three values was calculated, and then an average marginal gap formation was obtained for each group (yielding a total of 15 data points per group). During the marginal gap assessment, the operator was blinded to the type of the resin used on each specimen.

Thermal Aging

All specimens were submitted to thermocycling for 15,000 cycles in water baths between temperatures of 5°C and 55°C, with the dwell time set at 15 seconds (Thermocycler Machine, Erios, Sao Paulo, SP, Brazil). The marginal gap assessment was then performed. One operator prepared all of the specimens and conducted all of the laboratory procedures (MRA).

Residual Stress Calculation: FEA

A three-dimensional geometric model of a bovine tooth square section was modeled with 0.5 mm enamel (elastic modulus [E]=50 GPa and Poisson ratio [ν]=0.30)[40] and 9.5 mm dentin (E=14.7 GPa and ν=0.31)[40] using computer-aided design Rhinoceros software (version 4.0SR8; McNeel North America, Seattle, WA, USA). Two models were replicated following the same dimensions of the experimental test, resulting in two different cavity geometries. The restorations were modeled in the tooth bonding surfaces. Then, each model was duplicated, totaling four models, in which two received restorations with

Table 2: *Mean (SD) Marginal Gap Measurement (μm) Before and After Thermal Aging*

Restorative Material	Before		After	
	Large Extension Cavity	Small Extension Cavity	Large Extension Cavity	Small Extension Cavity
Nanofilled composite	13.01 (4.16)	14.47 (4.44)	14.65 (4.11)	15.42 (4.62)
Bulk-fill composite	10.33 (2.82)	10.44 (2.36)	12.07 (2.38)	11.47 (2.74)

a nano-filled composite (Filtek Z350 XT, 3M; E=13.45 GPz, v=0.17, and linear shrinkage=0.00033),[20] and the other two received restorations with a bulk-fill composite (Filtek Bulk Fill, 3M; E=13.46 GPa, v=0.18, and linear shrinkage=0.00025).[20]

The models were imported in STEP (Standard for the Exchange of Product Model Data) format into the analysis software (ANSYS 17.2, ANSYS Inc, Houston, TX, USA). The mesh was created with tetrahedral quadratic elements. The mechanical properties to characterize each structure/material were based on the literature. All materials were considered homogenous, elastic, and isotropic. In all models, the tooth-restoration interfaces were considered ideal. Polymerization shrinkage was simulated by thermal analogy.[10] The temperature was reduced by 1°C, and the linear shrinkage value (postgel shrinkage) was entered as the coefficient of linear thermal expansion. For the models restored with two increments, the stress was generated in two steps (one for each portion of the composite).

The maximum principal stress was used to express the tensile stress in the adhesive interface through colorimetric graphs. For each model, the stress peak was recorded in MPa for a qualitative comparison between the groups.

Statistical Analysis

The Kolmogorov-Smirnov test checked the normality of the data. Repeated-measures analysis of variance (ANOVA) was used to compare the marginal gap results among the four groups, followed by the Tukey test for multiple comparisons (α=0.05). The null hypotheses tested were 1) there is no significant difference in marginal gap formation among the restorations for the LE and SE cavity groups and 2) there is no significant difference in marginal gap formation between the bulk-fill and nano-filled composites, before and after aging.

RESULTS

Marginal Gap

Means and standard deviations of marginal gap measurements are presented in Table 2. The repeated-measures ANOVA revealed a significant effect only for the composite resin (df=1, F=4.09, p=0.04) and thermal aging (df=1, F=44.35, $p<0.001$) factors. There was no significant effect for the cavity extension factor (df=1, F=0.63, p=0.42) or for the interactions among the factors. Tukey test showed a lower marginal gap for the bulk-fill composite (Filtek Bulk Fill Posterior) compared with the nano-filled composite (Filtek Z350 XT). For all groups, gap formation increased significantly after thermocycling (Figure 2).

Finite Element Analysis

The polymerization shrinkage stress results for all groups are shown as colorimetric graphs in Figure 3. Different colorimetric fringes represent different stress magnitudes, visible in the horizontal scale of Figure 3. Hot colors mean higher stress values, while cold colors indicate lower stress values. Positive values indicate areas that were subjected to tensile stress. In general, the composite resin factor affected the stress distribution in the tooth, while the restoration with regular nano-filled composite resulted in a higher stress concentration. For all models, a higher stress concentration occurred at the enamel margin around the cavity, with a lower magnitude in the dentin surface inside the cavity. Regardless of the restorative material, the smaller cavities presented a lower stress concentration. The models' stress peak decreased in the following sequence: LE-N (17.0 MPa) > SE-N (15.0 MPa) > LE-BF (9.1 MPa) > SE-BF (8.2 MPa).

DISCUSSION

The resin restoration of NCCLs often fails because of marginal leakage and restoration loss.[1-4] This phenomenon is the result of a localized bond failure between the restorative material and the tooth substrate,[41] and it depends on the complex interactions among multiple factors.[5,8-10,13,14,18,19] For this reason, laboratory studies with a clinically relevant aspect are still conducted. Such models allow isolating the variable of interest, specimen standardization, lower cost and sample size, and time savings, and they are an alternative to testing an unviable laboratory setup *in vivo*.[6,19,34] Thus, the polymeriza-

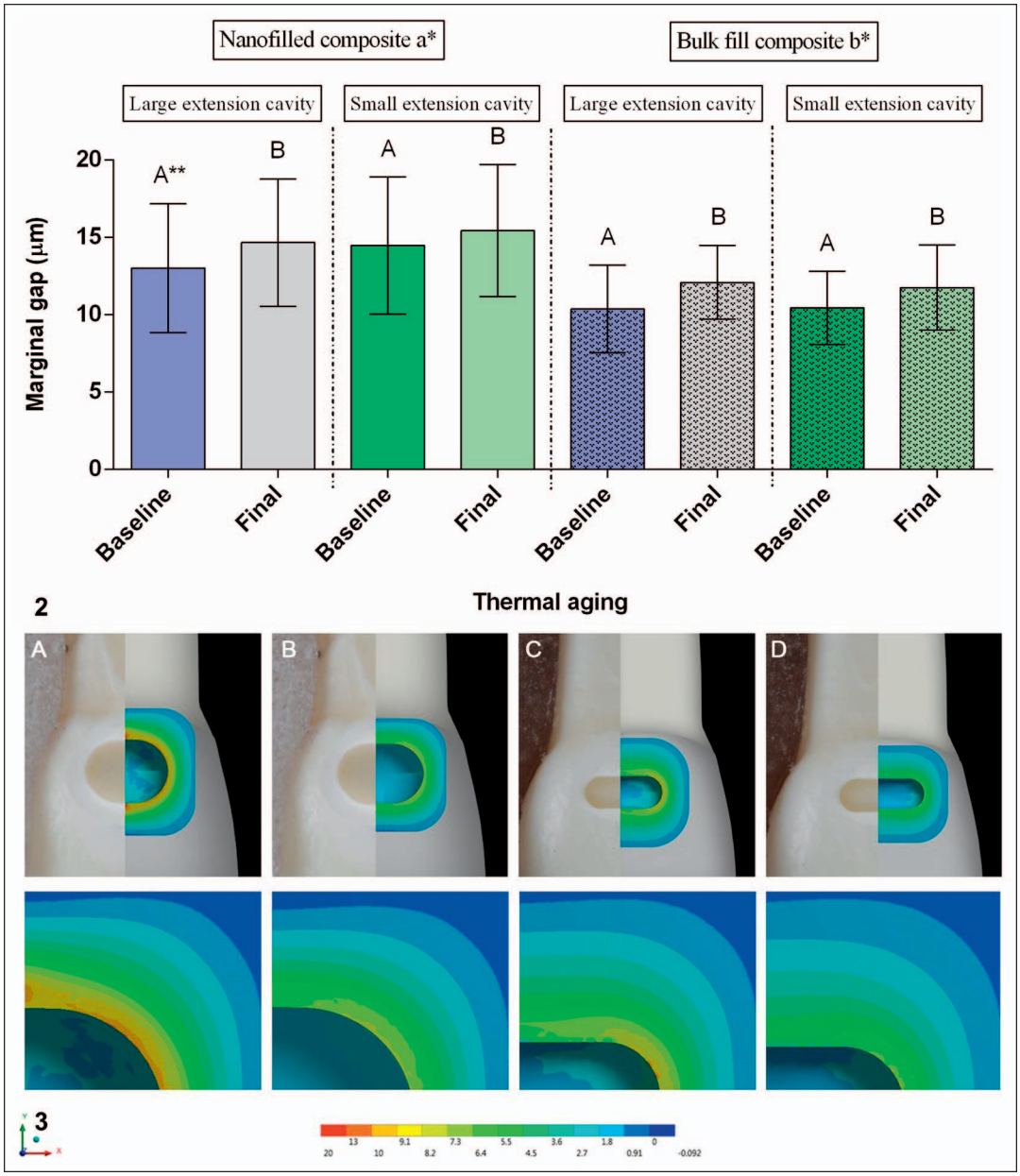

Figure 2. *Mean marginal gap measurement, with respective standard deviations, by group for each period investigated. *Different letters mean significant differences between the resin composites. **Different letters indicate significant differences before and after thermal aging (*$p<0.05$*). There was no significant difference between large and small extension cavities (*$p=0.42$*).*

Figure 3. *Polymerization shrinkage stress at the virtual tooth preparation according to the* in vitro *experimental design. (A): Large extension cavity restored with regular nano-filled composite (LE-N). (B): Large extension cavity restored with regular bulk-fill composite (LE-BF). (C): Small extension cavity restored with regular nano-filled composite (SE-N). (D): Small extension cavity restored with regular bulk-fill composite (SE-BF).*

tion shrinkage stresses promoted by the resin composites were assessed by marginal gap formation and biomechanical behavior. The thermocycling creates contraction/expansion stresses and accelerated chemical degradation,[42] simulating the clinical condition to which the restorations are exposed.[6] In this study, the thermal aging influenced the formation of the gaps at the adhesive interface, independent of the restorative material. It is interesting to note that the thermocycling protocol used in this study provoked an effect that mimicked long-term bonding effectiveness. Indeed, it has been suggested that 10,000 cycles give a close estimate of 1 year of clinical function.[42]

Two critical factors for the marginal gap formation were considered in this study: cavity extension and the restorative material. Thus, care was taken about tooth selection, cavity standardization, and restorative protocol. Teeth with structural defects were excluded because they influence the bonding.[19] The extensions of 2.9 mm and 1.4 mm were chosen based on a previous study of the prevalence of NCCLs,[43] in which the occlusogingival width of evaluated NCCLs ranged from 1 to 4 mm in most of the lesions (91%). In the present study, the purpose was to evaluate the influence of the cavity extension and, consequently, the volume of restorative material on the marginal gap formation and the theoretical polymerization shrinkage stress distribution in a tooth/restoration. Thus, two different sizes (large 2.9 mm and small 1.4 mm) within that previously reported range were determined. The use of a cutting machine allowed the adapted spherical bur to be moved precisely, and consequently, constant dimensions were obtained for each group. Clearfil SE Bond, a two-step self-etch adhesive, was chosen because it showed a good bonding performance in NCCLs, even without additional mechanical retention.[44] This adhesive system was used according to the manufacturer's instructions for a more effective bond. The teeth treated with Clearfil SE Bond were restored using the bulk-filled protocol for the LE-BF, SE-N, and SE-BF groups and the two oblique increments protocol for the LE-N group.

The gap marginal formation was similar between the restorations in the LE group and the SE group (Figure 2). It was previously suggested that restorations with the same substrate and depth, but with an increased diameter, would result in a higher volume and, consequently, an increase in the interfacial stress.[39] High stresses may contribute to the marginal gap formation.[8] In this study, a larger marginal gap was expected in the LE group because of the higher volume of restorative material, since this group presented an 11.6% greater magnitude of tensile stress peak calculated at the restoration margin compared with the SE group. Despite this, the influence of the volume for marginal gap formation may have been less pronounced compared with other variables of the cavity (geometry, C-factor, and substrate compliance) and composite resin properties (composition, filler content, resin flow, and E) because both cavities presented similar behavior during laboratory tests. However, the hypothesis cannot be ruled out that the areas with higher marginal stress concentration, mainly under loading, are more prone to interfacial damage.

Contrary to the results in the cavity extension, the restorative material employed had a significant influence on the marginal gap formation, before and after aging. Indeed, the composite resin properties (composition, filler content, resin flow, and E) affected polymerization shrinkage and, consequently, the marginal gap formation.[5,14,29,34] It is known that polymerization shrinkage has an inverse correlation with filler content and E,[5,14,15] meaning that the higher the filler content and E, the lower the polymerization shrinkage. Among the composites studied, Filtek Bulk Fill Posterior showed the lowest marginal gap formation and shrinkage stress, despite its having a relatively lower filler content (wt%) and similar E compared with Filtek Z350 XT. Filtek Bulk Fill Posterior is formulated with two novel methacrylate monomers (aromatic urethane dimethacrylate [AUDMA] and addition-fragmentation monomer [AFM]) designed to reduce volumetric shrinkage and shrinkage stress. The action mechanism of AUDMA, a monomer of high molecular weight, consists in reducing the number of reactive sites, thereby moderating the volumetric shrinkage and the stiffness of the developing and final polymer matrix, which is responsible for polymerization stress development. The presence of AFM has the potential to reduce polymerization shrinkage stress without prejudice to physical properties of the polymer.[45] According to the manufacturer, when the polymerization occurs, AFM first reacts into the developing polymer, leading to the formation of crosslinks between polymeric chains, like any methacrylate. A third reactive site of this monomer cleaves through a fragmentation process, in which a mechanism for the relaxation of the polymeric network is reached, allowing stress relief.

Regardless of the restorative material, when we observe the figures of the stress maps (Figure 3), it is shown that the region of highest stress concentration occurred in the enamel margin around the cavity compared with the dentin wall inside the cavity. This is in agreement with the results of Peutzfeldt and others[6] showing that the gaps are more visible in enamel than in dentin. However, the same study concluded that for a Class II cavity, the incremental technique with a regular composite is less susceptible to gap marginal formation than the single increment technique with a bulk-fill composite. It is noteworthy that the cavity of the present study presented enamel along its margin, different from the cavity of Peutzfeldt and others,[6] which contained margins in the dentin also. These findings are also supported by the observations of Alqudaihi and

others,[19] in which a regular composite incrementally placed in a Class I cavity showed less gap formation and better internal adaptation than the bulk-fill composite.

Clinically, the presence of a marginal gap can facilitate bacterial infiltration with subsequent degradation of the adhesive interface and dental structure, development of secondary caries, pulpal irritation, hypersensitivity, marginal staining, and durability of resin restorations.[1-4,19,29,46] Nevertheless, secondary caries lesions seem to be related to individual factors, and their development is linked to a gap larger than 7-13 μm.[47] For Maske and others,[47,48] the secondary caries lesions develop in gap size between 13 and 30 μm, while the threshold gap size for the secondary caries wall lesion would be about 30 μm independent of caries activity level of the patient. The gap measurement values of the four groups investigated in the present study (Figure 2) are in agreement with previously reported studies. This means that the gap generated can have a significant impact on the development of secondary caries. This outcome might be related to shrinkage stress, poor adhesion of the restorative material, and personal factors.[7,19,47]

The results of the present study are in agreement with other previous theoretical findings, which conclude that the use of a bulk-fill composite may reduce the undesirable effects of polymerization shrinkage.[6,19,20,32,46] However, these results should be carefully interpreted, since the cavities' margins were located only in the enamel, which is unfortunately not representative of NCCLs found clinically. Several factors, such as the adhesive system, the substrate type (enamel or dentin), the aging protocol, the photopolymerization protocol, and the loading of the restorations during masticatory function also have to be considered, since the cavity and restorative material are not the only factors that influence the clinical performance of a restoration.[6,11,18,19] It should be emphasized that bovine teeth were used in this study. Despite the similarity between chemical and physical properties of the bovine substrate (composition, density, and microhardness, for example) and human enamel,[49,50] and bovine teeth are the first choice to replace human teeth, it is important to consider the differences in the values of marginal gap and stress concentration that can be found if human teeth are used. Furthermore, another limiting factor is the finite element model with an isotropic composite resin, without bubbles or defects in the material, perfectly bonded in all cavities and with a linear behavior.

CONCLUSION

For the present study, the first null hypothesis was not rejected since the cavity extension did not influence the gap formation. However, the second null hypothesis was rejected because the bulk-fill composite showed less gap formation before and after aging and better stress distribution around the cavity margin than the nano-filled composite, regardless of the cavity's geometry. Based on this, and taking into account the study's limitations, further studies remain necessary to investigate questions that have not yet been answered, as well as the clinical behavior of a bulk-fill composite in Class V restorations.

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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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