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Aim and Scope

Operative Dentistry publishes articles that advance the practice of operative dentistry. The scope of the journal includes conservation and restoration of teeth; the scientific foundation of operative dental therapy; dental materials; dental education; and the social, political, and economic aspects of dental practice. Review papers, book reviews, letters and classified ads for faculty positions are also published.

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On The Cover

Capitol Reef National Park as seen through a dilapidated barn doorway, Teasdale UT, USA. Photo provided by Kevin Matis of Indianapolis, IN USA. Photo taken with a Nikon D70, 52mm f/9 at 1/320 sec exposure. © Operative Dentistry, Inc.

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Guest Editorial

American Board of Operative Dentistry Certification

Dr. David Jones, Vice President, ABOD

The American Board of Operative Dentistry (ABOD) is dedicated to elevating the art and science of Operative Dentistry by encouraging the study of and recognizing excellence in clinical practice. Created in 1980, ABOD was originally composed of 18 members selected by the Executive Council of the Academy of Operative Dentistry (AOD). Selection was based on professional reputation and membership in AOD, and included a wide geographical distribution and various practice settings.

The founding board members developed the existing organizational structure, a Constitution, and By-laws. They also established a rigorous certification program – including written, clinical, and oral examinations – designed to evaluate and establish expertise, not merely competence, in clinical judgment, diagnosis and treatment planning, and hands-on skill. These criteria are still used today to recognize those pursuing excellence in Operative Dentistry. Eligibility to challenge the board includes: Membership in the Academy of Operative Dentistry, four years of post-dental school clinical practice experience, and completion of a recognized master's level training program in Operative or Restorative Dentistry.

Since ABOD's founding nearly 35 years ago, only 87 dentists have been certified in Operative Dentistry. Current membership includes practicing dentists from the private sector, academia, and the U.S. military, as well as a growing number of international dentists, all distinguishing themselves as leaders in organized dentistry. Of the 120 dentists currently serving on the Review Board for the Operative Dentistry journal, 22 are ABOD members. Seven have received the AOD Award of Excellence, two have received the Hollenback Memorial Prize, and ten have served as AOD President. Within the military, two ABOD members have served as Flag Officers, four as commanding officers, and eight as specialty leaders. Nearly every ABOD member has held a dental school faculty appointment, some international, with ten serving as Department Chairs or Program Directors and two as Dean of their schools. Ongoing active participation in the functions of the board following certification is highly encouraged as well. Interest in ABOD certification has increased worldwide in recent years. In fact, the number of international applicants now far exceeds the number of American applicants.

The American Dental Association (ADA) has not recognized a new dental specialty for over a dozen years and Operative Dentistry does not currently fit qualifications for full specialty status. However, in 2010 the ADA created a new recognition status, Interest Area in General Dentistry. Interest areas are recognized by the ADA "to protect the public, nurture the art and science of dentistry, and improve the quality of care." The ADA will consider granting this status "in those fields where advanced knowledge and skills are essential to maintain or restore oral health." Application for Interest Area status involves much the same rigorous process as full specialty recognition and requires sponsorship by the Academy of Operative Dentistry as the parent organization. Designation as an interest area will formalize accreditation for advanced educational programs in Operative Dentistry and recognizes the contributions of Board Members. ABOD is the first organization to apply for this status. The ADA decision is expected to be made by mid-2015.

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The Board strives to emulate the integrity, professionalism, and pursuit of excellence demonstrated by the Founding members. ABOD Executive Council and Business Meeting sessions are open to any members who wish to attend and are held in conjunction with the annual Academy meeting. All AOD members are urged to consider pursuing ABOD certification. For complete information regarding eligibility and the certification process, please see the ABOD web page: https://www.academyofoperativedentistry.com/american.html. Questions can also be sent to the Secretary of the Board at ABOD_Sec@comcast.net.

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Clinical Technique/Case Report

A Case Report of Gingival Enlargement Associated With Invasive Cervical Resorption

MV Bal • Ş Yıldırım • I Saygun

Clinical Relevance

Invasive cervical resorption is a rare external dental resorption with unknown etiology. Lesions are mostly misdiagnosed as internal resorption or caries, which leads to erroneous treatments. So it should be noted that both clinical examination and radiological examination are important in the treatment of invasive cervical resorption lesions.

SUMMARY

Invasive cervical resorption (ICR) is a rare external dental resorption with unknown etiology; it progresses asymptomatically in the cervical area of the permanent teeth. Lesions are mostly misdiagnosed as internal resorption or caries, which leads to erroneous treatments. This case report presents the clinical and radiological diagnosis, as well as the results of treatment and 3-year follow-up in a 50-year-old female patient with gingival enlargement associated with ICR in tooth No. 25. Granulation tissue was removed by accessing the cervical resorption area through a flap operation. Following the endodontic treatment, the tooth was restored using composite resin and the hyperplastic lesion was excised. In conclusion, it should be kept in mind that clinical, radiological, and pathological evaluation in the differential diagnosis of localized hyperplastic lesions in the gingiva is of importance and that ICR could play a role in the etiology of these lesions.

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INTRODUCTION

Invasive cervical resorption (ICR) is a rare clinical condition of the permanent teeth that progresses asymptomatically and is defined as a destructive form of external dental resorption.[1] Pathological mechanisms in the etiology of ICR are not known definitively; however, dentoalveolar surgery, orthodontic interventions, periodontal disease and trauma caused by its treatment,[2] and intracoronal bleaching procedures are considered to be the factors likely to play a role in the development of ICR.[3] Furthermore, developmental defects such as cemental hypoplasia and hypomineralization are also considered to be predisposing factors. It has been reported that necrotic pulp–originated pathogens generally do not affect the occurrence and development of ICR; however, injury to the periodontium in the cervical area may contribute to the

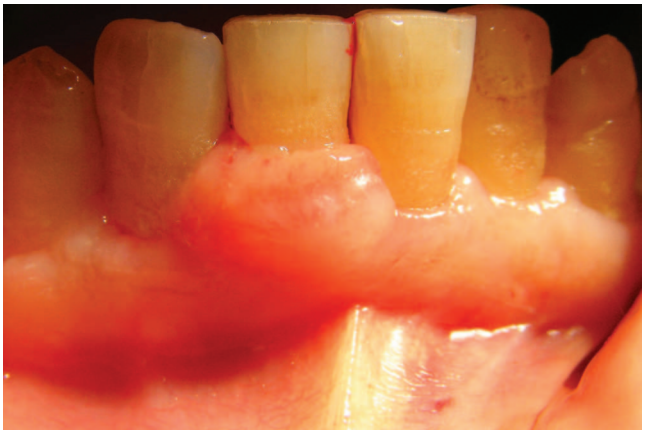

Figure 1. *Intraoral view of the lesion.*

development of lesions.[4,5] Lesions have been reported to occur without any determined etiological factor in approximately 15% of ICR cases.[6]

Histopathologically, ICR is defined as a localized resorptive process that commences in the coronal aspect of the root surface and develops in the zone of connective tissue attachment supported by alveolar bone below the epithelial attachment. This pathological process can extend to the pulp space following resorption of the cementum and dentin.[2] In the clinical examination of ICR, a lesion usually does not demonstrate any sign in the early period, and the proliferative tissue occupying the defect, which is caused by resorption, is hyperemic and prone to bleeding. The resorption extends into the coronal surface, weakening the enamel and causing a pinkish appearance. Sometimes the defect may be probed from the gingival margin; in that case, the dentin may appear intact.[7]

Surgical removal of fibrovascular tissue in the resorption area and repair of the defective area with a restorative material are recommended for the treatment of ICR.[8] The relation of resorption to the pulpal tissue is important to determine the need for endodontic therapy. The pulp is usually vital in the presence of a thin predentin layer between a ICR lesion and the pulp tissue. Considering this, it has been reported that removal of the fibrovascular tissue from the resorption area should be cautiously performed and pulp perforation should be avoided.[9]

This case report presents clinical and radiological diagnoses, as well as the results of treatment and 3-year follow-up, in a patient with gingival enlargement in the mandibular incisor, which is rarely encountered together with ICR.

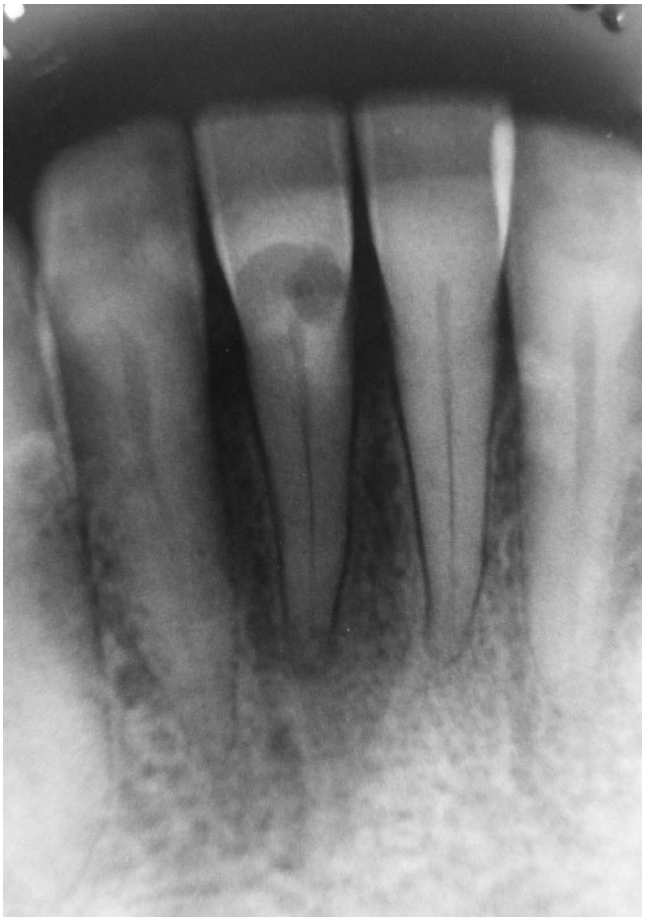

Figure 2. *Radiolucent image in the cervical region.*

CASE

A 50-year-old female patient who was admitted to the Dental Clinic of Bursa Military Hospital with gingival enlargement in the anterior mandible underwent an intraoral examination. Her history revealed that she noticed gingival enlargement and redness without pain approximately one and a half months ago. Systemic examination and consultations revealed controlled hypertension and a thyroid disorder. Her extraoral examination was unremarkable; intraoral examination showed poor oral hygiene and a gingival lesion of $7 \times 5 \times 5$ mm on the facial gingival margin of tooth No. 25 (Figure 1). The patient had no parafunctional habits and had not undergone orthodontic treatment. Probing the pocket depth showed a 5-mm pseudopocket in the vestibule of tooth No. 25; neither bleeding nor occlusal trauma was detected. Periapical radiography demonstrated a radiolucent image in the cervical region of tooth No. 25 (Figure 2). The tooth was determined to be non-vital upon vitality testing; a presumptive diagnosis of ICR was made. The

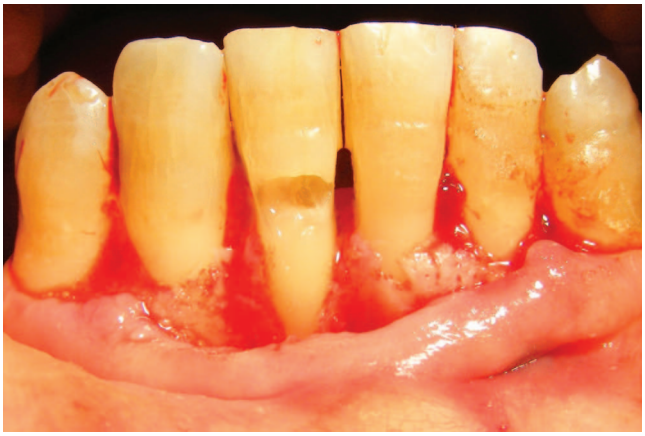

Figure 3. *Defect area in the cervical region and defective facial crown/root surface after removal of the granulation tissue.*

patient was informed about the planned treatment and her consent was obtained. Oral care training was provided for the patient, and a complete oral and dental surface cleaning was performed. The lesion was excised one week later under local anesthesia using a flap procedure and an appropriate route of entry into the cervical resorption area was created. Granulation tissue in the resorption area was removed; it was determined that the lesion extended to the pulp (Figure 3). The excised tissue was transferred to the pathology department. Histopathological examination showed remarkable fibrosis and hyalinization in the lesion area, as well as vascular proliferation, lymphocyte and plasmacyte infiltration below the ulcerated squamous epithelium (Figure 4). During the flap operation, the root canal was filled with gutta-percha (DiaDent Group International Inc, Seoul, Korea) and AH 26 sealer

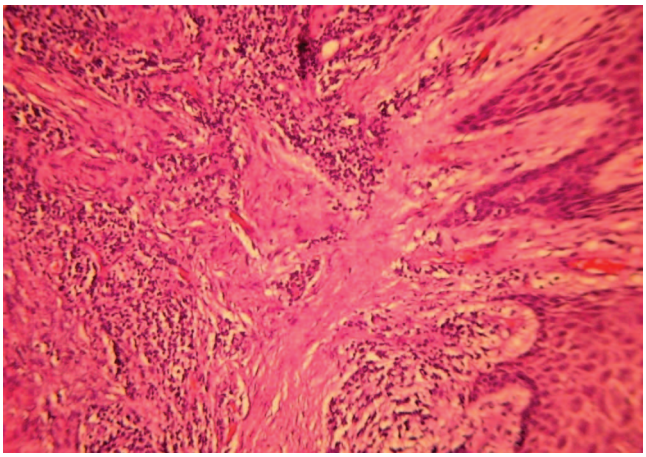

Figure 4. *Histopathological view of vascular proliferation, perivascular lymphocyte and plasmacyte infiltration, and fibrosis below the hyperplastic squamous epithelium (hematoxylin and eosin staining, 100×).*

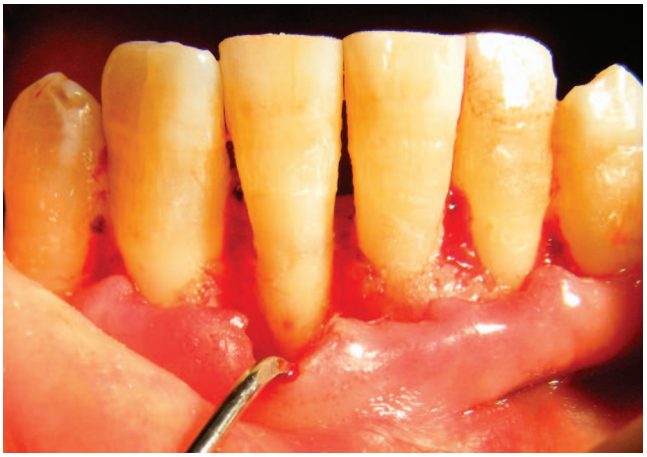

Figure 5. *Restoration of the defect area in the cervical region with composite resin.*

(Dentsply DeTrey GmbH, Konstanz, Germany) using a lateral condensation method. The cervical area of the tooth was restored with composite resin (Filtek Z250, 3M ESPE, St Paul, MN, USA) (Figure 5). The flap was closed and sutured following endodontic and restorative treatments.

The patient received amoxicillin + clavulanic acid (1000 mg, two times daily for 5 days). After being controlled in the first week, the patient was followed up at the sixth month and first, second, and third years after treatment. On the six month control visit, she had no relapse and her intraoral photographs (Figure 6) and radiographs (Figure 7) were obtained. On the control visits performed at the first, second, and third years after the endodontic and restorative treatments, no relapse was observed in the patient or in her intraoral photograph (Figure 8) and periapical radiograph (Figure 9).

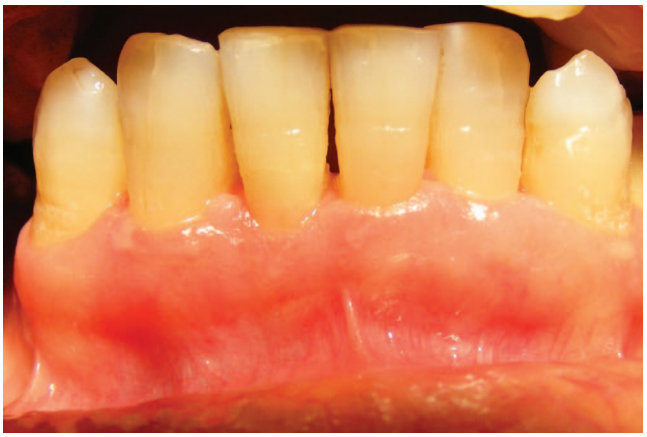

Figure 6. *Clinical view six months after the endodontic and restorative treatments.*

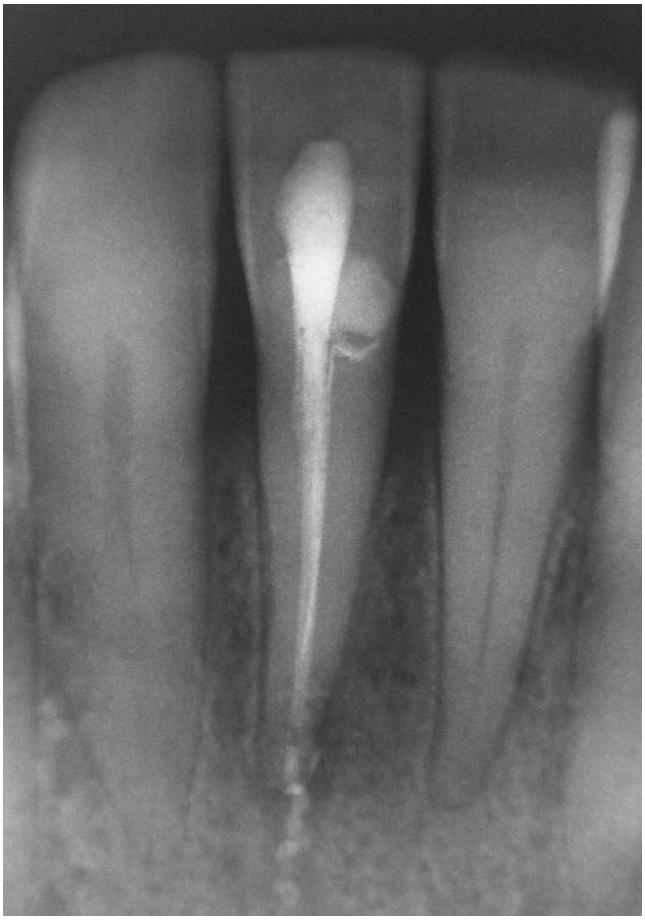

Figure 7. *Radiological image six months after the endodontic and restorative treatments.*

DISCUSSION

In the present case report, the diagnosis, treatment, and three-year follow-up results of localized gingival enlargement associated with ICR including the dentin, which is rarely encountered, are presented. The most common causes of localized gingival enlargement, which is defined as fibrous epulis, include subgingival plaque, tartar, and inappropriate restoration margins. In the present case, it was thought that cervical resorption might have been provoked by an inflammatory reaction in the periodontium due to inadequate plaque control. It was also thought that extensive hypoplasia in other teeth of the patient might also have played a role in the etiology. Natural cementum defects[10] or root surface damage[2] are being investigated as predisposing factors in the pathogenesis of cervical resorption. In his study, Heithersay[6] found no predisposing factor in 16.4% of 257 teeth with ICR and reported that developmental defects such as hypomineralization or hypoplasia of the cementum might play a role in the etiology. In the present case, however, developmental defects in the cementum could not be evaluated because the tooth was kept in the mouth and histopathological examination could not be performed.

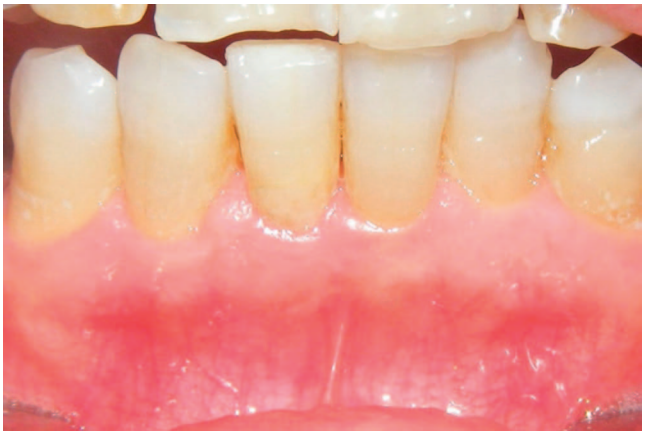

Figure 8. *Intraoral clinical view three years after the endodontic and restorative treatments.*

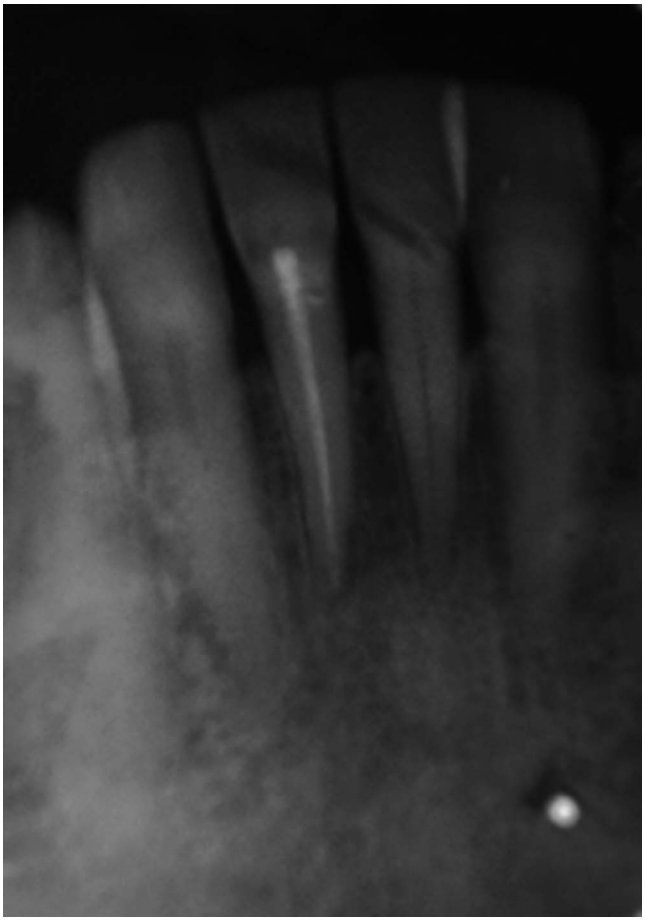

Figure 9. *Radiological image three years after the endodontic and restorative treatments.*

The cellular mechanism that causes ICR is quite complicated. It has been demonstrated that multinucleated giant cells or inflammatory cells, which have the potential to cause resorption, accumulate in the area due to numerous inflammatory mediators scattered into the medium caused by the inflammation or injury of hard dental tissues.[1] It has been reported that traumatic or bacterial stimulants lead to resorption by causing an inflammatory response in the periodontal ligament due to cervical changes in or local damage to the root surface, and thus, stimulating multinucleated giant cells having destructive activity.[11] In the present case, we thought that the patient had no traumatic stimulant; however, she had bacterial stimulants due to poor oral hygiene.

Etiological factors should be considered in the treatment of ICR. Elimination of these factors constitutes the first step of the treatment. In addition to dental extraction, various treatment options have been recommended in the literature including subgingival curettage[12] and calcium hydroxide administration to the defect in order to provide neutralization around the lesion.[13] In their study, Meister and others[14] recommended exposing the resorption defect by a surgical approach together with systemic antibiotic use and performing mechanical debridement. With the present patient, the gingival enlargement was excised by a flap operation and the granulation tissue was removed by reaching the resorption area in the cervical region. In addition to mechanical debridement, the patient received systemic antibiotic therapy. She did not relapse within the three-year follow-up period, which verified the success of the treatment method.

Knowing the vitality of the tooth in the treatment of ICR is crucial. Precise clinical and radiological examination and assessment of pulp vitality via vitality tests are of great importance for the selection of the treatment method in teeth with resorption. It is thought that endodontic treatment along with a surgical approach could prevent the bleeding complication that is likely to occur during root canal treatment in the presence of a necrotic pulp.[15] In addition, there are studies reporting that endodontic therapy performed before surgery prevents leakage of irrigation fluid from the canal into the periodontal ligament and surrounding tissues.[16] With the present patient, the surgical procedure was performed simultaneously with endodontic treatment in order to prevent both the leakage of irrigation fluid out of the tissue and bleeding complications. No clinical or radiological pathology related to endodontic treatment was observed during the three-year follow-up period.

In the literature, treatment options in the early period of ICR include topical application of trichloroacetic acid, root canal therapy without surgery, curettage of the tissue in the resorption area, restoration of this area with glass ionomer cement, and a careful follow-up during the healing period.[17] The basic principle of topical application of trichloroacetic acid is to create coagulation necrosis in the surrounding tissues. This chemical agent acts not only in the resorptive area but also in the interrelated canals and in the depth of the tissue.[18] Moreover, topical application of bisphosphonates, which are used for the treatment of osteoporosis, has also been suggested as another therapy option.[19] However, in the present case, no agent was applied to the resorption area in addition to surgical treatment.

In the literature, for restoration of a defect area in ICS, glass ionomer,[17,20,21] amalgam,[22-24] or composite resin[20,25] have been used. In the present case, we used composite resin for the restoration of the defect area in the cervical region because the defect area was above the gingival margin after excision of the gingival enlargement.

This case report presents the results of a surgical procedure that was performed simultaneously with endodontic treatment followed by a three-year follow-up period in a patient diagnosed with ICR. The results of the three-year follow-up revealed that early detection of ICR—detection of a defect area in a cervical region before progression into the root, which can be asymptomatic—with precise clinical and radiological examination would enhance the success of treatment. Endo-perio treatment options should be considered before deciding to extract the tooth.

Note

This case was presented as a poster at the Turkish Society of Periodontology, 40th Scientific Congress, May 14–16, 2010, İzmir, Turkey.

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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Biological Restorations as an Alternative to Reconstructing Posterior Teeth: A Case Report

NLG Albuquerque • JS Mendonça • CSR Fonteles
JC Pereira • SL Santiago

Clinical Relevance

Biological restoration using tooth fragments offers a viable restorative option for the clinician because it restores tooth function and esthetics with the use of a very conservative and cost-effective approach.

SUMMARY

This article reports on a three-year follow-up of two biological restorations performed on a 15-year-old female patient. After clinical evaluation, tooth fragments from extracted permanent molars were obtained from a Human Teeth Bank and were autoclaved, adjusted to the prepared cavity, and bonded to the remaining tooth structure with dual resin cement. The technical aspects are described and the benefits and disadvantages of biological restorations as an alternative treatment for rehabilitation of severely destroyed permanent molars are discussed.

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INTRODUCTION

Reconstructions of posterior teeth are still a challenge for restorative dentistry because of the absence of sufficiently resistant restorative materials with favorable biological properties compatible with dental tissues.[1] Currently, many different materials and techniques, such as resin composite as a direct or indirect restoration or porcelain, have been used to rehabilitate function and esthetics. Often, however, the use of clinical judgment and creativity is essential to modify existing techniques or even to create new ones. Therefore, deciduous and permanent teeth have been reused as an alternative to anatomically restore the lost structure.[1-4]

Since Buonocore's first introduction of the acid-etch technique in 1955,[5] biological restoration techniques of tooth fragments became a possibility. In 1978, Tenery[6] stated that the use of tooth structure

as a restorative material should always be considered as the first treatment alternative. In addition, several authors[2,7,8] have suggested the use of natural tooth fragments as an efficient method for restoring fractured anterior teeth.

The biological restoration technique comprises the use of adhesives, composites, resin cements, and human teeth, frequently those procured from a Human Teeth Bank (HTB). Thus, sufficient information, such as origin and preparation of the dental fragment, should be provided to patients and/or legal guardians in order to obtain informed consent.[1,9,10] This article describes a three-year follow-up in the clinical case of a 15-year-old female patient in whom the biological restoration technique with tooth fragments obtained from a HTB was the treatment elected to restore two permanent molars subjected to extensive amalgam restorations.

CASE REPORT

A healthy 15-year-old female patient presented to the Dental Clinic of the Bauru School of Dentistry, University of São Paulo (Brazil), seeking dental treatment. Clinical examination showed the absence of active caries lesions and two extensive amalgam restorations without marginal adaptation on the lower left (#19) and upper right (#3) first molar teeth. Treatment plan alternatives for the replacement of these restorations included 1) direct or indirect composite resin material, 2) porcelain inlay, or 3) biological restoration. These teeth presented no clinical signs or symptoms of pulp inflammation/degradation; hence, normal responses were observed to cold and percussion tests. In addition, the patient reported no sensitivity or spontaneous or induced pain associated with these teeth. The patient and legal guardians were informed of the advantages and disadvantages of each treatment option and elected the biological restoration technique as their first choice of treatment.

The mandibular left first molar was the first tooth to be restored (Figure 1). Following the administration of local anesthesia, the area to be restored was isolated with rubber dam and retentive areas were eliminated (Figure 2). An arch impression with an irreversible hydrocolloid material (Jeltrate Plus, Dentsply Ind. e Com. Ltda, Petrópolis, RJ, Brazil) was performed in order to obtain a plaster cast. The mesiodistal, cervico-occlusal, and buccolingual dimensions of the tooth were measured to facilitate the selection of an extracted tooth from the HTB with coronal length, height, and width that best fit the prepared tooth (Figure 3). The tooth was also matched by color during selection. The patient was released with a temporary restoration until the next session.

The selected dental specimen was decoronated and the coronal fragment adjusted with diamond points at high speed under air/water spray coolant until it fit the cavity. Articulating paper was interposed between the fragment and the cavity in the plaster cast to demarcate the areas that needed further adjustment. The extracted tooth had been previously sterilized by autoclaving, in accordance with biosecurity standards. At the second visit, prophylaxis was completed and the adaptation of the fragment to the tooth was checked (Figure 4). Acid-etching with a 37% phosphoric acid gel (3M/ ESPE, St Paul, MN, USA) was extended approximately 2 mm beyond the margin of the cavity for 15 seconds; the fragment was acid-etched for 15 seconds and was subsequently washed (Figure 5). Single Bond (3M/ESPE) adhesive system was applied in two consecutive layers on the tooth and fragment, and each side was light-cured for 20 seconds using a visible light-curing device (XL 3000, 3M/ESPE) (Figure 6). The fragment was bonded with a dual-cure, resin-based cement, shade A2 (Rely X ARC, 3M/ESPE) (Figure 7) and light-cured for 60 seconds. Small imperfections were corrected with light-curing composite resin (Filtek Z250, 3M/ESPE) and the occlusion checked with articulating paper. Figure 8a illustrates the final clinical aspects of the restoration and the three-year follow-up (Figure 8b).

After seven days, the patient was readmitted to perform the treatment in the maxillary right first molar (Figure 9), following the same standards used for the above procedure. Since cavity preparation was deeper, the cavity floor was protected with a calcium hydroxide cement layer (Dycal, Dentsply Ind. Com. Ltda), and a resin-modified glass ionomer cement base was used to replace dentin tissue (Vitrebond, 3M/ESPE). The steps for selection of a tooth compatible with the remaining tooth structure, cutting, and adaptation of the fragment to the plaster cast were the same as described previously. The prepared cavity received a temporary restoration, and at the second visit, the same steps described above were followed. Once treatment was concluded, oral hygiene instructions were provided and the need for periodic evaluations was emphasized. Figure 10a illustrates the final aspects of the restoration and the three-year follow-up (Figure 10b).

DISCUSSION

Biological restoration is a simple rehabilitating technique, with advantages that include 1) better

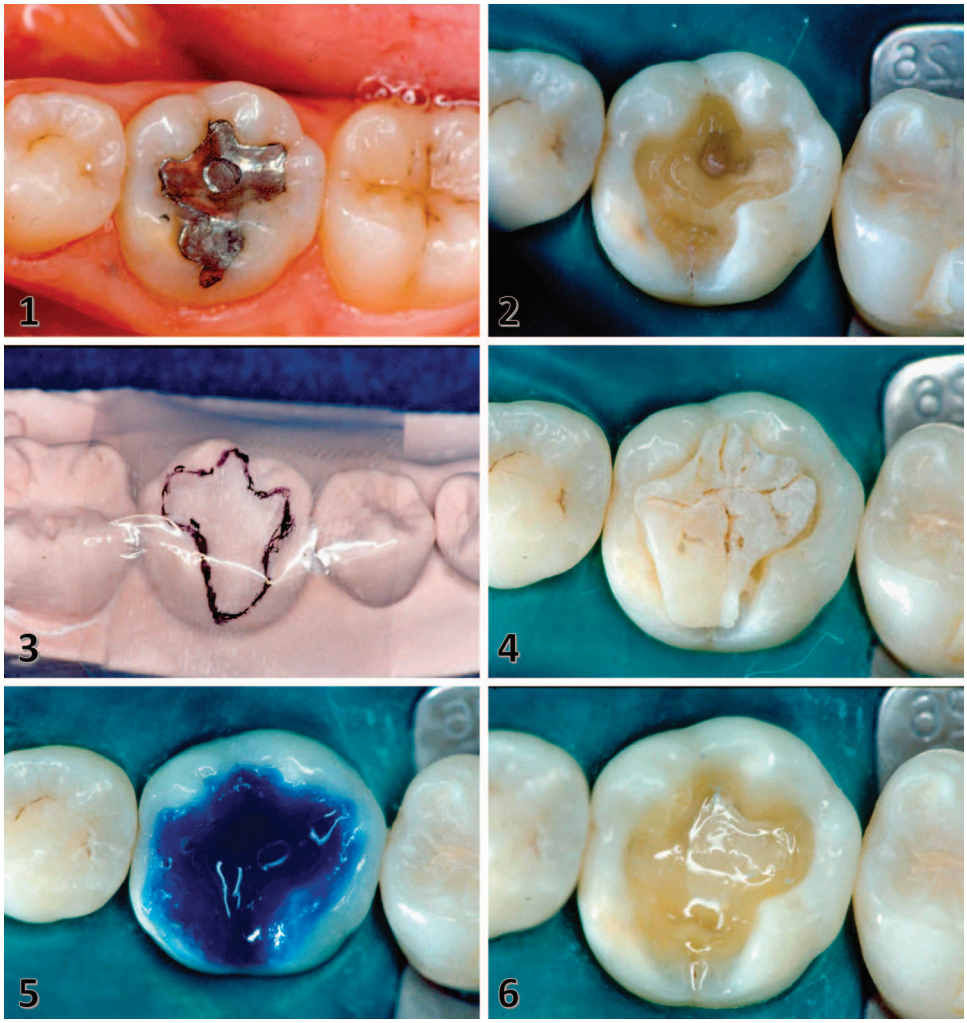

Figure 1. *Extensive amalgam restoration without marginal adaptation in the mandibular left first molar (#19).*
Figure 2. *The area to be restored was isolated with rubber dam, and retentive areas were eliminated.*
Figure 3. *The mesiodistal, cervicoocclusal, and buccolingual dimensions of the tooth were measured to facilitate the selection of an extracted tooth from the HTB with coronal length, height, and width that best fit the prepared tooth.*
Figure 4. *Checking the adaptation of the fragment.*
Figure 5. *Acid-etching with a 37% phosphoric acid gel for 15 seconds.*
Figure 6. *Application of the Single Bond (3M/ESPE) adhesive system in two consecutive layers.*

reconstruction of the dental margins, 2) minimal need for dental restorative material, 3) durability and preservation of the remaining tooth structure, 4) resilience comparable to that of the original tooth, and 5) excellent esthetic results compared to composite resins and stainless-steel crowns, providing good translucency.[1,2,7,8] In addition, this biological restoration allows maintenance of pulp vitality[11] and has low cost.[12] In spite of being simple, the technique requires professional expertise to adequately prepare and adapt the natural crowns to the cavity.[13] Disadvantages of the biological restoration technique include 1) the difficulty in selecting the fragment to adequately meet the natural tooth color, especially in cases involving partial destruction of the crown, and 2) the possibility of nonacceptance by the patient, as the technique involves a carrier of tooth fragment from another individual.[1] It is important to inform the patient or his/her parents (legal guardians) that prior to clinical use, tooth fragments are submitted to a rigorous sterilization process in an autoclave at 121°C for 15 minutes, ensuring all biosecurity standards.[14] The advantages and disadvantages of the technique and treatment alternatives must also be provided to enable patients to choose in an informed manner what they understand as the most appropriate treatment option in each case.

Although over the course of many years amalgam has been referenced as the material of choice to restore posterior teeth in different parts of the world, in spite of its relatively low and long-term cost effectiveness,[15,16] esthetic limitation remains a disadvantage. Likewise, gold inlays are still indicated for larger restorations that need support to withstand intense masticatory stress, presenting unsurpassable longevity with minimal wear on antagonists, and in contrast to amalgam, this material is not susceptible to corrosion.[17] Direct

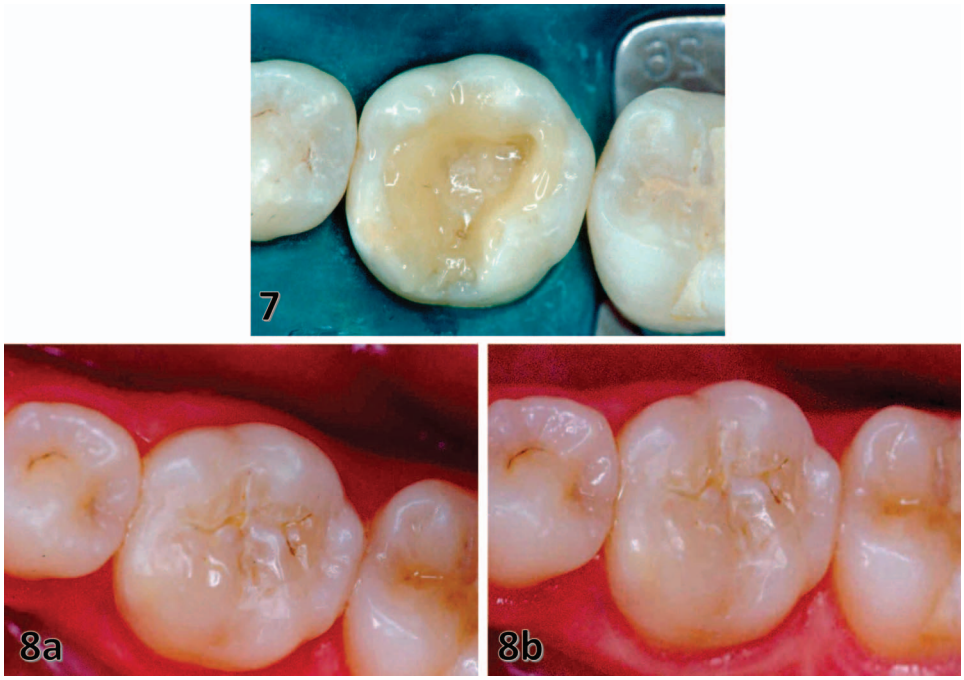

Figure 7. *The fragment was bonded with a dual-cured, resin-based cement.*
Figure 8. *Final clinical aspects of restoration (a) and three-year follow-up (b).*

composite resin restorations present the following advantages: 1) these restorations involve a low cost compared to other esthetic materials; and 2) these restorations involve single-session procedures and do not requirement temporary restorations, reducing chair time and dispensing a second session for cementation. In the present clinical case, the patient expressed a desire for an esthetic restoration. Hence, treatment options discussed with the patient and her parents included direct composite resin restorations, porcelain inlay, and biological restoration. Since no synthetic material is capable of replicating the esthetic characteristics or color stability of natural teeth,[18] composite resin restorations are less esthetic

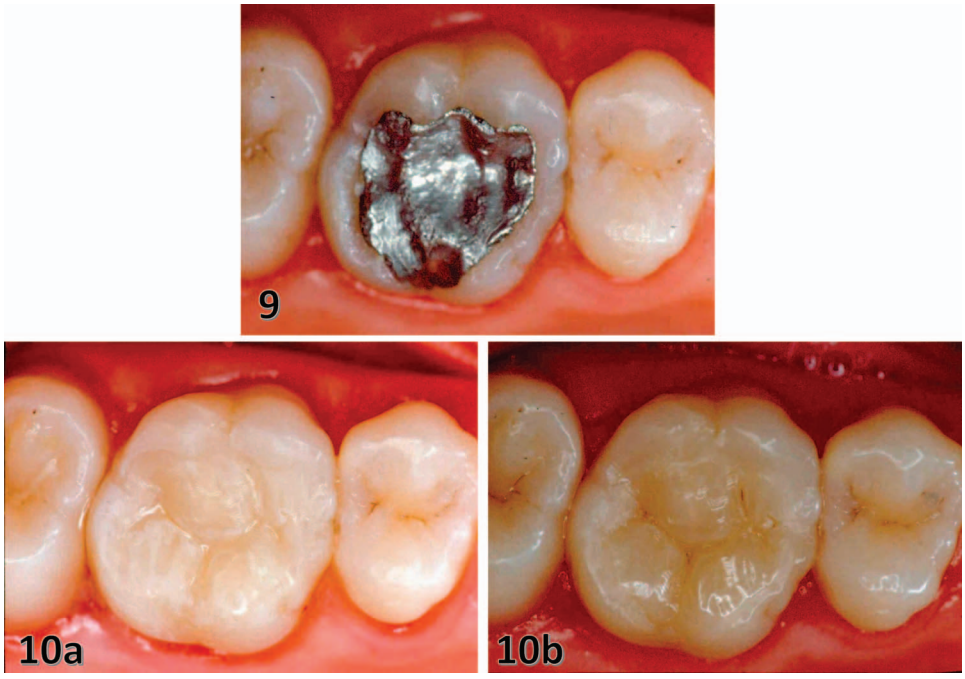

Figure 9. *Extensive amalgam restoration without marginal adaptation in the maxillary right first molar (#3).*
Figure 10. *Final clinical aspects of restoration (a) and three-year follow-up (b).*

compared to the biological restoration technique. In spite of a necessary laboratory stage, this technique requires a relatively short clinical time compared to other esthetic restorative procedures and offers superior physical properties compared to composite resins.[17] Porcelain inlay is a more expensive technique that may require greater tooth wear to provide an adequate dental preparation for indirect restoration. In young patients, it is desirable to preserve dental structure in order to avoid or postpone the progression toward endodontic treatment and porcelain-metal crown restorations or future tooth loss and implant rehabilitation, justifying the treatment alternative chosen for our 15-year-old patient. The biological restoration technique is a more conservative clinical approach, one that offers greater durability, better cost-effectiveness, and shorter chair time, which in turn allows natural results in terms of anatomic shape, surface shine, smoothness, and translucence of the enamel, when compared to other choices of treatment.

The first report in the literature of the use of fragments of extracted teeth as dental restorative materials was published in 1964 by Chosak and Eidelman,[19] and the expression "biological restoration" was first coined by Santos and Bianchi[20] in 1991. Busato and others[2] described the technique of using human teeth from Tooth Banks in large restorations, emphasizing the greater resistance of teeth restored with tooth fragments, as compared to restorations with composite resin materials. The author presented a two-year evaluation of a clinical case, showing that the biological restoration technique has extraordinary clinical potential and social impact. Tavano and others[8] presented the esthetic and functional rehabilitation of an upper left central incisor (#9) through homogeneous bonding of a dental fragment. Biological restoration was used to restore this incisor because the patient did not have the original tooth fragment itself. After a one-year follow-up, the results obtained were highly satisfactory. In 2010, Corrêa-Faria and others[1] reported a clinical case performed by means of biological restoration using homogeneous fragment bonding associated with biological posts to reconstruct an extensively fractured central maxillary incisor and after one year demonstrated excellent results. Carvalho and others[21] described a clinical case demonstrating the quality and functionality of a biological restoration performed to reestablish function and esthetics to a left maxillary first premolar (#24) that presented fracture of the entire buccal region. A 12-month follow-up indicated a stable restoration. In addition, this technique has also been described as an alternative to the reconstruction of extensively destroyed deciduous teeth.[13,22] Sanches and others[13] reported on two young children, aged four and five years, in whom biological restorations using tooth fragments were placed in primary molars with severely damaged crowns due to extensive carious lesions. The restorations were bonded to the remaining tooth structure with either adhesive system (case 1) or dual-cure, resin-based cement (Enforce, Dentsply Ind. Com. Ltda) (case 2) over a calcium hydroxide layer and a glass ionomer cement base. Periodical clinical and radiographic controls were carried out and the restored teeth were followed for four and three years, respectively, until exfoliation. Thus, biological restoration technique using tooth fragments has a practical clinical applicability and may present as an interesting treatment alternative when treating pediatric patients.

Currently, with the existence of HTBs and the characteristics of the adhesive materials, rehabilitation of extensively destroyed teeth with this technique became possible and quite feasible.[1,8,10] Therefore, there is a need to organize the functionality of HTBs standardizing autoclave sterilization for 40 minutes according to the American Dental Association[23] and the Centers for Disease Control and Prevention (CDC).[24] This method does not alter the physical properties of the dentinal tissues and does not compromise the goals and/or results of the application of these teeth in therapeutics.[23,24] In addition, the proper storage with saline solution, water, and disinfectants, as recommended by the CDC, is essential to maintaining the chemical, physical, and mechanical properties of these teeth.[24] All of these precautions must be followed carefully, thereby eliminating the possibility of transmission of pathogenic microorganisms.

After three years of clinical follow-up of two biological restorations placed on permanent first molars (#3 and #19) in a 15-year-old adolescent, acceptable clinical results were observed, with no signs of caries, migration of the fragments, or marginal infiltration, thus demonstrating satisfactory esthetics. Our results are in accordance with those of several studies[1,2,8,13,17,21,22] that showed successful outcomes of functional, esthetic, and the psychological aspects of patients. Therefore, biological restorations have considerable clinical applicability and may offer a highly biological option to restore teeth, reestablishing function and esthetics,

as long as standard of care and treatment options are carefully considered in all cases.

CONCLUSION

In conclusion, the restoration technique using biological fragments of teeth is a feasible restorative option for adolescents, showing excellent clinical applicability, in addition to great cost effectiveness, for the restoration of permanent molars with crowns containing extensive amalgam restorations required for replacement due to secondary caries or marginal failure.

Conflict of Interest

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

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Clinical Research

The Local Anaesthetic Effect of a Dental Laser Prior to Cavity Preparation: A Pilot Volunteer Study

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O Al Naimi • J Meechan

Clinical Relevance

Laser preconditioning has no role in producing alterations in pulpal response.

SUMMARY

Objectives: It has been suggested that laser preconditioning can produce dental anaesthesia. This study aimed to assess the response of the dental pulp to laser preconditioning.

Methods: The effects of laser preconditioning, sham laser (negative control), and composite curing light (positive control) on the response of the dental pulp to electric pulp testing was investigated in this double-blind crossover trial with six volunteers. The Er,Cr:YSGG laser or curing light was shone on a premolar tooth in a sweeping motion for 30 seconds (in the sham treatment, the laser was not activated) in blindfolded volunteers subjected to a consistent aural stimulus. Treatment method at each visit was randomized and performed by a researcher not involved in pulp testing. Teeth were pulp tested twice initially by another member of the research team to get baseline readings, immediately following the treatment, and thereafter every two minutes for 10 minutes. Results were analyzed using analysis of variance and an independent-sample *t*-test.

Results: There were no significant differences in pulpal response between treatments ($p>0.05$).

Conclusion: Laser preconditioning did not affect pulpal response as measured by an electronic pulp tester. Laser preconditioning did not result in any pain or noticeable symptoms for both teeth and soft tissues.

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INTRODUCTION

Although there is some evidence to show that laser tooth preparations can be achieved without local anaesthesia,[1,2] there are still no scientific data to support laser preconditioning as effective or neces-

sary. It has been suggested that shining the Waterlase (Biolase Europe GmbH, Paintweg, Germany) when it is set at its minimum output on the tooth surface induces local anaesthesia.[3] Most of the studies conducted on the clinical application of this laser system as a replacement for the conventional dental drill have been sponsored by the laser's manufacturer or have design weaknesses.[3] To ensure that preconditioning causes no surface enamel damage or an unacceptable rise in the pulp's temperature, we have carried out pilot laboratory tests on extracted teeth.[4] No physical damage was visible on the tooth when the laser was used. This was assessed using both visual assessment and electron microscopy. In addition, measurements recorded in the pulp chambers of extracted teeth showed less temperature rise than that induced by a composite curing light.[5] Such lights are used routinely in restoring teeth with resin composite; the additional temperature rise due to polymerization will be much higher than the light used by itself.[6]

In addition to allowing pain-free cavity preparation, any local anaesthetic effect produced by laser preconditioning could be useful in diagnosis, as it might produce single-tooth anaesthesia, a feature not usually afforded by conventional dental local anaesthetic techniques. Therefore, any potential local anaesthetic effect is worth investigating.

The primary aim of this volunteer study was to assess the response of the dental pulp to preoperative conditioning with the laser set at its minimum power setting (0.25 W). A secondary aim was to determine if this preoperative conditioning produced any unwanted effects. The null hypothesis was that pretreatment with a laser did not differ from pretreatment with a curing light or placebo in the changes in pulpal response to electronic stimulation.

SUBJECTS AND METHODS

Study Design

This study was a crossover trial with six healthy adult volunteers. Each subject received a different treatment at each of three visits. Neither the volunteer nor the researcher measuring the response of the tooth knew which treatment had been given at any particular visit. An unrestored vital first or second premolar tooth (upper or lower) was used as the test tooth, and a similar tooth on the other side of the jaw was used as a control to assess the functioning of the electronic pulp tester. Test and control teeth were randomly allocated. The primary outcome measure was the pulpal response, which was assessed using the electronic pulp tester (EPT). The secondary measure was the subjective assessment of sensation encountered during and after laser or control preconditioning. All active and control pretreatments were applied by the same clinician, and the pulpal responses were recorded by another researcher who was blind to the pretreatment used. Each subject required three visits at least one week apart, with the treatment method at each visit (whether a test or control treatment) selected at random. All subjects had received each treatment at the end of the trial.

Regulatory Approval

Regulatory approval was obtained from the following authorities: a medical research ethics committee (Newcastle & North Tyneside Local Research Ethics Committee 1) and Trust (Research & Development).

Volunteer Selection

Volunteers were selected from the staff/student population of the Newcastle Dental Hospital and Newcastle University. Six subjects were required to have unrestored vital first or second premolar teeth in one quadrant of the jaw (upper or lower) and a similar tooth on the other side of the jaw that was used as a control. EPT tests were performed to assess the response of the pulps of the test and control teeth.

Operators

There were two operators. The chief researcher performed all pretreatment procedures, and the second operator carried out the pulp tester measurements.

Test Method

A Millennium Er,Cr:YSGG laser system (Biolase Europe GmbH, Paintweg, Germany) was used for the active treatment. The laser light was shone on the buccal and the lingual surfaces of the test teeth in a sweeping motion for 30 seconds at 4-mm distance. A mechanical spacer was used to maintain a constant distance from the tooth surface and the laser tip (Figure 1). The laser system was used at the minimum power setting (0.25 W) without cooling. Exposure times were measured using a stopwatch.

Control Methods

A calibrated quartz composite curing light QHL 75 (Dentsply Caulk, Milford, DE, USA), emitting a

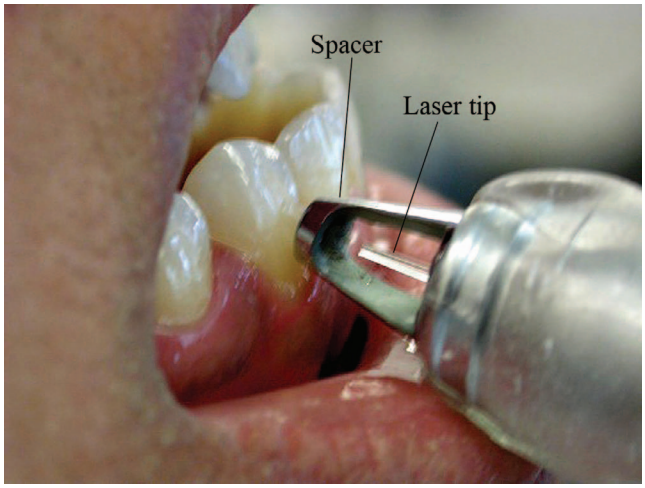

Figure 1. *Mechanical spacer applied to the tooth surface.*

power output of 1500 mW/cm^2, was used for the positive control test. The curing light was calibrated (Coltolux Light Meter, Coltene/Whaledent, Sussex, UK) before and after each set of experiments. The curing light was used on the buccal and the lingual surfaces of the target tooth for 30 seconds in a sweeping motion and at 0-mm distance. The negative control was a sham laser hand piece (ie, not switched on). During exposure to both types of control, the laser device was still switched on to produce the characteristic laser "popping sound" without being actively used. A second curing light machine (not actively used) was also switched on during all tests so that a mixture of sounds was audible. The subjects were blindfolded during treatment. The calibrations of the curing light were checked at the beginning and end of the experiment and were found to be constant.

Pulpal Response Assessment

The standard outcome measure used to determine the presence of anaesthesia in local anaesthetic trials is response to electronic stimulation.[7] An Analytical Pulp Tester (Vitality scanner, Kerr, Analytical Endodontics, Redmond, WA, USA) was used by a researcher who was blind to the selected treatment (test or control). Tooth surfaces were carefully isolated and dried with cotton wool. The pulp tester tip was covered with a conducting medium (Prophylaxis paste, Kemdent, Swindon, UK) and then applied on the mid-surface of the occlusal one-third of the buccal surface of the tooth.[8] To complete the circuit, the volunteer was instructed to hold the handle of the pulp tester device and to raise his or her hand at the occurrence of any sensation. Numeric values corresponding to the intensity of stimulation were displayed on the device and recorded. The maximum output of the device corresponded to a reading of 80. No response to this maximum reading is considered successful anaesthesia. The batteries of the device were replaced by a new set on a weekly basis. Both teeth (test and control) were pulp tested twice initially to get the baseline readings. Single readings were then taken immediately following the treatment and then every two minutes for 10 minutes. The test tooth was pulp tested first during each testing cycle.

Screening for Symptoms

Volunteers were given a questionnaire to record how the tooth and surrounding tissues felt during each preconditioning treatment. The discomfort level was recorded on a 100-mm visual analogue scale (VAS) with the anchors being "no pain" and "unbearable pain." Volunteers were asked to place a vertical mark on the scale where it best described his or her pain level.

Statistical Analysis

The pulp tester readings of the test and the control treatments were subjected to analysis of variance (ANOVA) and post hoc Tukey multiple comparison test to compare the results at each two-minute interval from baseline to 10 minutes, a Student independent-sample t-test was used to compare the baseline EPT readings of individual pairs of teeth (test and control). VAS results were measured in millimeters and presented as whole numbers. The software SPSS 15.0 (SPSS Inc, Chicago, IL, USA) was used. The level of significance was set at $p<0.05$.

RESULTS

The six volunteers (two males and four females) had a mean age of 33 (range 27–41). Although there were minor variations in baseline readings between individual pairs of teeth (test and control) there were no significant differences as a group ($t=0.8$, $p=0.425$). The mean and standard deviation of the pulpal response assessment over time are shown in (Table 1). There were no significant differences in the changes in pulpal response between treatments at any time (ANOVA, $p>0.05$). Laser preconditioning did not achieve anaesthesia in any of the volunteers as measured by no response to an EPT reading of 80. All teeth responded to the pulp tester at every reading. None of the subjects experienced any pain greater than zero as measured by VAS at any time during the trial either for teeth or for soft

Table 1: *Means ± Standard Deviation of EPT Pulpal Response Measurements Over Time*

Time (Minutes)	Laser (n=6)	Sham Laser (n=6)	Curing Light (n=6)
Baseline	37 ± 10	37 ± 8	34 ± 5
0	39 ± 14	39 ± 7	38 ± 7
2	42 ± 11	39 ± 7	37 ± 5
4	40 ± 11	38 ± 6	41 ± 7
6	38 ± 9	38 ± 6	40 ± 8
8	40 ± 8	39 ± 6	40 ± 7
10	38 ± 10	39 ± 5	38 ± 7

tissues. No symptoms were noted related to the procedure except for one volunteer who reported the noise and the smell as the only causes of discomfort.

DISCUSSION

The literature on the local anaesthetic effect of dental lasers is sparse. It has been reported that cavity preparation using lasers can be achieved without local anaesthesia.[1,2] However, it is not clear whether laser preconditioning is necessary or effective as a local anaesthetic method. This study was aimed to assess the response of the dental pulp to preoperative conditioning with the laser when set at its minimum power setting. It has been shown that pulpal anaesthesia is considered achieved if the maximum reading of 80 on the pulp tester does not generate discomfort.[9-11] The data obtained from the current study would suggest that laser preconditioning did not achieve a local anaesthetic effect, as all volunteers reported a response to an electronic pulp stimulus at an average reading of 38.7 immediately following laser preconditioning. Indeed, the responses of the dental pulp following laser and both control treatments were similar (Table 1). The electronic pulp tester is technique sensitive and has a number of limitations and requirements. All EPT measurements undertaken in this study were performed by the same operator to ensure that the application method involving isolation technique and application site was consistent. Since the different conducting media influence the responses gained from electronic pulp testing,[12] the same conducting medium was used throughout the trial to ensure that a maximum steady current passed from electrode to tooth.

The number of volunteers was small in this study. There is a lack of relevant data in the literature on which to base a power calculation to inform a sample size. Even with small numbers, it is clear that preconditioning does not influence the outcome of electronic pulp testing. Nevertheless, while preconditioning did not affect the response to electronic pulp testing, we cannot be sure that it has no effect on reducing discomfort during cavity preparation.

Matsuda and others[13] reported that several types of laser (Ar^+ Nd:YAG and He-Ne) have the potential to block nerve conduction, likely through sodium and potassium channels "by means of heat or photochemical effects." Such reductions appear to be power, wavelength, and time dependent. Furthermore, low-intensity lasers[14] and high-intensity lasers[15] have been used for the treatment of dentin hypersensitivity, but the exact mechanism by which lasers might affect pulp is still not known, and the safety of the procedure is still being investigated. Although there was no effect in the present study on electronic pulp test response, the possibility remains that continued laser irradiation, particularly at higher power, may have a local anaesthetic effect during cavity preparation. In order to determine if laser preconditioning produces any local anaesthetic effect, a further clinical trial with an active intervention is needed. A number of factors that may influence pulp sensitivity have been identified, such as the clinician's approach to the patient[16] and hypertension.[17] In the current study, we were careful to adopt a calming approach to the patients, and no patients diagnosed with hypertension were included in the study.

The VAS, which is widely used in pain studies,[18] was chosen to assess pain intensity and to allow parametric analysis of the data. All the volunteers recorded no discomfort during and at the end of all three visits. One subject reported the noise and the smell from the electric motors of the curing light and the laser units as being unpleasant.

CONCLUSIONS

- Laser preconditioning of teeth prior to cavity preparation did not provide a local anaesthetic effect in adults as measured with an electronic pulp tester. There were no significant differences

in pulpal response following laser irradiation compared to control treatments.
- Preconditioning did not result in any pain or noticeable symptoms at any time in the trial for both teeth and soft tissues regardless of the type of pretreatment as assessed by subjective measures, except for one subject who reported the noise and the smell as unpleasant.

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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Four-year Randomized Clinical Trial to Evaluate the Clinical Performance of a Glass Ionomer Restorative System

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Clinical Relevance

The clinical effectiveness of Equia and Gradia Direct Posterior was acceptable in Class 1 and Class 2 cavities subsequent to four-year evaluation.

SUMMARY

Objective: The aim of this study was to evaluate the clinical performance of a glass ionomer restorative system compared with a microfilled hybrid posterior composite in a four-year randomized clinical trial.

Methods: A total of 140 (80 Class 1 and 60 Class 2) lesions in 59 patients were either restored with a glass ionomer restorative system (Equia, GC, Tokyo, Japan), which was a combination of a packable glass ionomer (Equia Fil, GC) and a self-adhesive nanofilled coating (Equia Coat, GC), or with a microfilled hybrid composite (Gradia Direct Posterior, GC) in combination with a self-etch adhesive (G-Bond, GC) by two experienced operators according to the manufacturer's instructions. Two independent examiners evaluated the restorations at baseline and at one, two, three, and four years postrestoration according to the modified US Public Health Service criteria. Polyvinyl siloxane impression negative replicas at each recall were observed under scanning electron microscopy (SEM) to evaluate surface characteristics. The statistical analyses were carried out with McNemar, Pearson Chi-square, and Cochran *Q*- tests ($p<0.05$).

Results: After four years, 126 (76 Class 1 and 50 Class 2) restorations were evaluated in 52 patients, with a recall rate of 88.1%. None of the restorations showed trends to downgrade in anatomical form, secondary caries, surface texture, postoperative sensitivity, and color

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match ($p>0.05$). Significant differences in marginal adaptation and discoloration were found at four years compared to baseline for both restorative materials for Class 1 and Class 2 restorations ($p<0.05$). Only one Class 2 Equia restoration was missing at three years (3.9%), and another one was missing at four years (7.7%) ($p>0.05$). SEM evaluations were in accordance with the clinical findings.

Conclusions: The use of both materials for the restoration of posterior teeth exhibited a similar and clinically successful performance after four years.

INTRODUCTION

Over the course of the last several decades, an increasing variability of dental restorative materials has conquered the market. The concepts in restorative dentistry are also changing, and adhesive dentistry has steadily gained in importance. Today, the modern operative dentistry focus is on minimal removal of tooth tissue and on application of adhesive restorative materials that possibly perform therapeutic action on demineralized dentin. Those requirements are perfectly matched by glass ionomer cements (GICs).[1-3]

GICs are clinically attractive dental materials and have certain unique properties that make them useful as restorative and adhesive materials. Since the introduction of GICs by Wilson and Kent,[4] many modifications of these materials have been performed over the years. Despite having advantages such as adhesion to moist tooth structure and base metals, anticariogenic properties due to the release of fluoride, thermal compatibility with tooth enamel, and biocompatibility and low toxicity, GICs suffer from low fracture toughness and a higher rate of occlusal wear compared to other restorative materials, such as amalgam and modern composite restorative materials.[1-3,5]

Today, highly viscous GICs achieve superior physical properties compared to traditional GICs by optimizing polyacid and particle size distribution, resulting in a high cross-linkage in the GIC matrix.[1] Recently, a new restorative concept has been marketed (Equia, GC, Tokyo, Japan): a system consisting of a highly viscous conventional GIC (Equia Fil, formerly known as Fuji IX GP Extra) combined with a novel nanofilled coating material (Equia Coat, formerly known as G-Coat Plus).[6] This self-adhesive, nanofilled resin coating, which provides a high hydrophilicity combined with an extremely low viscosity, accounts for the perfect seal of a GIC surface. Compounded nanofillers are thereby intended to protect the system against abrasive wear. This is of importance in the first months until the GIC is completely matured and able to withstand the intraoral stresses. The coating acts as a glaze, further increasing the esthetic properties.[1,6,7]

Reviews have indicated that to date no study regarding the long-term clinical success of this new restorative system has been reported. Therefore, the purpose of this study was to evaluate the clinical performance of this highly viscous conventional GIC restorative system (Equia System/GC) and a microfilled hybrid resin composite (Gradia Direct Posterior/GC) as a comparison material using modified US Public Health Service (USPHS) criteria. The null hypothesis was that under the conditions of this study there would be no difference in the clinical performance of the two restorative materials for the criteria assessed.

METHODS AND MATERIALS

In this four-year randomized controlled clinical study, the GIC restorative material (Equia System, GC, Tokyo, Japan) and a microfilled hybrid composite resin (Gradia Direct Posterior, GC, Tokyo, Japan) were compared. These materials are described in Table 1.

Study Population and Sample Size

Following the approval of the study by the Ethical Committee of Hacettepe University, Ankara, Turkey (protocol HEK 09/112-10), a group of patients seeking routine dental care and recruited by the Hacettepe University, School of Dentistry, Department of Restorative Dentistry were screened, and a total of 59 patients satisfying the inclusion and exclusion criteria were selected. Inclusion criteria were as follows: a patient presenting with 1) a need for at least two but not more than four posterior tooth-colored restorations; 2) the presence of teeth to be restored in occlusion; 3) teeth that were symptomless and vital; 4) a normal periodontal status; and 5) a good likelihood of recall availability. Exclusion criteria were as follows: 1) partly erupted teeth; 2) absence of adjacent and antagonist teeth; 3) poor periodontal status; 4) adverse medical history; and 5) potential behavioral problems. The average age of patients was 24 years (range, 15-37 years). All patients participated voluntarily and were required to provide written informed consent.

Table 1: *Description of Materials Used in this Study*

Material	Type	Manufacturer	Composition
Equia Fil	Conventional glass ionomer cement	GC, Tokyo, Japan	Powder: 95% strontium fluoroalumino-silicate glass, 5% polyacrylic acid
			Liquid: 40% aqueous polyacrylic acid
Equia Coat	Low-viscosity nanofilled surface coating resin	GC, Tokyo, Japan	50% Methyl methacrylate, 0.09% camphorquinone
Gradia Direct Posterior	Microfilled hybrid composite	GC, Tokyo, Japan	Urethane dimethacrylate co-monomer matrix, silica, pre-polymerized fillers, fluoroalumino-silicate glass
G-Bond	All-in-one dentin/enamel bonding agent	GC, Tokyo, Japan	40% Acetone, 20% distilled water, 15% 4-methacryloxy-ethyltrimellitate anhydride, 10-20% urethane dimethacrylate, 10% triethyleneglycoldimethacrylate

Restoration Placement

Two experienced dentists placed 80 Class 1 and 60 Class 2 restorations, totaling 140 restorations in 59 patients (Table 2). The filling materials Equia or Gradia Direct Posterior were randomized over these two cavity groups using a table of random numbers.[8] Before treatment, initial periapical radiographs of the teeth to be treated were taken and vitality test scores were recorded. Cavities were prepared using diamond fissure burs (MS Rounded Edged Cylinder Bur [835R-012-4], Diatech, Heerbrugg, Switzerland) at high speed with water-cooling. Hand instruments and slow-speed tungsten carbide burs were used to remove the caries. Local anesthesia was applied to patients complaining about pain or sensitivity to prevent discomfort during restorative procedures.[9] Conservative cavity design was used according to the principals of minimal invasive dentistry. None of the cavity preparations involved one or more cusps. All of the gingival margins included sound enamel. No beveling was applied to the cavity walls. $CaOH_2$ cavity liner (Life Regular Set, Kerr Corporation, Romulus, MI, USA) was applied where needed as base material. An ivory type matrix system (Hahnenkratt, Königsbach-Stein, Germany) was used for Class 2 cavities. All of the cavities were either restored with glass ionomer restorative system (Equia, GC), which is a combination of a packable glass ionomer (Equia Fil, GC) and a self-adhesive nanofilled coating (Equia Coat, GC) or microfilled hybrid composite (Gradia Direct Posterior, GC) in combination with a self-etch adhesive (G-Bond, GC), according to the manufacturer's instructions.

Glass Ionomer Restorations

The dentin and enamel of cavities were conditioned with 20% polyacrylic acid for 20 seconds (Cavity Conditioner, GC), washed, and briefly dried. Equia Fil was injected into the cavity. Isolation was maintained using cotton rolls and a saliva ejector. After the passage of the manufacturer's recommended setting time of 2.5 minutes, the restoration was trimmed and polished wet using high-speed fine diamonds (Diatech, Swiss Dental, Heerbrugg, Switzerland). After the restoration was briefly dried, Equia Coat was applied and photocured for 20 seconds using a photo-curing light (Radii Plus, SDI, Bayswater, Australia).

Composite Resin Restorations

After the enamel and dentin were conditioned with G-Bond (GC) using a microtip applicator, left undisturbed for five to 10 seconds, and then dried thoroughly for five seconds with oil-free air under air pressure, Gradia Direct Posterior resin was applied with the incremental technique (2-mm thick layers) and light-cured for 20 seconds. Finally, the restoration was shaped with finishing diamonds and silicon instruments (Hi Luster Plus Polishing System, KerrHave, Bioggio, Switzerland).

Table 2: *Distribution of the Restorative Materials Among Dental Arches*

Restorative Materials	Maxillary				Mandibular				Total
	Premolar		Molar		Premolar		Molar		
	Class 1	Class 2	Class 1	Class 2	Class 1	Class 2	Class 1	Class 2	
Equia	8	8	10	8	4	8	18	6	70
Gradia Direct Posterior	9	7	9	9	5	5	17	9	70
Total	17	15	19	17	9	13	35	15	140

Table 3: *Recall Rates of Patients*

Restorative Materials	Baseline		1 Year		2 Years		3 Years		4 Years	
	Class 1	Class 2	Class 1	Class 2	Class 1	Class 2	Class 1	Class 2	Class 1	Class 2
Equia	33	26	32	25	33	22	33	21	32	20
Gradia Direct Posterior	33	26	32	25	33	22	33	20	32	20
Total	59		57		55		53		52	
(%)	100		96.6		93.2		89.8		88.1	

One week after restoration placement (baseline), patients were recalled and restorations were examined clinically. Direct clinical evaluation of restorations was performed using the modified USPHS criteria[10] by two independent investigators using mirrors, probes, bitewing radiographs, and intraoral photographs. Patients were recalled at one, two, three, and four years for assessments of the restorations using the same criteria as at baseline. At each recall, the same two calibrated evaluators, who were blinded to the restoratives used for cavities and patients, examined the restorations. When disagreement occurred during the evaluation, the final decision was made by consensus of both examiners.

Scanning Electron Microscopy (SEM) Analysis

At each recall, impressions of each air-dried, cotton-roll–isolated tooth were taken from one patient selected randomly from each group with polyvinyl siloxane impression material and used as negative replicas for morphological observation with SEM. The replicas were gold sputter-coated and observed under SEM (JSM-6400 SEM, JEOL, Tokyo, Japan) at 50× and 200× magnifications for surface morphology and marginal integrity.

Statistical Analysis

Statistical analysis was performed with SPSS 15.0 software. To compare the performance of restorative materials according to USPHS criteria over the study period, the McNemar test was used. Within each material group, further analysis was done using the Pearson Chi-square test to distinguish the differences between different cavity types for marginal adaptation and marginal discoloration. The Cochran *Q*-test was then used to compare the marginal adaptation and marginal discoloration scores of each material with baseline scores for each cavity type to evaluate the changes of each dependent group by the time. The level of significance was set at $p<0.05$ for all tests.

RESULTS

After four years, 126 (76 Class 1 and 50 Class 2) restorations in 52 patients were evaluated and scored according to the USPHS criteria. The recall rate of the patients was 88.1% at four years (Table 3). The overall clinical recall rate of restorations at the four-year recall was 90%. Fourteen (four Class 1 and 10 Class 2) original restorations could not be evaluated at four years because seven patients (11.9%) had moved away. Table 3 also shows the number of recalls at one, two, three, and four years.

After four years, success rates for Class 1 Equia, Class 1 Gradia Direct Posterior, and Class 2 Gradia Direct Posterior were 100%, whereas the failure rate was 7.7% for only Class 2 Equia restorations. Only one Class 2 restoration had to be replaced as a result of marginal fracture at three years and one at four years. No significant change over time was found for the anatomical form, color match, secondary caries, postoperative sensitivity, surface texture, and retention for either restorative material ($p>0.05$).

Clinically acceptable (Bravo) moderate marginal discolorations were noted for both materials at one year (three [7.7%] Class 1 and two [6.9%] Class 2 Equia restorations and three [7.7%] Class 1 and two [6.9%] Class 2 Gradia Direct Posterior restorations), two years (three [7.7%] Class 1 and two [7.7%] Class 2 Equia restorations and five [12.9%] Class 1 and five [18.6%] Class 2 Gradia Direct Posterior restorations), three years (three [7.7%] Class 1 and two [8%] Class 2 Equia restorations and five [12.9%] Class 1 and five [18.6%] Class 2 Gradia Direct Posterior restorations), and four years (two [5.3%] Class 1 and two [8.4%] Class 2 Equia restorations and five [13.2%] Class 1 and five [19.3%] Class 2 Gradia Direct Posterior restorations) (Table 4a,b). Equia Class 1 restorations exhibited a significant difference starting at two years, whereas Class 2 restorations showed significant differences starting at three years ($p<0.05$) for marginal discolorations. Both Class 1 and Class 2 Gradia Direct Posterior restorations exhibited significant changes starting

Table 4a: *Clinical Evaluation Scores of the Equia Restorations at Baseline and at One, Two, Three, and Four Years*

USPHS Criteria	USPHS Scores	Equia, No. (%)									
		Class 1 (N=40)					Class 2 (N=30)				
		Baseline	1 Year	2 Years	3 Years	4 Years	Baseline	1 Year	2 Years	3 Years	4 Years
Anatomical form	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	26 (100)	25 (100)	24 (100)
	Bravo	0	0	0	0	0	0	0	0	0	0
	Charlie	0	0	0	0	0	0	0	0	0	0
Color match	Alfa	40 (100)	39 (100)	38 (97.4)	38 (97.4)	37 (97.3)	30 (100)	29 (100)	26 (100)	25 (100)	24 (100)
	Bravo	0	0	1 (2.6)	1 (2.6)	1 (2.7)	0	0	0	0	0
	Charlie	0	0	0	0	0	0	0	0	0	0
Marginal discoloration	Alfa	40 (100)	36 (92.3)	36* (92.3)	36* (92.3)	36* (94.7)	30 (100)	27 (93.1)	24 (92.3)	23* (92)	22* (91.6)
	Bravo	0	3 (7.7)	3 (7.7)	3 (7.7)	2 (5.3)	0	2 (6.9)	2 (7.7)	2 (8)	2 (8.4)
	Charlie	0	0	0	0	0	0	0	0	0	0
Marginal adaptation	Alfa	40 (100)	33 (84.6)	31* (79.5)	31* (79.5)	31* (81.5)	30 (100)	25 (86.2)	22 (84.6)	19* (76)	18* (75)
	Bravo	0	6 (15.4)	8 (20.5)	8 (20.5)	7 (18.5)	0	4 (13.8)	4 (15.4)	6 (24)	6 (25)
	Charlie	0	0	0	0	0	0	0	0	0	0
Secondary caries	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	26 (100)	25 (100)	24 (100)
	Charlie	0	0	0	0	0	0	0	0	0	0
Postoperative sensitivity	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	26 (100)	25 (100)	24 (100)
	Bravo	0	0	0	0	0	0	0	0	0	0
	Charlie	0	0	0	0	0	0	0	0	0	0
Surface texture	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	26 (100)	25 (100)	24 (100)
	Bravo	0	0	0	0	0	0	0	0	0	0
	Charlie	0	0	0	0	0	0	0	0	0	0
Retention	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	26 (100)	25 (96.1)	24 (96)
	Charlie	0	0	0	0	0	0	0	0	1 (3.9)	1 (4)

Abbreviation: USPHS, US Public Health Service.
** Indicates significant difference in comparison with baseline according to Cochrane Q-test ($p<0.05$).*

at two years ($p<0.05$) (Table 4b). However, the McNemar test indicated no significant difference between the two restorative materials in terms of marginal discoloration at any recall period ($p>0.05$) (Table 5). In addition, there were no significant differences between the marginal discoloration scores of Class 1 and Class 2 cavities for either restorative material at one, two, three, and four years ($p>0.05$) (Table 6).

Moderate marginal adaptation was also noted for both materials at one year (six [15.4%] Class 1 and four [13.8%] Class 2 Equia restorations and seven [18%] Class 1 and five [17.3%] Class 2 Gradia Direct Posterior restorations), two years (eight [20.5%] Class 1 and four [15.4%] Class 2 Equia restorations and 10 [25.7%] Class 1 and eight [29.7%] Class 2 Gradia Direct Posterior restorations), three years (eight [20.5%] Class 1 and six [24%] Class 2 Equia restorations and 10 [25.7%] Class 1 and eight [29.7%] Class 2 Gradia Direct Posterior restorations), and four years (seven [18.5%] Class 1 and six [25%] Class 2 Equia restorations and 10 [26.4%] Class 1 and eight [30.8%] Class 2 Gradia Direct Posterior restorations) (Table 4a,b). Equia Class 1 restorations showed moderate changes starting at two years, whereas Class 2 restorations showed changes starting at three years ($p<0.05$). Gradia Direct Class 1 restorations exhibited significant changes starting at one year, whereas Class 2 restorations did at two years ($p<0.05$) (Table 4b). The differences between two restorative materials were not statistically significant at any recall period in terms of marginal adaptation ($p>0.05$) (Table 5). Additionally, no significant difference was observed between Class 1 and Class 2 cavities within each restorative material at one, two, three, and four years ($p>0.05$) (Table 6).

No patient at any time interval experienced pain or sensitivity from the restored teeth, and no incidence of secondary caries was observed.

The SEM observations of one representative GIC restoration and one composite resin are shown in Figures 1 and 2, respectively. The SEM evaluation of the replicas of the restorations demonstrated the occlusal surface characteristics. Both materials

Table 4b: *Clinical Evaluation Scores of the Gradia Direct Posterior Restorations at Baseline and at One, Two, Three, and Four Years*

USPHS Criteria	USPHS Scores	Gradia Direct Posterior, No. (%)									
		Class 1 (N=40)					Class 2 (N=30)				
		Baseline	1 Year	2 Years	3 Years	4 Years	Baseline	1 Year	2 Years	3 Years	4 Years
Anatomical form	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	27 (100)	27 (100)	26 (100)
	Bravo	0	0	0	0	0	0	0	0	0	0
	Charlie	0	0	0	0	0	0	0	0	0	0
Color match	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	27 (100)	27 (100)	26 (100)
	Bravo	0	0	0	0	0	0	0	0	0	0
	Charlie	0	0	0	0	0	0	0	0	0	0
Marginal discoloration	Alfa	40 (100)	36 (92.3)	34* (87.1)	34* (87.1)	33* (86.8)	30 (100)	27 (93.1)	22* (81.4)	22* (81.4)	21* (80.7)
	Bravo	0	3 (7.7)	5 (12.9)	5 (12.9)	5 (13.2)	0	2 (6.9)	5 (18.6)	5 (18.6)	2 (19.3)
	Charlie	0	0	0	0	0	0	0	0	0	0
Marginal adaptation	Alfa	40 (100)	32* (82)	29* (74.3)	29* (74.3)	28* (73.6)	30 (100)	24 (82.7)	19* (70.3)	19* (70.3)	18* (69.2)
	Bravo	0	7 (18)	10 (25.7)	10 (25.7)	10 (26.4)	0	5 (17.3)	8 (29.7)	8 (29.7)	8 (30.8)
	Charlie	0	0	0	0	0	0	0	0	0	0
Secondary caries	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	27 (100)	27 (100)	26 (100)
	Charlie	0	0	0	0	0	0	0	0	0	0
Postoperative sensitivity	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	27 (100)	27 (100)	26 (100)
	Bravo	0	0	0	0	0	0	0	0	0	0
	Charlie	0	0	0	0	0	0	0	0	0	0
Surface texture	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	27 (100)	27 (100)	26 (100)
	Bravo	0	0	0	0	0	0	0	0	0	0
	Charlie	0	0	0	0	0	0	0	0	0	0
Retention	Alfa	40 (100)	39 (100)	39 (100)	39 (100)	38 (100)	30 (100)	29 (100)	27 (100)	27 (100)	26 (96)
	Charlie	0	0	0	0	0	0	0	0	0	0

Abbreviation: USPHS, US Public Health Service.
** Indicates significant difference in comparison with baseline according to Cochrane Q-test ($p<0.05$).*

exhibited successful surface characteristics, with the absence of significant wear, surface porosities, cracks, and marginal gap formation during the four-year evaluation.

DISCUSSION

The advantages of GICs as restorative materials are clearly reflected by the literature.[11] The most significant advantages of these materials include their chemical adhesion to dentin and enamel, release of fluoride, the high tolerance of tissues, and biocompatibility. However, the lack of resistance to abrasion and their poor esthetics are two of the reasons why they are not used frequently by most professionals. In the late 1990s, the conventional GIC was overtaken by the highly viscous GIC, which has a faster setting time and notably higher strengths.[11] A new generation of highly dispersed nanofilled resin coating that has been recently introduced claims to increase the resistance of the GIC and improve marginal sealing and the esthetics

Table 5: *McNemar Test Results: Comparisons of Equia and Gradia Direct Posterior Restorations Are Presented for Marginal Discoloration and Marginal Adaptation*[a]

Criteria Assessed	Material	Evaluation Periods, *p*-Value			
		1 Year	2 Years	3 Years	4 Years
Marginal discoloration	Equia	1.00	1.00	1.00	0.637
	Gradia Direct Posterior	1.00	0.775	0.775	0.759
Marginal adaptation	Equia	1.00	0.845	0.985	0.764
	Gradia Direct Posterior	1.00	1.00	1.00	0.637

[a] *The level of significance was set at* $p < 0.05$.

Table 6: *Pearson Chi-square Test Results Present the Differences Between Cl1 and Cl2 Cavities Within Each Material Group for Marginal Discoloration and Marginal Adaptation*[a]

Criteria Assessed	Cavity Type	Evaluation Periods, *p*-Value			
		1 Year	2 Years	3 Years	4 Years
Marginal discoloration	Class 1	1.00	0.5	0.5	0.5
	Class 2	1.00	0.25	0.25	0.25
Marginal adaptation	Class 1	1.00	0.5	0.5	0.5
	Class 2	1.00	0.125	0.5	0.5

[a] *The level of significance was set at* $p < 0.05$.

of the restoration.[7] Several *in vitro* tests had proved the positive influence of this coating on the fracture strength and the early wear on GIC.[12-14]

Although a variety of clinical trials with different types of GICs as permanent restorative materials were carried out in primary molars only,[15-17] very few prospective studies or long-term trials are published about GICs in permanent premolars and molars.[18] Retrospective studies primarily reported disappointing results when GICs were applied in average cavities and slightly better results when minimum intervention cavities were restored with conventional GIC.[18]

This clinical trial was conducted on permanent posterior teeth both in Class 1 and Class 2 caries lesions of young patients with the average age of 24 years. The clinical efficacy of the systems tested was determined by evaluating the anatomical form, color match, marginal discoloration, marginal adaptation, secondary caries occurrence, and retention at one year and annually for four years. In most of the restorations evaluated, only a few changes were noted from baseline to the four-year evaluation visit.

The American Dental Association requires a retention rate of at least 90% of the restorations placed after 18 months to obtain full acceptance.[19] In this study, at the end of two years, the success rates for both of the restorative materials were 100%. Because of the retention loss of two Equia Class 2 restorations, the overall clinical success rate was 97.1% (100% for Class 1 and 92.3% for Class 2 restorations) for the Equia System after four years. Therefore, both restorative materials evaluated in the present study demonstrated good clinical performance and full acceptance. The results of this study disagreed with those reported by Hickel and others,[20] who reviewed annual failure rates of stress-bearing cavities in posterior primary teeth and determined median annual failure rates of 0-25.8% for GIC. They reported fractures to represent the main reason for failure in Class 2 restorations, with a higher load situation compared to Class 1 restorations. Although a variety of clinical trials with GICs as permanent restorative materials were carried out in Class 1 cavities,[11-13] there are limited data showing the performance of GIC in Class 2 cavities. As the Equia restorative system was introduced with the claim that it could be used both in moderate Class 1 and Class 2 cavities, in this study, the intent was to evaluate the performance of this restorative system separately for the restoration of Class 1 and Class 2 cavities.

The clinical assessment of the loss of anatomical form of restorations is essentially an indication of the

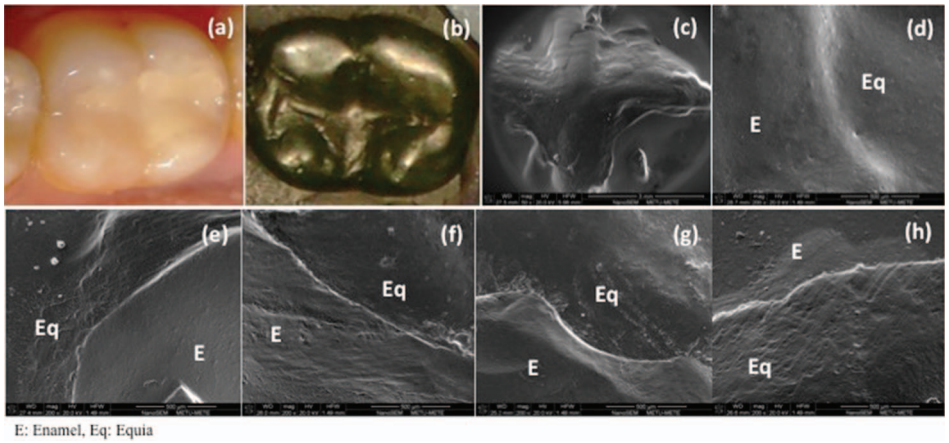

Figure 1. *A representative Equia Cl1 restoration. This figure includes a representative clinical picture (a), a negative replica (b); a SEM photomicrograph of the occlusal view of the restoration, 50× (c); and the occlusal contact area shared by enamel and restoration at baseline, 200× (d), 12 months 200× (e), 24 months 200× (f), 36 months 200× (g), and 48 months 200× (h). E = enamel; Eq = Equia.*

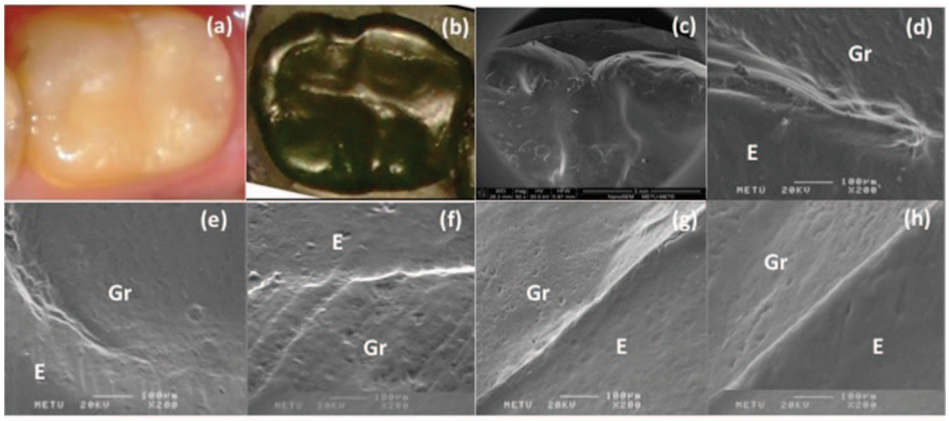

Figure 2. *A representative Gradia Direct Posterior CI1 restoration. This figure includes a representative clinical picture (a); a negative replica (b); a SEM photomicrograph of the occlusal view of the restoration 50× (c); and the occlusal contact area shared by enamel and restoration at baseline 200× (d), 12 months 200× (e), 24 months 200× (f), 36 months 200× (g), and 48 months 200× (h). E = enamel; Gr = Gradia Direct Posterior.*

restored surface exhibiting morphological alterations due to wear. The alpha scores of the criteria anatomical form and retention in almost all restorations throughout the study period showed that the wear on the restoration morphology and retention was barely visible to the naked eye. Therefore, both of the materials have smooth surface textures, successful anatomic form, and retention. SEM examinations also supported the clinical observations.

The absence of failures due to secondary caries after four years could be due to good oral hygiene status of the patients. The time and attention devoted to the restoration placement techniques and the clinically acceptable properties of the restorative materials that minimize the hydrostatic dentin fluid movement might explain the lack of postoperative sensitivity after four years. Postoperative sensitivity has been attributed to several factors, such as operative trauma, desiccation, leakage, and other sources.[21-23] The ability of the coating in Equia restorations and the adhesive in the Gradia Direct Posterior restorations played roles in reducing sensitivity.

In contrast to the previous reports,[18] in the present study, Class 2 restorations recorded as not having lost the interproximal contact could be explained by the special attention given to the appropriate use of the matrix in building up the proximal part of the restorations. Only two retention losses in Class 2 Equia restorations may be related to cyclic stress resulting in occlusal-proximal marginal fracture, weakening the proximal points in the evolving periods of three and four years.

In the present study, marginal discoloration was moderate and was observed in few restorations. The staining appeared only as superficial discoloration (Bravo score), and although it was not significantly different, it mainly occurred in Gradia Direct Posterior restorations rather than in the Equia restorations. This might be due to the adhesive system used. The application of the self-etch adhesive system might have led to a compromise concerning adhesion to the cavosurface margins. Studies have shown that self-etch adhesive systems and the all-in-one adhesives were less effective than total etch systems in terms of dentin and enamel bond strength.[24-26] The discolorations could also be due to food consumption or related to pigment absorption from dietary habits and antagonist teeth during mastication.

Color match was within the alpha range and the color stability of both restorative materials was good, indicating no mismatch in color, shade, or translucency between the restorations and adjacent teeth during the four years of clinical service.

There are only a few clinical studies describing the clinical performance of the Equia System. These clinical reports are mainly abstracts from research meetings. Friedly and others[27] examined retrospectively the performance of Fuji IX GP Extra in posterior restorations over 24 months and reported that volume loss was proportional to the cavity size. However, all restorations were assessed as satisfactory. Gurgan and others[28-30] showed that the 12-, 24-, and 36-month performance of the Equia System was similar to that of the resin composite. Turkun and Kanic[31] compared the Equia System to Riva-conventional GIC and found no difference in performance. Basso[32] used Fuji IX GP Extra in a four-center study, and after a mean follow-up of 18 months, 100% of the Class 1 restorations were successful. In a six-center study, Khandelwal and others[33] also evaluated the Equia System after an evaluation time of 24 months. They reported 88.8%

success in Class 1 cavities and a visible and perceptible roughness in 11.5% of restorations with less than 1% of marginal disintegration.

The only published data were reported by Diem and others.[7] They used Fuji IX Extra (Equia Fil) with and without coating (G-Coat Plus/Equia Coat) and also compared the Equia System with a microfine hybrid resin composite in first premolars of 11-12-year-old children with the ART technique. The study was carried out under field conditions. At the end of three years, the color match of all of the restorations was assessed as 'good,' with no significant differences among materials. Moderate marginal staining was noted, and marginal adaptation loss was minimal for all restorations. They concluded that Fuji IX GP Extra either with or without the coating showed acceptable clinical performance but that the application of G-Coat Plus to Fuji IX GP Extra was beneficial in reducing wear in occlusal cavities.

In the present study, Equia System either in Class 1 or Class 2 cavities exhibited significantly good clinical outcome over the observation period of four years. Therefore, the null hypothesis was accepted, because there was not a distinct difference between Equia Fil-GIC and Gradia Direct Posterior microfilled hybrid composite resin. However, further long-term clinical studies are required to confirm the results of the present clinical trial.

This clinical trial furthermore included SEM analyses to demonstrate the micromorphologic features of the restoration surface. The presented observations on the microscopic level have supported the clinical observations of the occlusal surface on marginal adaptation and surface texture.

The introduction of GIC was connected with the hope of being able to replace amalgam. Especially in Europe, this was an interesting aspect because amalgam was more and more disregarded during the 1990s, with many amalgam restorations having been replaced by GICs. Therefore, GICs might turn out to be the more reliable restorative material in minimally invasive dentistry based on adhesive techniques. However, these materials still offer opportunities for improvement. Several attempts to improve their mechanical parameters are still underway, and some forecast a promising future for GIC as a dental filling material with extended indications.

CONCLUSION

The highly viscous GIC restorative system, Equia, and microfilled hybrid resin composite, Gradia Direct Posterior, showed acceptable clinical performance according to modified USPHS criteria assessed in Class 1 and Class 2 cavities over the course of four years.

Conflict of Interest

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

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Sealing Composite With Defective Margins, Good Care or Over Treatment? Results of a 10-year Clinical Trial

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Clinical Relevance

Sealing the defective margins of the composite resin restorations improves the margins of restorations.

SUMMARY

Purpose: The objective of this study was to clinically evaluate sealed composite restorations after 10 years and compare their behavior with respect to controls.

Methods and Materials: The cohort consisted of 20 patients aged 18 to 80 years with 80 composite restorations. All participants in the sealing and no-treatment groups presented with clinical features for the marginal adaptation that deviated from the ideal and were rated Bravo (United States Public Health Service criteria). Composites with Alfa values for the marginal adaptation were used as the positive control.

Results: The marginal adaptation behavior was similar between the sealing and control (+) groups, with a high frequency of Bravo values

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in the 10th year (80% and 51%, respectively). Most of the no-treatment (-) group maintained the Bravo values (91%) for 10 years, although some restorations (9%) progressed to Charlie values. The anatomy parameter differed significantly between the first and 10th years, with deterioration in all three groups ($p<0.05$). The secondary caries parameter had a similar behavior in the three groups ($p>0.05$).

Conclusions: Sealing the margins of the composite resin restorations had no significant effect compared with the control groups, under the conditions of this study. Sealing the restorations substantially improved the marginal staining and marginal adaptation parameters, although by the tenth year they were similar to the group without intervention.

INTRODUCTION

The longevity of restorations is determined by the most common causes of failure: secondary caries, fracture, and marginal adaptation problems.[1,2] Clinicians have traditionally taken a mechanical approach with respect to restorations, replacing those that could have been treated with minimal intervention. This approach saves both time and healthy tissue.[3]

The most frequently reported causes for failure for adhesive restorations are marginal adaptation and retention loss, for which a high potential has been reported due to *in vivo* degradation of the adhesive bonding of the composite resins. Because Classes I through IV have macromechanical retention, retention loss is less clinically evident for those Classes than for Class V restorations. Therefore, marginal adaptation becomes an important sign of the adhesive degradation in composite restorations.[4]

The problems of marginal adaptation have been shown to be associated with the occurrence of secondary caries and the eventual loss of the restoration. All marginal deterioration and loss of substance, either at the expense of tooth structure or due to composite resin degradation, ultimately generates a risk of restoration failure. Gaps larger than 400 μm are associated with caries adjacent to restorations, especially at the gingival margin.[5]

Sealing defective margins of composite restorations appears to be a quick, inexpensive, and simple solution to improve the marginal integrity and prevent further problems. After etching the enamel and surface of the composite, the resin sealant penetrates the surface to adhere micromechanically. The resulting retention values typically support the masticatory functional load. It is also known that the mass of sealant will decrease over time, as the small proportion of filler does not support functional wear. However, the portion that remains on the tooth-restoration interface is maintained.[6]

Repair is defined as the partial removal of a restoration, which allows better examination and diagnosis of the underlying tissue, then the removal of carious tissue, and finally making the composite resin restoration.[7] There is a lack of quality evidence to support repair procedures. However, it is accepted and recommended as a fast, inexpensive, and minimally invasive treatment.[8,9] Also, it is important to remark that the seal is focused on solving small 1-mm minor imperfections on the margins of restorations without removing tooth structure or restoration, or filling a small gap.[10]

There are reports of sealed restorations that have been maintained over time with acceptable clinical results. Sealed restorations are an intervention that improves the marginal adaptation, which then progressively deteriorates over time. One report covered a seven-year timespan for amalgam restorations that were sealed at the margins.[10] Therefore, it is important to understand what occurs to composite restorations after 10 years of follow-up.

The objective of this study was to clinically evaluate sealed composite restorations after 10 years and compare their behavior with respect to controls.

METHODS AND MATERIALS

Study Design

A cohort of 20 patients between 18 and 80 years of age (mean 28.35 years; 35% men, 65% women) with 80 composite restorations (Class I: 45; Class II: 35) (Figure 1) were recruited at the Operative Dentistry Clinic at the Dental School of the University of Chile. All participants in the sealing and no-treatment (-) groups presented with clinical features for the marginal adaptation that deviated from the ideal and were rated Bravo according to the modified United States Public Health Service (USPHS) criteria.[11,12] Composites with Alfa values for marginal adaptation were used as positive controls. All patients signed informed consent forms and completed registration forms. The selection criteria are summarized below.

General inclusion criteria included:

- patients with more than 20 teeth;
- restorations in functional occlusion, with an opposing natural tooth;

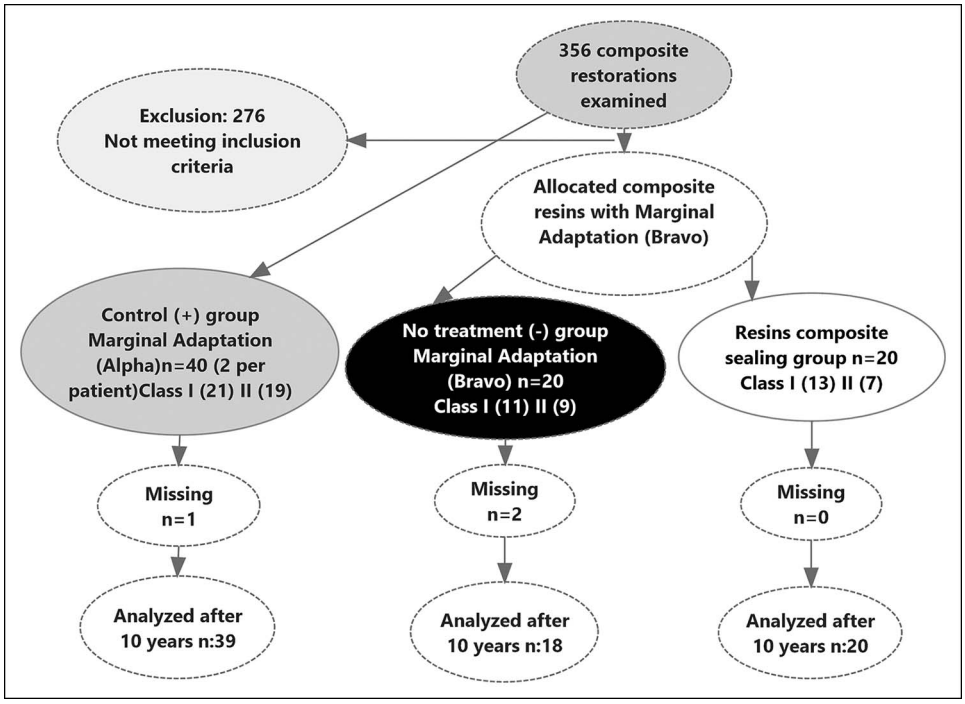

Figure 1. *Flow diagram of study phases and group distributions.*

- asymptomatic restored tooth;
- at least one proximal contact area with a neighboring tooth;
- patients older than 18 years;
- patients who agreed and signed a consent form for participating in the study; and
- area outside of the restoration failure in good condition.

General exclusion criteria included:

- patients with contraindications for regular dental treatment based on their medical history;
- patients with xerostomia or who are taking medication that significantly decreased salivary flow;
- patients at a high risk of caries;
- patients with psychiatric or physical diseases, which interfered with oral hygiene; and
- resin-based restorations with localized marginal deficiencies >1 mm and/or secondary caries or major defects adjacent to the restorations.

Inclusion criteria for the allocated groups (Figure 1) included patients with localized marginal defects of less than 1 mm (Bravo Ryge criteria) on composite restorations that were clinically judged to be suitable for sealing according to the USPHS criteria.

Inclusion criteria for the positive control group (Figure 1) included composite resins with Alfa values for the marginal adaptation criteria.

Treatment Group Criteria

Fifty-eight patients and 356 restorations were initially evaluated and assigned in accordance with the modified USPHS criteria,[11] and 80 were selected based on the inclusion criteria. Restorations with marginal defects (>0.5 mm and <1 mm) and/or marginal staining (Bravo) were randomly assigned to the sealing (n=20) or no-treatment (n=20) groups. The randomization was performed by the PASS software (NCSS, LLC, Kaysville, UT, USA). Patients who had at least four Class I or Class II posterior composite restorations were examined. Two restorations with <1 mm defects (Bravo) longitude corroborated by the North Carolina 15 periodontal probe (Hu-Friedy Mfg. Co, Chicago, IL,USA) at the margins were randomly assigned to be sealed or left untreated (-). Two other composite restorations that had excellent margins (Alfa) acted as a positive control. The patient was considered the statistical unit in this study (n=20).

Restoration Assessment

The quality of the restorations was scored in accordance with the modified USPHS criteria. The Cohen kappa interexaminer coefficient was 0.74 at the first year and 0.87 after 10 years for two examiners (JM and EF) who underwent calibration exercises each year. In the first, second, third, fourth, fifth, and 10th year, the examiners independently assessed the restorations for anatomic form,

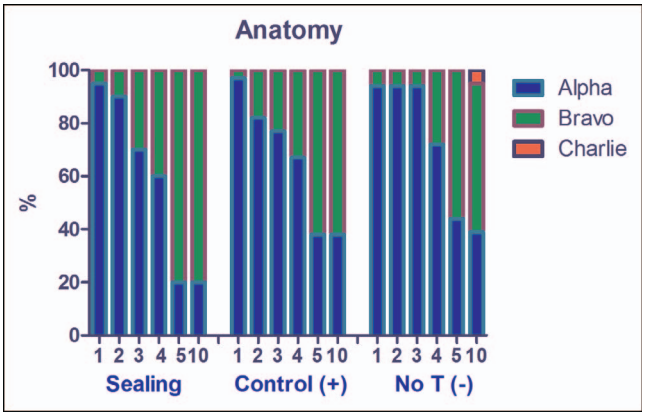

Figure 2. *Anatomy observations separated by groups and quality evaluation expressed as a percentage of USPHS/Ryge criteria per year.*

secondary caries, marginal staining, and marginal adaptation, both directly by tactile and visual examinations with mouth mirror number five and explorer number 23 (Hu Friedy) and indirectly by radiographic examination (bitewing). A third clinician (GM), who also underwent the calibration exercises, made the final decision if a difference was recorded between the two examiners and an agreement could not be reached.

Treatment Groups

Sealing—For this group, defective areas were acid etched with 35% phosphoric acid for 15 seconds, and then a resin-based sealant (Clinpro Sealant, 3M ESPE, St Paul, MN, USA) was applied to the area and polymerized with a photocuring unit (Curing Light 2500, 3M ESPE) for 40 seconds. Rubber dam isolation was used for this procedure.

Positive Control—Composite resins with Alfa values for the marginal adaptation criteria were used as the positive control and were made with resin composite (Filtek Supreme, 3M ESPE).

No Treatment—Composite resin restorations that had marginal defects, but were clinically acceptable, did not receive treatment.

Statistical Analysis

The sample size was defined by setting a beta error rate of 0.2. A Wilcoxon test was performed for comparisons between the same groups with a significance level of 0.05. A Friedman test was utilized for multiple comparisons between different years of the same group. The Kaplan-Meier survival curves were calculated, and Mantel-Cox test was used for the comparison of the curves. The statistical analysis was performed using SPSS 21.0 (IBM, New York, NY, USA) and GraphPad Prism version 6.00 for Windows (GraphPad Software, La Jolla, CA, USA, www.graphpad.com).

Caries Risk Assessment

A graphical computer program (Cariogram, Malmö Högskola, Malmo, Sweden) was used to assess the risk of caries for the individual patients.[13] The results also indicated where targeted actions to improve the situation would have the best effect. This analysis was performed only for select patients from the study, according to the recommendations of the local ethics committee.

RESULTS

The recall of this cohort of patients at 10 years was 100%. The distribution according to caries risk patients was medium caries risk 80% (n=18) and low risk 20% (n=2); three missing restorations (dropout=3.75%) were lost by orthodontic treatment. Due to local ethics committee requirements at the time this trial was initiated, including high caries risk patients proved to be impossible because the sealing was considered an experimental treatment at that time. This was considered a study limitation.

The anatomic criteria showed a similar trend in the three groups, with primarily Alfa values after the first year, which at the 10th year had deteriorated to Bravo values of 80%, 62%, and 56% for the sealing, control (+), and no-treatment (-) groups, respectively. The no-treatment (-) group also had a 5% frequency of Charlie values (Figure 2).

The secondary caries behavior was similar between the groups, with 11% of the sealing and no-treatment (-) groups having Charlie values in the 10th year, while the control group (+) had only Alfa values (Figure 3).

Regarding marginal staining, the frequency of Bravo values was similar in the sealing and no-treatment groups after 10 years, with 65% and 56%, respectively; only 15% of the control (+) group had Bravo values. The frequency of Charlie values was similar for all three groups in the 10th year, fluctuating between 5% and 6% (Figure 4).

The marginal adaptation behavior was also similar between the sealing and control (+) groups, with a high frequency of Bravo values in the 10th year (80% and 51%, respectively). Most of the no-treatment (-) group maintained the Bravo values (91%) for 10 years, although some restorations (9%) progressed to Charlie values (Figure 5).

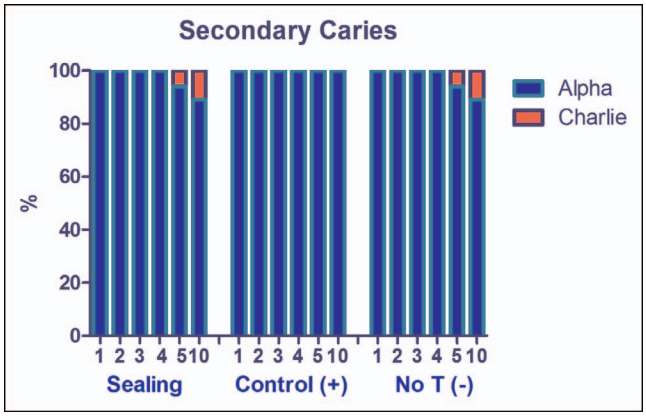

Figure 3. *Secondary caries observations separated by groups and quality evaluation expressed as a percentage of USPHS/Ryge criteria per year.*

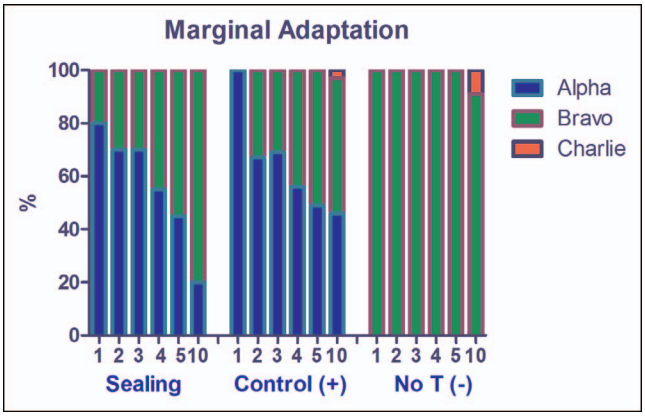

Figure 5. *Marginal adaptation observations separated by groups and quality evaluation expressed as a percentage of USPHS/Ryge criteria per year.*

Utilizing Wilcoxon tests to compare the parameters between the first and 10th years, the marginal adaptation parameter was significantly different in two groups, with deterioration occurring over time. The marginal adaptation behavior in the sealing and control (+) groups had similar levels of deterioration in the 10th year ($p<0.05$), and there was a statistically significant difference between the first and 10th years ($p<0.05$), whereas there was no significant difference over the 10 years for the no-treatment (-) group ($p>0.05$). The anatomy parameter differed significantly between the first and 10th years ($p<0.05$), with deterioration in all three groups. There were no statistically significant differences between the first and the 10th year for the secondary caries parameter ($p>0.05$).

Comparing the different years with Friedman tests, the marginal staining and anatomy parameters were similar for the three groups, and all had statistically significant differences ($p<0.05$). The marginal adaptation parameter was significantly different in the sealing and control (+) groups ($p<0.05$), but there were no significant differences in the no-treatment (-) group ($p>0.05$). There were also no statistically significant differences in the three groups for the secondary caries parameter ($p>0.05$).

In the Kaplan-Meier survival analysis, the sealing group and no treatment group (-) exhibited exactly the same behavior in terms of failures of restorations per year: first, fourth, and 10th year (Figure 6). The control group (+) had a curve with one less failure, and showed failures in the fifth and 10th year. However, the Mantel-Cox analysis revealed no significant difference between the curves with a log-rank of $p=0.336$. Survival rates for the three groups were high, but the most striking fact was that the control group showed the same results as the sealing group (Table 1). All restorations that failed were Class II composite resins.

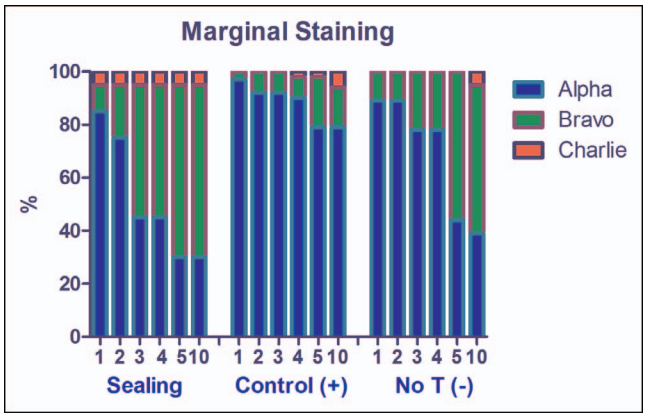

Figure 4. *Marginal staining observations separated by groups and quality evaluation expressed as a percentage of USPHS/Ryge criteria per year.*

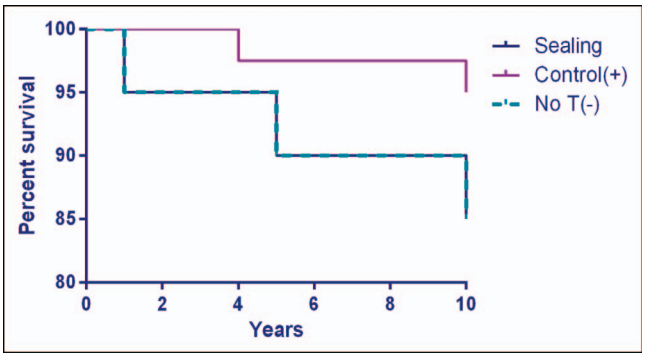

Figure 6. *Kaplan-Meier survival curves for the clinical evaluation. Log-rank,* p*=0.3368.*

Table 1: *Survival Rates of Groups Specifying Only the Years of Failures Expressed as a Percentage*

	1 Year	4 Year	5 Year	10 Year
Sealing	95%	95%	90%	85%
Control (+)	100%	97.5%	97.5%	95%
Control (-)	95%	95%	90%	85%

DISCUSSION

This trial was prospective, randomized, and blinded clinical work following patients for 10 years with regard to the behavior of composite restorations with defective margins that were sealed or left without intervention in comparison to a group with excellent margins at the first year. Sealing the margins of a restoration is a fast, low-cost, minimal intervention that could solve existing small (<1 mm) defects, which was one of the inclusion criteria for this study.

There are several clinical studies that show repair, which means a correct solution for localized defects under a certain indication.[14,15] In the work of Opdam and others,[16] repairs were in larger defects and bigger reconstructions and were perhaps therefore less successful. The question that we sought to answer in this study was whether to seal minor defects or only monitor them over time. According to the results and considering the conditions and limitations of this work, it appears that monitoring restorations with minimal defects is completely permissible.

The sealant used in this trial was a light-polymerizing resin base shown in a meta-analysis by Kuhnisch and others[17] to have retention rates of 77.8% at three years, 80.4% at four years, and 83.8% at five years, which suggests it could still be partially present on the composite resins after 10 years, the length of our study. According to several systematic reviews, pit and fissure sealants are effective measures to prevent caries in young children. Knowledge regarding composite restorations is far from complete, and the question is whether the sealant achieved its goal and remained in the composite. The frequency of new caries lesions after 10 years was similar in the sealing and no-treatment (-) groups, but the control (+) group had no caries lesions appear in the course of 10 years. Thus, the clinical decision passed, because if the group that began the study with Alfa marginal adaptations did not have caries lesions after five years, and the other two groups did, we could propose that the seal was completely effective in preventing new caries lesions around composite resins for up to four years, and if necessary, the composite resin could be resealed in this period.

The results for this cohort of restorations coincide with those of Gordan and others[10] for amalgam restorations sealed for seven years. There are no other reports of sealed composite resins with a longer follow-up.

Clearly, the question of deciding how to treat a composite resin restoration with marginal adaptation problems has not been resolved. The criteria used for the clinical assessment are very important because, in this case, the Ryge criteria lack accuracy for finding differences when evaluating sealing and probably this could be solved with the criteria proposed by Hickel and others.[18]

Marginal adaptation problems represent one of the most important causes for replacing composite restorations, and on many occasions, a replacement leads to more damage to healthy tissue.[19,20] The marginal adaptation problems can entail an increased risk to the pulp of an injury, endodontic treatment, weakening of the structure, and ultimately tooth loss.[21-23]

Many studies support the use of a sealant as a minimally invasive treatment to seal minimal marginal defects between composite resins and the enamel. However, most of these trials have been *in vitro* and do not examine the clinical behavior of the seal over time.[24,25]

The anatomy parameter showed a similar pattern in all three groups, and the comparisons using Wilcoxon and Friedman tests were statistically significant ($p<0.05$), which means the form of the restorations showed similar deterioration in the 10th year. This parameter could be considered a "control," providing evidence of the deterioration of the composite resins over time.[26]

The presence of secondary caries is the most critical parameter that defines whether the seal was able to prevent the emergence of new caries adjacent to the restorations and increase their longevity. Despite losing two restorations for caries in the fifth and 10th years in the sealing group, the behavior of the sealed restorations over the years was similar to that of the controls, which validates the mechanical seal as a preventive therapy in this cohort of composite resins.

There was a similar pattern between the sealing and no-treatment (-) groups in the marginal staining, which differed from the control (+) group pattern that showed a higher frequency of Alfa

values in the 10th year. This means the initial state of the margins is related to the appearance of marginal staining in the future. Compared to the first year, all three groups showed statistically significant differences, although the multiple comparisons of all years had no statistically significant differences.

Survival curves were very similar, with log-rank of 0.336, which means that there was no difference among the three survival curves. It was not possible to calculate the half-life of composite resins due to the low rate of failure. This result presumes that this cohort of restorations should be evaluated at a time to get a clearer idea of whether there was an influence of the sealed margins on the time and the relationship of the sealing to failure. As the patient cohort was medium or low caries risk, it is possible that few of these restorations would fail from this cause. It would accordingly be very interesting to conduct future studies with populations of individuals with a high risk of dental caries to measure the actual influence of sealing margins on composite restorations. Such studies could also evaluate other less self-cleaning surfaces with greater local risks, including sealed margins on interproximal areas, or even in the cervical cavity margin of proximal boxes, which will provide a challenge for clinical dentists in the future. The sealant does not increase the bond strength, applying it on the margins of composite resin restorations does not mean there is an adhesive reinforcement of the restoration surface area.[27]

The protocol of this study was to seal without applying an acid conditioning adhesive, which implies, according to current evidence, that if this protocol had been used, adhesive results could have been better.[28] For the clinical dentist, it is important to consider this option in decision-making along with improved clinical techniques to obtain best possible results.

It is important to note that for the marginal adaptation parameter, more than 50% of the sealed composites had good Alfa values after 4 years, while more than 80% had deteriorated to Bravo values after 10 years, although they remained clinically acceptable restorations.[12,29-31] The take-away point to understand is at what stage the restorations can be conveniently resealed to ensure lowering the risk of mechanical and biological sealing defects.

There are very few clinical reports regarding sealed composite restorations, but previous reports for this cohort of resin restorations agree that the use of pit and fissure sealant in minimum marginal defects increases the life of the composite.[12,29,30,32] The use of pit and fissure sealant has been considered a good preventive agent for use against the development and progression of pit and fissure caries. Sealants have also been used to successfully arrest occlusal caries lesions.[33,34] In comparison to untreated restorations, our study shows an improvement in the marginal adaptation of defective restorations sealed with the pit and fissure sealants after five years. The results are also similar to restorations that were replaced, thus questioning the need for replacement when sealant is a viable treatment option.[31]

Although, there were many Bravo values for the marginal adaptation criteria in the sealed group by the 10th year, there were few secondary caries in the group, which may be due to the preventive action of the sealant. Having a Bravo value in a sealed restoration does not mean that the mass of the sealant was completely lost, only that, at least at one point of the restoration, the margin retains the explorer. Despite this evidence, there is contradiction in the control group (-) where increased deterioration was not detected in marginal adaptation at 10 years, which may be due to limited explanation given by the amplitude of Bravo criteria of the USPHS and could be better explained by the evaluation criteria proposed by Hickel and others.[18]

These results are consistent with those of Kuper and others[35] that suggest that irrespective of the size of the gap, the caries risk is more important in the formation of new lesions. When risk is high, even a gap size of only 68 μm may allow for development of a secondary wall lesion next to a composite restoration.[35]

This study commenced examinations after the first year, and there are no records assessing the sealing immediately after the restoration, which we consider to be a limitation of this work. However, we believe that the only parameter that might have changed markedly would have been the marginal adaptation. Furthermore, it is important to emphasize that the sealing was made by professors of restorative dentistry at the University of Chile calibrated for this procedure, which ensured the reliability of the protocol.

Despite not considering the type of restorations (Class I or Class II) when forming the groups, which we consider a great limitation of this trial, we believe that the Class had no influence on the results. The evaluation in this trial was considering only the

occlusal surface of the restorations. The sample size of this study was low; subsequent results at baseline showed data that likely helped to obtain an estimate of an adequate sample. Considering the differences obtained in the different study groups, a sample size of more than 100 would be required to reach statistical significance.

Another limitation is that the presence of the white sealant prevented blind evaluations of the restorations for this group, which could have influenced the evaluators, although they were blind for the other two groups (no treatment and control).

Although the evaluation at the 10th year indicates that no-treatment (-) and sealing groups had a similar situation, it is important to note that the measuring instrument used has very wide ranges that are not sensitive to small changes in quality of the margins; for example, the Ryge Bravo criteria might consider a marginal defect from 200 μm to even 3 mm. Therefore, it is important to know next assessments in time because without knowing the extent of the defects, it could be assumed that initial small defects might increase in size, and according to Ryge criteria, it would be impossible to detect differences until they reach a Charlie value.

Even though the Kaplan-Meier curve results were similar, it is important to explain that this curve represents a dichotomous analysis without considering differences between excellent or damaged, but clinically acceptable restorations. This is better represented on the percentage charts, which could guide the clinical dentist to make the decision of resealing or repairing the sealant after a particular time to achieve a marginal maintenance of excellence in resin composite restorations.

CONCLUSIONS

- Sealing the margins of the composite resin restorations had no significant effect, under the conditions of this study in the 10th year.
- Sealing the restorations substantially improved the marginal staining and marginal adaptation parameters, though by the 10th year they were similar to the group without intervention, considering the limitations of evaluation.
- After 10 years, the three groups, with the parameters studied, remained clinically acceptable.

Human Subjects Statement

This study was conducted in accordance with all the provisions of the local human subject oversight committee guidelines and policies. The approval code for this study is Project PRI-ODO-0207. This study was conducted at the University of Chile.

Conflict of Interest

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

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Laboratory Research

Efficiency of Dual-Cured Resin Cement Polymerization Induced by High-Intensity LED Curing Units Through Ceramic Material

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Clinical Relevance

When polymerizing dual-cured resin cements through all-ceramic restorations, high-intensity light-emitting diode curing units are recommended because they can compensate for the reduction in the intensity of the light reaching the cement and achieve adequate polymerization with a shorter irradiation period.

SUMMARY

Objective: This study aimed to evaluate the ability of high-intensity light-emitting diode (LED) and other curing units to cure dual-cured resin cement through ceramic material.

Methods: A halogen curing unit (Jetlite 3000, Morita), a second-generation LED curing unit (Demi, Kerr), and two high-intensity LED curing units (PenCure 2000, Morita; Valo, Ultradent) were tested. Feldspathic ceramic plates (VITABLOCS Mark II, A3; Vita Zahnfabrik) with thicknesses of 1.0, 2.0, and 3.0 mm were prepared. Dual-cured resin cement samples (Clearfil Esthetic Cement, Kuraray Noritake Dental) were irradiated directly or through one of the ceramic plates for different periods (5, 10, 15, or 20 seconds for the high-intensity LED units and 20, 40, 60, or 80 seconds for the others). The Knoop hardness test was used to

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determine the level of photopolymerization that had been induced in the resin cement. Data were analyzed by one-way analysis of variance and Dunnett's post-hoc test to identify test-control (maximum irradiation without a ceramic plate) differences for each curing unit ($p<0.05$).

Results: For all curing units, the curing conditions had a statistically significant effect on the Knoop hardness numbers (KHNs) of the irradiated cement samples ($p<0.001$). In general, the KHN decreased with increasing plate thickness and increased as the irradiation period was extended. Jetlite 3000 achieved control-level KHN values only when the plate thickness was 1.0 mm. At a plate thickness ≥2.0 mm, the LED units (except for PenCure 2000 at 3.0 mm) were able to achieve control-level KHN values when the irradiation time was extended. At a plate thickness of 3.0 mm, irradiation for 20 seconds with the Valo or for 80 seconds with the Demi were the only methods that produced KHN values equivalent to those produced by direct irradiation.

Conclusion: Regardless of the type of curing unit used, indirect irradiation of dual-cured resin cement through a ceramic plate resulted in decreased KHN values compared with direct irradiation. When the irradiation period was extended, only the LED units were able to achieve similar KHN values to those observed under direct irradiation in the presence of plates ≥2.0-mm thick. High-intensity LED units require a shorter irradiation period than halogen and second-generation LED curing units to obtain KHN values similar to those observed during direct irradiation.

INTRODUCTION

Resin cements are commonly used in all-ceramic restorations because of their low solubility, high bond strength, and superior mechanical properties, all of which contribute to the reinforcement of ceramic restorations.[1-4] Adequate curing of resin cement during ceramic restoration is a very important factor in obtaining adequate physical and biological properties.[5,6] When the restoration is thicker than 1.5-2.0 mm and/or its opacity inhibits light transmission, the use of dual-cured resin cements is advocated.[7-9] Dual-cured resin cements have been developed in an attempt to combine the properties of chemical-cured and light-cured materials, thereby providing adequate polymerization in deeper and/or shadowed regions and a shorter setting time.[3] It is also important that dual-cured resin cements are irradiated sufficiently at the time of their application because occlusal adjustment and polishing, during which the restoration is subjected to mechanical stress, are usually performed immediately after the cementation.

During light-curing through a ceramic material, the irradiance of the transmitted light decreases as the thickness of the ceramic material increases.[10] Thus, the actual irradiance reaching the dual-cured resin cement underneath the ceramic material is reduced to a certain extent, depending on the irradiance of the light-curing unit and the thickness, type, and opacity of the ceramic material.[9,11] Reductions in irradiance could adversely affect the physicochemical properties of dual-cured resin cements because they can lead to reductions in cement polymerization, which might not be completely counteracted by the chemical-curing abilities of the resins.[12]

Halogen lights are the most frequently used light sources for inducing polymerization in resin-based dental materials.[13] They emit a continuous spectrum of light, though only a small part of the spectrum is useful for curing. Other wavelengths are filtered out to prevent undesirable side effects.[14] Even after the filtration, however, halogen lights deliver several unwanted wavelengths of light that are highly absorbed by dental materials, resulting in the heating of the tooth and resin during the curing process.[15] Other drawbacks include a decline in irradiance over time, a limited curing depth, and a need for a longer exposure period.[13]

Recently developed light-emitting diode (LED) lights offer a much narrower emission spectrum (a bandwidth of about 20 nm centered on 470 nm),[16] and the spectrum falls closely within the absorption range of camphorquinone, the most frequently used photoinitiator in resin composites.[16] In general, LED lights have the following advantages: extended lifetimes of more than 10,000 hours, little light output degradation over time, and resistance to shock and vibration.[16] Today, most light-curing units use single-peak blue LEDs, which usually have higher irradiances than conventional halogen lights.[13,16] The manufacturers of these high-intensity LED curing units claim that they can reach irradiances of up to 2,000-3,200 mW/cm^2 depending on the chosen mode.[13] More recently, high-intensity third-generation LED curing units have become commercially available. These units are equipped with multiple diodes (violet/blue diodes, polywave)

Table 1: *Materials Used in the Study*

Materials	Manufacturer	Shade	Lot No.	Composition[a]
VITABLOCS Mark II	Vita Zahnfabrik	A2	26630	Fine particle feldspar ceramic: SiO_2 (56-64), Al_2O_3 (20-23), Na_2O (6-9), K_2O (6-8), CaO (0.3-0.6), TiO_2 (0.0-0.1)
Clearfil Esthetic Cement	Kuraray Noritake Dental	A2	016ABA	Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, silanated barium glass filler, colloidal silica, accelerator, others
				Paste B: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated barium glass filler, colloidal silica, benzoyl peroxide, dl-camphorquinone, initiators, pigments, others

Abbreviations: Bis-GMA, bisphenol A diglycidyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate.
[a] *Weight percentage is in parentheses.*

and, thus, are effective not only at curing cements containing camphorquinone but also at curing those containing its alternatives.[13] In addition, many of the newer high-power LED units are said to require shorter irradiation periods;[13,16] however, the ability of these devices to polymerize dual-cured resin cement through ceramic restorations has not been fully investigated.

Thus, the purpose of this study was to evaluate (using microhardness measurements) the ability of high-intensity LEDs, second-generation LEDs, and halogen curing units to induce polymerization in dual-cured resin cement through ceramic plates.

METHODS AND MATERIALS

A block of feldspathic glass ceramic material (VITABLOCS Mark II, shade A2, size I12, lot 26630, Vita Zahnfabrik, Bad Säckingen, Germany) was used to produce the ceramic plates (Table 1). From the block of ceramic material, plates 12.0 mm long and 10.0 mm wide were cut using a low-speed diamond saw (Micro-cutter 201, Maruto, Tokyo, Japan). Both sides of the ceramic plates were then polished under water cooling with a polishing device (Struers A/S, Marumoto Struers, K.K., Denmark) and silicon carbide papers (FEPA P, Marumoto Struers, K.K., Denmark) of descending grit size (#320 to #1200). During the aforementioned polishing, the thickness of the ceramic plates was monitored with a digital micrometer (Mitutoyo PK-1012, Mitutoyo, Kanagawa, Japan), and the plates were polished until the following thicknesses were reached: 1.0, 2.0, and 3.0 mm.

Four different light-curing units were tested: a conventional halogen unit (Jetlite 3000, Morita, Tokyo, Japan), a second-generation LED curing unit (Demi, Kerr, Orange, CA, USA), and two high-intensity LED curing units (PenCure 2000, Morita; Valo, Ultradent, South Jordan, UT, USA) were tested (Table 2). All of the curing units were used in maximum power mode. The emission spectra and the irradiance of each unit were measured with a laboratory-grade spectroradiometer (USR-45DA-14, Ushio, Tokyo, Japan),[17] either without ceramic plates (at a thickness of 0 mm) as a control or through a ceramic plate with a thickness of 1.0, 2.0, or 3.0 mm. The plate was placed between the tip of the curing unit and the aperture of the spectroradiometer, and the resultant light output was detected and recorded using the analytical software supplied with the device. All the results were expressed as the means of five measurements, and distribution of the irradiances (in mW/cm^2) was calculated in 380–525 nm ranges using the analytical software.

A dual-cured resin cement (Clearfil Esthetic Cement, clear, lot 016ABA, Kuraray Noritake Dental, Tokyo, Japan; Table 1) was used in this experiment. To prepare the resin cement specimens, a clear glass slab was used as a supporting surface on top of a black background that decreased the

Table 2: *Curing Units Used in the Study*

Curing Unit	Manufacturer	Type	Serial No.	Light Intensity,[a] mW/cm^2
Jetlite 3000	Morita	QTH	2010889	>400
Demi	Kerr	LED	752020284	1100
PenCure 2000	Morita	LED	2B0085	2000
Valo	Ultradent	LED	V18955	3200

Abbreviatiions: LED, light-emitting diode; QTH, quartz-tungsten-halogen.
[a] *According to the manufacturer.*

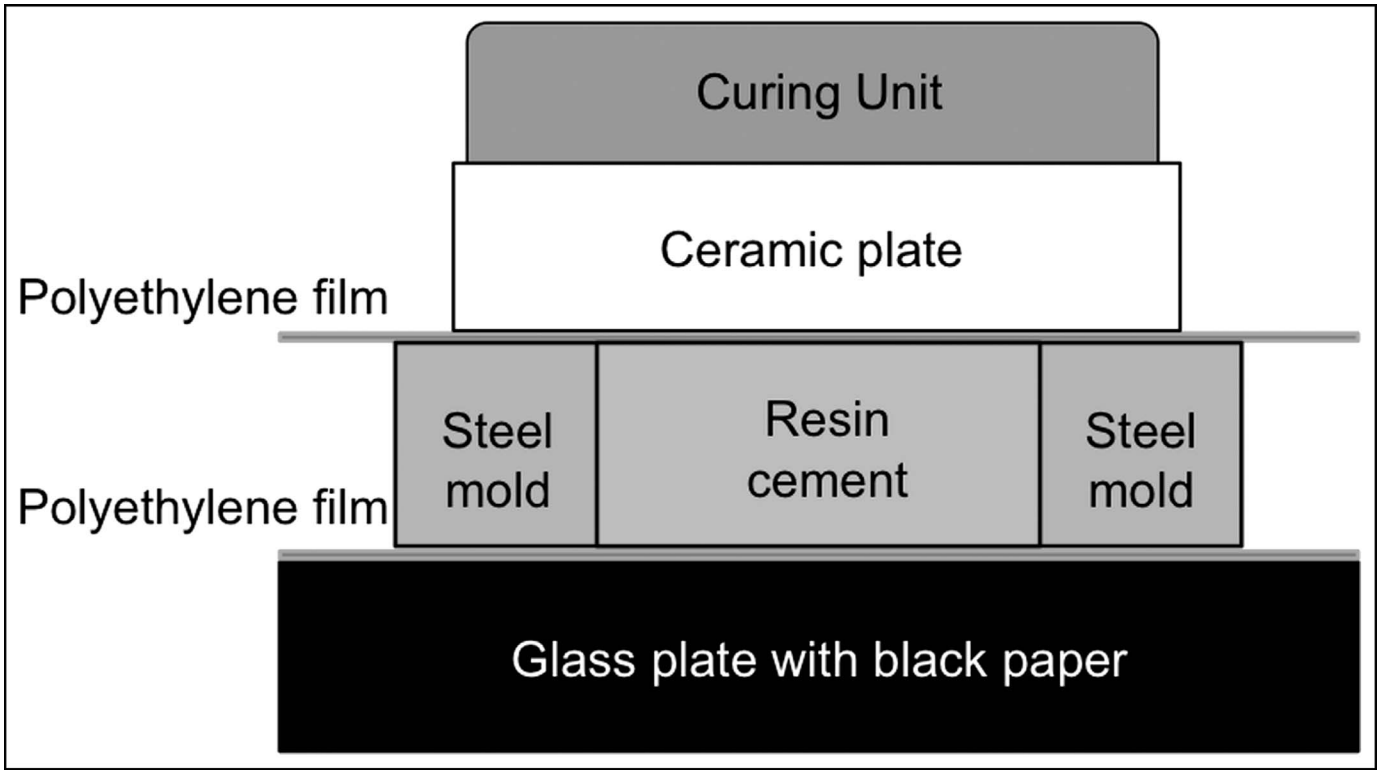

Figure 1. *Preparation of resin cement specimens.*

reflectivity of the underlying surface. A stainless steel mold (internal dimension: 12 mm wide, 2 mm deep, and 2 mm high) was placed on the glass slab. The dual-cured resin cement was mixed using mixing tips according to the manufacturer's instructions and used to fill the mold after a small amount of the mixed paste had been discarded to ensure that equal amounts of the two pastes were pushed out of the mold. A polyethylene film (GC, Tokyo, Japan) was then placed on the top and bottom of the resin cement to isolate it from the ceramic plate and glass slab.

Light-curing was performed either through the polyethylene film (as a control) or through a ceramic plate with a thickness of 1.0, 2.0, or 3.0 mm placed on top of the polyethylene film, whilst the tip of the curing unit was in contact with the ceramic plate (Figure 1). For the Jetlite 3000 and Demi units, the curing time was set at 20, 40, 60, or 80 seconds (n=5, each). The latter three time periods were achieved by performing the appropriate number of 20-second irradiation periods. For the PenCure 2000 and Valo, in which the maximum irradiation period was set to 3 seconds when the device was in maximum power mode to prevent heat generation, the curing time was set to 5 (3 + 2), 10 ([3 × 2] + [2 × 2]), 15 (3 × 5), or 20 (3 × 6 + 2) seconds (n=5, each). The minimum irradiation period was set according to the recommendation of the manufacturer of the cement used. The maximum irradiation period (80 seconds for the Jetlite 3000 and Demi and 20 seconds for the PenCure 2000 and Valo) was only used when the 3.0-mm-thick ceramic plates were used. As a result, a total of 260 resin cement specimens were evaluated. All of the specimens were stored in lightproof containers in distilled water at 37°C for 24 hours.

A microhardness tester (MVK-E, Akashi Co Ltd, Tokyo, Japan) was used to produce microindentations on the surface of the resin cement specimens. The microindentations were then measured to determine the microhardness of the resin cement specimens, which indicated the extent of the polymerization that had been induced in the resin cement. Knoop hardness measurements were performed under a load of 50g for 15 seconds. For each examined cement sample, three indentations were made in a longitudinal section of the sample taken from a region located 100 μm from the sample's surface (Figure 2). In addition, the distance of the indentations from the specimen's center was set at ≥1.0 mm. The mean of the five measurements was recorded as the Knoop hardness number (KHN) (Figure 2).

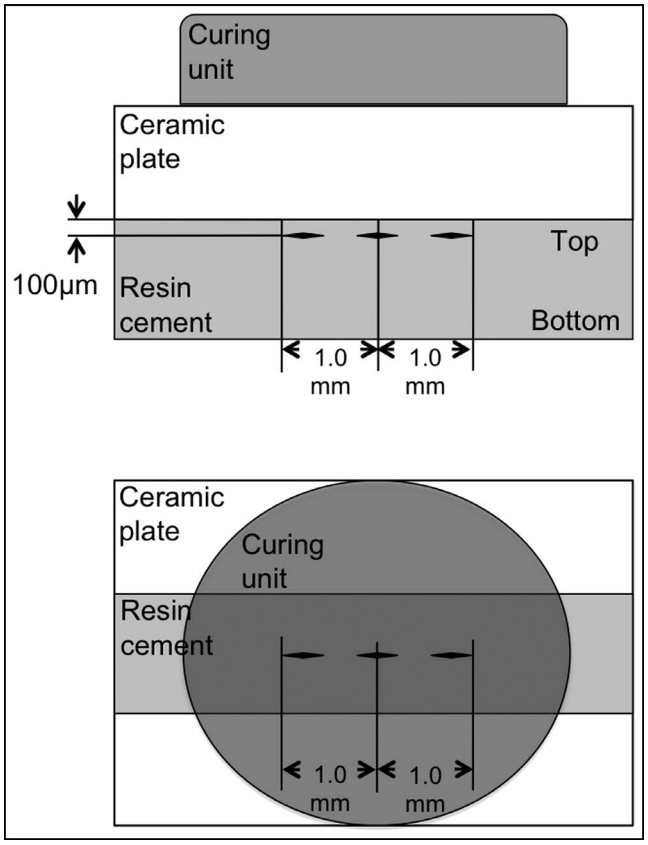

Figure 2. *Position of Knoop hardness measurement (top: side view; bottom: top view).*

The KHN data for the four curing units were analyzed by one-way analysis of variance using the SPSS Base 10.0 statistical software package (SPSS Inc, Tokyo, Japan). The curing condition (13 levels) was treated as an independent variable. Multiple comparisons analysis was performed with Dunnett's test to identify test-control (maximum irradiation without a ceramic plate) differences. All statistical tests were performed using a significance level of $\alpha=0.05$.

RESULTS

Figure 3 shows spectral distributions obtained for each curing unit in the absence or presence of ceramic plates with different thicknesses. Jetlite 3000 showed broad spectra with a mild peak around 480 nm. Both Demi and Pencure 2000 had distinct single-peak spectra in the wavelength range of 440 to 460 nm. Valo showed dual-peak spectra around 400 nm and between 440 and 470 nm. Almost all emissions were detected in the wavelength range between 380 and 525 nm. Light transmittance values calculated by integrating the irradiance values in the 380–525 nm ranges are shown in Table 3. In the absence of a ceramic plate, the irradiance values exhibited the following order: Jetlite 3000 < Demi < PenCure 2000 < Valo. The overlaid ceramic plates severely reduced the irradiance of the transmitted light; that is, the irradiance values were reduced to approximately 1/3, 1/5, and 1/10 of the relevant positive-control value by the plates with a thickness of 1.0, 2.0, and 3.0 mm, respectively.

The KHN values of the resin cement samples after polymerization had been performed with each curing unit are shown in Table 4. One-way analysis of variance demonstrated that the curing condition had a statistically significant effect on microhardness, regardless of the curing unit used ($p<0.001$). In general, the KHN values decreased as the plate thickness increased and increased as the curing period was extended. When the Jetlite 3000 was used, control-level KHN values were only obtained at a plate thickness of 1.0 mm. The specimens cured with the Demi exhibited significantly reduced KHN values at a plate thickness $\geq$2.0 mm and required 80 seconds to produce control-level KHN values at a plate thickness of 3.0 mm. When irradiation was performed with the PenCure 2000, significantly decreased KHN values were observed after 5 seconds' irradiation at all plate thicknesses, and when a plate thickness of 3.0 mm was used, the KHN values were still significantly decreased even after 20 seconds' irradiation. The specimens irradiated with the Valo also showed significantly decreased KHN values after 5 seconds' irradiation at all plate thicknesses. At a plate thickness of 3.0 mm, the Valo required 20 seconds to polymerize the cement to a sufficient extent to produce control-level KHN values.

DISCUSSION

The mechanical properties of resinous materials are dependent on the degree of conversion of the resin matrix, which can be assessed by several methods.[18] Fourier transform infrared spectrometry (FT-IR) and laser Raman spectroscopy, which directly measure the degree of conversion, are regarded as sensitive methods; however, they are time consuming and expensive.[19] Thus, indirect methods, such as assessments of the depth of cure and microhardness testing,[20] are used as practical techniques for assessing the effects of different exposure conditions.[21,22] In this study, we used microhardness testing because it is the most commonly used technique for measuring the degree of conversion of resin cements.[23] It has been reported that surface hardness measurements

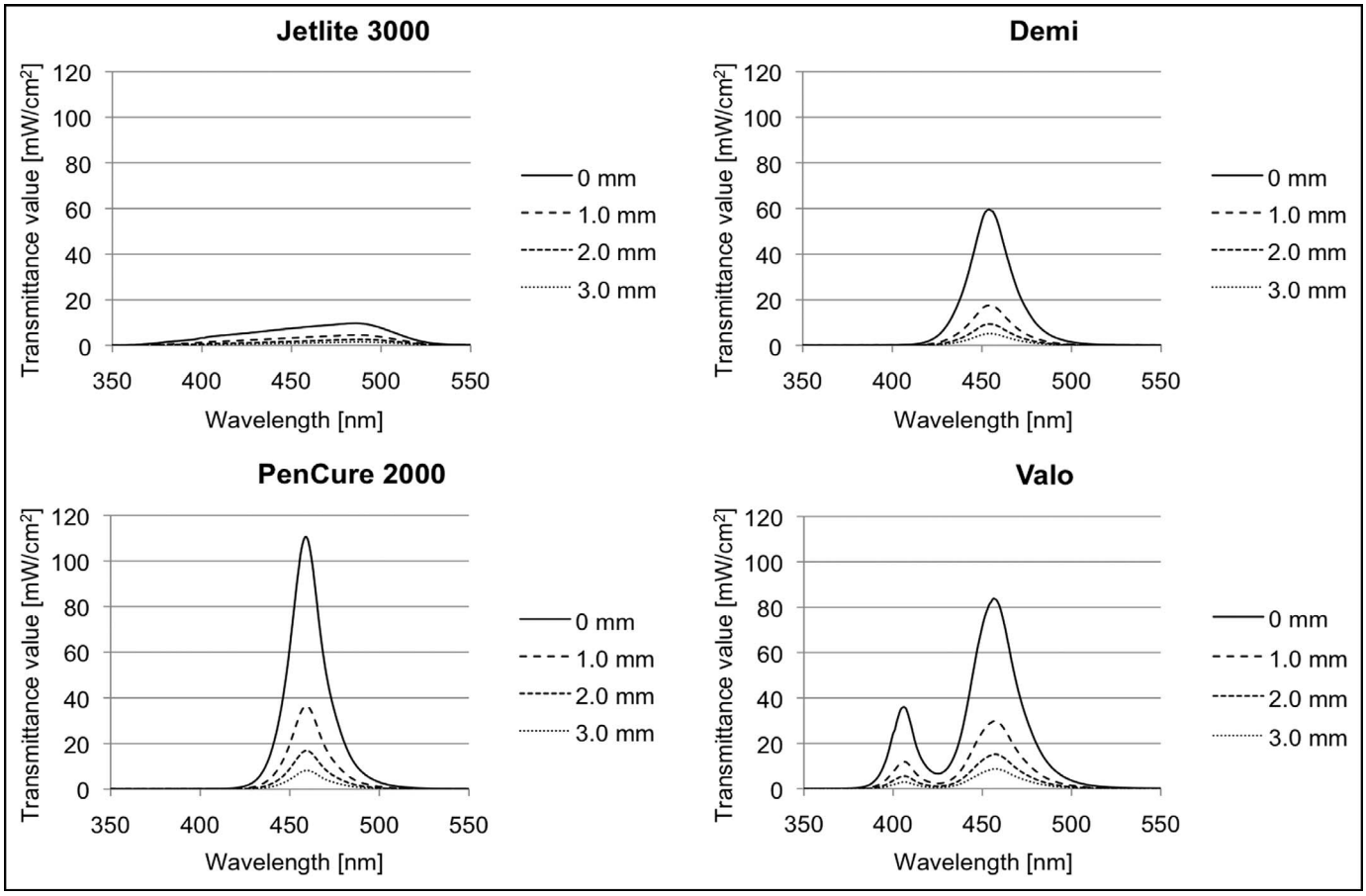

Figure 3. *Spectral distribution of light transmittance values (mW/cm$^{2)}$ of four light-curing units through various thicknesses of ceramic plates.*

produce similar results with FT-IR spectroscopy and that hardness testing is more able to detect small changes in the degree of conversion than FT-IR after the network has been cross-linked.[24]

In this study, we measured the irradiance of the four curing units with a laboratory-grade spectroradiometer.[17] The results demonstrated that light intensity values obtained were equal or higher than those stated by the manufacturer, whereas they decreased as the plate thickness increased (Table 3; Figure 3). All the units showed a peak around 470 nm and thus are compatible to camphorquinone. On the other hand, lights emitted from dental curing units are known to be inhomogeneous,[25,26] which could have some influence on the present results. This point requires further investigation, although one study has reported that LED units emit more homogenous light, and thus yield more uniform distribution of surface hardness (KHN) of a resin composite, than halogen units.[25]

Ceramic thickness is regarded as a critical factor determining the amount of light transmission thorough all-ceramic restorations,[10] although other factors, such as crystalline structure and light refractive index, also have an effect.[27,28] The results of the present study confirm that ceramic thickness has a strong effect on the irradiance of transmitted light;[10] that is, ceramic disks 2.0 and 3.0 mm thick reduced irradiance by approximately 80% and $\geq$90%, respectively, regardless of the light-curing unit used (Table 2). Similar results were obtained in a previous study, in which leucite-reinforced glass ceramic (IPS Empress CAD, Ivoclar Vivadent, Schaan, Liechtenstein) and lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent) caused reductions in irradiance of 81.9%-83.3% and 85.8%-86.5%, respectively, at a thickness of 1.5 mm and reductions of more than 95% at a thickness of 3.0 mm.[11]

In a clinical setting, a large proportion of all-ceramic crowns are 1.0-3.0 mm thick.[29,30] Furthermore, posterior inlay/onlay restorations have to be at least 1.5 to 2.0 mm thick, and the thickness of the ceramic can increase to 3.0 mm in the proximal box.[31] Thus, a thickness of 3.0 mm is clinically

Table 3: *Integrated Light Transmittance Values (Standard Deviation) (mW/cm^2) of Four Curing Units Through Various Thicknesses of Ceramic Plates*

Curing Unit	Ceramic Thickness			
	0 mm	1.0 mm	2.0 mm	3.0 mm
Jetlite 3000	860 (86)	384 (9)	210 (8)	116 (1)
Demi	1812 (108)	538 (55)	284 (23)	157 (13)
PenCure 2000	2790 (79)	927 (52)	431 (12)	210 (10)
Valo	3337 (88)	1181 (15)	595 (16)	337 (13)

relevant, and clinicians should consider the negative effects of light attenuation on the polymerization of dual-cured resin cements.[8] The chemically cured properties of dual-cured resin cements might compensate to some extent for decreased light transmission;[7] however, the actions of chemical catalysts might not be sufficient to allow maximum monomer conversion.[6,32] According to our unpublished data, KHN value of non-light-cured Clearfil Esthetic Cement was 24.6 ± 1.8, and this value is comparable to that obtained for specimens cured with Jetlite 3000 (20 and 40 seconds) through a ceramic plate 3.0 mm thick (Table 4). This may indicate that, in the presence of a plate 3 mm thick, the halogen unit achieves little effect on resin polymerization unless irradiation time is extended.

The duration of irradiation is another important factor affecting the curing of dual-cured resin cement through ceramic materials.[33] This is explained by radiant exposure (J/cm^2); that is, the product of irradiance (mW/cm^2) and light-curing time (s),[34] which indicates that an increase in light-curing time could be used to compensate for a reduction in irradiance.[34-37] The present results demonstrated that as the thickness of the ceramic plates increased, a longer irradiation period was required to produce positive control-level (no ceramic plate) KHN values. Moreover, previous studies have reported that different combinations of irradiance values and light-curing periods that resulted in similar radiant energy levels produced similar material properties, such as surface hardness,[10,32] degree of conversion,[32-35,37] and flexural strength.[36] Taken together, these findings support the notion that longer irradiation periods are required for the adhesive luting of all-ceramic restorations in order to compensate for attenuated irradiance and to provide sufficient radiant energy for the adequate polymerization of resin cements.

To induce adequate monomer conversion in light-cured composite resin materials, 40 seconds' curing at an irradiance level of 400 mW/cm^2 is considered to be sufficient for direct irradiation.[38] However, it is apparent that this curing protocol should be modified according to the ceramic material (thickness, composition, shade, etc) and curing unit (light source, irradiance, etc) used. The data presented here indicate that working time can be shortened by the use of high-intensity units. However, in the presence of ceramic material ≥2.0 mm thick, the curing time required for adequate polymerization is markedly longer than the time recommended by the manufacturers, indicating that it is necessary to extend the curing time. A recent study has shown that many practitioners use halogen lights with power outputs <300 mW/cm.[2,39] Thus, the polymer-

Table 4: *Mean (Standard Deviation) Microhardness (Knoop Hardness Number) Values for the Resin Cement Samples Irradiated With Each Curing Unit for Various Curing Periods*

Ceramic Thickness	Curing Time, s	Curing Unit			
		Jetlite 3000	Demi	PenCure 2000	Valo
0 mm	20 or 5	46.3 (2.0)	48.2 (2.1)	43.4 (1.8)*	46.7 (1.3)
	40 or 10	47.8 (2.4)	47.8 (3.4)	48.0 (1.9)	47.0 (3.1)
	60 or 15 (control)	49.2 (1.3)	48.7 (1.9)	48.1 (0.9)	48.2 (1.5)
1.0 mm	20 or 5	38.0 (0.7)*	47.6 (0.9)	41.7 (1.6)*	44.7 (0.6)*
	40 or 10	46.0 (0.8)	48.3 (0.9)	47.1 (0.9)	48.4 (1.2)
	60 or 15	48.0 (1.4)	49.4 (0.7)	47.2 (0.9)	48.9 (0.9)
2.0 mm	20 or 5	37.9 (0.9)*	38.2 (1.5)*	38.3 (1.4)*	39.1 (1.4)*
	40 or 10	38.9 (2.8)*	46.6 (2.2)	43.7 (2.0)*	44.7 (3.7)
	60 or 15	44.0 (3.4)*	46.4 (1.3)	46.1 (1.9)	47.9 (2.4)
3.0 mm	20 or 5	24.3 (0.8)*	35.6 (0.8)*	32.1 (2.3)*	32.6 (1.0)*
	40 or 10	25.3 (0.8)*	38.8 (1.5)*	33.7 (1.3)*	34.6 (1.8)*
	60 or 15	28.0 (0.8)*	42.3 (1.2)*	34.7 (1.2)*	40.4 (1.4)*
	80 or 20	29.3 (1.4)*	46.6 (1.1)	35.9 (2.2)*	48.8 (0.8)

* *Significantly different compared with the control duration (ceramic thickness: 0 mm, curing periods: 60 and 15 seconds, respectively).*

ization efficiency achieved with high irradiance lights might be even more significant than was demonstrated in the present study.

The radiant energy theory also indicates that increasing irradiance by using a high-power light unit shortens the light-curing period by increasing the irradiance of the curing light, which results in more photons available per unit time for absorption;[40] thus, more photoinitiator molecules react with amines, and more free radicals are available for polymerization.[41] Therefore, recently developed high-power curing units (eg, the PenCure 2000 and Valo) are considered to achieve adequate polymerization (as indicated by positive control-level KHN values in this study) within a shorter exposure period than traditional units.

It is worth noting that in the present study the KHN values of the cement samples did not always reach the positive control level, even after the maximum curing duration (four times the minimum light-curing period). In particular, when a plate thickness of 3.0 mm was used, only the Demi and Valo achieved positive control-level KHN values after the maximum curing period (80 seconds and 20 seconds, respectively). These findings indicate that a high-intensity light is required to compensate for the light attenuation that occurs as light passes through a thick ceramic material, even when the irradiation period is extended.[19] In this regard, high-intensity LED curing units might have advantages over conventional halogen units. In the present experimental conditions, however, the PenCure 2000 did not produce positive control-level KHN values in the presence of ceramic plates 3 mm thick. This might have been because, even in high power mode, the shortened irradiation period decreased the amount of radiant energy delivered by the PenCure 2000 to a level that was insufficient for achieving adequate polymerization.

All curing units in this study can produce a painful burning sensation if the light-curing tip is inadvertently placed in contact with surrounding soft tissues when the curing time is extended over the manufacturer's recommendation. An extensive curing time could also cause cytotoxic effects.[42] Thus, care should be taken to avoid direct contact of soft tissues with high-intensity curing ights. Regarding pulpal damage, however, an *in vitro* study reported that light curing a resin cement under feldspathic ceramic caused a temperature increase of 3°C,[43] which is smaller than that necessary to damage the pulp (5.5°C).[44] This suggests a possible insulation effect of the ceramic material. Moreover, the temperature increase in a clinical situation might be smaller because of the effect of blood circulation in the pulp chamber. Nevertheless, clinicians should take the harmful effects into account, and avoid inadvertently extended irradiation of high-intensity LED lights. Air-cooling of the tooth during irradiation may be recommended.

The results of the present study indicate that high-intensity curing units are recommended for polymerizing dual-cured resin cement through all-ceramic restorations because they are able to achieve adequate resin cement polymerization within a shorter period than halogen and second-generation LED curing units. However, even high-intensity LED curing units might require an extended curing period to induce sufficient polymerization in such cements.

CONCLUSION

Regardless of the type of curing unit used, indirect irradiation through a ceramic plate decreased the KHN values of the dual-cured resin cement tested. In the presence of ceramic plates $\geq$2.0 mm thick, only the LED units achieved KHN values similar to those produced by direct irradiation, even when the irradiation period was extended. High-intensity LED units require a shorter irradiation period to obtain KHN values similar to those produced by direct irradiation compared with halogen and second-generation LED curing units.

Conflict of Interest

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

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Effect of Cleansing Methods on Saliva-Contaminated Zirconia—An Evaluation of Resin Bond Durability

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Clinical Relevance

Considering the bond strength data and absence of adhesive failures, it can be stated that an appropriate cleaning method (ie, a zirconium-oxide–based paste) can be helpful in restoring the resin bond strength to saliva-contaminated zirconia.

SUMMARY

The aims of this study were to investigate 1) the influence of cleansing methods after saliva contamination and 2) aging conditions (thermocycling and water storage) on zirconia shear bond strength (SBS) with a resin cement. One hundred and eighty zirconia specimens were sandblasted with 50 μm aluminum oxide particles, immersed in saliva for one minute (with the exception of the control group, [C]), and divided into groups according to the cleansing method, as follows: water rinse (W); 37% phosphoric acid gel (PA); cleaning paste (ie, Ivoclean®) containing mainly zirconium

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oxide (IC); and 70% isopropanol (AL). Scanning electron microscopy was done to qualitatively evaluate the zirconia surface after each cleansing method. For the SBS test, resin cement buttons were bonded to the specimens using a dedicated jig. SBS was evaluated according to standard protocols after 24 hours, 5000 thermal cycles (TC), or 150 days of water storage. Statistical analysis was performed using two-way analysis of variance and Tukey test ($p<0.05$). Data showed a significant effect for the 150 days of water storage, TC, and 24 hours of water storage (150 days < TC < 24 hours). Group comparisons showed that PA < AL and W < IC and C. SBS ranged from 10.4 to 21.9 MPa (24 hours), from 6.4 to 14.8 MPa (TC), and from 2.9 to 7.0 MPa (150 days). Failure analysis revealed a greater percentage of mixed failures for the majority of the specimens and a smaller percentage of adhesive failures at the ceramic-resin cement interface. Our findings suggest that Ivoclean® was able to maintain adequate SBS values after TC and 150 days of storage, comparable to the uncontaminated zirconia.

INTRODUCTION

Current advances in computer-aided design/computer-aided manufacturing technology have facilitated and expanded the use of high-toughness yttria-stabilized tetragonal zirconia ceramics (Y-TZP) as frameworks for fixed-partial dentures (FPDs) and more recently as full-contour restorations.[1-5] Regrettably, apart from its superior mechanical properties, when contrasted with glassy-matrix ceramics, and the finer esthetic and biocompatibility characteristics, as opposed to those of metallic FPD frameworks, the achievement of a durable adhesive bonding to structural Y-TZP ceramics remains a very difficult task.[6-20]

Meanwhile, another major issue pertaining to bonding of ceramic restorations relates to the potential of contamination before cementation. Zirconia shows a strong affinity toward the phosphate group found in saliva and other fluids.[21] After sandblasting and clinical try-in procedures, zirconia may become contaminated with saliva and/or blood, which reacts with the zirconia surface and makes bonding a challenge.[21] X-ray photoelectron spectroscopy (XPS) revealed that the organic coating formed after saliva contamination resisted complete removal with water rinsing, isopropanol, or phosphoric acid.[21] Nonetheless, while numerous studies[6-18] have shown an immediate (24-hour) increased bond strength between zirconia and resin cements after various surface conditioning methods, the potential contamination of the intaglio surface prior to cementation, as well as the maintenance of high bond strength values after long-term storage periods and/or thermocycling (TC) regimens, should be the primary goal. The null hypotheses tested were that 1) the cleansing methods would not negatively influence zirconia bonding; and 2) the aging conditions (ie, TC and 150 days of water storage) would not damage the bond strength between zirconia and resin cement.

METHODS AND MATERIALS

Specimen Preparation

One hundred and eighty zirconia (Diazir®, batch P02286, Ivoclar-Vivadent, Amherst, NY, USA) specimens (12×13×3 mm^3) were obtained from full-contour zirconia blocks with a diamond wafering blade mounted in a precision saw machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). Specimens were sintered at 1500°C according to the manufacturer's instructions in a high-temperature furnace (Lindberg/Blue M, Asheville, NC, USA).[22,23] Specimens were embedded in acrylic resin (Bosworth Fastray™, Bosworth Company, Durham, UK), wet-finished with 600-1200-grit silicon carbide papers (LECO Corporation, Saint Joseph, MI, USA), and cleaned in an ultrasonic bath in distilled water for five minutes. All specimens were sandblasted with 50 μm aluminum oxide particles (Patterson Dental Supply Inc, batch 3150313, St Paul, MN, USA) for 30 seconds, under 2.8 bars and from a distance of approximately 10 mm.[24,25] Next, the specimens were rinsed with water, air-dried, and randomly distributed into five groups (N=36), as follows: control (C)—no saliva contamination; water rinse (W)—specimens were immersed in stimulated human saliva (IRB approval 1105005588) for one minute at 37°C, rinsed with water from a multifunction syringe (MFS) for 15 seconds, and then air-dried[21]; phosphoric acid (PA)—contamination with saliva followed by 37% phosphoric acid (Total Etch, batch R51858, Ivoclar-Vivadent) cleansing for 60 seconds, rinsed with water from MFS for 15 seconds, and air-dried[26,27]; Ivoclean® (IC)—contamination with saliva and cleansing with a commercially available cleaning paste (Ivoclean, batch R53033, Ivoclar-Vivadent), according to the manufacturer's instructions (briefly, it was applied on the bonding surface with a microbrush for 20 seconds and then rinsed with water from MFS); and isopropanol (AL)—contamination with saliva and immersion in 70% isopropa-

Table 1: *Materials, Manufacturer, Batch Number, and Composition*

Materials		Manufacturer	Batch No.	Composition
Zirconia	Diazir Full-Contour	Ivoclar-Vivadent, Amherst, NY, USA	P02286	Y-TZP
Phosphoric acid	Total Etch 37%	Ivoclar-Vivadent, Amherst, NY, USA	R51858	Distilled water, phosphoric acid (85%), thickener, pigments
Clean paste	Ivoclean	Ivoclar-Vivadent, Schaan, Liechtenstein	R53033	Zirconium oxide, water, polyethylene glycol, sodium hydroxide, pigments, additives
Silane	Monobond Plus	Ivoclar-Vivadent, Amherst, NY, USA	R50513	Alcohol solution of silane methacrylate, phosphoric acid, methacrylate and sulfide methacrylate
Resin cement	Multilink Automix	Ivoclar-Vivadent, Amherst, NY, USA	S04093	Dimethacrylate, HEMA, barium glass, ytterbium trifluoride, spheroid mixed oxide

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; Y-TZP, yttria-stabilized tetragonal zirconia ceramics.

nol for two minutes and rinsed with water from MFS for 15 seconds and air-dried. Two additional zirconia specimens were prepared to assess the surface morphology after the different cleaning methods (ie, groups C, W, PA, IC, and AL). Briefly, zirconia specimens were mounted on Al stubs, sputter-coated with Au-Pd alloy, and imaged at various magnifications using a scanning electron microscope (SEM, JSM-6390, JEOL, Tokyo, Japan).

All bonding procedures were carried out immediately after the contamination and cleansing steps. The same individual bonded all the study specimens. The materials, manufacturers, compositions, and batch numbers are listed in Table 1.

Bonding Procedure

After the specimens received the assigned cleansing regime, a silane agent (Monobond Plus, batch R50513, Ivoclar-Vivadent) was applied with a brush and left undisturbed for one minute, and then the solvent was air-dried. Resin cement buttons (ca 2.15 mm in height and 2.38 mm in diameter) were fabricated using a specially fabricated jig (Ultradent, South Jordan, UT, USA) with a cylindrical Teflon mold over each zirconia specimen. The resin cement (Multilink® Automix, batch S04093, Ivoclar-Vivadent) was applied into the mold and then photopolymerized (Demi L.E.D. Dental Curing Light, Kerr Corporation, Middleton, WI, USA), following the manufacturer's instructions. The curing light intensity was measured before bonding procedures (ca 1200 mW/cm^2) using a radiometer (Cure Rite, Curing light meter, Caulk, Dentsply International Inc, Milford, DE, USA).

Aging Conditions

The specimens (N=36) of each group were assigned into three subgroups (n=12), as follows: 1) no aging (ie, the specimens were kept in water for 24 hours at 37°C before testing); 2) TC: the specimens were thermocycled before testing (5000 cycles, 8°C to 48°C, dwell time of 30 seconds, transfer time of 10 seconds)[28]; and 3) water storage: the specimens were kept in water at 37°C for 150 days before testing. The water was changed every other week. No evidence of any bacterial and/or fungal growth was seen; however, the pH was not monitored.

Shear Bond Strength and Failure Analysis

Shear bond strength (SBS) was determined using a dedicated jig (Ultradent) attached to the Universal Testing Machine (ElectroPuls E3000 All-Electric Test Instrument, Instron Industrial Products, Grove City, PA, USA). The load was applied to the adhesive interface until failure at a crosshead speed of 1 mm/min. The maximum stress to produce fracture was recorded (N/mm^2=MPa). The fractured interfacial zones on the zirconia specimens were examined under optical microscopy, and the mode of failure was identified as follows: cohesive resin cement—cohesive failure in the resin cement; Cohesive-ceramic—cohesive failure in the ceramic; and mixed—adhesive failure combined with cohesive failure in the resin cement, adhesive—within any of the substrates or interfaces.[10] Representative specimens were examined under a scanning electron microscope (SEM) (JSM-6390, JEOL, Tokyo, Japan). Images were taken after sputter coating the specimens with gold at different magnifications.

Statistical Analysis

Two-way analysis of variance was used to examine the effects of both the cleansing method and the aging condition on SBS. Comparisons were adjusted for multiple testing using the Tukey method, with an overall significance level of 5%. The SBS data were found to have a log-normal distribution, so the

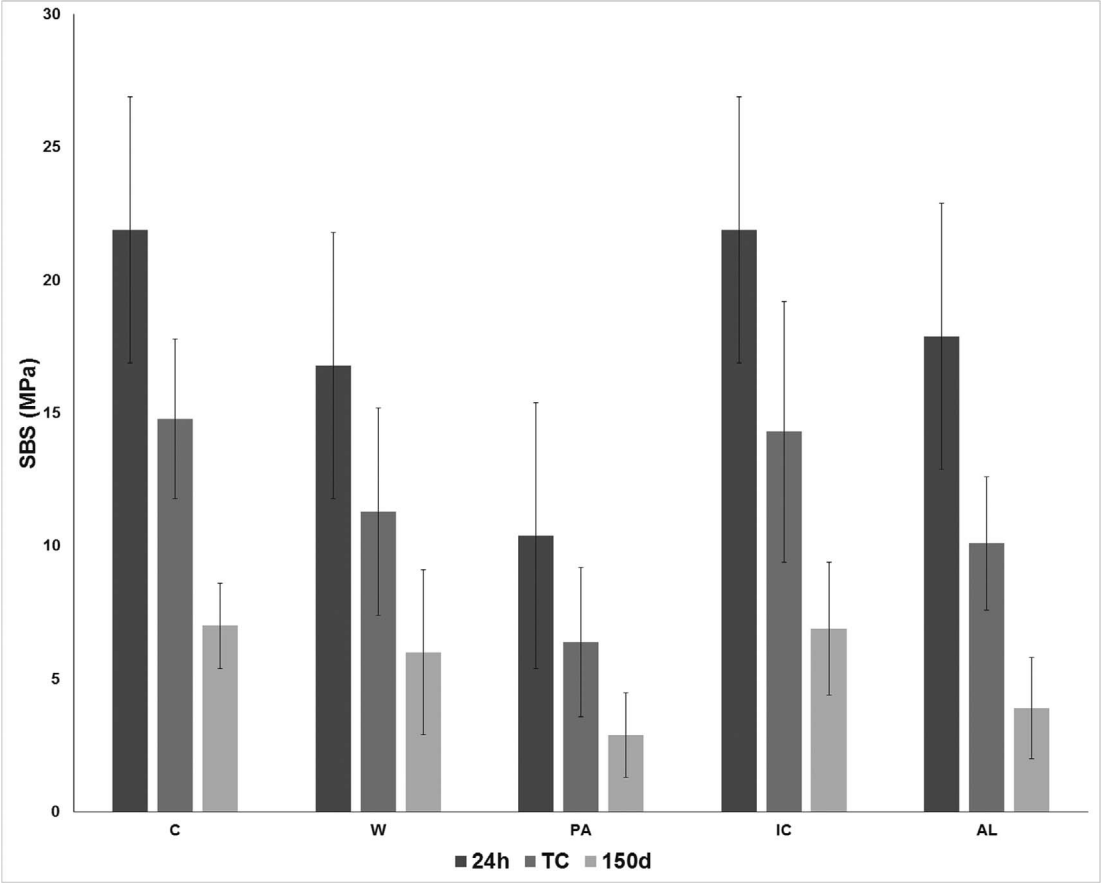

Figure 1. *SBS values after 24 hours, thermocycling, and 150 days.*

analyses were performed on the transformed data. The means along with the 95% confidence intervals were calculated using the transformed data and were then converted back to the original scale to allow the results to be more easily interpreted.

RESULTS

Figure 1 and Table 2 show means and standard deviations of SBS (in MPa). The interaction between groups and the effect of TC and water storage was not significant (p=0.47), indicating that the

Table 2: *Mean (95% Confidence Interval) Shear Bond Strength (SBS; MPa)*[a]

Groups	C		W		PA		IC		AL	
24 h	20.5	a	16.4	a	10.1	a	21.5	a	17.4	a
	(16.1, 26.1)		(14.1, 19.0)		(8.6, 11.9)		(18.9, 24.4)		(14.7, 20.6)	
	A		AB		B		A		A	
TC	14.5	a	10.7	a	5.9	a	13.5	a	9.8	b
	(12.7, 16.6)		(8.6, 13.3)		(4.4, 7.8)		(10.8, 16.8)		(8.4, 11.4)	
	A		A		B		A		AB	
150 d	6.8	b	5.1	b	2.5	b	6.5	b	3.5	c
	(5.8, 7.9)		(3.4, 7.7)		(1.7, 3.6)		(5.2, 8.3)		(2.5, 4.8)	
	A		AB		C		A		BC	

Abbreviations: AL, isopropanol; C, control; IC, Ivoclean cleaning paste; PA, phosphoric acid gel; TC, thermocycling; W, water rinse.
[a] *Lowercase letters in the same column imply statistical similarity among conditions within the groups ($p<0.05$). Uppercase letters in the same row imply statistical similarity among groups within condition ($p<0.05$).*

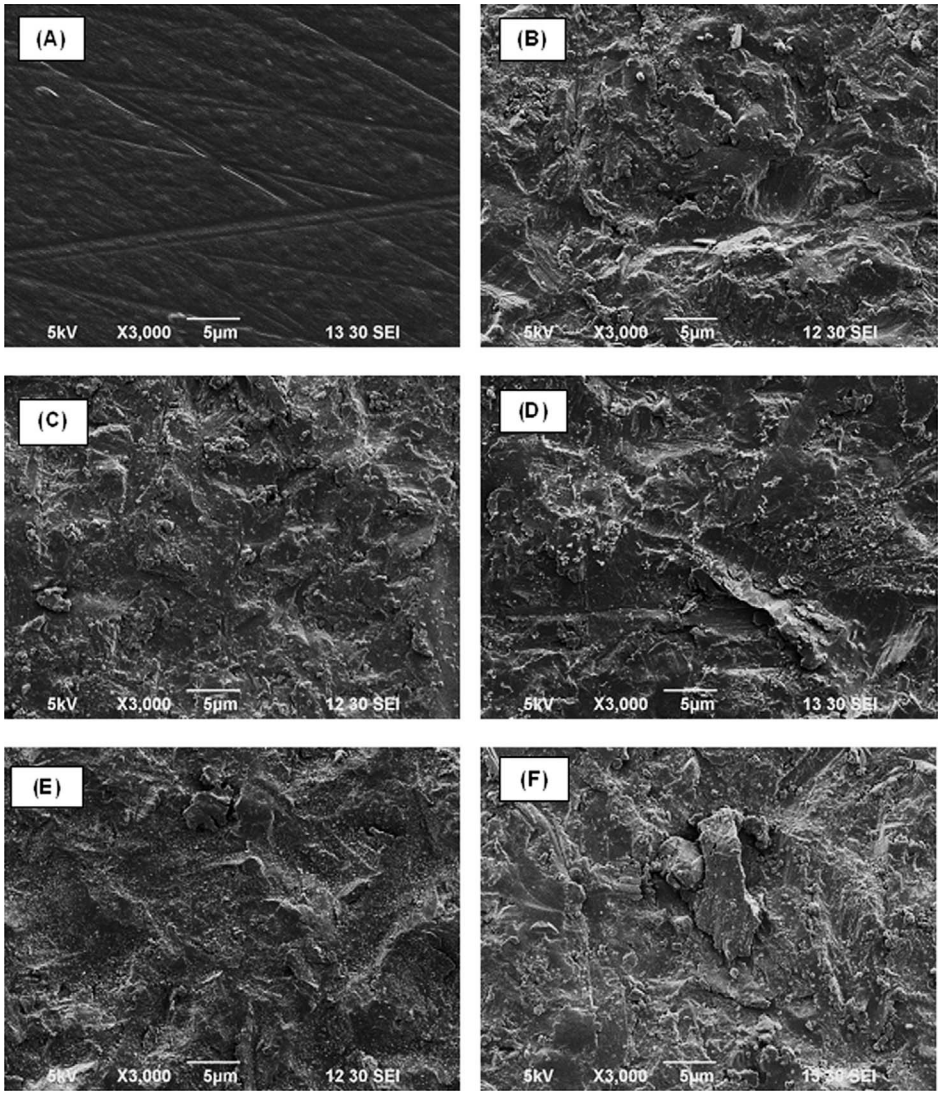

Figure 2. *(A-F) Representative SEM micrographs (3000× magnification) of (A) FCZ surface; (B) FCZ surface after sandblasting, sb; (C) FCZsb after saliva contamination, c; (D) FCZsbc and cleaned with H_3PO_4; (E) FCZsbc and cleaned with Ivoclean®; and (F) FCZsbc and cleaned with isopropanol.*

condition comparisons are valid for all groups and that the group comparisons are valid for all conditions. The effect of TC and water storage comparisons showed the following results: 150 days < TC < 24 hours. The overall group comparisons showed that phosphoric acid < isopropanol and water < cleaning paste and the control group. Figure 2 displays representative SEM micrographs for the zirconia surface morphology after the different cleaning regimens. No obvious morphological differences can be seen among the sandblasted groups (2B-F).

Table 3: *Percentage of Failure Modes Observed in Groups After Shear Bond Strength (SBS) Testing*

	Group														
Condition	**Control, %**			**Water,%**			**H_3PO_4, %[a]**			**Ivoclean, %**			**Isopropanol, %**		
Failures	**Adhesive**	**Cohesive**	**Mixed**	**Adhesive**	**Cohesive**	**Mixed**	**Adhesive**	**Cohesive**	**Mixed**	**Adhesive**	**Cohesive**	**Mixed**	**Adhesive**	**Cohesive**	**Mixed**
24 h	16	0	84	16	0	84	33	0	67	0	0	100	0	0	100
TC	7	0	93	25	0	75	7	0	93	0	0	100	0	0	100
150 d	0	0	100	0	0	100	7	0	93	0	0	100	0	0	100

[a] *Group H_3PO_4: two pretest failures during thermocycling (TC).*

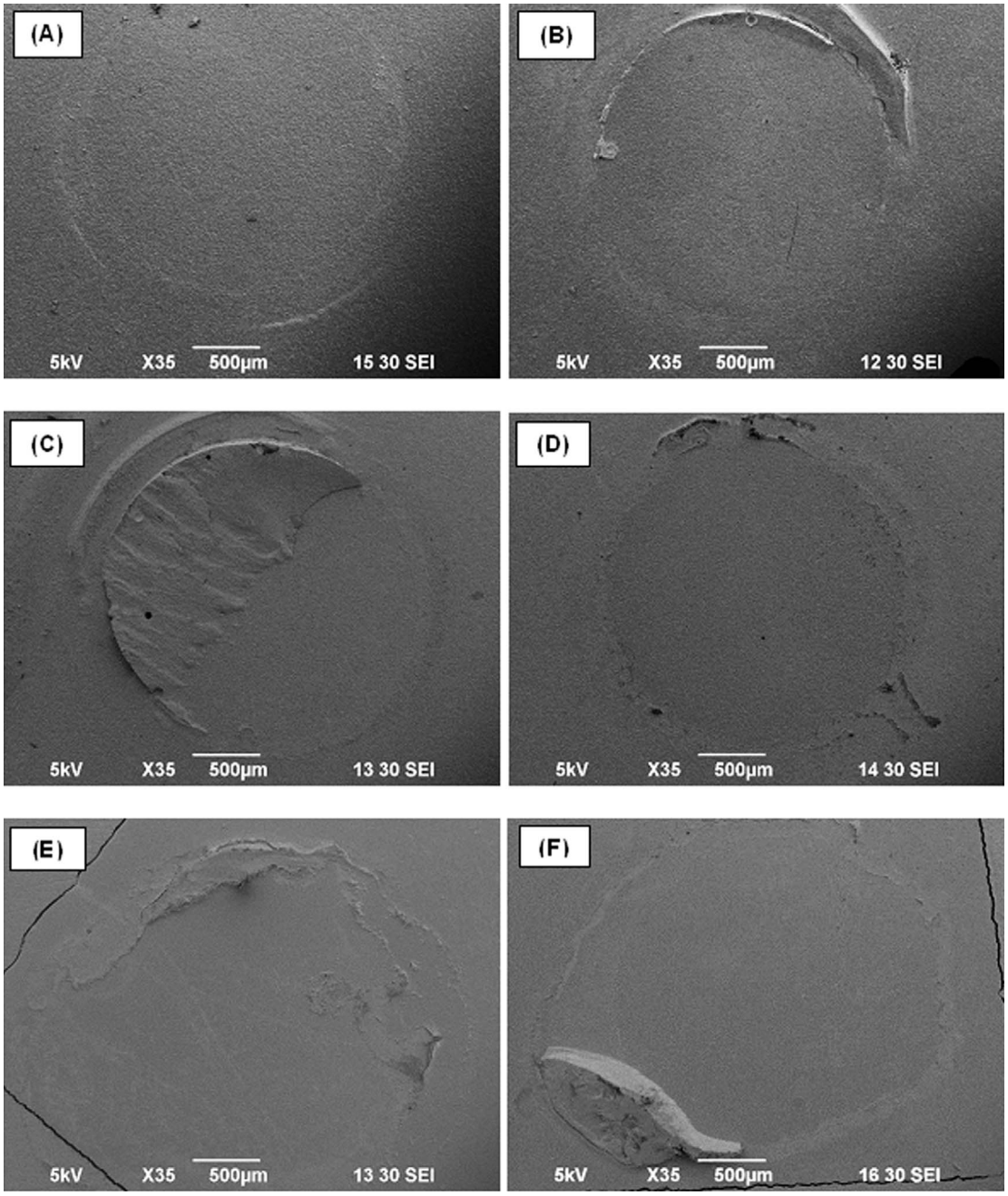

Figure 3. *Representative SEM micrographs of the debonded FCZ surface. Saliva, 24 hours (A): The failure mode was classified as adhesive; Ivoclean, 24 hours (B): The failure mode was classified as mixed with a small amount of composite resin cement on the FCZ surface. Saliva TC (C): The failure mode was classified as mixed with a significant amount of composite resin cement on the FCZ surface; Ivoclean TC (D): The failure mode was classified as adhesive. Saliva, 150 days (C): The failure mode was classified as mixed; and Ivoclean, 150 days (D): The failure mode was classified as mixed.*

Failure analysis revealed a larger percentage of mixed failure (M) for the majority of the specimens and a smaller percentage of adhesive failure at the ceramic-resin cement interface (Table 3). The water group presented a few mixed failures with small amounts of resin cement. Figure 3 shows representative SEM micrographs of the failure modes of the group that utilized the zirconium-based cleaning paste (Ivoclean®) vs control at the three conditions tested.

DISCUSSION

The challenge in promoting a strong, reliable bond between the intaglio (ie, the internal surface of zirconia restorations to resin luting agents) lies in

achieving a surface free of the contaminants that often result from intraoral try-in procedures. Previous studies have reported on different cleansing protocols, such as water,[21] alcohol (70%-96% isopropanol),[21,29] phosphoric acid (35%-37%),[21,27,29,30] and additional airborne particle abrasion (Al_2O_3).[21,31] Here, we evaluated the effect of water, H_3PO_4, isopropanol, and a fairly new cleaning paste (Ivoclean®) on the resin/zirconia SBS bond durability. The results of the present study led us to accept the null hypothesis that the cleansing method would not negatively influence zirconia bonding (Table 2) and to reject our second hypothesis, since a significant effect of the aging, especially after 150 days of water storage, promoted a significant reduction in bond strength. The group comparisons after 24 hours showed that all groups presented lower results after 150 days, except group AL, which presented statistically differences after TC and after 150 days.

It is worth mentioning that prior studies[21,30] reported that water rinsing may not be effective to remove some saliva contaminants from the zirconia surface.[21] Studies using XPS showed that H_3PO_4 seems to be an effective cleansing method with which to remove organic contaminants from saliva and blood,[21,27,29] although it, leaves phosphorous residues that could negatively impair bonding ability.[27] As a result, the adhesion between zirconia and resin cement was shown to decrease, consequently changing the surface energy,[21] being unable to reestablish the original bond strength value of the uncontaminated zirconia surface,[30] a finding that is in agreement with the results of the present study. Accordingly, this film associated with water storage and TC changes the bonding interface, which can explain some adhesive failures (Figure 3) presented in groups cleaned with water and H_3PO_4.[30]

Some authors[21] have suggested that an additional particle abrasion may provide good bonding results after contamination, comparable to that seen in groups without contamination. However, the use of a second particle abrasion could be controversial as a result of the potentially deleterious effect on zirconia phase transformation that could possibly weaken the zirconia ceramic.[32]

Several testing methodologies, namely macroshear, microshear, macrotensile, and microtensile tests, have been suggested for evaluation of the bond strength of resin-based materials to dental ceramics where load is applied in order to generate stress at the adhesive joints until failure occurs. Hence, for the test to measure the bond strength values between an adherent and a substrate accurately, it is crucial that the bonding interface should be the most stressed region, regardless of the test methodology being employed. Shear tests have been criticized for the development of nonhomogeneous stress distributions in the bonded interface. On the other hand, conventional tensile tests also present some limitations, such as the difficulty of specimen alignment. Even though the microtensile test allows better specimen alignment and a more homogeneous stress distribution, during cutting procedures the adhesive joint may suffer from early debonding, yielding to high numbers of pretest failures, especially with a zirconia substrate.[33] There is still no consensus in the dental literature with regard to the best surface conditioning method for adequate adhesion of the resin cement to highly crystalline, oxide-based ceramics, but SBS can be useful in ranking materials or systems rapidly. The best outcome could then be tested with more sophisticated methods.

A fairly new cleaning agent called Ivoclean®, which is an alkaline suspension of zirconium oxide particles (ZrO_2), has recently entered the market. In the present study, the Ivoclean group showed bond strength results comparable to those of the control group after TC and water storage. Even though TC and water storage (150 days) reduced the SBS values, the results showed that the Ivoclean and control groups maintained similar SBS values. On the basis of the present study, additional studies, for example, one that makes use of chemical composition analyses through XPS, are suggested to understand the mechanism of Ivoclean® on the saliva-contaminated zirconia surface.

CONCLUSIONS

In conclusion, our findings suggested that a cleansing protocol for zirconia ceramics must be considered after exposure to saliva. The zirconium-based cleaning paste applied on the contaminated zirconia surface is the most effective method, being comparable with the effectiveness of the uncontaminated zirconia control group.

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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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Polymerization Shrinkage and Depth of Cure of Bulk-Fill Resin Composites and Highly Filled Flowable Resin

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Clinical Relevance

As an alternative to conventional resin composites for direct posterior restorations, the use of new strategic composites that speed up the restorative procedure should be guided by careful attention to case selection and operative procedure.

SUMMARY

The aim of this study was to evaluate the polymerization behavior and depth of cure (DOC) of recently introduced resin composites for posterior use: highly filled flowable composite and composites for bulk fill. A highly filled flowable (G-aenial Universal Flo [GUF]), two bulk-fill flowables (Surefil SDR Flow [SDR] and Venus Bulk fill [VBF]), and a bulk-fill nonflowable composite (Tetric N-Ceram Bulk fill [TBF]) were compared with two conventional composites (Tetric Flow [TF], Filtek Supreme Ultra [FS]). Linear polymerization shrinkage and polymerization shrinkage stress were each measured with custom-made devices. To evaluate DOC, the composite specimen was prepared using a mold with a hole of 4 mm depth and 4 mm internal diameter. The hole was bulk filled with each of the six composites and light cured for 20 seconds, followed by 24 hours of water storage. The surface hardness was measured on the top and the bottom using a Vickers microhardness (HV) indenter. The linear polymerization shrinkage of the composite specimens after photo-initiation decreased in the following order: TF and GUF > VBF > SDR > FS and TBF ($p<0.05$). The polymerization shrinkage stress of the six composite groups decreased in the following order: GUF > TF and VBF > SDR > FS and TBF ($p<0.05$). The mean bottom surface HV of SDR and VBF exceeded 80% of the top surface HV (HV-80%). However, the bottom of GUF and TBF failed to reach HV-

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80%. A highly filled flowable (GUF) revealed limitations in polymerization shrinkage and DOC. Bulk-fill flowables (SDR and VBF) were properly cured in 4-mm bulk, but they shrank more than the conventional nonflowable composite. A bulk-fill nonflowable (TBF) showed comparable shrinkage to the conventional nonflowable composite, but it was not sufficiently cured in the 4-mm bulk.

INTRODUCTION

Because composite has shown a level of success as a restorative material, there have been continuous efforts to improve its physical and mechanical properties and the operating techniques used to apply it.[1-3] Even so, complications related to polymerization shrinkage stress and curing depth still cause significant reluctance to use them. Not only will this polymerization shrinkage stress be trapped within the material itself, but it also will exert forces on the adhesive interfaces of the dentin.[4,5] Consequently, this shrinkage stress could lead to abundant clinical problems such as microleakage, marginal gap formation, recurrent caries, pulpal irritation, and maybe even tooth loss.[2] The decrease in the degree of conversion is also a nuisance, compromising the physical properties and increasing elution of the monomer. It might lead to postoperative sensitivity and result in premature failure of the composite restoration.[6,7]

Various clinical strategies have been suggested to reduce the restorative complications in direct posterior composite restoration; these include an incremental layering technique, the use of a flowable lining layer, and the modulation of the photoinitiation mechanism.[1,8,9] Among them, incremental layering is the standard of care for placement of resin composites in cavity preparations exceeding 2 mm, by virtue of the sufficient exposure of the entire increment to the curing light, as well as the reduction of the volume of the contracting material.[10,11] Despite those strategies having been shown to be effective in improving the longevity of restorations,[11-13] clinicians still desire easier and quicker composite restorations with less shrinkage. Significant advances have been made in composite formulations that target less shrinkage and are more user friendly; these include 1) highly filled flowable, 2) bulk-fill flowable, and 3) bulk-fill nonflowable composites.

Flowable composite was introduced in the 1990s, and it was promoted because it is injectable, which is regarded as a desirable handling property and allows simplification of the placement procedure.[14-16] Typically, flowable composite has a lower filler content and higher volume of resin matrix when compared with nonflowable composite, so the first-generation flowable composite was applied as a cavity liner or Class V restoration due to the low elastic modulus. However, the recent generations of flowable composite (G-aenial Universal Flo [GUF], GC Co, Milford, DE, USA) have higher filler content and are claimed to have improved mechanical properties; thus, they are indicated not only as a cavity liner but also for larger posterior restorations.[17] The latest version of flowable composites for simplifying the restorative procedure is the bulk-filling posterior flowable. Surefil SDR Flow (SDR; Dentsply Caulk, Milford, DE, USA) and Venus Bulk fill (VBF; Heraeus Kulzer GmbH, Hanau, Germany) are intended to be placed and bulk-cured in one increment up to 4 mm. The matrix composition of these two bulk-fill flowables is based on modified urethane dimethacrylate (UDMA). The manufacturer of SDR says that it differs from conventional composites by incorporating stress-decreasing resin technology, which comprises a high molecular weight polymerization modulator in the matrix structure. This unique molecular structure contributes to the delay of the gel point, which represents an increase of viscosity through network formation, and it allows for a greater pregelation-phase time.[18] In terms of the depth of cure (DOC), these new-generation flowable composites showed satisfactory results in 4-mm increments after 20 seconds of photo-polymerization, which is recommended by the manufacturer.[19]

Along with the bulk-fill flowables, the bulk-fill nonflowable composite, Tetric N-Ceram Bulk-fill (TBF; Ivoclar Vivadent, Schaan, Liechtenstein), was recently launched with the claim that it would substitute for not only the conventional nonflowable composite but also for the bulk-fill flowables that are required for the final 2 mm when using the incremental layering technique. According to the manufacturer‘s information, this new composite will achieve full-depth bulk fill up to 4 mm without a superficial capping layer, unlike the bulk-fill flowables. The manufacturer states that TBF contains a shrinkage stress reliever to minimize polymerization shrinkage; this is a modified unique filler partially functionalized with silanes.

Up to now, an incremental layering technique has been the standard procedure in direct posterior composite restorations to reduce polymerization shrinkage stress and achieve adequate DOC.[11,20] Yet, recent advances in composite technology for

Table 1: *Materials, Manufacturers, and Chemical Composition of Matrix and Filler*

Product (Code)	Type	Manufacturer, Batch No.	Matrix System
Tetric N-Flow (TF)	Flowable	Ivoclar Vivadent, Schaan, Liechtenstein N03326	Bis-GMA, UDMA, TEGDMA
SDR (SDR)	Bulk-fill flowable	Dentsply Caulk, Milford, DE, USA 100831	Modified UDMA, EBPDMA, TEGDMA
Venus Bulk Fill (VBF)	Bulk-fill flowable	Heraeus Kulzer GmbH, Hanau, Germany 010100	UDMA, EBPDMA, TEGDMA
G-aenial Universal Flo (GUF)	High-viscosity flowable	GC Co., Milford, DE, USA 1108032	UDMA, Bis-MEPP, TEGDMA
Filtek Supreme Ultra (FS)	Nano-composite (nonflowable)	3M ESPE, St Paul, MN, USA N367463	Bis-PMA, DUDMA, Bis-GMA, TEGDMA
Tetric N-Ceram Bulk Fill (TBF)	High-viscosity bulk-fill composite (nonflowable)	Ivoclar Vivadent, Schaan, Liechtenstein R52452	Bis-GMA, DMA

Abbreviations: Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; Bis-MEPP, 2,2-Bis (4-methacryloxypolyethoxyphenyl) propane; DUDMA, diurethane dimethacrylate; EBPDMA, ethoxylated bisphenol-A dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] Prepolymer includes monomer, glass filler and ytterbium fluoride.
Note: Material information as supplied by manufacturer.

direct posterior restoration have been targeted as alternatives to the incremental layering technique. Hence, the overall evaluation of the mechanical properties and polymerization shrinkage of various strategic composites including highly filled flowables, bulk-fill flowables, and bulk-fill nonflowable composites is mandatory.

This study evaluated and compared the recently developed resin composites that are targeted as an alternative to composite restoration with the incremental layering technique, regarding their polymerization behavior and DOC. The following null hypotheses were evaluated: 1) There would be no differences in polymerization shrinkage behavior (including shrinkage strain/stress) between bulk-fill composites and conventional composites; and 2) there would be no differences in DOC between tested resin composites.

METHODS AND MATERIALS

Six brands of resin composite materials were analyzed. The chemical composition and manufacturers' information about the materials are listed in Table 1.

Linear Polymerization Shrinkage Measurement

The linear polymerization shrinkage of the composite specimens was measured using a custom-made linometer (R&B Inc, Daejeon, Korea). A fixed amount of composite was pressed between a glass slide and aluminum disk to produce specimens 1.5 mm in thickness and 4.5 mm in diameter. The tip of the linear variable differentiated transducer (LVDT) sensor (R&B Inc) was placed on the center of the glass slide with constant pressure and was set to the zero point. The light-curing unit was positioned on the custom-made light-curing unit station to ensure a constant 2-mm distance from the glass slide. An LED light-curing unit (Bluephase, Ivoclar Vivadent) was used at a light intensity of 700 mW/cm^2. The specimen was then polymerized for 40 seconds. During the light curing, the displacement distance of the disk was measured every 0.5 seconds for 120 seconds; the displacement was caused by the linear shrinkage of the composite material.

Polymerization Shrinkage Stress Measurement

The polymerization shrinkage stress of the composite specimens was measured using a custom-made device (R&B Inc). To do so, 0.3 g of the composite was carried to the acrylic disk of the measuring device. The steel rod of the device was positioned 1 mm above the acrylic disk to ensure a constant thickness of the specimen. The specimen was then polymerized for 40 seconds. As it was light cured toward the light source, the polymerization shrinkage stress of the composite specimen was measured by a load cell connected to the metal rod and computer. The data were recorded every 0.5 seconds for 180 seconds.

Depth of Cure by Vickers Microhardness

An opaque poly-acrylic mold (Dentsply Caulk), 4 mm long with an internal diameter of 4 mm, was used to prepare the composite specimens. The mold was placed on a glass slide covered with a Mylar strip, then the composite was filled in bulk for each

Table 1: Extended.

Product (Code)	Filler System	Filler Load (wt%/vol%)	Flexural Modulus (GPa)
Tetric N-Flow (TF)	Barium glass, ytterbium fluoride, and silica	63.8/43	5.3
SDR (SDR)	Barium aluminofluoride borosilicate glass	68/44	5.0
Venus Bulk Fill (VBF)	Barium aluminofluoride borosilicate glass, ytterbium fluoride, and silica	65/38	3.6
G-aenial Universal Flo (GUF)	Silica, strontium glass	69/50	7.95
Filtek Supreme Ultra (FS)	Zirconia/silica	78.5/63.3	11
Tetric N-Ceram Bulk Fill (TBF)	Barium alumino silicate glass, prepolymer filler[a]	80 (including 17% prepolymers)/60	4.5

material. The upper surface of the mold was filled with composite and was covered with a Mylar strip, followed by a glass slide. Then, the specimen was polymerized for 20 seconds, keeping the tip of the light-curing unit in contact with the 1.2-mm-thick glass slide to ensure a constant distance from the specimen. After polymerization, each specimen was removed from the mold. The specimens were stored in distilled water for 24 hours at room temperature. Subsequently, the top and bottom surface hardness of each 4-mm high specimen were measured using the Vickers microhardness instrument (HMV-2, Shimadzu, Kyoto, Japan). The measuring indenter, the Vickers pyramid, was pressed to the composite specimen using a load of 4.903 N for five seconds. The surface Vickers hardness (HV) was measured at three points of each specimen to minimize measurement errors within a specimen. The DOC, usually acknowledged as the thickness of the composite that is adequately polymerized or rather as the depth where HV equals the surface value multiplied by an arbitrary ratio, usually 0.8 (HV-80%), was calculated.[21,22] Therefore, each specimen HV of the lower surface was compared with the upper surface value and was noted when it dropped below HV-80%.

Statistical Analysis

The results of the present study were analyzed by using SAS 9.2 (SAS Inc, Cary, NC, USA). A one-way analysis of variance was applied to examine the significance of the differences in polymerization shrinkage strain occurring in 120 seconds and stress in 180 seconds. Pearson correlation analysis was used to compare the correlation between polymerization shrinkage strain and stress. The differences in microhardness between the top and bottom surfaces within each material were compared using paired *t*-tests. Scheffé and Bonferroni comparison tests were used to isolate statistical significance at the 95% confidence level.

RESULTS

Linear Polymerization Shrinkage Measurement

The patterns of linear polymerization shrinkage of the six composite materials after photo-initiation are presented in Figure 1. The amount of polymerization shrinkage of the composite specimens after photo-initiation decreased in the following order: Groups TF and GUF > VBF > SDR > FS and TBF ($p<0.05$). In all groups, the shrinkage graph curve was steep in the first 20 seconds, which coincides with the polymerization time, followed by a gradual increase.

Polymerization Shrinkage Stress Measurement

The patterns of shrinkage stress of the six composite materials after photo-initiation are shown in Figure 2. The polymerization shrinkage stress of six composite groups decreased in the following order:

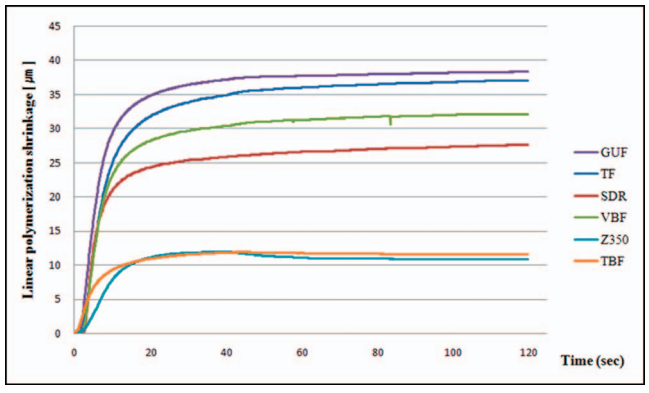

Figure 1. *Comparison of the linear polymerization shrinkage, average curves (n=8).*

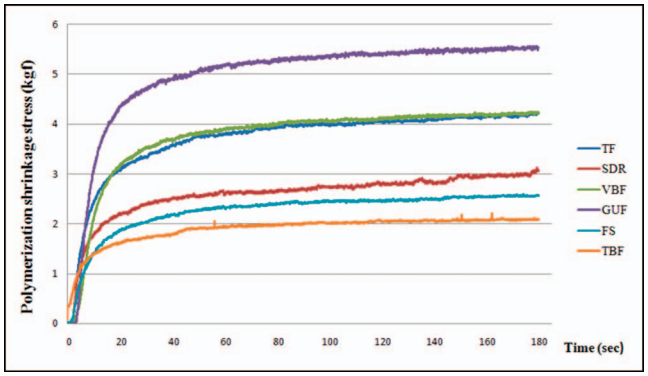

Figure 2. *Comparison of the polymerization shrinkage stress, average curves (n=8).*

Groups GUF > TF and VBF > SDR > FS and TBF ($p<0.05$).

Pearson correlation analysis showed that with respect to the shrinkage strain, shrinkage stress correlated significantly ($r=0.890$). The mean amount of linear shrinkage and the polymerization shrinkage stress for each test group are summarized in Table 2.

Depth of Cure by Vickers Microhardness

The statistical analysis of HV on the top and the bottom surfaces and the bottom-to-top ratio for each test group are presented in Table 3. All of the composites except VBF showed significantly lower HV values for the bottom compared with the top surface ($p<0.05$). Among the bulk-fill composites, the bottom surface HV of SDR and VBF, which were the bulk-fill flowables, exceeded HV-80%. However, TBF and GUF, which were bulk-fill nonflowable and highly filled flowable, respectively, failed to reach HV-80%.

Table 2. *Mean (Standard Deviation) Amount of Linear Shrinkage and Polymerization Shrinkage Stress for Each Test Group (n=8)*

Material	Linear Shrinkage (μm)	Shrinkage Stress (kgf)
TF	35.75 (2.71)A[a]	4.02 (0.37)A
SDR	25.36 (1.49)B	3.08 (0.16)B
VBF	32.14 (1.75)C	4.34 (0.35)A
GUF	38.38 (1.28)A	5.59 (0.41)C
FS	11.13 (1.15)D	2.48 (0.15)D
TBF	11.57 (1.48)D	2.09 (0.20)D

Abbreviations: FS, Filtek Supreme Ultra; GUF, G-aenial Universal Flo; TBF, Tetric N-Ceram Bulk Fill; TF, Tetric N-Flow; VBF, Venus Bulk Fill.

[a] *The measured linear shrinkage and the shrinkage stress were within 120 and 180 seconds, respectively. Different small cap letters indicate statistical differences between the groups (p<0.05). The Pearson correlation coefficient between shrinkage strain × stress was 0.890 (r=0.890, p=0.000).*

Table 3. *Comparison of Vickers Surface Hardness (HV) of Top and 4-mm Bottom, as well as Depth of Cure (HV-80%), for Each Test Group (n=8)*

Material	Top	4-mm Bottom	Bottom-to-Top Ratio
TF	35.36 (4.62)A	26.36 (6.90)B	0.74
SDR	32.14 (1.42)A	30.28 (1.73)B	0.94
VBF	30.55 (1.17)A	29.95 (1.16)A	0.98
GUF	48.54 (5.39)A	23.75 (1.51)B	0.49
FS	87.30 (6.41)A	67.21 (4.96)B	0.77
TBF	49.05 (3.82)A	37.83 (5.73)B	0.77

[a] *The samples were light polymerized for 20 seconds and stored for 24 hours at room temperature in distilled water. HV is detailed in mean and standard deviations. Different small cap letters indicate statistical differences between the top and 4-mm-depth bottom HV (p<0.05). A ratio of bottom-to-top surface microhardness over 0.80 indicates adequate DOC.*

DISCUSSION

In the current study, the polymerization shrinkage behavior and mechanical properties of recently introduced resin composites including highly filled flowables, bulk-fill flowables, and bulk-fill nonflowable composite were investigated and compared with conventional resin composites.

The flowable composites SDR and VBF, which are intended to bulk-fill, showed lower polymerization shrinkage than the conventional flowable composite. The nonflowable composites, TBF and FS, presented no significant differences in polymerization shrinkage (Figures 1 and 2). Thus, the first null hypothesis was partially rejected.

The two nonflowable composites (TBF and FS) showed significantly lower linear polymerization shrinkage compared with the flowable composites (Figure 1). In general, the flowable composite had a lower inorganic filler content and higher volume of resin matrix as compared with the nonflowable composite, and it usually exhibited a greater amount of polymerization shrinkage.[23] The flowable composites intended for bulk filling (SDR and VBF) showed lower linear shrinkage than the conventional flowable composite (TF) (Figure 1). SDR exhibited the least linear polymerization shrinkage in the tested flowables for 120 seconds, with an average of 25.36 μm (Table 2), although its shrinkage was greater than that of the two nonflowable composites. This might be attributed to the modified polymer chains of the bulk-fill flowables, which are very flexible in the pregelation phase.[24] This highly stress-relieving internal monomer might delay the gel point, which could allow more time to compensate for the shrinkage; consequently, polymerization shrinkage would be reduced.[18,19]

Polymerization shrinkage induces shrinkage stress during the curing of the resin composites. In our study, a strong correlation was observed between the shrinkage strain and stress (Table 2). The magnitude of the polymerization shrinkage stress has been found to be dependent on volumetric polymerization shrinkage and polymer elastic modulus,[9,25] whereas polymerization shrinkage is related to the degree of conversion and initial reactive group concentration.[4,26] Generally, increasing the filler load in the resin matrix results in reduction of overall shrinkage of the composite due to the reduced availability of the monomer for the curing reaction. But it also may result in a high elastic modulus of the material, which can lead to high shrinkage stress.[14,27] The present results of the shrinkage stress test showed an inverse relationship between the filler load and flexural modulus (Table 1). Concerning the polymerization shrinkage stress, which relies on the volumetric shrinkage and elastic modulus, the order of the stress value of our study was similar to the order of the estimated shrinkage strain value multiplied by each material's flexural modulus. These results coincide with those of a previous study, which hypothesized that a relevant influence of the material's stiffness on stress development was present.[28]

Of the flowable composites, SDR showed the least polymerization shrinkage stress, although it has a relatively high filler load and elastic modulus (Table 1). The modified matrix containing the shrinkage modulator might incorporate it to control the polymerization kinetics. Among the tested bulk-fill materials, TBF, which is a bulk-fill nonflowable composite, showed the least polymerization shrinkage stress. Several factors might have affected the results. First, this material contained a shrinkage stress reliever, which is a special filler functionalized with silane.[29] The manufacturer stated that the shrinkage stress reliever features a lower modulus of elasticity so that it acts like a microscopic spring, attenuating the forces generated during shrinkage.[29] Second, the material included prepolymerized fillers. Resin composites typically show a relatively low elastic modulus with the use of prepolymerized filler particles.[30]

Microhardness has been suggested as a way to examine the DOC of photo-activated resin composite. According to Bouschlicher,[31] a value over 0.80 in bottom-to-top surface microhardness indicates adequate DOC. The HV values are highly dependent on the size, weight, and volume of the filler particles as well as on the chemical composition of the composite when the test instrument produces larger indentations than the size of the filler.[32] Consequently, the HV values in our study present the average microhardness of the fillers and matrix, and for this reason, the HV value should not be considered a mechanical property and should be compared only within the same material.

In our study, the bottom surface HV values of SDR and VBF, which are bulk-fill flowables, exceeded HV-80%. The HV values of the bottom-to-top ratio of TBF, a bulk-fill nonflowable, and GUF, a highly filled flowable, were less than 0.80 (Table 3). Thus, the second null hypothesis was partially rejected.

The favorable DOC results of SDR and VBF might be attributed to the translucent matrix being highly conducible to light transmission and the incorporation of a functional photoactive group in the methacrylate matrix.[33] Previous studies[19,34] reported that bulk-fill flowables exhibited large filler size with dominant polygonally shaped features compared with conventional flowable resin composites, as seen with a scanning electron microscope. The filler load was slightly increased, but the filler-matrix interface was assumed to be decreased, due to the bigger size of the filler particle. Hence, it allows more curing light to transmit through the composite and improve the DOC.

TBF, a bulk-fill nonflowable composite, also contains a translucent filler and matrix that allow the light to pass through the material.[29] In addition, it includes Ivocerin (Ivoclar Vivadent), which is described as a germanium-based photo-initiator. According to the manufacturer, Ivocerin has a higher photo-curing activity than camphorquinone, due to its higher absorption in the region between 400 and 450 nm.[35,36] Furthermore, it can be used without the addition of an amine as coinitiator and forms at least two radicals able to initiate the radical polymerization; thus, it is more efficient than camphorquinone/amine systems with only one radical having that capability.[36,37] However, in our study, TBF presented no difference in the HV value of the bottom-to-top ratio compared with the conventional nonflowable composite, FS (Table 3). A recent study[7] was consistent with our result; it investigated the DOC of several resin composites including TBF using the ISO 4049 method and 80%-HV depth method. The author reported that the TBF specimens showed a low DOC, calculated from the bottom-to-top surface microhardness; this might have resulted in the hardness of TBF, which dropped drastically after the measurement of the superficial surface (0.1 mm).

Although the bulk-fill flowables, SDR and VBF, are indicated for restoration in bulk up to a 4 mm thickness, the manufacturers commonly recommend that these materials be covered with a 2-mm-thick capping layer by using conventional nonflowable composites.[24,37] This step is mandatory not only for reinforcing the surface hardness but also for preventing subsequent water sorption of the composite material. Recent research indicated that the composites intended for bulk fill, including SDR and VBF, are more susceptible to water deterioration in comparison with conventional composites, causing creeping deformation of the composites.[38] SDR and VBF commonly incorporate UDMA instead of bisphenol A-glycidyl methacrylate (Bis-GMA), and the matrix contents are increased to control the consistency.[18] A high-content UDMA matrix exhibits low viscosity that contributes to the void-free bulk-fill restoration. Yet it is also known as a high water-sorptive composite matrix compared with Bis-GMA or triethyleneglycol-dimethacrylate (TEGDMA).[39] The absorbed moisture may expand the matrix and induce crazing and hygroscopic expansion; it could elute the residual monomers, resulting in dimensional change of the composite restoration, and weaken the mechanical properties.[40,41] Furthermore, the results of the present study showed that the polymerization shrinkage of bulk-fill flowables was higher than that of the conventional nonflowable composite. Considering these limitations, their use as the first increment in Class II restorations as a dentin and proximal enamel replacement might result in poorer physical properties than restoration with a conventional composite. To minimize the configuration factor and restorative water sorption, the inner core of the cavity should be filled with a bulk-fill flowable first, and the placement of the conventional nonflowable composite on the outer capping layer would result in better restorative integrity in a Class II restoration. Until now, no study has been available on their use in Class II restorations without a capping layer, so this needs further investigation.

GUF was not intended to be placed in one bulk-increment, but was targeted to alternate with the resin composite for mild to moderate cavity restoration with flowable composite texture. GUF has been promoted for its unique consistency, called "injectable composite," with a glossy surface similar to that of microfill resin composite.[42] The manufacturer suggested that it could be applied not just as a cavity liner or for a small cavity but also for a larger cavity or stress-bearing area in a posterior tooth because its mechanical properties are comparable with those of conventional nonflowable composites. However, the polymerization shrinkage of GUF exceeds not only that of nonflowable composites but also that of flowable composites (Table 2). Regarding DOC, GUF exhibited the lowest HV value at the bottom surface (Table 3). Thus, its use as the alternative for conventional resin composites in an extensive posterior cavity could cause restorative failure, and it is relevant to limit the clinical indications with careful consideration.

Despite the limitations of this study, bulk-fill flowables (SDR and VBF) were properly cured in 4-mm increments, but they showed more shrinkage than conventional nonflowable composite. The bulk-fill nonflowable (TBF) showed comparable shrinkage to that of conventional nonflowable composite, but it was not sufficiently cured in 4-mm increments. The highly filled flowable (GUF) revealed, due to its polymerization shrinkage and DOC, its limitation as an alternative to conventional nonflowable composite. Further study of real restorations and long-term clinical evaluation are required for final evaluation of the suggested results.

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Conflict of Interest

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

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Influence of a Repeated Preheating Procedure on Mechanical Properties of Three Resin Composites

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Clinical Relevance

There is a lack of information about the effect of repeated preheating cycles on the mechanical properties of resin composites. It could be of high clinical interest to assess whether dental clinicians can steadily adopt preheating procedures without compromising composite mechanical strength.

SUMMARY

The aim of this study was to assess the flexural strength, flexural elastic modulus and Vickers microhardness of three resin composites prepared at room temperature or cured after one or repeated preheating cycles to a temperature of 39°C. Three resin composites were evaluated: Enamel Plus HFO (Micerium), Opallis (FGM), and Ceram X Duo (Dentsply DeTrey). For each trial, one group of specimens of each material was fabricated under ambient laboratory conditions, whereas in the other groups, the composites were cured after 1, 10, 20, 30, or 40 preheating cycles to a temperature of 39°C in a preheating device. Ten rectangular prismatic specimens (25 × 2 × 2 mm) were prepared for each group (N=180; n=10) and subjected to a three-point bending test for

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flexural strength and flexural modulus evaluation. Vickers microhardness was assessed on 10 cylindrical specimens from each group (N=180; n=10). Statistical analysis showed that, regardless of the material, the number of heating cycles was not a significant factor and was unable to influence the three mechanical properties tested. However, a significant main effect of the employed material on the marginal means of the three dependent variables was detected.

INTRODUCTION

Chairside warming of resin-based restorative materials, prior to placement and contouring, is one of the recent trends in composite application. Preheating reduces viscosity and increases flowability, which facilitates better adaptation to cavity walls.[1,2] This may result in superior marginal adaptation,[3,4] reduce microleakage, and thus enhance the durability of restorations.[5,6] The increase in temperature of a composite enhances both radical and monomer mobility, resulting in a high degree of monomer conversion[7,8] as well as an improvement of the polymerization rate.[9] As a result, more highly cross-linked polymer networking and improved mechanical and physical properties may be anticipated.[9] Daronch and others[7,9] calculated the conversion rate of a preheated composite and found that by heating the resin composites to 140°F (60°C), the conversion rate increased between 31.6% and 67.3% and therefore that less polymerization time was required. Deb and others[2] showed that the cytocompatibility of composites after preheating remains unaffected. Preheating may be achieved by placing compules or syringes of the resin composite material into commercially available preheating devices that operate at a temperature range of 39°C-68°C.[10] Some *in vitro* studies using commercially available resin composites indicate superior surface hardness and greater depth of cure for preheated composites.[1,11,12] However, in a recent *in vivo* study, Rueggeberg and others[13] showed that a warmed composite lost heat quickly once removed from the heating device and inserted into a tooth preparation. It is estimated that when a composite is heated up to 60°C and removed from the device, the temperature is reduced by 50% after 2 minutes and 90% after 5 minutes.[14] Therefore, it is clinically important to evaluate the influence of preheating under a nonisothermal condition, to simulate the real clinical scenario.[3]

Many studies[1,2,15] have disclosed that preheating protocols did not have any harmful effect on the mechanical properties of resin composite materials. However, all the *in vitro* studies in the literature have compared the mechanical properties of resin composites cured at room temperature (RT) with those of the same materials cured after a preheating cycle to a determined temperature. Only two studies analyzed the effect of repeated preheating and cooling cycles as well as extended periods of preheating on composite cure.[10,14] This information could be of extreme importance because the same composite syringe can clinically undergo numerous preheating cycles before it is completely consumed. On these bases, it could be of high interest to assess whether the mechanical properties of a cured composite can be affected by repeated preheating cycles in a preheating device operating at 39°C that improves the ease of handling and composite placement.

The aim of this *in vitro* study was to assess the flexural strength, flexural modulus, and Vickers microhardness of three different resin composites prepared at RT or cured after 1, 10, 20, 30, or 40 preheating cycles to a temperature of 39°C. The formulated null hypotheses were that mechanical properties would not show significant differences among 1) the different resin composites or among 2) the number of preheating cycles.

METHODS AND MATERIALS

Three resin composites were evaluated in this study: Enamel Plus HFO (Micerium, Avegno, Genova, Italy; HFO group), Opallis + (FGM, Produtos Odontológicos, Joinville, Brazil; OPA group), and Ceram X Duo + (Dentsply DeTrey GmbH, Konstanz, Germany; CER group). Their specifications are given in Table 1. Specimens were fabricated for two different mechanical tests: 180 beam-shaped specimens were prepared for the three-point bending test (N=180), and 180 disc-shaped specimens were subjected to the Vickers microhardness (VH) indentation test (N=180). For each test, one group of specimens of each material was fabricated under ambient laboratory conditions (21°C ± 1°C), whereas in the other groups the composites were cured after 1, 10, 20, 30, or 40 preheating cycles to a temperature of 39°C in a commercially available preheating device (ENA HEAT composite heating conditioner, Micerium; batch no. SN C1102004).

Preliminary tests were carried out on the three materials to evaluate the heating and cooling times needed at RT (21°C ± 1°C). Temperature variations of the materials were monitored with a digital multimeter equipped with a temperature microprobe

Table 1: *Summary of the Resin Composites Tested*

Material (Group)	Shade	Composition	Total Content of Filler	Particle Size	Classification	Batch Number	Manufacturer
Enamel Plus HFO (HFO)	UD3	UDMA, Bis-GMA, 1,4-butandioldimethacrylate Glass filler, highly dispersed silicone dioxide	75% by weight (53% by volume).	Glass filler: mean particle size of 0.7 μm; highly dispersed silicone dioxide: mean particle size of 0.04 μm	Microhybrid	2009000372	Micerium, Avegno, Genova, Italy
Opallis + (OPA)	EA3	Bis-GMA monomers, Bis-EMA, TEGDMA, UDMA Barium-aluminum, silanized silicate, silicon dioxide, camphoroquinone, accelerators, stabilizers, pigments	78.5% to 79.8% by weight (57% by volume)	Between 40 nm and 3.0 μm with a mean particle size of 0.5 μm	Microhybrid	80172310008	FGM Produtos Odontológicos, Joinville, Brazil
Ceram X Duo + (CER)	D3	Methacrylate modified polysiloxane, dimethacrylate resin Fluorescence pigment, UV stabilizer, stabilizer, camphorquinone, ethyl-4(dimethylamino)-benzoate, barium-aluminum-borosilicate glass, methacrylate functionalized silicon dioxide nanofiller, iron oxide pigments and titanium oxide pigments, aluminum sulfosilicate pigments	76% by weight (57% by volume)	Organically modified ceramic nanoparticles (mean 2.3 nm) and nanofillers (mean 10 nm) combined with conventional glass fillers of ~1 μm	Nanoceramic	1112001219	Dentsply DeTrey GmbH, Konstanz, Germany

Abbreviations: UDMA, diurethane dimethacrylate; Bis-GMA, iso-propyliden-bis (2(3)-hydroxy-3(2)-4(phenoxy)propyl)-bis (methacrylate) or bisphenol A diglycidyl methacrylate; Bis-EMA, bisphenol A diglycidyl methacrylate ethoxylated; TEGDMA, triethylene glycol dimethacrylate.

(GBC KDM 350, KON EL CO SpA, Milano, Italy). The composites needed a maximum of 10 minutes to reach a temperature of 39°C. The same time was required to return the composites to 21°C. As a consequence, in this study each preheating cycle consisted of 10 minutes of composite heating in a heating device and 10 minutes of composite cooling at RT. The same heating unit was used to heat all the composite syringes tested in this study.

Three-Point Bending Test

Ten specimens for each group (n=10) were prepared using a stainless-steel mold with the dimensions recommended by the ISO 4049/2000 specification (25 × 2 × 2mm) and positioned over a polyester strip.[10] The materials were inserted into rectangular molds at RT (control groups) or after 1, 10, 20, 30, or 40 preheating cycles. Each preheating cycle consisted of 10 minutes of composite heating in the heating device set at 39°C and 10 minutes of composite cooling at RT. After the last heating cycle, the heated samples were immediately packed into the molds, covered by an acrylate strip, and smoothed with a glass slide to achieve a uniform surface finish. Overlapping sections of the composite were then successively light cured for 20 seconds (Bluephase C8, with 800 mW/cm^2 output; Ivoclar Vivadent AG, Schaan, Liechtenstein). Polymerization was performed by placing the curing unit tip in direct contact with the glass slide upper surface and perpendicular to the composite specimens. The proper output and the LED efficiency of the light-curing unit were checked every 10 samples using the built-in digital radiometer of a T-LED Anthos light-curing unit (T-LED; Anthos SRL, Imola, Italy). The final temperatures of the composites before insertion into the mold were gauged with the digital multimeter (GBC KDM 350). The mean time between removing the composite from the heating device and light polymerization was approximately 40 seconds for all tests. After irradiation, any flash material on the specimens was carefully removed by gently abrading it with 320-grit abrasive paper. Specimen dimensions were checked again by measuring them

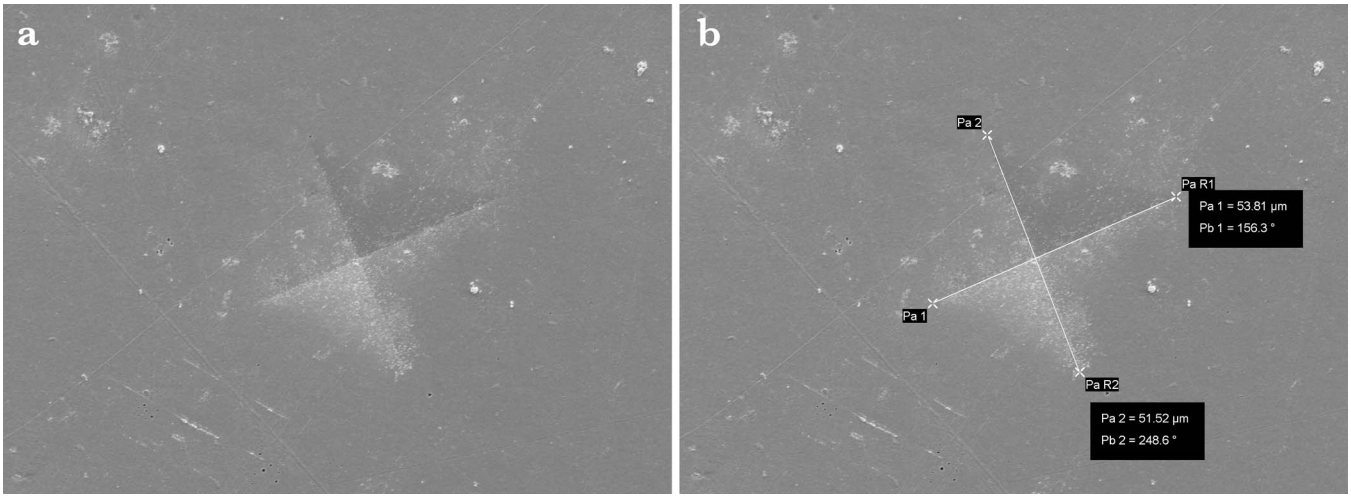

Figure 1. *Scanning electron micrograph showing a Vickers hardness (VH) indentation (a) and the measurement of its diagonals (b) on one specimen from the HFO group with no heating cycles.*

with a digital caliper (series 500 Caliper, Mitutoyo America Corp, Aurora, IL, USA). The specimens were placed into deionized water at 37°C for 24 hours. A three-point bending test was then performed using a computer-controlled universal testing machine (LLOYD LR 30K, Lloyd Instruments Ltd, Fareham, UK) at a crosshead speed of 0.5 mm/min and with a 20-mm-span distance; the load-deflection curves were recorded with PC software (Nexygen-Ondio Version 4.0, Lloyd Instruments. The fracture load (N) of the specimens was measured. Flexural strength (MPa) and flexural modulus (MPa) were then calculated for each specimen.

VH Measurement

For VH evaluation, composite pastes were placed into cylindrical molds with a 10-mm inner diameter and 2 mm high. The materials were employed at RT (control groups) or after 1, 10, 20, 30, or 40 preheating cycles. With each one of the three resin composites under investigation, 60 samples were manufactured (N=180), 10 at RT and 10 for any group of preheating cycles (n=10). Composite layering was carried out in one single increment. To achieve in all samples flat and smooth top surfaces, the uncured paste was placed inside the mold in slight excess and covered with a transparent polyester film followed by a microscope glass. Pressure was then applied to displace the excess material, and light curing was performed through the glass for 40 seconds (800 mW/cm^2 output), maintaining the curing unit tip in direct contact with the glass slide upper surface and perpendicular to the composite specimens. The proper output and the LED efficiency of the unit were checked every 10 samples using the built-in digital radiometer of a T-LED. The final temperatures of the composites before insertion into the mold were gauged with the digital multimeter (GBC KDM 350). The mean time between removing the composite from the heating device and light polymerization was approximately 40 seconds for all tests. The obtained specimens were stored at RT in black film canisters for 24 hours before subsequent procedures. VH readings were recorded on the top smooth surface of the specimens. Vickers indentations were produced by applying a 1 N load for 10 seconds using a universal testing machine with a 500 N load cell (Lloyd LR 30K, Lloyd Instruments) provided with a standard 136° Vickers diamond indenter (item #17, Affri, Induno Olona, Varese, Italy).[16] Scanning electron microphotographs (EVO 50 XVP LaB6, Carl Zeiss, Cambridge, UK) were taken at different magnifications in order to measure the linear extent of the diagonal indentations (Figures 1 through 3). Subsequently, VH numbers were calculated considering the measured diagonals (mm), and the predetermined applied load was expressed in kilograms-force (1.0204 kg). For each specimen, the mean value of three VH readings performed at approximately 2 mm distance from one another was used as raw datum.

Statistical Analysis

Data were statistically analyzed. Two-way analysis of variance (ANOVA) tests were performed to analyze the influence of the two factors (number of heating cycles and restorative material) on the mean values of the three dependent variables under

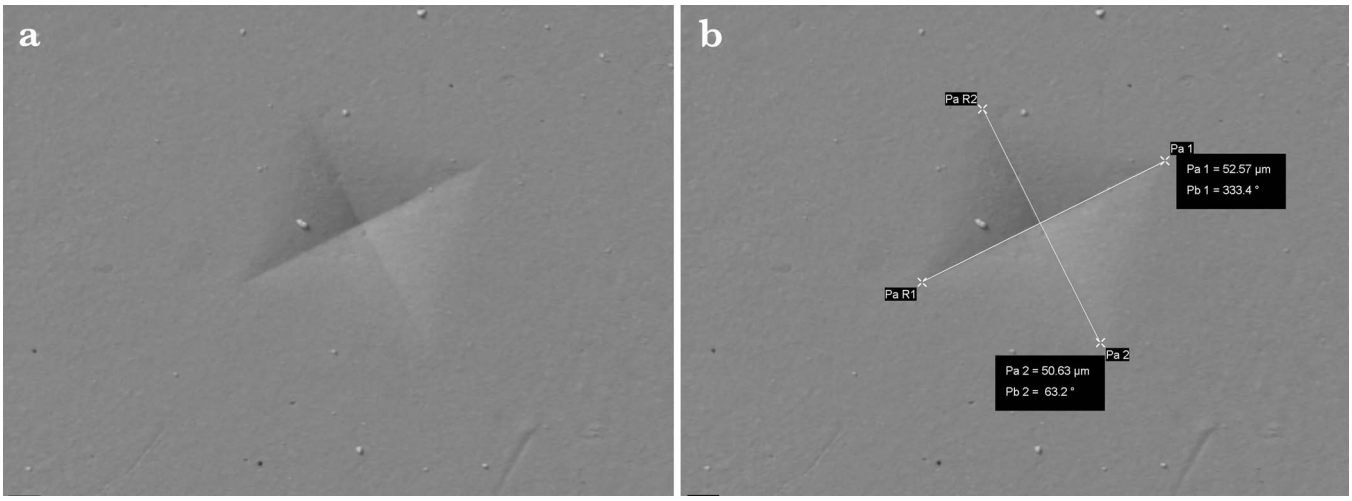

Figure 2. *Scanning electron micrograph showing a Vickers hardness (VH) indentation (a) and the measurement of its diagonals (b) on one specimen from the OPA group with 30 heating cycles.*

investigation (flexural strength, flexural modulus, and VH). Multiple comparisons were carried out according to the Tukey method. Considering each material separately, two-variable linear regression analyses were performed to investigate the presence of a linear relationship between the number of heating cycles and each mechanical property under evaluation. The number of cycles was assumed as the explanatory variable; the observed values for flexural strength, flexural modulus, and VH were the dependent variables. The sample regression function coefficients (intercept and slope) were calculated according to the ordinary least squares (OLS) method. ANOVA tables were computed to test the null hypothesis that the explanatory variable had no significant influence on each specific dependent variable; subsequently, the r^2 coefficient of determination (R^2) was calculated as the ratio between the regression sum of squares (RSS) and the total sum of squares (TSS) (R^2 = RSS/TSS). Values of p lower than 0.05 were considered statistically significant in all tests.

RESULTS

The two-way ANOVA tests showed that, regardless of the material, the number of heating cycles was not a significant factor and was unable to influence flexural strength, flexural modulus, and VH values. However, a significant main effect of the material factor on the marginal means of the three dependent variables was detected. There was no statistically

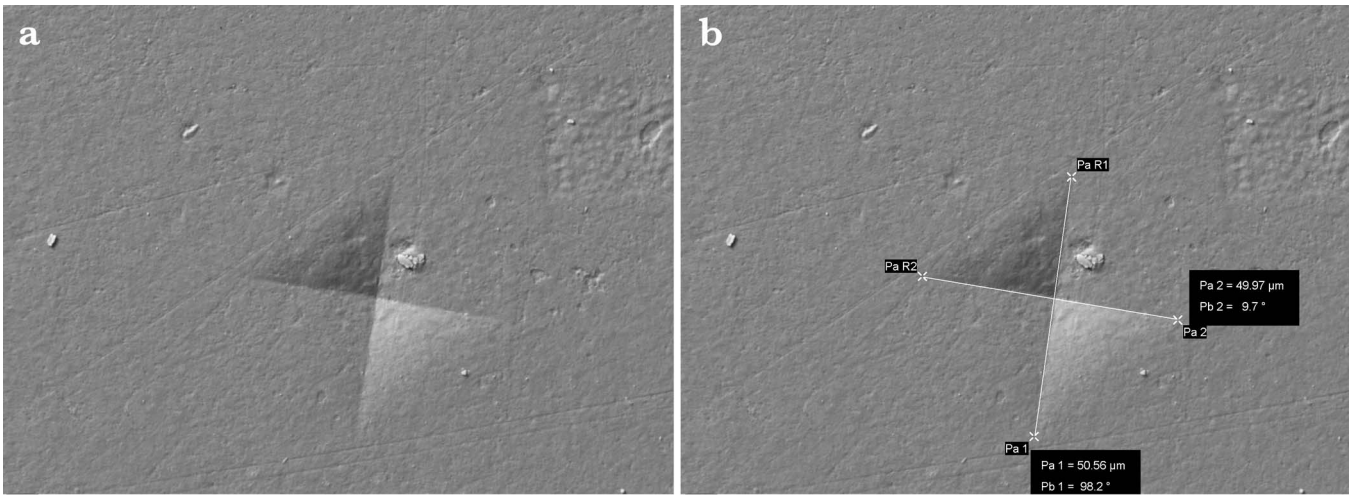

Figure 3. *Scanning electron micrograph showing a Vickers hardness (VH) indentation (a) and the measurement of its diagonals (b) on one specimen from the CER group with 30 heating cycles.*

Table 2: *Values of* p *Achieved From Two-Way Analysis of Variance Tests That Were Performed to Evaluate the Effect of the Two Factors (Material and Number of Heating Cycles) and of Their Interaction on the Mean Values of the Three Variables Under Investigation (Flexural Strength, Flexural Modulus, and Vickers Hardness)*[a]

	Flexural Strength	Flexural Modulus	Vickers Hardness
Factor	Material ($p<0.001$)	Material ($p<0.001$)	Material ($p<0.001$)
Factor	Cycles ($p=0.691$)	Cycles ($p=0.278$)	Cycles ($p=0.099$)
Interaction	Material × cycles ($p=0.532$)	Material × cycles ($p=0.814$)	Material × cycles ($p=0.572$)

[a] *Values of* $p<0.05$ *were considered statistically significant.*

significant interaction (Table 2). Mean values, marginal means, and standard deviations achieved in the different groups are shown in Tables 3 through 5.

Following regression analysis, the observed R^2 values were generally rather low, ranging between 0.003 and 0.209. All the R^2 values are given in Figure 4 together with the graphical representation of the corresponding sample regression functions.

The ANOVA tables for the performed simple linear regression analyses showed that almost all the calculated regression functions were not able to adequately account for the observed variability in the dependent variables ($p>0.05$). Concerning the VH in the OPA group, a statistically significant regression function was detected ($p=0.002$): its coefficients were 65.324 (intercept) and 0.144 (slope).

DISCUSSION

This study showed that the flexural strength, the flexural modulus, and the VH of the three composites tested were not significantly affected by the adopted repeated composite preheating technique. Within the predetermined confidence level set at 95%, only the VH variability observed for the OPA group could be statistically correlated to the increasing number of preheating cycles using a linear regression function. It should not be neglected, however, that the calculated slope (0.144) was very close to zero, leading to an almost horizontal regression line. According to this model, therefore, a great number of heating cycles would be necessary to determine even a small change in the VH values. Concerning the other mechanical properties tested and all the remaining resin composites under investigation, it was not possible to determine regression functions that could adequately correlate the observed variability to the number of preheating cycles.

The composites had a similar behavior after 1, 10, 20, 30, and 40 prewarming cycles to a temperature of 39°C in the sense that the mechanical characteristics were not significantly affected if compared with the unheated groups. Studying the effect of prewarming on mechanical properties can provide useful information to practitioners who are considering using this technique to increase flowability of composite materials. In a clinical situation, warming the composite reduces its viscosity, allowing the material to be injected into the preparation rather than manipulating it into the preparation with hand instruments.[17] The warm composite technique allows handling characteristics similar to those of a flowable composite without sacrificing the benefits of superior mechanical, wear, and polymerization shrinkage properties associated with the use of heavily filled restorative composite.[2] The reduced

Table 3: *Flexural Strength Mean Values (Standard Deviations) Achieved in Different Groups*[a]

Flexural Strength (MPa)	Heating Cycles						Overall
	0	1	10	20	30	40	
HFO	104.6	102.0	104.8	111.5	106.1	84.5	102.2[2]
	(24.2)	(23.3)	(20.0)	(17.1)	(22.0)	(22.7)	(22.4)
OPA	111.9	117.8	122.2	116.6	118.9	120.2	117.9[1]
	(18.0)	(25.1)	(16.4)	(24.3)	(17.8)	(19.9)	(19.9)
CER	104.4	100.4	100.1	103.5	97.1	100.6	101.0[2]
	(23.5)	(15.3)	(18.2)	(23.2)	(22.6)	(16.0)	(19.4)
Overall	107.0 A	106.7 A	109.0 A	110.5 A	107.4 A	101.8 A	—
	(21.6)	(22.4)	(20.1)	(21.7)	(22.2)	(24.1)	—

[a] *Same letters indicate no statistically significant differences among the levels of the heating cycles (reading horizontally). Different superscript numbers indicate significant differences among the levels of composite employed (reading vertically).*

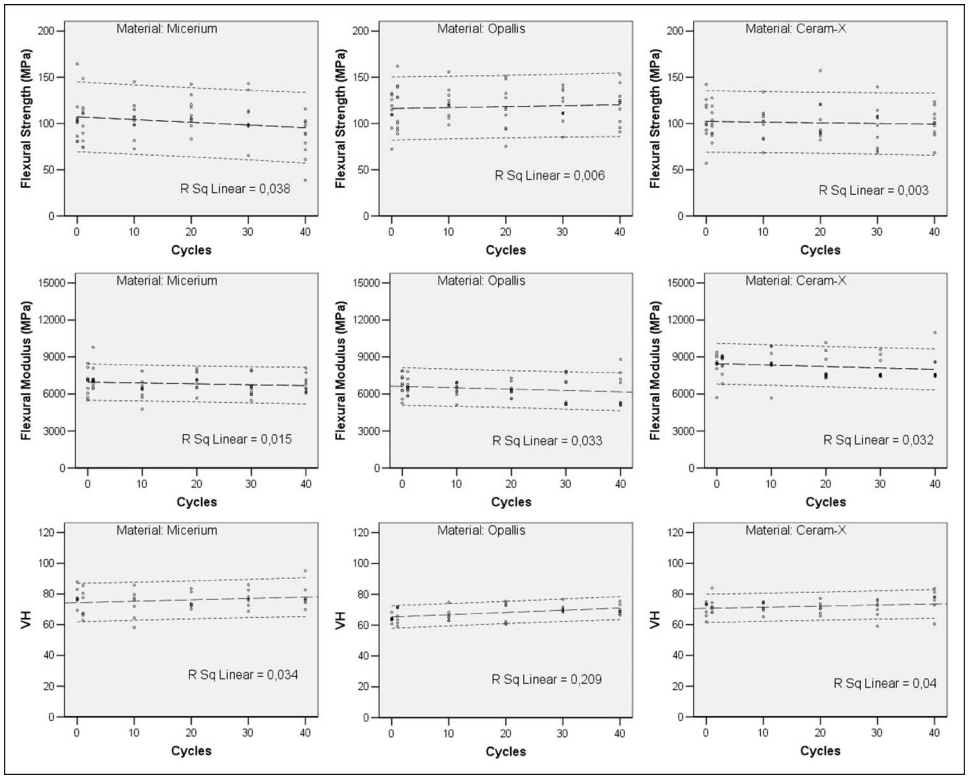

Figure 4. *Scatter plots and linear regression functions (showing also the 95% confidence intervals) obtained looking at the number of preheating cycles as the independent variable (horizontal axis) and the observed flexural strength, flexural modulus, and the Vickers hardness (VH) values as the dependent variables (vertical axes). Each material was considered separately. R Sq Linear = r^2 coefficient of determination (R^2).*

viscosity also allows for improved wetting of cavity walls compared with RT heavily filled restorative composites. This in turn provides for improved adaptation to cavity walls and decreased gap formation.[3] Viscosity of composite resin pastes is a complicated phenomenon, especially when the factor of heat is introduced.[18] The extent of viscosity change may be attributed to many factors, including resin composition, filler content, and shade. Thus, because of the wide variety in chemistry and composition of resin composites currently used, a great variation in the viscosity of these materials in response to evaluated temperatures may be expected.[15] With increasing molecular weight and the greater potential for hydrogen bonding, viscosity of the resin component will increase.[19,20] Also, increases in chain length and extent of side chain structures (branching) will tend to increase viscosity as polymer chains become more entangled.[19] Likewise, with heating, sufficient energy must be given to overcome these obstacles (hydrogen bonding and chain entanglement) to allow molecules freedom to move in a less hindered sheering pattern with respect to one another. Filler particle content, shape, and size also influence composite resin paste flow.[18] In general, the filler loading level, the filler surface contour, and

Table 4: *Flexural Modulus Mean Values (Standard Deviations) Achieved in Different Groups*[a]

Flexural Modulus (MPa)	Heating Cycles						Overall
	0	1	10	20	30	40	
HFO	6904.0	7327.9	6366.6	7072.4	6561.1	6811.2	6840.5[2]
	(979.6)	(972.7)	(807.6)	(731.7)	(802.1)	(688.8)	(862.5)
OPA	6737.3	6576.1	6390.7	6343.2	6337.6	6187.8	6428.8[3]
	(894.6)	(594.0)	(557.4)	(528.9)	(1225.0)	(1360.1)	(899.8)
CER	8376.6	8486.0	8528.4	8091.4	8013.8	8079.5	8262.6[1]
	(1015.2)	(752.2)	(1179.0)	(1029.9)	(832.4)	(1118.5)	(978.6)
Overall	7339.3 A	7463.4 A	7095.2 A	7169.0 A	6970.8 A	7026.1 A	
	(1194.7)	(1103.2)	(1338.9)	(1055.4)	(1204.9)	(1323.1)	

[a] *Same letters indicate no statistically significant differences among the levels of the heating cycles (reading horizontally). Different superscript numbers indicate significant differences among the levels of composite employed (reading vertically).*

Table 5: *Vickers Hardness Mean Values (Standard Deviations) Achieved in Different Groups*[a]

Vickers Hardness	Heating Cycles						Overall
	0	1	10	20	30	40	
HFO	78.2	72.5	73.4	75.3	77.2	78.8	75.9[1]
	(5.8)	(8.6)	(9.4)	(5.1)	(5.8)	(8.2)	(7.3)
OPA	64.1	66.4	66.6	68.6	70.9	70.0	67.8[3]
	(2.2)	(5.2)	(4.1)	(6.8)	(2.8)	(3.2)	(4.7)
CER	70.1	72.5	71.3	71.1	70.5	75.8	71.9[2]
	(4,8)	(5,5)	(3,5)	(3,8)	(6,0)	(7,6)	(5,4)
Overall	70.8 A	70.4 A	70.4 A	71.6 A	72.9 A	74.9 A	—
	(7.3)	(6.9)	(6.6)	(5.8)	(5.8)	(7.4)	—

[a] *Same letters indicate no statistically significant differences among the levels of the heating cycles (reading horizontally). Different superscript numbers indicate significant differences among the levels of composite employed (reading vertically).*

the distribution of filler size impacts the ability of particles to easily slide past one another. Heating would not directly affect the glassy particle itself because, within the temperature range imparted at clinically relevant temperatures, the viscosity of the filler particle (a ceramic) remains unchanged. Coatings on the filler particle could affect the ease with which a filler particle would move in the warmed resin fluid. Particles not silanated would be more difficult to move than those that are coated, as silanization imparts better resin wetting and, thus, ease of fluid movement around the particle.[20] An additional advantage of heating the resin composite is that preheated light-curing composites can be easily used as luting agents for porcelain veneers[22,23] or indirect composite restorations[24] in place of dual-curing materials.[25,26]

There is a general consensus in the literature on the absence of harmful effects of preheating procedures on the mechanical properties of resin composites.[2,3,15] In a recent study, Osternack and others[27] concluded that composite hardness was not affected by precooling or preheating procedures. However, the majority of previous studies did not consider repeated preheating cycles. Daronch and others[14] reported that neither prolonged preheating nor 10 repeated continuous preheating cycles (cycles of 15 minutes from RT to 60°C) affected the degree of conversion of preheated composites compared with composites maintained at RT. However, in a recent study, D'Amario and others[10] concluded that highly repeated preheating cycles (40 preheating cycles to a temperature of 45°C) seem to negatively influence the flexural strengths of three commercially available resin composites; this seems to be the only study that takes into account more than 10 preheating cycles. Since in clinical use a standard composite syringe can be used to fill more than 20 cavities, especially if a multishade layering technique is steadily adopted, the authors concluded that the adoption of single-use composite compoules instead of syringes would be considered preferable if a preheating procedure to a temperature of 45°C were steadily adopted. In contrast, the present study showed that even highly repeated cycles of preheating to a temperature of 39°C did not negatively influence the mechanical properties of the resin composites tested. The effect of warming at 39°C in this study was considered sufficient to obtain an increased flowability and a better adaptation of the composites. In contrast with other studies that reported a slightly lower composite temperature compared with that of the heating source,[10,14] in this study all the composites achieved a maximum temperature of 39°C after 10 minutes with the preheating device preset to 39°C.

CONCLUSIONS

The tested preheating procedure did not negatively influence the mechanical properties of the resin composites even when highly repeated. Further studies might be needed to assess the clinical relevance of the other variables connected to the repeated preheating and cooling cycles. Based on these findings and within the limitations of the study, dental clinicians can steadily adopt this preheating procedure without compromising the mechanical strengths of the heated composites.

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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Bulk-Fill Resin Composites: Polymerization Contraction, Depth of Cure, and Gap Formation

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Clinical Relevance

The filling of deep, wide cavities with bulk-fill resin composites is appealing. However, in Class II cavities some bulk-fill resin composites result in larger gaps on dentin walls than observed for a conventional resin composite.

SUMMARY

The bulk-filling of deep, wide dental cavities is faster and easier than traditional incremental restoration. However, the extent of cure at the bottom of the restoration should be carefully examined in combination with the polymerization contraction and gap formation that occur during the restorative procedure. The aim of this study, therefore, was to compare the depth of cure, polymerization contraction, and gap formation in bulk-fill resin composites with those of a conventional resin composite. To achieve this, the depth of cure was assessed in accordance with the International Organization for Standardization 4049 standard, and the polymerization contraction was determined using the bonded-disc method. The gap formation was measured at the dentin margin of Class II cavities. Five bulk-fill resin composites were investigated: two high-viscosity (Tetric EvoCeram Bulk Fill, SonicFill) and three low-viscosity (x-tra base, Venus Bulk Fill, SDR) materials. Compared with the conventional resin composite, the high-viscosity bulk-fill materials exhibited only a small increase (but significant for Tetric EvoCeram Bulk Fill) in depth of cure and polymerization contraction, whereas the low-viscosity bulk-fill materials produced a significantly larger depth of cure and polymerization contraction. Although most of the bulk-fill materials exhibited a gap formation similar to that of the conventional resin composite, two of the low-viscosity bulk-fill resin composites, x-tra base and Venus Bulk Fill, produced larger gaps.

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INTRODUCTION

Bulk-filling techniques have become more widely used following the development of materials with improved curing,[1,2] controlled polymerization contraction stresses,[3,4] and reduced cuspal deflection.[5] Using this approach, the number of increments required to fill a cavity is reduced in comparison with traditional incremental filling techniques. In contrast to the maximum 2-mm increments recommended for conventional resin composites, manufacturers recommend 4- or 5-mm increments of the bulk-fill resin composites. The use of the bulk-fill technique undoubtedly simplifies the restorative procedure and saves clinical time in cases of deep, wide cavities. However, the data available for these materials are currently limited,[6] and therefore further laboratory studies are required in order to provide insight into likely clinical outcomes.

The use of thicker increments in bulk-fill resin composites is due to both developments in photoinitiator dynamics and their increased translucency,[7] which allows additional light penetration and a deeper cure.[8,9] Other than the improved depth of cure, recently developed bulk-fill resin composites exhibit lower polymerization contraction stress and contraction rates than hybrid and flowable resin composites.[3] However, a higher modulus of elasticity and increased plastic deformation suggest that the interfacial stress accumulation generated when using these bulk-fill materials, as well as the resulting consequences such as cuspal deflection and marginal gaps, may be difficult to predict.[3]

Gap formation may result from excessive contraction stresses at the interface between the restoration and the tooth,[5,10,11] which can be a consequence of the polymerization rate of the material[12] and the magnitude of polymerization contraction.[11,13] Additionally, contraction stresses are influenced by the composition and filler content of the resin composite,[1,13,14] its elastic modulus,[12,15] and its ability to flow, and thus compensate for the stresses generated during polymerization.[11-13,16] The degree of conversion[12,13,17] as well as depth of cure[18] of the material are also likely to influence the development of stresses, which may affect the quality of the bond at the interface of restorations. In materials with increased polymerization contraction, the interfacial stresses are more likely to be higher than can be compensated for by relaxation of the material [16] and cuspal deflection.[5,19,20] If these interfacial stresses exceed those that can be supported by the adhesive layer, gap formation will occur,[21-23] thus compromising the adhesive reinforcement of the tooth structure. Additionally, if the resin composite has limited depth of cure, it is likely to generate less contraction stress around the cavity walls and margins, thus possibly disguising an improved marginal adaptation due to poor polymerization. The complexity of interaction between some of these factors[1,13,15] may be further aggravated in cavities with an increased C-factor[24,25] or in the deeper and wider cavities, which are often encountered in the occlusal and approximal surfaces of posterior teeth.

Earlier research has demonstrated lower cuspal deflection after restoration of mesio-occlusodistal (MOD) cavities with two bulk-fill materials when compared with a nanohybrid resin composite.[5] This corroborates the previously reported findings of lower polymerization contraction stresses for a bulk-fill resin composite.[3] Finally, under fatigue testing, similar marginal integrity was observed in MOD cavities restored with one type of bulk-fill material and conventional resin composites.[26] Despite the positive results reported from previous studies, bulk-fill resin composites are somewhat recent materials with varied composition and handling characteristics, and thus have different physical properties.[2,3,6,27-30] Additionally, the availability of newer bulk-fill materials justifies further investigations because the overall properties of resin composite materials are usually composition-dependent.[6,27] Therefore, the aim of this study was to investigate the polymerization contraction, depth of cure, and gap formation of bulk-fill resin composites. The null hypotheses investigated were that 1) the polymerization contraction, 2) the depth of cure, and 3) the gap formation of bulk-fill resin composites are similar to those observed for a conventional resin composite.

METHODS AND MATERIALS

The polymerization contraction and depth of cure of high-viscosity (Tetric EvoCeram Bulk Fill, Ivoclar Vivadent, Schaan, Liechtenstein; SonicFill, Kerr Corporation, Orange, CA, USA) and low-viscosity (x-tra base, Voco GmbH, Cuxhaven, Germany; Venus Bulk Fill, Heraeus Kulzer GmbH, Hanau, Germany; SDR, Dentsply Caulk, Milford, DE, USA) bulk-fill resin composites were compared with a conventional resin composite (Tetric EvoCeram, Ivoclar Vivadent). The investigated materials were extruded from their respective capsules with the help of a manual applicator, with the exception of SonicFill, which due to its higher viscosity was extruded using its respective sonic handpiece (Kavo SonicFill, Kavo Dental GmbH, Biberach, Germany) attached to pressurized air.

Table 1: *Investigated Restorative Materials and Their Composition According to Information Provided by the Respective Manufacturers*

Composite	Monomers	Fillers
Venus Bulk Fill, Heraeus, Lot: 010031	Urethane dimethacrylate Ethoxylated bisphenol A dimethacrylate	Barium glass Ytterbium trifluoride Silicon dioxide (65 wt%, 38 vol%)
SDR, Dentsply Caulk, Lot: 1106281	Modified urethane dimethacrylate Ethoxylated bisphenol A dimethacrylate Triethyleneglycol dimethacrylate	Barium glass Strontium glass (68 wt%, 45 vol%)
x-tra base, Voco, Lot: 1137400	Dimethacrylates	Inorganic fillers (75 wt%)
Tetric EvoCeram Bulk Fill, Ivoclar Vivadent, Lot: P48869	Urethane dimethacrylate Bisphenol A dimethacrylate	Barium glass Ytterbium trifluoride Mixed oxide Prepolymer (79-81 wt%, 60-61 vol%)
SonicFill, Kerr, Lot: 3739797	Ethoxylated bisphenol A dimethacrylate Bisphenol A dimethacrylate Triethyleneglycol dimethacrylate	Barium glass Silicon dioxide (83.5 wt%)
Tetric EvoCeram, Ivoclar Vivadent, Lot: P40104	Urethane dimethacrylate, Bisphenol A dimethacrylate	Barium glass Ytterbium trifluoride Mixed oxide Prepolymer (82-83 wt%)

Polymerization Contraction

Polymerization contraction of the investigated materials (Table 1) was assessed with the bonded-disc method.[31] Triplicates were conducted for each investigated material. Standard amounts of the different materials (0.22 ± 0.02 g), which corresponded approximately to one application capsule, were inserted on top of a glass plate attached to a metallic ring. On top of the ring, a thin glass lamina was positioned. A linear variable differential transformer (LVDT; 7DCDT-100, Hewlett-Packard, Waltham, MA, USA) connected to a power output of 5 V rested on the surface of the thin glass lamina. A light-emitting-diode device (950±50 mW/cm^2, bluephase, Ivoclar Vivadent) placed underneath the glass lamina was used to light-activate the investigated materials for 20 seconds. When light-activation was initiated, the materials contracted and deformed the glass lamina, thus resulting in displacement of the LVDT. The displacement of the LVDT was registered at two, five, 20, and 60 minutes after irradiation in a plotter (LKB Bromma 2210 2-channel recorder, Bromma, Sweden). Values of vertical linear displacement of the LVDT after 60 minutes were converted to polymerization contraction (strain measured as a percentage) using the formula:

$$e_{\%} = (d_p \times 100)/(c_1 \times c_2 \times L_0) \quad (1)$$

where $e_{\%}$ = strain (%); d_p = displacement of plotter tip on graph paper (m); c_1 = LVDT scale factor (V/m); c_2 = plotter scale factor (m/V); and L_0 = original length of sample (m).

In order to calculate the strain from the displacement of the plotter tip on the graph paper, it was necessary to determine the values of the variables c_1, c_2, and L_0. The value of c_1 was obtained by performing a calibration of the LVDT. This calibration involved measuring the voltage output of the LVDT while displacing the LVDT rod in controlled increments using a micrometer head. A linear regression was performed on these points, which led to a value of c_1 equal to 951 V/m (R^2=0.9999). The value of c_2 corresponded to the scale factor set on the plotter and was equal to 4 m/V. The original sample length L_0 corresponded to the thickness of the metallic ring in the experimental setup and was equal to 1.93 mm. Substituting these values of the variables into equation (1) demonstrated that 1 mm of displacement on the graph paper corresponded to a polymerization contraction of 0.26 μm, or the equivalent strain of 0.014%. The error associated with these measurements was approximately 2%.

Depth of Cure

The depth of cure of the investigated materials (Table 1) was assessed according to International Organization of Standards 4049.[32] Each material was inserted in a metallic mold with an orifice of 4 mm in diameter and 12 mm in depth. The mold was pressed between polyester strips covered by glass slides and placed on white filter paper. The material was light-activated (950±50 mW/cm^2, bluephase, Ivoclar Vivadent) from the upper orifice during 20 seconds. Each specimen was removed from the mold, and the uncured material in the bottom was scraped

off with a plastic spatula. The height of the hardened material was measured in the center of the specimen with a micrometer (Carl Mahr GmbH, Esslingen, Germany), and this value was divided by two in order to determine the depth of cure. Triplicates were conducted for each investigated material.

Gap Formation

Gap formation was assessed in Class II cavities (vertical slot cavities) in extracted human molars using a method modified from Dewaele and others.[23] The teeth were extracted for therapeutic reasons; the research complies with the Use of Anonymous Human Biological Material Act on Research Ethics Review of Health Research Projects (from June 14, 2011), the National Committee on Health Research Ethics, Denmark. A total of 96 standardized cavities were prepared under water cooling in the approximal surfaces of the molars, with these dimensions (±0.5 mm): width, 4 mm; height, 6 mm; depth, 2 mm. The teeth were then divided into six groups (n=16): five experimental and one control. In the experimental groups, the cavities were filled with a bottom layer of the bulk-fill materials and an occlusal layer of the conventional resin composite. Cavities in the control group were filled incrementally with the conventional resin composite. Because the manufacturers recommend an occlusal coverage of the low-viscosity bulk-fill materials with a conventional resin composite to offer improved esthetics and mechanical performance,[27] the same condition was reproduced for all the investigated materials, including the high-viscosity bulk-fill materials (although this is not a recommendation from the manufacturers).

The enamel and dentin surfaces of each cavity were etched with 37.5% phosphoric acid (Gel Etchant, Kerr Italia Srl, Scafati, Italy) for 30 and 10 seconds, respectively. After rinsing for 15 seconds, the excessive water was removed without dehydrating the dentin. The primer (Optibond FL, 1 Prime, Kerr Italia Srl) was actively applied in the cavity, followed by air-drying for five seconds. The adhesive (Optibond FL, 2 Adhesive, Kerr Italia Srl) was then actively applied, air-dried for three seconds, and light-activated for 20 seconds (950±50 mW/cm^2, bluephase, Ivoclar Vivadent). In the experimental groups, a 4-mm increment of the designated bulk-fill resin composite (Table 1) was inserted into the cavity and against a metallic matrix (Hawe Contoured Matrices, KerrHawe SA, Bioggio, Switzerland) and then light-activated for 20 seconds. This increment was subsequently covered by a 2-mm increment of the conventional resin composite (Tetric EvoCeram, Ivoclar Vivadent) that was light-activated for 20 seconds. The control group was restored with four oblique increments of the conventional resin composite (Tetric EvoCeram), each light-activated for 20 seconds.

After the restorative procedure, the teeth were stored in water for 10 minutes prior to preparation for the gap analysis. Specimens were not subjected to thermocycling or cyclic loading, so that the effect of the restorative material alone could be assessed. The gap formation between the restorative materials and the dentin was assessed in faciolingual (n=6) or mesiodistal (n=10) sections. Each section was sequentially ground with wet paper discs #220, #500, and #1000 (Labopol-1, Struers A/S, Rødovre, Denmark) and polished with aluminum oxide powder to obtain a flat and regular surface. After polishing, each section was rinsed with pressurized water, dried with absorbent paper, and then analyzed in the light microscope (Orthoplan, Ernst Leitz GmbH, Wetzlar, Germany) under 510× magnification. The dentin-restoration interface was analyzed at seven sites in the faciolingual sections (Figure 1): the midgingival wall, the faciogingival and linguogingival angles, and two sets of points along the dentinal facial and lingual walls where the largest gaps and its corresponding direct opposite locations were observed. In the mesiodistal cuts, six reference points were used to analyze the dentin-restoration interface (Figure 1): the gingival cavosurface margin, half the distance of the gingival wall, the axiogingival angle, and respectively one-fourth, one-half, and three-quarters of the height of the dentinal axial wall. The size of the gaps in the different locations was measured using a reference scale visible in the objective of the microscope. A mean gap was calculated for each individual section, and an average gap formation was obtained for each investigated material from the combined mesiodistal and faciolingual sections.

Statistical Methods

Polymerization contraction and depth of cure were analyzed by a one-way analysis of variance test and the Tukey honestly significant difference (HSD) post hoc test. Due to their lack of normal distribution, gap measurements were analyzed by the Mann-Whitney U-test, each group being compared against the control group. Possible correlations between the investigated properties were analyzed using the Pearson test. The level of significance was 5%.

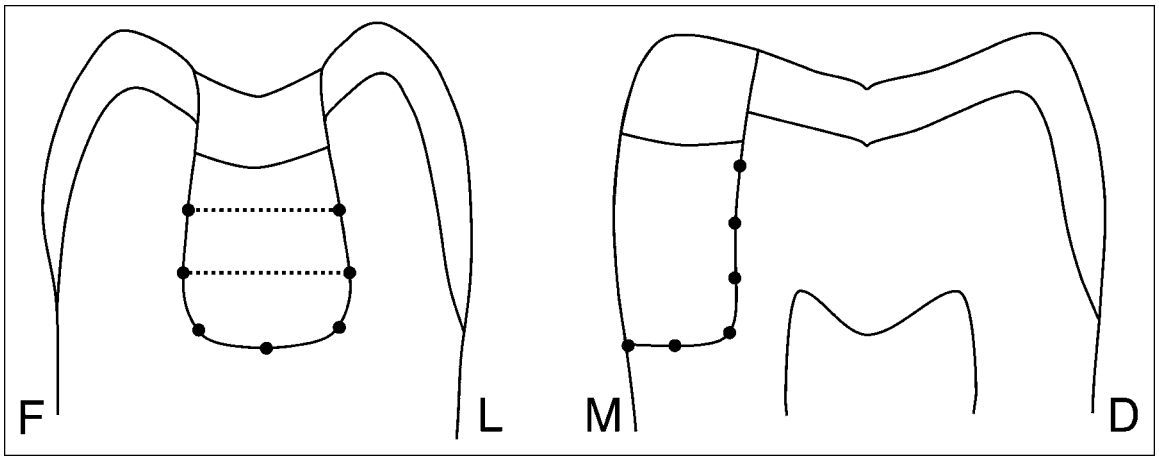

Figure 1. *The schematic drawing represents with dots the locations where the gaps between the restorative materials and dentin were assessed, both in faciolingual (FL) or mesiodistal (MD) sections.*

RESULTS

Significantly different polymerization contraction (Figure 2) was observed between the investigated materials ($p<0.001$). The conventional resin composite Tetric EvoCeram presented the lowest polymerization contraction, not significantly different from SonicFill ($p=0.061$) but significantly lower than Tetric EvoCeram Bulk Fill ($p=0.001$, Table 2). The low-viscosity bulk-fill resin composites demonstrated higher polymerization contraction: SDR and x-tra base showed an intermediate behavior, whereas Venus Bulk Fill presented the highest polymerization contraction ($p<0.001$, Table 2).

Depth of cure (Figure 3) was, in general, improved for the bulk-fill resin composites when compared with the conventional resin composite ($p<0.001$). SonicFill demonstrated depth of cure statistically similar to that of the conventional resin Tetric EvoCeram ($p=0.056$; Table 2). The low-viscosity bulk-fill resin composites x-tra base and Venus Bulk Fill demonstrated significantly higher depth of cure compared with the low-viscosity bulk-fill resin composite SDR ($p<0.001$) or the high-viscosity bulk-fill resin composite Tetric EvoCeram Bulk Fill ($p<0.001$) (Table 2).

In general, the low-viscosity bulk-fill resin composites investigated in this study demonstrated higher polymerization contraction and depth of cure. A significant positive correlation was identified between the polymerization contraction and the depth of cure of the investigated materials ($r^2=0.806$, $p<0.001$).

Gap formation (Figure 4) was significantly larger for x-tra base ($p=0.005$) and Venus Bulk Fill ($p=0.016$) when compared with the conventional resin composite (Tetric EvoCeram) (Table 2). No significant difference in gap formation was observed between the conventional resin composite and SDR ($p=0.880$), Tetric EvoCeram Bulk Fill ($p=0.925$), or SonicFill ($p=0.243$) (Table 2).

Gap formation was positively correlated with depth of cure of the investigated materials ($r^2=0.736$, $p=0.029$). No significant correlation was observed between gap formation and polymerization contraction of the investigated resin composites ($r^2=0.599$, $p=0.71$). However, because a pattern was identified within the results and one particular material seemed to stand out from the others, a second Pearson correlation test was conducted, this time excluding SDR. With the exclusion of SDR, a strong positive correlation was identified between gap formation and polymerization contraction (excluding SDR: $r^2=0.975$, $p=0.002$), thus indicating that this material has a different behavior from the other investigated materials (Figure 5).

DISCUSSION

Gap formation is the consequence of an interaction among several factors,[1,13,15] which adds complexity to understanding this phenomenon. This study focused on two of the factors involved in gap formation: polymerization contraction and depth of cure.

Because the polymerization contraction of most of the bulk-fill materials was higher than that of a conventional resin composite (Figure 2), the first null hypothesis was rejected. The low-viscosity bulk-fill resin composites containing lower filler volume (Venus Bulk Fill, SDR, and x-tra base) demonstrated higher polymerization contraction values. Converse-

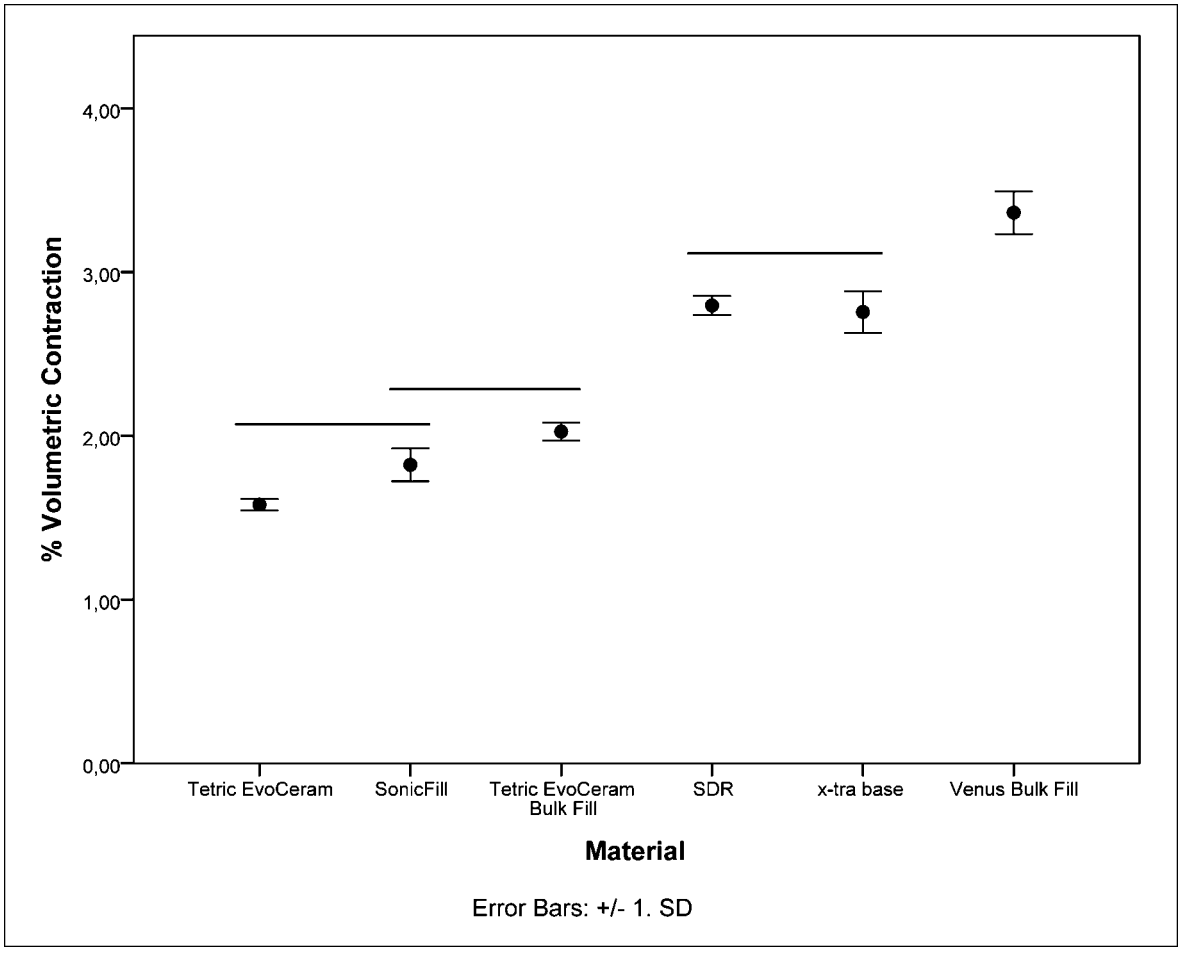

Figure 2. *Polymerization contraction (%) for the investigated materials obtained 60 seconds after light-activation using the bonded-disc method. The horizontal lines indicate the homogeneous grouping obtained from the Tukey HSD post hoc test.*

Table 2: *Average Contraction (%) and Depth of Cure (mm), With Respective Standard Deviations, as well as Median Dentin Gap Formation and Range (μm) for Each Investigated Resin Composite*

Composite	Contraction, %[a]	Depth of Cure, mm[a]	Gap, μm[b]
Venus Bulk Fill, Heraeus	3.36 (0.13) D	5.57 (0.28) D	Median, 10.2*
			Range, 3.6-31.7
SDR, Dentsply Caulk	2.80 (0.06) C	4.34 (0.15) C	Median, 6.1
			Range, 3.3-33.0
x-tra base, Voco	2.76 (0.13) C	5.68 (0.21) D	Median, 9.3*
			Range, 5.2-36.6
Tetric EvoCeram Bulk Fill, Ivoclar Vivadent	2.03 (0.05) B	3.82 (0.08) BC	Median, 6.6
			Range, 3.2-21.1
SonicFill, Kerr	1.83 (0.10) AB	3.43 (0.07) AB	Median, 7.1
			Range, 3.9-18.0
Tetric EvoCeram, Ivoclar Vivadent	1.58 (0.04) A	2.90 (0.28) A	Median, 6.2
			Range, 3.0-12.3

[a] *For contraction and depth of cure, different letters represent significant differences (Tukey HSD post hoc test, $p<0.05$).*
[b] *Median dentin gap formation and range (μm) from the combined mesiodistal and faciolingual sections of teeth restored with the investigated materials.*
* *For dentin gap formation, indicates significant differences between the bulk-fill resin composites and the conventional composite (Mann-Whitney U-test, $p<0.05$).*

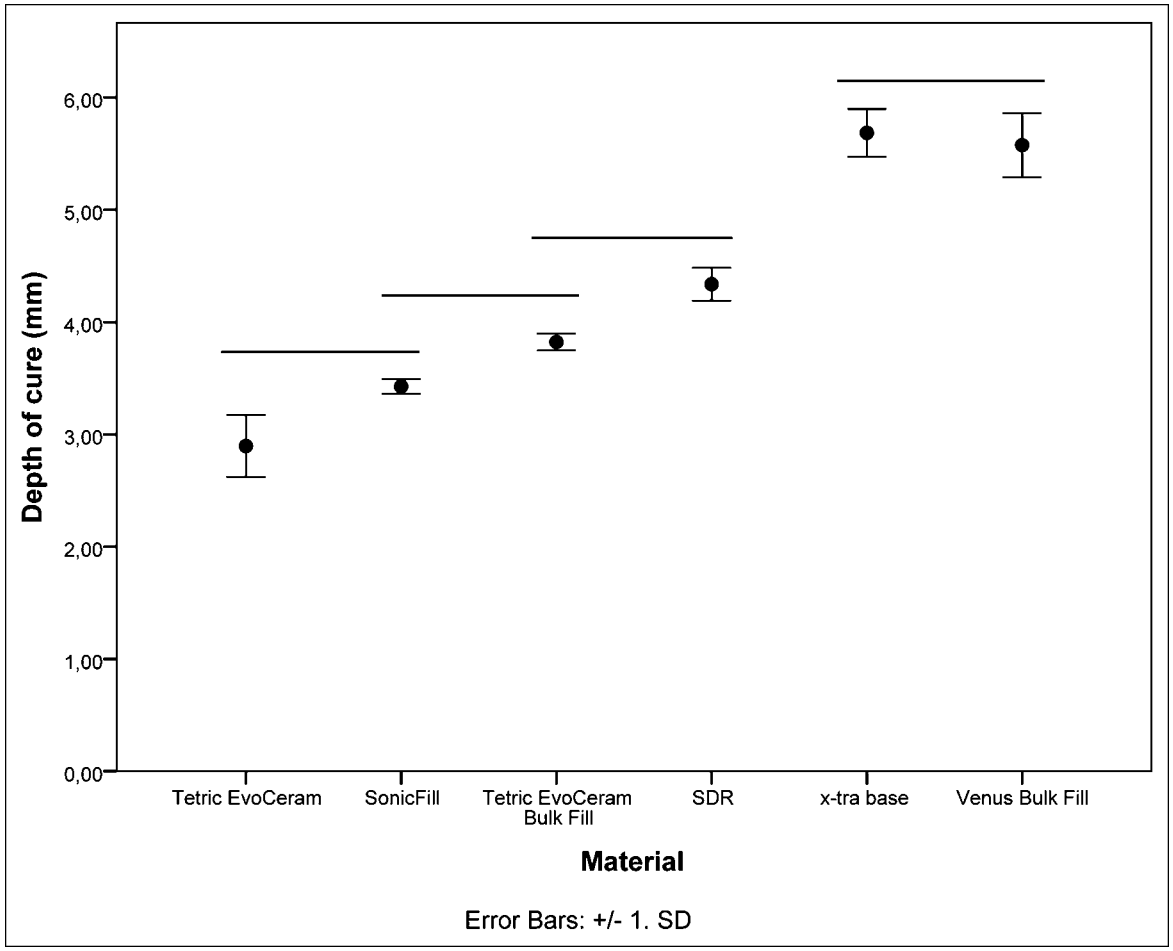

Figure 3. *Depth of cure (mm) of the investigated materials according to ISO 4049. The horizontal lines indicate the homogeneous grouping obtained from the Tukey HSD post hoc test.*

ly, high-viscosity bulk-fill resin composites with higher filler fraction (SonicFill and Tetric EvoCeram Bulk Fill) presented polymerization contraction values closer to the conventional resin composite (Tetric EvoCeram). An increase in the filler content can, to a certain extent, reduce the polymerization contraction[13,14] due to the decrease in the monomer content in relation to the filler-to-monomer ratio. In general, the polymerization contraction of all the investigated materials was between 1.58% and 3.36%, which is considered acceptable when compared with the polymerization contraction of the resin composites currently available on the market.

The depth of cure for most of the bulk-fill materials was improved when compared with the conventional resin composite (Figure 3). Nevertheless, the second null hypothesis was partially accepted due to the fact that SonicFill demonstrated a depth of cure statistically similar to that of the conventional resin composite Tetric EvoCeram. With a mean depth of cure of 3.43 mm, SonicFill also failed to comply with the requirement from ISO 4049, which states that the individual values for depth of cure of a material shall be no more than 0.5 mm below the value stated by the manufacturer.[32] The manufacturer of SonicFill states that the material has adequate depth of cure up to 5-mm increments based on hardness and degree of conversion data; yet, this study followed the ISO 4049. Because the method proposed by ISO 4049 tends to overestimate the depth of cure when compared with hardness profiles,[8,33] especially for bulk-fill resin composites,[8] it is surprising that SonicFill did not perform better in the current study. Tetric EvoCeram Bulk Fill also showed a depth of cure slightly lower than the value advertised by its manufacturer, as has been previously reported.[8] However, together with the other investigated low-viscosity bulk-fill resin composites (SDR, Venus Bulk Fill, and x-tra base), Tetric EvoCeram Bulk Fill demonstrated higher depth of cure when compared with the conventional resin

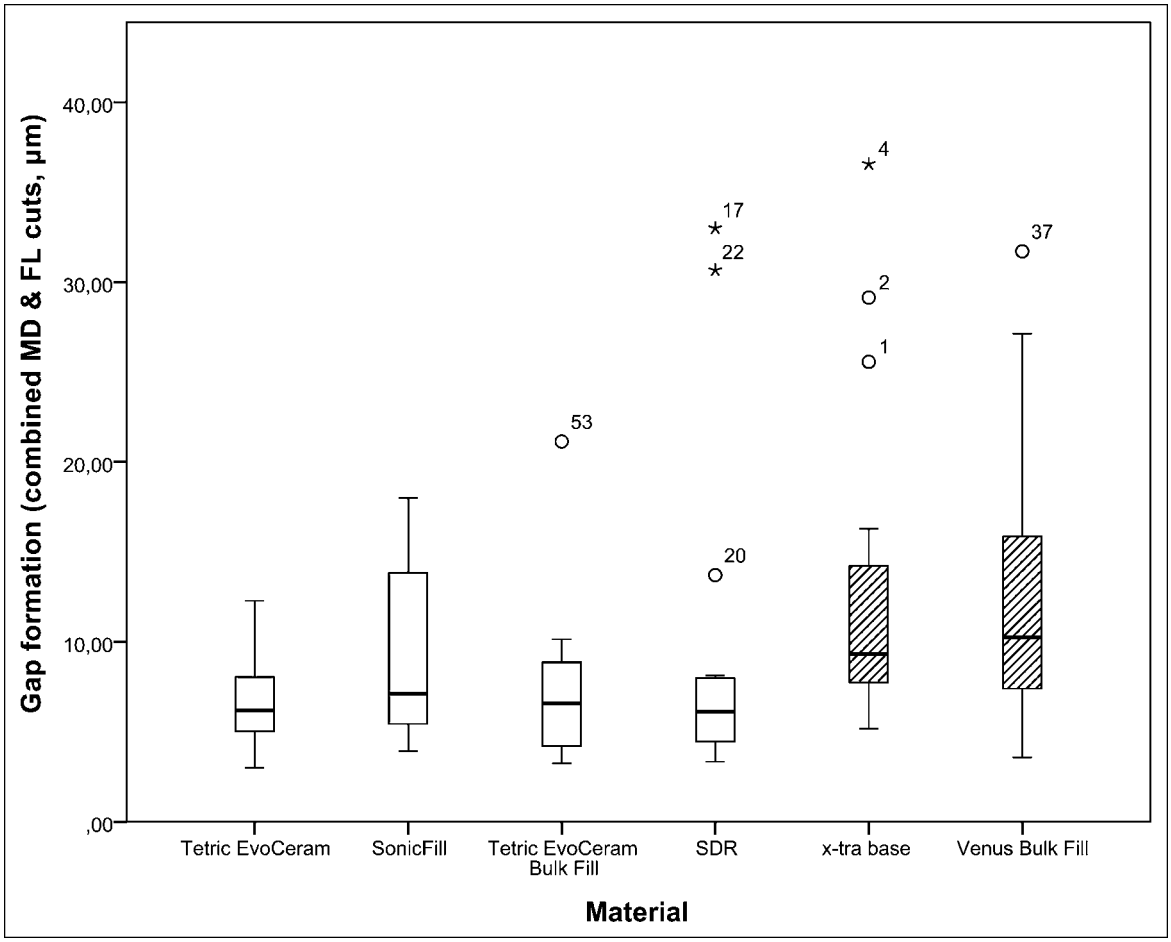

Figure 4. *Gap formation (median, minimum, and maximum, in µm) between the investigated materials and the dentin from combined mesiodistal (MD) and faciolingual (FL) sections. The striped boxes identify the materials for which gap formation was significantly larger than that of the conventional resin composite (Tetric EvoCeram), according to the Mann-Whitney U-test.*

composite. Higher depth of cure has been reported earlier for bulk-fill resin composites[8,9] due to improvements in their initiator system[8] and increased translucency.[7,8]

Among the many factors involved in gap formation, the quality and compliance of the adhesive bond play an important role in maintaining good contact between the resin composite and the cavity walls.[1,11,13,24-26,34] This is most critical in the absence of enamel, which was the case in the gingival margins of the cavities examined in this study. Therefore, a recognized, good-quality bonding system[34] was used that minimized the chance of gap formation due to poor bonding and allowed examination of the role of restorative materials in gap formation. It should be emphasized, however, that different outcomes may result from diverse bonding systems,[26] and perhaps a distinct behavior would have been observed had other bonding systems been investigated in this study.

Despite the use of a bonding system of recognized quality, none of the restorations were gap-free, as shown previously.[11] Some of the bulk-fill materials resulted in wider gaps than those observed for a conventional resin composite (Figure 4), despite their lower contraction stresses and the lower flexural modulus reported in an earlier study.[4] Therefore, the third null hypothesis was rejected. In the present study, the gaps were wider at the gingival walls, which is in accordance with previous data.[35] Gap formation was observed in all specimens, although to different extents. This is in contradiction with the data published by Roggendorf and others,[26] who observed predominantly gap-free margins in the absence of thermomechanical loading. Possible explanations for the different results, other than the different methods used for analyzing the gaps, are that the previously mentioned authors used MOD cavities. The increased compliance of an MOD cavity, when compared with the vertical slot cavities

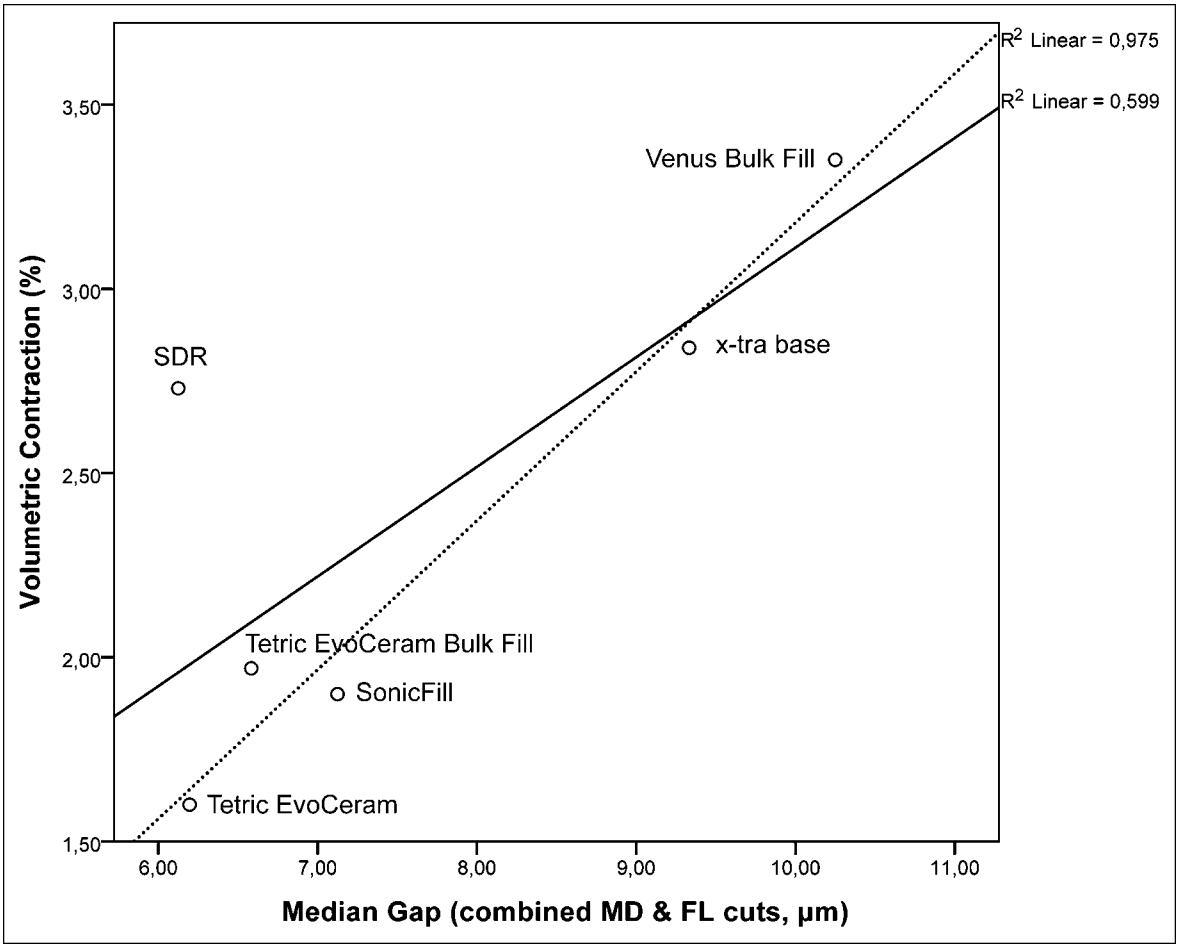

Figure 5. *Linear fit for gap formation (median from combined mesiodistal and faciolingual sections) and polymerization contraction (%) from the Pearson correlation test for all investigated materials (solid line, $r^2=0.599$) and with the exclusion of SDR (dotted line, $r^2=0.975$).*

used in this study, is a consequence of the flexibility of the cusps and the possibility of cuspal deflection.[5,13,19] The vertical slot, on the other hand, is a more rigid model with less mobility of the cavity walls, and induced stresses are therefore more likely to result in the rupture of the bonding, with subsequent gap formation.

A further analysis of our results demonstrated that the high-viscosity bulk-fill resin composites with reduced polymerization contraction (SonicFill and Tetric EvoCeram Bulk Fill) resulted in similar gap formation when compared with the conventional resin composite. It is acknowledged that polymerization contraction of a material is not the sole factor involved in the development of contraction stresses[12,15] and gap formation around cavity margins.[5,10,11] This fact was confirmed in part during this study: When all of the investigated materials were taken into account, no correlation was observed between polymerization contraction and gap formation. Nevertheless, polymerization contraction is one of the most important factors[13] affecting the development of contraction stresses,[11,12,14] which may to a certain extent help to explain gap formation. The results from the present study further support the fact that the polymerization contraction plays a role in stress development, and consequent gap formation, around cavity margins. Indeed, when a second Pearson correlation test excluding SDR was performed between gap formation and polymerization contraction, a significant and strong correlation was present (Figure 5). A strong linear correlation between polymerization contraction and its resulting stresses has been previously reported for most resin composites by Kleverlaan and Feilzer.[14]

Despite the higher polymerization contraction of SDR when compared with Tetric EvoCeram, SonicFill, and Tetric EvoCeram Bulk Fill, its gap formation was not significantly higher. Previous results for Tetric EvoCeram Bulk Fill have demon-

strated lower contraction stresses than for a conventional resin composite.[4] Positive results regarding gap formation were reported earlier for SDR, in a thermomechanical loading setup, when compared with conventional resin composites using different adhesive systems.[26] Other than the previously reported reduced polymerization contraction stresses,[3-5] a possible explanation for the positive results around SDR margins may be its lower flexural modulus[3,4,27] combined with its slower contraction rate,[3,4] which allowed the material to partially counteract the effect of polymerization contraction, thus resulting in gap formation similar to that of the conventional resin composite. The elastic modulus of resin composites has been considered an important aspect for both the polymerization contraction and development of polymerization contraction stresses.[4,12,14,15] Furthermore, a direct relationship between polymerization stress,[21,22] polymerization contraction,[23] and marginal integrity has been demonstrated *in vitro*. Additionally, in a current ongoing clinical study, restorations made with the recently developed bulk-fill resin composite SDR covered with a conventional resin composite were not yet significantly different from restorations made with a conventional resin composite following the three-year evaluation.[36]

The majority of studies regarding the development of polymerization contraction stress have been reported through mechanical testing[13] or indirectly through measurements of cuspal deflection[20] combined with microleakage assessments.[5,19] In the present study, an indirect appraisal of the polymerization contraction stresses generated by bulk-fill resin composites was assessed by gap formation. Although the application of laboratory results in clinical practice is limited or maybe uncertain, the results from this study further corroborate perspectives from previous research[2-5,8,25-27] that point out bulk-fill resin composites as promising restorative alternatives. However, further laboratory and long-term clinical investigations of bulk-fill resin composites remain necessary[6,36] before we can conclude that this new category of materials performs as well as the conventional resin composite.

CONCLUSIONS

Within the limitations of the present study, it is possible to conclude that the investigated high-viscosity bulk-fill resin composites (Tetric EvoCeram Bulk Fill and SonicFill) demonstrated, to some extent, polymerization contraction values and gap formation similar to the conventional resin composite, although their depth of cure was marginally below the values claimed by their respective manufacturers. Conversely, some of the investigated low-viscosity bulk-fill materials (x-tra base and Venus Bulk Fill) demonstrated higher contraction and unfavorably larger gap formation despite improved depth of cure, when compared with the conventional composite. One particular bulk-fill material (SDR) had improved depth of cure and comparatively low gap formation despite higher polymerization contraction.

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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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Fracture Resistance and Microleakage of Endocrowns Utilizing Three CAD-CAM Blocks

HM El-Damanhoury • RN Haj-Ali • JA Platt

Clinical Relevance

Fracture resistance and mode of failure of CAD/CAM-fabricated monoblock endocrowns varies widely between materials. Clinicians should be cautious with material selection for endocrown restorations.

SUMMARY

This study assessed marginal leakage and fracture resistance of computer-aided design/computer-aided manufacturing (CAD/CAM) fabricated ceramic crowns with intracoronal extensions into the pulp chambers of endodontically treated teeth (endocrowns) using either feldspathic porcelain (CEREC Blocks [CB], Sirona Dental Systems GmbH, Bensheim, Germany), lithium disilicate (e.max [EX], Ivoclar Vivadent, Schaan, Liechtenstein), or resin nanoceramic (Lava Ultimate [LU], 3M ESPE, St Paul, MN, USA).). Thirty extracted human permanent maxillary molars were endodontically treated. Standardized preparations were done with 2-mm intracoronal extensions of the endocrowns into the pulp chamber. Teeth were divided into three groups (n=10); each group was restored with standardized CAD/CAM fabricated endocrowns using one of the three tested materials. After cementation with resin cement, specimens were stored in distilled water at 37°C for one week, subjected to thermocycling, and immersed in a 5% methylene-blue dye solution for 24 hours. A compressive load was applied at 35 degrees to long axis of the teeth using a universal testing machine until failure. Failure load was recorded, and specimens were examined under a stereomicroscope for modes of failure and microleakage. Results were analyzed using one-way analysis of variance and Bonferroni *post hoc* multiple comparison tests (α=0.05). LU showed significantly ($p<0.05$) higher fracture resistance and more favorable fracture mode (ie, fracture of the endocrown without fracture of tooth) as well as higher dye penetration than CB and EX. In conclusion, although using resin

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nanoceramic blocks for fabrication of endocrowns may result in better fracture resistance and a more favorable fracture mode than other investigated ceramic blocks, more microleakage may be expected with this material.

INTRODUCTION

Restoration of endodontically treated teeth continues to be a challenge in reconstructive dentistry. A common protocol of restoring such teeth has been to build up the tooth with a post and core to aid the retention of an overlying crown. This can be achieved through a direct approach using a prefabricated intraradicular post followed by a direct core material or through an indirect post and core restoration for teeth with more extensive loss of tooth structure. However, many clinical and laboratory studies have reported that placing a post will contribute to the retention of the core portion of the restoration but may have a weakening effect on the root.[1-5] The use of intraradicular post and cores is complicated by the necessity to prepare an adequate ferrule, which reduces the risk of failure through root fracture.[6] Failure of post and core systems may be due to different mechanical behaviors relative to tooth structure in response to intraoral cyclic stresses.[7] This failure can be classified as repairable failure (favorable fracture) or nonrepairable failure (catastrophic fracture) that requires extraction of the tooth and subsequent prosthetic replacement.[8,9]

With the increasing popularity of adhesive dentistry, a shift in treatment decisions toward more conservative modalities has been observed, and the need for conventional post and cores has become less clear.[10] Ceramic inlays, onlays, and endocrowns have been introduced as alternative restorations for endodontically treated molars, depending on the availability of remaining tooth structure.[11-14] Initially proposed by Pissis[15] in 1995, endocrowns are a type of restoration consisting of the entire core and crown as a single unit (ie, monoblock). Endocrowns use the available surface of the pulp chamber axial walls as macroretentive resources and adhesive resin cement as a means of micromechanical retention.[16] Additionally, this type of restoration is made available through computer-aided design/computer-aided manufacturing (CAD/CAM) technology, which provides the possibility for chair-side design and fabrication.

Endocrowns are especially indicated in cases of inadequate clinical crown length, insufficient interocclusal space, and extensive loss of dental tissues that do not allow the use of an adequate ferrule.[17] Moreover, endocrowns have the advantage of preserving tooth structure, reducing the need for auxiliary macroretentive features, and saving patient's and operator's time due to fewer clinical steps and absence of the laboratory procedures needed for fabricating conventional crowns. This approach has shown promising results and comparable short-term survival when compared to post, core, and crown systems.[18-22]

A wide collection of ceramic materials has been available for CAD/CAM technology, ranging from relatively weak feldspathic ceramic and leucite glass ceramic to high-strength lithium disilicate glass ceramic and zirconium oxide.[23] Most recently, a resin nanoceramic has been introduced for permanent CAD/CAM fabricated restorations.[24,25] Ultrastructure, physical, and mechanical properties of available CAD/CAM materials vary widely, and, accordingly, their mechanical behavior in the tooth-restoration complex is expected to vary as well.[26,27]

With the intent of increasing the amount of information about the biomechanical behavior of these materials when used for endocrowns, the present study evaluated the microleakage, fracture resistance, and failure modes of three types of CAD/CAM fabricated restorations when they were submitted to an oblique compressive force.

METHODS AND MATERIALS

Tooth Collection and Preparation

Thirty freshly extracted human permanent maxillary first and second molars with approximately similar mesiodistal/buccolingual dimensions and root length were collected after patients' informed consents were obtained under a protocol approved by the institutional review board and in conformity with the university's guidelines for handling biological tissues. Teeth were ultrasonically cleansed of calculus and soft tissues, stored in a 1% chloramine-T solution at 4°C, and used within one month. Teeth were sectioned parallel to the occlusal surface at 2 mm above the cementoenamel junction (CEJ) to remove occlusal tooth structure and to deroof the pulp chamber.

Endodontic Procedures

Removal of pulp tissues was done with an endodontic reamer, and determination of root canal lengths was done radiographically with endodontic files inserted in the canals. Standardized canal enlargement was performed with an engine-driven rotary NiTi system (ProTaper, Dentsply Maillefer, Ballaigues, Switzer-

Table 1: *The Materials Tested in This Study*

Code	Material	Manufacturer	Batch Number	Ceramic Type	Fracture Toughness (MPa $m^{1/2}$)[a]	Modulus of Elasticity (GPa)[a]
CB	CEREC Blocks	Sirona Dental Systems	108290	Aluminosilicate (feldspathic) ceramic	1.4 (0.2)	45.0 (0.5)
EX	e.max CAD	Ivoclar Vivadent	P84622	Lithium disilicate glass ceramic	2.6 (0.3)	81.0 (3.3)
LU	LAVA Ultimate	3M ESPE	N333039	Resin nanoceramic	2.0 (0.2)	12.8 (1.0)

[a] *Values obtained from manufacturer's data.*

land) using a crown-down technique; 1% NaOCl was used as an irrigant for 10 seconds between each file. Root canals were obturated with a thermoplasticized gutta-percha (Calamus Dual, Dentsply Maillefer, Woodinville, WA, USA) and root canal sealer (AH 26 sealer, Dentsply Maillefer) according to the manufacturer's instructions, providing a standardized filling procedure.

The superior aspect of the gutta-percha material was removed using a small carbide bur to 1 mm below the orifice of each canal, then flowable resin composite (Filtek Z350XT flowable, 3M ESPE, St Paul, MN, USA) was used to fill the canals up to the level of the pulp chamber.

Endocrown Preparation

The teeth were individually fixed in fast-cure acrylic resin (Fastray, Harry J. Bosworth Co, Skokie, IL, USA) using polyvinyl chloride rectangular molds. The roots were embedded in resin up to 2 mm below the CEJ (simulated bone level). Intracoronal height of the prepared walls was reduced to 2.0 mm, measured from the internal cavity margin to the floor of the pulp chamber, using a periodontal graded probe.

A standardized cavity preparation was performed in all teeth limited to removal of undercut areas of the pulp chamber and alignment of its axial walls with an internal taper of 8-10 degrees using a tapered diamond coated stainless-steel bur with a rounded end (G845KR, Edenta, Basel, Switzerland) held perpendicular to the pulpal floor. All internal line angles were rounded and smoothed using the same type of bur. The axial walls were prepared from the pulpal side to provide for a standardized cavity wall thickness of 2.0 $\pm$ 0.2 mm measured with a digital caliber (Mitutoyo IP 65, Kawasaki, Japan) having a precision of 0.001 mm.

Endocrown Fabrication and Thermocycling

CAD/CAM ceramic endocrowns were fabricated with a CEREC AC system by using the software package provided (CEREC 3D, version 3.8, Sirona Dental Systems GmbH, Bensheim, Germany). All endocrowns were designed to have similar occlusal anatomy by using the biogeneric reference option as well as having the same occlusogingival height. Teeth were randomly distributed into three equal groups (n=10) according to the block material: feldspathic block ceramic (CB), lithium-disilicate blocks (EX) and resin nanoceramic blocks (LU). Tested materials are listed in Table 1.

Before cementation, the marginal adaptation of the endocrowns was checked using a Measurescope (UM-2, Nikon, Tokyo, Japan), and any specimen with a marginal gap >40 microns was rejected and replaced with a new specimen. Intaglio surfaces of each endocrown were treated according to the manufacturer's instructions for the respective block material. Etching with 5% hydrofluoric acid gel (IPS Ceramic Etching Gel, Ivoclar Vivadent, Schaan, Liechtenstein) was done for 60 seconds for CB or 20 seconds for EX, then rinsed for 60 seconds with running water and dried for 30 seconds with oil-free, moisture-free air. Intaglio surfaces of LU crowns were sandblasted with $\leq$25-μm aluminum oxide particles (MicroEtcher CD, Danville Materials, San Ramon, CA, USA), then sand was removed with alcohol and dried with oil-free, moisture-free air. A ceramic primer containing silane coupling agent (Monobond Plus, Ivoclar Vivadent) was applied to the intaglio surfaces of all endocrowns and allowed to dry for 60 seconds.

Prepared tooth surfaces were etched with 37% phosphoric acid–etching gel for 15 seconds, rinsed for 20 seconds, and dried with oil-free air for another 5 seconds. Dentin primer (Syntac, Ivoclar Vivadent) was applied for 15 seconds and dried thoroughly for 10 seconds, then dentin adhesive (Syntac, Ivoclar Vivadent) was applied for 10 seconds and dried thoroughly for another 10 seconds. Adhesive resin (Heliobond, Ivoclar Vivadent) was applied and air blown to a thin layer for 15 seconds. All specimens were cemented with dual cure resin cement (Variolink II, Ivoclar Vivadent) under a constant load of 50 g for 30 seconds. Excess material was removed

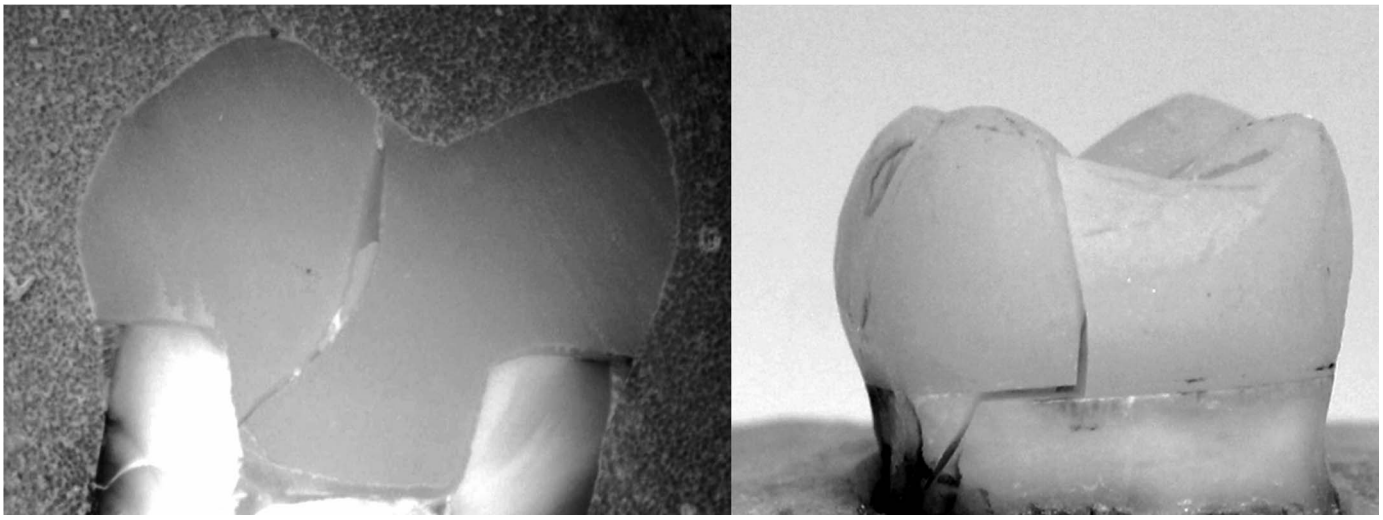

Figure 1. *Crack of endocrown/tooth complex above margin of bone level simulation (type III, acceptable failure) characteristic for feldspathic ceramic crowns (CB) with little penetration of the dye materials at the margin.*

with the help of a microbrush. Restoration margins were covered with a glycerine gel (Liquid Strip, Ivoclar Vivadent) to prevent oxygen inhibition of polymerization. The resin cement was light activated at each surface for 20 seconds using a light-emitting diode curing unit (Demetron A.1, Kerr/Sybron, Orange, CA, USA) with a 12-mm-diameter curing-light tip in standard mode with irradiance output of 1000 ± 50 mW/cm^2 held at a surface-tip distance of 0.5 mm. Output intensity was monitored after every fifth specimen using a handheld radiometer (Kerr/ Model 100, Demetron Research, Orange, CA, USA). Margins of the restorations were finished with sandpaper polishing discs (Sof-Lex, 3M ESPE).

Specimens were stored in double-distilled water at 37°C for one week to allow for bonded interface maturation. Specimens were subjected to 5000 thermal cycles between two water baths of 5°C and 55°C with a dwell time of 30 seconds at each temperature (Thermocycler, Willytec, Munich, Germany). After thermocycling, the entire surface of each specimen was covered with two coats of varnish up to 1 mm from the crown margins. Teeth were soaked in an aqueous solution of 5% methylene blue dye for 24 hours at 37°C. Following dye exposure, the teeth were rinsed thoroughly with a water syringe for 30 seconds.

Fracture Resistance Testing

Each mounted tooth was placed in a two-dimensional precision vice (FT-USV80, Firstec Inc, Osaka, Japan), positioned at an angle of 35 degrees between the long axis of the tooth and the loading jig in a universal testing machine (Sintech Renew 1123, TestWorks 4.08, MTS, Eden Prairie, MN, USA) with a 2.5-kg load cell. Force was applied through a stainless-steel ball (2.5 mm in diameter) representing the antagonist tooth. Load was applied to the incline of the palatal cusp at a crosshead speed of 0.5 mm/min. The fracture load needed to cause failure of the specimen, which was signaled as a peak in the load-displacement tracing, was recorded in newtons (N). Mode of fracture was examined for each specimen and categorized according to the following descriptions:

- Type I: complete or partial debonding of the endocrown without fracture (favorable failure)
- Type II: fracture of the endocrown without fracture of the tooth (favorable failure)
- Type III: fracture of the endocrown/tooth complex above the height of bone level simulation (acceptable failure)
- Type IV: fracture of the endocrown/tooth complex below the height of bone level simulation (catastrophic failure)

Microleakage Testing

The fractured coronal portion of the specimens were reassembled and embedded in fast-cure resin (Fastray, Harry J. Bosworth Co). Resin blocks were allowed to polymerize for 24 hours. Each specimen was sectioned buccolingually with a slow-speed diamond precision saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) under water cooling, producing five sections from each tooth. The two outermost sections were discarded, and the middle three tooth sections were used for dye penetration evaluation.

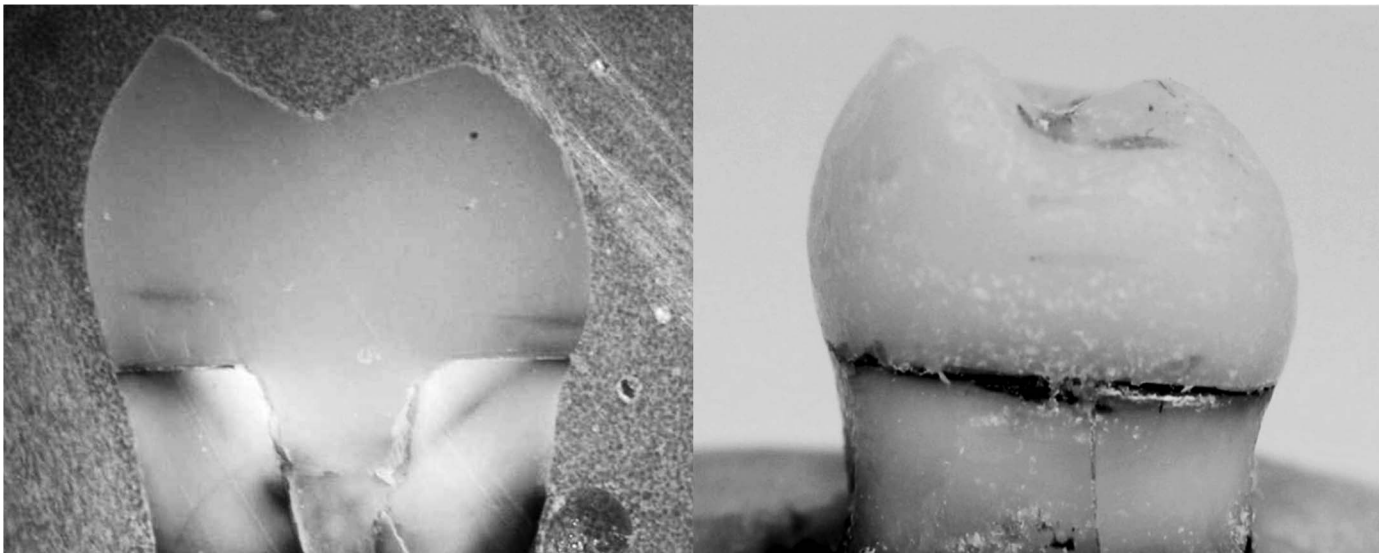

Figure 2. *Crack of endocrown/tooth complex below margin of bone level simulation (type IV, catastrophic failure) characteristic for lithium disilicate (EX) crowns with little penetration of the dye materials at the margin.*

A digital multiaxis dimensional measurement device (Quadra-Chek 200, Metronics Inc, Bedford, NH, USA) connected to a Measurescope (UM-2, Nikon) was used to measure the depth of dye penetration with the help of a built-in digital camera (Digital Microscope Camera, Model DMC 1, Polaroid, PLR Ecommerce, LLC, Minneapolis, MN, USA) and fiber-optic light at a magnification of 90×. Dye penetration at the tooth/luting agent interface at both the buccal and the lingual margins of each section was measured in millimeters, and dye penetration for each tooth was calculated from the average of all the readings of the three sections (Figures 1 through 3).

Statistical Analysis

Results were analyzed with statistical software (SPSS version 20.0, SPSS Inc, Chicago IL, USA) using a one-way analysis of variance (ANOVA) and Bonferroni *post hoc* multiple comparison tests (α=0.05).

RESULTS

The means, standard deviations, and 95% confidence interval levels for both fracture resistance and dye penetration for the three investigated CAD-CAM blocks are presented in Table 2. ANOVA revealed that there was a statistically significant difference between the groups ($p<0.05$) for both fracture resistance and dye penetration. The Bonferroni test (Table 3) indicated that there was a significantly higher ($p<0.05$) mean fracture resistance value for LU (1583.28 ± 170.55 N) when compared to both CB and EX (1340.92 ± 97.80 and 1368.76 ± 237.34 N, respectively). There was no significant difference between mean fracture resistance of EX and CB. Additionally, the mean dye penetration values of LU (2.80 ± 0.19 mm) were found to be significantly higher ($p<0.05$) than those of CB and EX (1.111 ±

Table 2: *Means, Standard Deviations,95% Confidence Intervals (in Parentheses) of the Dye Penetration and Fracture Resistance of Different CAD/CAM Blocks*

	Material	Mean	Standard Deviation	95% Confidence Interval for Mean		Minimum	Maximum
				Lower Bound	Upper Bound		
Dye penetration (mm)	CB	1.11	0.19	0.98	1.24	0.92	1.37
	EX	1.91	0.14	1.81	2.01	1.70	2.06
	LU	2.80	0.18	2.67	2.93	2.51	2.94
Fracture resistance (N)	CB	1340.92	97.80	1270.96	1410.88	1240.05	1494.48
	EX	1368.77	237.34	1198.99	1538.55	811.36	1746.28
	LU	1583.28	170.55	1461.27	1705.28	1316.89	1746.28

Table 3: *Results of Bonferroni* Post Hoc *Test From Multiple Comparison of Dye Penetration and Fracture Resistance of Different CAD/CAM Blocks*

Dependent Variable	(I) Block Type	(J) Block Type	Mean Difference (I − J)	Standard Error	Significance
Dye penetration	CB	LU	−1.690*	0.076	0.000
		EX	−0.802*	0.076	0.000
	EX	CB	0.802*	0.076	0.000
		LU	−0.888*	0.076	0.000
	LU	CB	1.690*	0.076	0.000
		EX	0.888*	0.076	0.000
Fracture resistance	CB	LU	−242.358*	79.574	0.015
		EX	−27.848	79.574	1.000
	EX	CB	27.848	79.574	1.000
		LU	−214.510*	79.574	0.036
	LU	CB	242.358*	79.574	0.015
		EX	214.510*	79.574	0.036

** The mean difference is significant at the 0.05 level.*

0.185 and 1.91 ± 0.14 mm, respectively), which were also found to be significantly different.

Modes of failure of the three tested CAD/CAM blocks are presented in Table 4. The results showed that 50% of the CB specimens exhibited acceptable fracture type (Figure 1) and 30% catastrophic fracture. High prevalence of catastrophic fracture (70% type IV) was demonstrated by EX, as shown in Figure 2. Meanwhile, LU exhibited a higher occurrence of favorable fracture modes (20% type I and 60% type II), as demonstrated in Figure 3.

DISCUSSION

This *in vitro* study simulates the compromised situation of extensive loss of tooth structure, which does not readily allow for the use of the ferrule effect in crown preparation. Under such circumstances, endocrowns take advantage of recent developments in adhesives, ceramics, and CAD/CAM technologies in an approach that is based mainly on a decay-oriented design concept.[28] This concept is built on a minimally invasive preparation that preserves maximum amounts of tooth surface for bonding and where extensive macroretention designs are no longer a prerequisite. The utilization of the available space inside the pulp chamber adds to the stability and retention of the restoration and reduces the operational errors possible during post-space preparation.[20] It has been assumed that through establishing adhesion, the occlusal stresses that occur during function are transmitted to the walls of the pulp chamber. The deeper the pulp cavity and resulting intracoronal extension, the greater the surface area that can be utilized for adhesive retention and transmission of masticatory forces.[16]

Table 4: *Modes of Failure (%) of Feldspathic Porcelain (CB), Lithium Disilicate (EX), and Resin Nanoceramic (LU)*

Material	Mode of Failure %			
	Type I	Type II	Type III	Type IV
CB	10	10	50	30
EX	0	0	30	70
LU	20	60	20	0

In an attempt to exclude the effect of variances in the intracoronal extensions of the endocrowns, a standardized cavity design following guidelines by Pissis[15] was used. The preparations were done to allow for an intracoronal extension of 2 mm. This minimal extension allowed for testing endocrown/tooth systems with minimal remaining tooth structure, in other words, the ability of the remaining tooth structure to retain the restoration and the ability of the adhesive restoration to reinforce the remaining weakened tooth structure. A previous study had reported clinical evaluation of endocrowns with intracoronal extensions varying from 1 to 4 mm, corresponding to variances in pulp chamber depth.[17] Yet no studies report the effect of the dimension of the intracoronal extension on fracture resistance and modes of failure. One study reported that the possible failure of the endocrown was associated with the height of the endocrown itself (position of the finish line) and the height level of the applied force on the crown (contact with opposing teeth) rather than the concept of the endocrown itself.[29] Therefore , in the present study, variability in endocrown dimensions was controlled using the

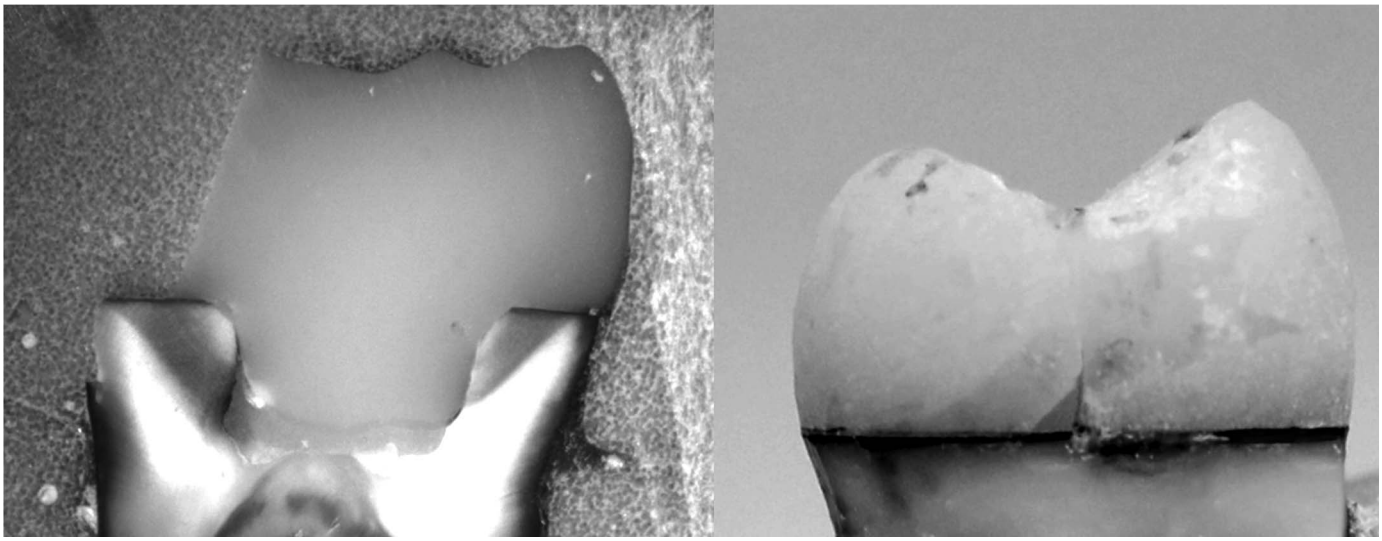

Figure 3. *Crack of endocrown without fracture of tooth (type II, favorable failure) characteristic for nanocomposite (LU) crowns with deep penetration of the dye material.*

Cerec technology, which allowed the fabrication of standardized restoration size, shape, and cuspal inclines and hence standardizing the point of load application during testing.

In complex multilayered restorations, such as cemented ceramic restorations, several factors contribute to the mechanical behavior of the restoration/tooth system. The intrinsic strength of each component of the system (ie, tooth, adhesive system, luting cement layer, and restoration), the thickness of the restorative material, the ratios of elastic moduli between the restoration material, the luting cement and dentin, and finally the quality of the adhesive interface between these layers in terms of bond strength and presence of micro- or nanoleakage are all factors that play a role in the behavior of such restorations.[31] Results of the present study showed a significantly higher mean fracture resistance value for LU when compared to EX and CB. These results were in agreement with another study by Heo and others,[30] who reported significantly higher impact fracture resistance and fewer cases of complete fracture of LU when compared to lithium disilicate.

The unique composition of LU allows the material to have a modulus of elasticity (12.8 GPa) similar to that of dentin (5.5-19.3 GPa).[32] The modulus of elasticity influences the susceptibility to fracture of a cemented ceramic restoration since materials with more compatible elastic moduli tend to bend under load and distribute stresses more evenly, while rigid materials with different elastic moduli, such as lithium disilicate, produce stress concentrations at critical areas that might cause catastrophic failures.[33,34] Failure modes reported in this study support such an explanation, as none of the LU specimens showed catastrophic failure modes, while 80% had favorable modes of failure. On the other hand, 70% of the EX specimens had catastrophic failure modes.

Moreover, weak bond strength between restorations and resin cements could lead to a nonhomogeneous distribution of forces that could result in cohesive failure of the resin cement. Multiple authors have evaluated the bond strength of feldspathic and lithium disilicate–based glass ceramic to composite resin or resin cements using tensile, microtensile, shear, and microshear mechanical tests[23,35-37] and concluded that lithium-disilicate glass ceramic exhibits significantly higher bond strengths than feldspathic ceramics independent of surface conditioning, which is attributed mainly to its unique crystalline microstructure. Another study reported higher bond strength to resin cement and more favorable modes of failure of LU when compared to feldspathic porcelain monoblocks.[38] These findings can provide understanding for the results of the current study, as the bond strength of LU to composite resin is expected to be better than that found with ceramics.[39,40] The presence of resin matrix in LU blocks should facilitate bonding to resin composite luting materials, resulting in more uniform stress distribution when compared to feldspathic and reinforced ceramics and therefore better fracture resistance. It is worth mentioning that although the application of a single monotonic load

to cause failure does not represent the clinical situation, in which repetitive cyclic fatigue loading is characteristic, the setting of this study provided a controlled environment that allows comparing the behavior of materials under the applied circumstances.

Thermocycling and application of mechanical loading are widely accepted methods when testing for *in vitro* microleakage and fracture resistance to simulate aging and stress at the adhesive interface.[41] Exposure of the hybrid layer to hot water during thermocycling can affect the adhesive layer by accelerating the hydrolysis of unprotected collagen and extracting poorly polymerized resin. Additionally, stresses are generated at the adhesive interface during thermocycling due to the difference in the coefficient of thermal expansion between the restorative materials and the tooth structure. The linear coefficient of thermal expansion has been suggested as an important factor that influences microleakage.[42,43] This factor is influenced by the composition of the restorative material. A greater difference in the linear coefficient of thermal expansion between tooth and restorative material leads to the generation of excessive stresses with temperature fluctuation that may result in microcracks that propagate along the bonded interface, causing a gap to form.

In the present study, LU showed a significantly higher dye penetration than CB and EX. Unlike the other ceramics, LU contains 80% nanoceramic particles embedded in a highly cured resin matrix (20%).[32] It is thought that this unique composition results in a higher coefficient of thermal expansion in comparison to that of ceramic materials and dentin, which in sequence would exaggerate the effect of thermocycling on margin quality of this material and therefore could result in greater microleakage.[44]

One limitation of this study was the use of one type of adhesive and luting cement system. The use of other systems may have resulted in different outcomes. Additionally, cyclic fatigue, bond strength data, and the effect of the endocrown intracoronal extension dimension on the fracture resistance and pattern of failure were not evaluated. Therefore, more studies are needed to investigate the effect of these variables on the mechanical behavior of endocrown restorations.

CONCLUSION

In comparison to feldspathic and lithium disilicate ceramics, the higher fracture resistance and more favorable failure of resin nanoceramic, may favor its use for endocrown restoration of endodontically treated teeth with extensive loss of tooth structure. However, higher amounts of microleakage may jeopardize the long-term performance of this material.

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Note

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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 Ceramic Thickness and Ceramic Materials on Fracture Resistance of Posterior Partial Coverage Restorations

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Clinical Relevance

This study demonstrated lithium disilicate glass ceramic significantly improved fracture resistance when compared to a leucite-reinforced glass ceramic, even at a thickness below the manufacturer's suggested minimum. The use of lithium disilicate may have advantages in clinical situations of minimal occlusal clearance.

SUMMARY

This study evaluated the influence of ceramic thickness and ceramic materials on fracture resistance of posterior partial coverage ceramic restorations. Forty extracted molars were allocated into four groups (n=10) to test for two variables: 1) the thickness of ceramic (1 mm or 2 mm) and 2) the ceramic materials (a lithium disilicate glass-ceramic [IPS e.max] or leucite-reinforced glass ceramic [IPS Empress]). All ceramic restorations were luted with resin cement (Variolink II) on the prepared teeth. These luted specimens were loaded to failure in a universal testing machine, in the compression mode, with a crosshead speed of 1.0 mm/min. The data were analyzed using two-way analysis of variance and the Tukey Honestly Significantly Different multiple comparison test (α =0.05). The fracture resistance revealed a significant effect for materials ($p<0.001$); however, the thickness of ceramic was not significant ($p=0.074$), and the interaction between the thickness of ceramic and the materials was not significant ($p=0.406$). Mean (standard deviation) fracture resistance val-

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ues were as follows: a 2-mm thickness of a lithium disilicate bonded to tooth structure (2505 [401] N) revealed a significantly higher fracture resistance than did a 1-mm thickness of leucite-reinforced (1569 [452] N) and a 2-mm thickness of leucite-reinforced ceramic bonded to tooth structure (1716 [436] N) ($p<0.05$). There was no significant difference in fracture resistance values between a lithium disilicate ceramic at 1-mm thickness (2105 [567] N) and at 2-mm thickness. Using a lithium disilicate glass ceramic for partial coverage restoration significantly improved fracture resistance compared to using a leucite-reinforced glass ceramic. The thickness of ceramic had no significant effect on fracture resistance when the ceramics were bonded to the underlying tooth structure.

INTRODUCTION

All-ceramic restorations have been shown to demonstrate superior optical properties over ceramic metal restorations.[1-3] In addition, the mechanical requirements of adhesively retained all-ceramic restorations can typically be met with less tooth reduction than is required of metal-ceramic or all-ceramic cohesively retained restorations. While the concept of using ceramic in posterior teeth dates back to the late 1800s, it was not considered practical until the advent of adhesive protocols. In addition, adhesively retained restorations negate the need to extend preparations to sound tooth structure purely for establishing resistance and retention form. Preservation of sound tooth structure is of interest because loss of tooth structure involves biologic compromise.[4] As the physical requirements of a restoration align to provide greater respect for the biologic requirements of a restored tooth, there is a greater chance for long-term success of the tooth/restoration partnership. For this reason, adhesively retained ceramic onlays warrant consideration as an effective means of restoring posterior teeth. Previous studies revealed the high survival rate of partial coverage glass ceramic to measure between 92% and 97% during observation periods extending up to five years[5-9] and to measure 94% to 98% at the seven- and eight-year intervals, respectively.[10] Despite the high survival rate of restorations, fracture of the ceramic material is the most frequently reported complication resulting in failure. Investigations of clinically failed all-ceramic restorations have shown that the failure stresses depend on their mechanical properties.[11,12]

Occlusal thickness requirements for traditional ceramics dictate a 2-mm minimum to achieve optimal compressive strength. Undulations present in tooth anatomy are impractical to mirror in kind during tooth preparation. Establishing clearance for a 2-mm minimum thickness of restorative material in all areas, including fossa and fissures, results in areas of tooth reduction that exceed 2 mm in other areas, such as cuspal inclines. Increased tooth reduction directly relates to greater probability of enamel loss; enamel is critical to the long-term success of adhesively retained restorations.[13] Dimensional loss of enamel with preparation can be exaggerated in the already-worn dentition. A posterior partial coverage restoration with minimal thickness of ceramic has the potential to conserve and protect tooth structure, preserve enamel, and safeguard pulpal vitality while achieving the desired esthetic results.[14,15] However, reduced thickness of occlusal ceramic may have a negative effect on the mechanical properties of the material since the strength of ceramic is inversely related to the square of the ceramic thickness.[16,17] Several all-ceramic materials have been used to fabricate these partial coverage adhesively retained restorations. The heat-pressed technique was introduced to dentistry in the early 1990s as an innovative processing method for all-ceramic restorations. Applying this technique, a leucite-reinforced glass ceramic (IPS Empress, Ivoclar Vivadent Inc, Amherst, NY, USA) can be processed into various shapes (veneer, inlay, onlay, or single crown). More recently, a lithium disilicate-reinforced glass ceramic (IPS e.max, Ivoclar Vivadent) with further improved physical and mechanical properties was developed. Often improved physical properties of ceramics are countered by less favorable optical properties. However, as a result of the favorable optics (shade and translucency) of lithium disilicate, it can be used to create esthetically acceptable full-contour restorations. The purpose of this study was to evaluate the influence of ceramic materials and their thickness on fracture resistance of posterior partial coverage restorations. The null hypothesis was that fracture resistance of posterior partial coverage restorations would not be affected by the choice of ceramic materials (a lithium disilicate–reinforced ceramic or a leucite-reinforced ceramic) or the ceramic thickness (1 mm or 2 mm).

METHODS AND MATERIALS

Selection and Preparation of Teeth

Forty extracted human molar teeth were selected. Teeth were included based on the specific criteria

that they were intact and lacked cracks or fractures in the crown, contained no evidence of caries, and had no prior restorations. The teeth were cleaned of surface debris, disinfected in 0.5% sodium hypochlorite, and kept in distilled water until the study began. The occluso-cervical and mesio-distal dimensions of the teeth were measured three times using a dial caliper accurate to within 0.1 mm (Masel Dental Dial Caliper; Masel, Carlsbad, CA, USA), and the averages were determined. The teeth were ranked according to the decreasing mesio-distal dimension. The ranked teeth were divided into four groups: the first tooth was assigned to group 1, the second to group 2, the third to group 3, the fourth to group 4, the fifth to group 4, the sixth to group 3, the seventh to group 2, and the eighth to group 1. This procedure was repeated until each group had a sample size of 10 teeth. Each group was assigned to one of the four test groups with a combination of ceramic thickness and ceramic materials. The teeth were attached with sticky wax (Kerr sticky wax; Kerr, Orange, CA, USA) to a dental surveyor rod (J.M. Ney Co, Bloomfield, CT, USA) on a vertically prepared surface so that the long axis of the teeth would be parallel to the surveyor rod. The teeth were lowered into a copper cylinder and positioned in the center of the cylinder with the buccal cemento-enamel junction 3 mm above the top of the copper-mounting cylinder. Premixed autopolymerizing resin (Pattern Resin; GC America, Alsip, IL, USA) was injected into the cylinder until it was completely full. After acrylic resin polymerization, the dental surveyor rod was detached, and the specimens of teeth were stored in distilled water at room temperature. The mounted teeth were allocated into four groups (n=10) and restored with a leucite-reinforced ceramic (IPS Empress; Ivoclar Vivadent) or a lithium disilicate ceramic (IPS e.max; Ivoclar Vivadent) as follows: 1) IPS e.max 1-mm (EX-1): 2-mm occlusal reduction restored with 1 mm of a lithium disilicate ceramic; 2) IPS e.max 2-mm (EX-2): 2-mm occlusal reduction restored with 2 mm of a lithium disilicate ceramic; 3) IPS Empress 1-mm (EMP-1): 2-mm occlusal reduction restored with 1 mm of a leucite-reinforced ceramic; and 4) IPS Empress 2-mm (EMP-2): 2-mm occlusal reduction restored with 2 mm of a leucite-reinforced ceramic. A clinician prepared the teeth in all groups according to preparation designs previously described in the literature.[13] All teeth received a 2-mm occlusal reduction, maintaining cusp steepness of 45° relative to the occlusal surface (Figure 1). All specimens were prepared using a high-speed electric handpiece and diamond rotary cutting instrument under cool-water irrigation. An impression of each prepared tooth was made with light-body and heavy-body vinyl polysiloxane using a dual-phase single-stage technique, according to the manufacturer's instructions.

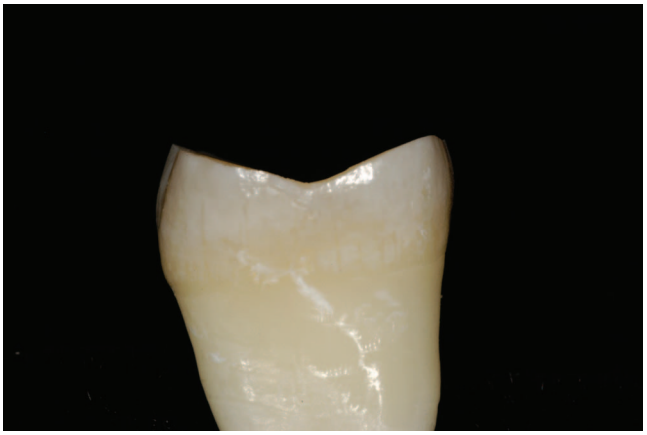

Figure 1. *All teeth received a 2-mm occlusal reduction, maintaining cusp steepness of 45° relative to the occlusal surface.*

Fabrication of Ceramic Restorations

After 24 hours, all dies were poured using vacuum-mixed die stone (Fuji Rock; GC America). Accuracy of die stone and water were ensured by measuring and dispensing from an automated system (Smart Box; AmannGirrbach, Vorarlberg, Austria). Die stone was allowed to set for 24 hours to ensure uniform hardness. All dies were carefully removed and model trimmed and sealed with die sealer (MS1; Harvest Dental, Brea, CA, USA). The 40 posterior partial coverage restorations were waxed and carefully measured using a digital caliper to ensure uniform thickness for each test group. Wax patterns were sprued and invested per manufacturer instructions—Ceravety (Shofu Incorporated; San Marcos, CA, USA) for IPS e.max restorations and Speed vest (Ivoclar Vivadent) for IPS Empress restorations. All pressings were done utilizing a speed burn-out technique (20-30 minutes after investing, rings were placed in a preheated burnout oven at 843°C). All restorations were divested using 50-μm particle size aluminum oxide. IPS e.max exhibits a strong reaction layer after pressing, which requires ultrasonic treatment for 10 minutes utilizing Invex liquid (Ivoclar Vivadent), followed by additional sandblasting with 50-μm aluminum oxide to insure complete removal of the reaction layer. The sprues were separated from the restorations and the restorations were measured for accuracy. The restorations fit the dies with minor adjustment; this was due to the accuracy of investment materials and the smooth,

nonretentive nature of the test patterns. Restorations were carefully measured with a microcaliper and adjusted where necessary using a fine diamond bur (ZR8881.FGL.016, Komet USA, Rock Hill, SC, USA) and water in order to achieve the desired test thicknesses. A thin layer of glaze paste was applied to each restoration. The restorations were placed on a pillow tray and then baked in a ceramic furnace (Programat P700; Ivoclar Vivadent) following the manufacturer's instructions (785°C for IPS e.max and 790°C for IPS Empress).

All sets of restorations were acid-etched according to the manufacturer's recommendations (IPS Ceramic Etching Gel; Ivoclar Vivadent): for 60 seconds for the leucite-reinforced ceramic and for 20 seconds for the lithium disilicate ceramic. The by-products were eliminated from the internal aspects of the restorations by immersing them in isopropyl alcohol and placing them in an ultrasonic cleaner for five minutes.

Cementation Procedure

Specimen cementation included mechanical debridement using aluminum oxide abrasion (PrepStart; Danville Engineering, San Ramon, CA, USA) with a particle size of 27 μm at 0.28 MPa at a distance of 2 mm from the tooth surfaces.[18] The prepared teeth were etched for 15 seconds with 37% phosphoric acid (Etch-37; Bisco Inc, Schaumburg, IL, USA), rinsed for 10 seconds, and dried sparingly. The dentin primer of a fourth-generation adhesive system (All-Bond II; Bisco) was applied to specimens according to manufacturer instructions and gently air-dried. A thin layer of unfilled resin (D/E Resin; Bisco) was applied to the specimens. The internal surfaces of the ceramic restorations were cleaned with 37% phosphoric acid for 60 seconds, dried, and silanated with ceramic primer (Scotch Bond Primer; 3M ESPE, St Paul, MN, USA). The ceramic veneers were luted with light-polymerizing composite resin cement (Variolink II; Ivoclar Vivadent). The restorations were seated with finger pressure and light-polymerized with a wave length of 480 nm and a light intensity of 1100 mW/cm^2 (±10%) for a five-second burst (Optilux 501; Kerr), and then the excess was removed to simulate intraoral conditions. Specimens were then polymerized for 40 seconds on all surfaces for a total of 120 seconds. Any remaining excess was removed with a scalpel blade (Bard Parker #12; Becton Dickinson and Co, Franklin Lakes, NJ, USA), and all restoration margins were polished with fine polishing disks (Sof-Lex; 3M ESPE). The bonded specimens were stored at room temperature with 100% relative humidity for 48 hours prior to fracture testing.

Measurement of Fracture Resistance

Each specimen was mounted on a metal holder in a universal testing machine (Model 5585H; Instron Corp, Norwood, MA, USA), equipped with a 10-kN load cell at a crosshead speed of 1.0 mm/min. All of the specimens were tightened and stabilized to ensure that the loading stainless-steel ball of 6-mm diameter was positioned on the central occlusal surface of ceramic onlays. A 6-mm-diameter stainless-steel ball, the size of which was similar to that of a molar cusp, was positioned on the central fossa of the occlusal surface of the restoration to simulate an occlusal contact point of an antagonist tooth.[19] A load was applied until catastrophic failure occurred. The ultimate load to failure was recorded in newtons (N), and the means and standard deviations (SDs) were calculated. The fractured surfaces were then examined to obtain the catastrophic mode of failure. All restorations were inspected under 10× magnification. The catastrophic failure was classified in accordance with one of the following criteria: a cohesive failure not involving tooth (Type I), a cohesive failure involving any interface (Type II), a cohesive failure involving the crown (root preserved) (Type III), and a fracture involving root (Type IV). Parametric statistical analyses were performed at a 95% confidence interval using statistical software (SAS V.9.1, SAS Institute Inc, Cary, NC, USA). Groups were analyzed using a two-way analysis of variance with ceramic thickness and ceramic material as the variables, followed by a Tukey Honestly Significantly Different multiple comparison test to evaluate differences among the testing groups.

RESULTS

Ceramic materials had a significant effect on the fracture resistance values ($p<0.001$); however, the thickness of ceramic was not significant ($p=0.074$), and the interaction between the thickness of ceramic and ceramic material was not significant ($p=0.406$). The highest mean (SD) fracture resistance was obtained from a 2-mm thickness of a lithium disilicate glass ceramic (2505 ± 401 N), followed by a 1-mm thickness (2105 ± 567 N) of a lithium dislilicate glass ceramic (Figure 2). This represents approximately 15% to 30% more than the lowest mean obtained from those groups of a 1-mm thickness (1569 ± 452 N) and a 2-mm thickness (1716 ± 436 N) of teeth restored with a leucite-reinforced glass ceramic. There was no significant

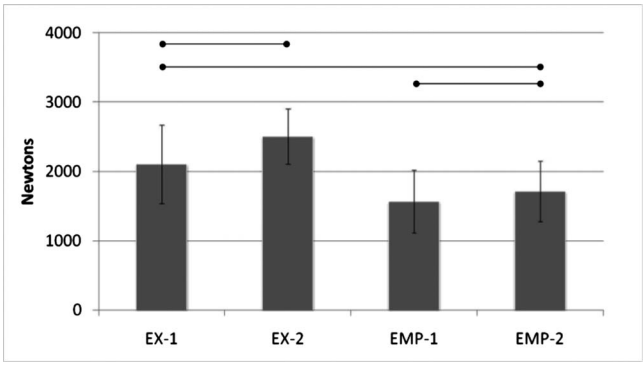

Figure 2. *Schematic representation of the mean and standard deviation of load to failure in each testing group. Note that the same running bar showed no significant difference among the groups.*

difference in fracture resistance between the 2-mm thickness group and the 1-mm thickness group in both ceramic materials. The mode of failure in the majority of the specimens involved ceramic fracture with a cohesive failure not involving tooth (Type I) (Figure 3A) and ceramic fracture with a cohesive failure involving the interface (Type II) (Figure 3B). Several fractured ceramic specimens involved coronal tooth structure but were not catastrophic (Type III). None of the ceramic specimens revealed root fracture in conjunction with failure (Table 1).

DISCUSSION

Restoration longevity depends not only on the ceramic material used but also on adherence to strict bonding protocols and the characteristics of the remaining tooth structure. Considering the brittle nature and limited flexural strength of glass ceramics, adhesive cementation with resin cements must be used to increase the fracture resistance of the restoration.[20,21] An enamel substrate favorably influences the predictability of bonded restorations.[22,23] A previous study[13] demonstrates that the amount of remaining circumferential enamel to retain posterior partial coverage should be at least 1 mm. In the present study, all specimens were prepared with 2-mm occlusal reduction, providing a relatively consistent adhesive substrate. Certainly the underlying tooth structure contributed to the strength of the ceramic to resist fracture upon loading. The contributions of the underlying tooth structure being relatively even, clearly the stronger the properties of the ceramic material itself, the better the fracture resistance of the tooth/restoration complex. Lithium disilicate and leucite-reinforced glass ceramics are both recommended for use in posterior restorations and therefore were selected for the present study. Previous studies[24,25] of lithium disilicate demonstrate a flexural strength of approximately 400 MPa and a fracture toughness value of 3.3 MPa $\times$ $m^{1/2}$, which are almost three times the values of a leucite-reinforced glass ceramic. As a result of the increased crystallinity of lithium disilicate ceramic, the material provides a tighter interlocking matrix in its structure and prevents the propagation of microcracks. Because of the improvement in mechanical properties, it is deemed a material that can withstand higher masticatory forces and provide improved clinical performance.[26,27] The present finding was consistent with those of previous reports. The results of the present study support the first null hypothesis, since there was no significant difference in load to failure values for ceramics of different thickness (1 mm or 2 mm). The results of the study reject the second null hypothesis, since there was a significant

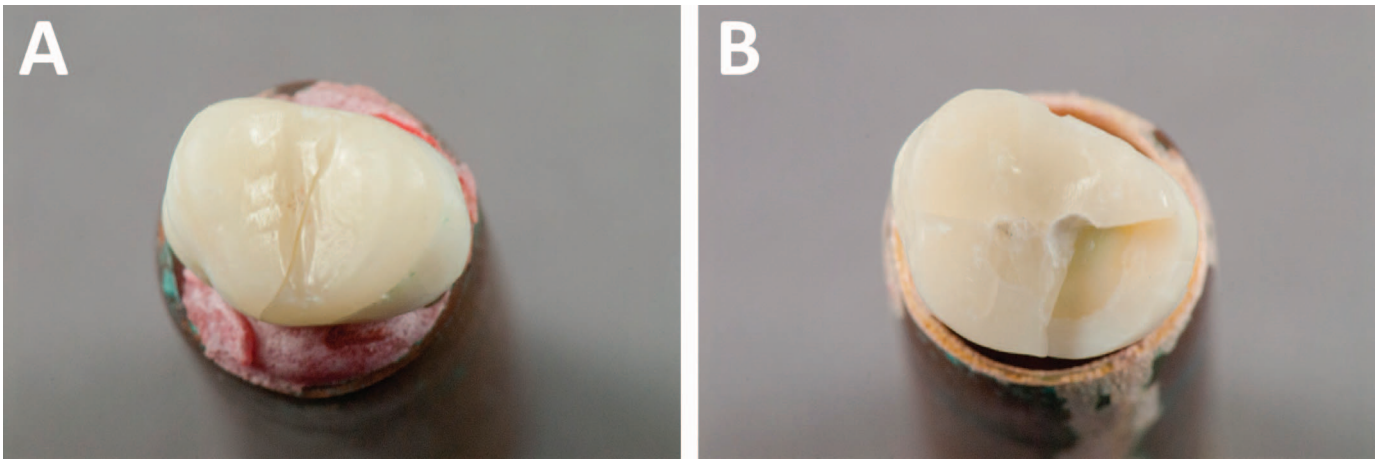

Figure 3. *Representative photographs of failed specimens: A, a cohesive failure not involving the tooth (Type I); B, a cohesive failure involving any interface (Type II).*

Table 1: *Mode of Failure in All Testing Groups. Note a Cohesive Failure Not Involving Tooth (Type I), a Cohesive Failure Involving Any Interface (Type II), a Cohesive Failure Involving the Crown (Root Preserved) (Type III), and a Fracture Involving Root (Type IV)*

Group	Type and Thickness of Ceramic	Mode of Failure			
		I	II	III	IV
EX-1	1-mm thickness lithium disilicate	7	3	0	0
EX-2	2-mm thickness lithium disilicate	5	2	3	0
EMP-1	1-mm thickness leucite-reinforced	8	1	1	0
EMP-2	2-mm thickness leucite-reinforced	7	2	1	0

difference in load to failure for different ceramic materials (lithium disilicate glass ceramic or leucite-reinforced glass ceramic).

In summary, the fracture resistance of posterior partial coverage restorations is affected relative to the choice of ceramic materials, but not relative to ceramic thickness. In general, 1.5 mm of occlusal thickness is recommended as a minimum for lithium disilicate ceramics. Previous studies[28] reported that the thickness of monolithic ceramic material had no effect on the failure distribution. Change in thickness would create minimal influences on overall flexural strength of the material. The results of the present study corroborate this finding and suggest that the thickness might be decreased to 1 mm when the ceramic is lithium disilicate–reinforced glass and when it is effectively bonded to the underlying tooth structure. This finding does not suggest a change in generalized preparation design; there is still a need to have adequate space in which to reproduce esthetic and functional anatomy. Instead, the results support the clinical acceptability of lithium disilicate glass ceramic as thin as 1 mm in thickness in areas of minimal occlusal clearance. Several specimens revealed fractured tooth structure after load. A causative reason for this could be that the load to failure was high enough to exceed the proportional limit of the tooth. It must also be considered that extracted human teeth offer large variation in quality and that the standardization of this type of specimen is difficult. Based on the results of load to failure, it is unlikely that the masticatory system approaches the type of loads used in the study unless the patient is the victim of blunt-force trauma. The average human bite forces for posterior teeth have been reported in the literature[29] to be a maximum of 500-600 N.

There are several limitations to the present study. The study was limited solely to loading in a vertical vector. Evaluation with varied loading vectors may provide information that more accurately reflects the dynamic nature of the oral environment. In addition to the traditional heat-pressed technique, CAD/CAM fabrication techniques are available in today's market. The leucite-reinforced and lithium disilicate glass ceramic restorations made with different fabrication techniques (heat pressed or CAD/CAM) can be considered for clinical trials to compare and confirm the effect of fabrication process on ceramics with similar compositions. However, this study provides further information on the *in vitro* strength of dental ceramics used for posterior partial coverage restorations, at the same time acknowledging the need for future studies that incorporate varied fabrication protocols and the challenges of the oral environment.

CONCLUSIONS

Within the limitations of this *in vitro* study, a lithium disilicate glass ceramic for partial coverage restorations significantly improved fracture resistance compared to a leucite-reinforced glass ceramic. The thickness of ceramic had no significant effect on fracture resistance when ceramics were bonded to the underlying tooth structure.

Acknowledgement

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Conflict of Interest

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

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Analysis of Anticaries Potential of Pit and Fissures Sealants Containing Amorphous Calcium Phosphate Using Synchrotron Microtomography

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Clinical Relevance

The combination of fluoride and resin sealants containing amorphous calcium phosphate was highly effective at preventing the demineralization of enamel.

SUMMARY

The aim of this study was to analyze the anticaries potential of pit and fissure sealants containing amorphous calcium phosphate (ACP) by synchrotron microtomography. Bovine enamel blocks (4×4 mm; n=50) were selected through surface hardness (Knoop) analysis. Slabs were obtained through cross-

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sections taken 1 mm from the border of the enamel. Five indentations, spaced 100 μm apart, were made 300 μm from the border. Ten specimens were prepared for each tested material (Ultraseal XT plus TM, Aegis, Embrace, Vitremer and Experimental Sealant). The materials were randomly attached to the sectioned surfaces of the enamel blocks and fixed with sticky wax. The specimens were submitted to pH cycling. After that, the surface hardness (SH_1) was determined, and the blocks were submitted to synchrotron microcomputed tomography analysis to calculate the mineral concentration (Δg_{HAp} cm^{-3}) at different areas of the enamel. The comparison between the SH_1 and Δg_{HAp} cm^{-3} showed a correlation for all groups (r=0.840; p<0.001). The fluoride groups presented positive values of Δg_{HAp} cm^{-3}, indicating a mineral gain that was observed mainly in the outer part of the enamel. The ACP showed mineral loss in the outer enamel compared with fluoride groups, although it inhibited the demineralization in the deeper areas of enamel. The combination of two remineralizing agents (fluoride and ACP) was highly effective in preventing demineralization.

INTRODUCTION

Amorphous calcium phosphate (ACP) has been identified as a possible precursor in the formation of hydroxyapatite. Dental applications based on the unique characteristics of ACP have been proposed, and it has been shown that the properties of ACP are enhanced when used with similar products that have anti-demineralizing and remineralizing potential.[1] The systems developed use casein phosphopeptides (CPPs) to stabilize the calcium phosphate ions at high concentrations; these include amorphous nanocomplexes designated CPP-ACP on the enamel surface.[2] Incorporating CPP-ACP into glass-ionomer cements improved the anticariogenic potential of this material without adversely affecting its mechanical properties.[3,4] Recent studies show that composites containing ACP can release supersaturated levels of calcium and phosphate ions in proportions favorable for apatite formation.[5-8] In these composites, the addition of ACP led to failures due to degraded mechanical strength; thus, they are not indicated as restorative or lining materials but can be adequate as pit and fissure sealants.[5,9]

As a sealant, two studies (*in vitro* and *in situ*) showed that the ACP sealant presented the same capacity of remineralization as the fluoride sealant.[7,10] When the sealant product contained ACP and fluoride (ACP-F), there was no improvement in its remineralizing effect.[7] However, it is important to determine if the ACP sealant has the ability to inhibit enamel demineralization because caries is a dynamic process that involves demineralization and remineralization. If it is determined that sealants with ACP-F act as a favorable alternative process for remineralization of the enamel compared with resin agents that contain only fluoride, practitioners would have an additional method for preventing dental caries. The purpose of this study was to analyze the anticaries potential of an ACP sealant and an ACP-F sealant using a pH-cycling model and synchrotron microtomography.

METHODS AND MATERIALS

Preparation and Selection of Enamel Blocks

Enamel blocks (4 mm×4 mm×3 mm; n=50) were obtained from bovine incisor teeth that were stored in 2% formaldehyde solution with a pH of 7.0 for 30 days.[11] The enamel surface of the blocks was then serially polished, and the slabs were cross-sectioned at 1 mm from the border (Figure 1A), resulting in specimens with an area of 12 mm^2.[11] The blocks were subjected to surface hardness (Knoop) analysis (SH) using a microhardness tester (Shimadzu MicroHardness Tester HMV-2000, Shimadzu Corp, Kyoto, Japan) with a Knoop diamond under a 25g load for 10 seconds.[11] Five indentations spaced 100 μm from each other were made at a distance of 300 μm from the enamel sectioned border (Figure 1B). Enamel blocks with hardness values between 330 and 370 kgf/mm^2 were selected.

Sample Preparation and Enamel Block Adaptation

Ten samples were prepared for each tested material (Table 1) using a metallic matrix (4 mm×2 mm×1 mm) (Figure 1C) following the manufacturer's instructions, with the exception of the Vitremer (3M ESPE, St Paul, MN, USA), which had a diluted mix at 1/4 the powder to liquid ratio.[11,12] Polymerization of the materials was performed with a VIP unit (BISCO, Schaumburg, IL, USA) for 40 seconds on both sides of the specimen, using a light intensity of 500 mW/cm^2. After the sample preparation (Figure 1D), the materials were randomly attached to the sectioned surfaces of the bovine enamel blocks (Figure 1E) and fixed with sticky wax (Kota Industria and Comércio Ltda, São Paulo, Brazil) (Figure 1F).[11] The specimens (enamel block +

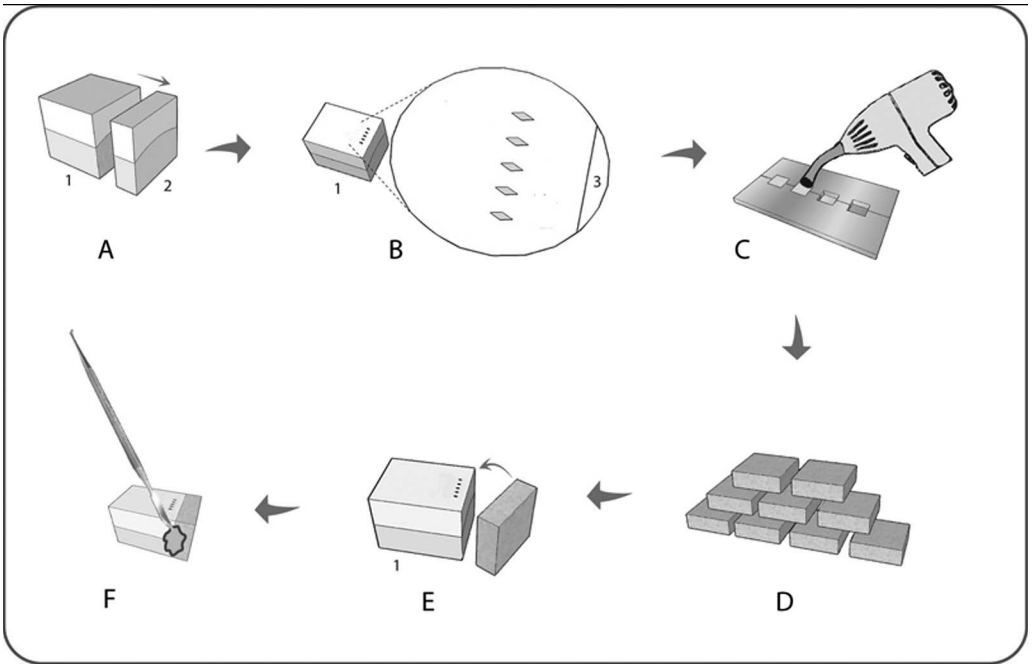

Figure 1. *Schematic presentation. (A) Section of the block (1. block 3×4 mm used in the research; 2. Piece of the block 1×4 mm discarded). (B) Five indentations at 300 μm from the enamel sectioned border (3). (C) Polymerization of sample. (D) Samples. (E) Samples adapted onto the enamel blocks (3). (F) Samples fixed with wax.*[11]

sample) were then coated with an acid-resistant varnish, except for the enamel and sample surface.

pH Cycling

The effect of the material in interfering with the dynamic caries process was evaluated. To simulate the demineralization and remineralization process *in vitro*, all specimens were immersed in demineralizing solution for six hours (2.0 mmol/L Ca and P, 0.075 mol/L acetate buffer, 0.04 ppm F, 2.2 mL/mm^2 of the enamel surface, pH 4.7) and remineralizing solution for 18 hours (1.5 mmol/L Ca, 0.9 mmol/L P, 0.15 mol/L KCl, 0.02 mol/L Tris buffer, 0.05 ppm F, 1.1 mL/mm^2 of the enamel surface, pH 7.0) for five days at 37°C. The specimens were then submerged in the remineralizing solution for an additional two days before the surface hardness analysis.[11]

Surface Hardness Analysis

The surface hardness analysis was performed using a Shimadzu HMV-2000 (Shimadzu Corp) microhardness tester and a Knoop diamond under a 25g load for 10 seconds. Five indentations spaced 100 μm from each other were made on the slab surface at 300 μm from the sectioned enamel border (Figure 1B).[11] After the pH cycling, the final surface hardness (SH_1) was measured, following the aforementioned methodology.

Analysis of Synchrotron Microcomputed Tomography

Five blocks of each group were sectioned longitudinally and transversely; the samples (1.5 mm×2.0 mm) were them submitted to synchrotron microcomputed tomography at an Advanced Photon

Table 1: *Identification of the Tested Material*

Material	Manufacturer	Classification	Batch No.	Group
Ultraseal XT plus	Ultradent Products Inc, South Jordan, UT, USA	Light-cured resin sealant	B 0997	Resin no-F
Aegis	Bosworth Company, Skokie, IL, USA	Light-cured ACP resin sealant	0407-397	Resin ACP
Experimental sealant	Bosworth Company, Skokie, IL, USA	Light-cured ACP and F resin sealant	HJB6-203A	Resin ACP-F
Embrace	Pulpdent Corporation, Watertown, MA, USA	Light-cured F resin sealant	040923	Resin F
Vitremer	3M/ESPE, St Paul, MN, USA	Resin-modified glass ionomer cement	22015	Ionomer

Abbreviations: ACP, amorphous calcium phosphate; F, fluoride.

Table 2: *Analysis of Surface Hardness (SH_1), Integrated Loss of Subsurface Mineral (Δg_{HAp} cm^{-3}), and Integrated Differential Mineral Concentration (ΔIML) According to Groups*[a]

Groups	SH_1 (kgf/mm^2)	Δg_{HAp} cm^{-3}	ΔIML, g_{HAp} cm^{-3}	
			Zone A (2.8–33.6 μm)	Zone B (36.4–89.6 μm)
Resin no-F	47.9^{a} (20.8)	–15.6^{a} (4.5)	–19.8^{Aa} (5.5)	–7.9^{Aa} (10.2)
Resin F	285.6^{b} (21.3)	0.3^{b} (1.9)	0.7^{Ab} (4.0)	0.6^{Aa,b} (6.7)
Resin ACP	94.7^{c} (45.1)	–7.3^{c} (1.3)	–3.0^{Ab} (4.4)	3.8^{Ab} (9.4)
Resin ACP-F	213.9^{d} (37.0)	2.5^{b} (3.7)	1.8^{Ab} (4.6)	–1.0^{Aa,b} (3.9)
Ionomer	288.9^{b} (19.8)	10.5^{d} (2.3)	5.4^{Ab} (3.8)	2.6^{Ab} (5.4)

Abbreviations: ACP, amorphous calcium phosphate; F, fluoride.
[a] Distinct superscript lowercase letters indicate statistical significance in each analysis (Student-Newman-Keuls test; $p<0.05$). Distinct superscript capital letters indicate the differences between zones A and B in each line (Student-Newman-Keuls test; $p<0.05$).

Source 2-BM bending magnet station (Argonne National Laboratory, Argonne, IL, USA). X-ray photons with an energy of 20 keV were provided by a double multilayer monochromator.[13] The detector system consisted of a 12-bit, CoolSNAP 2K × 2K CCD camera (Princeton Instrument, New Jersey, USA) coupled with an optical lens (2.5×) to a CdWO4 single-crystal phosphor. Views were recorded every 0.25° from 0° to 180° and were normalized for the detector and beam nonuniformities. Specimens were reconstructed on a 2K × 2K grid of isotropic voxels, side length ~2.8 μm. The analysis was based on mineral concentrations calculated from the linear attenuation coefficient (μ) and described as mass of pure hydroxyapatite (ρ=3.15 g cm^{-3}) per unit volume of tissue (g_{HAp} cm^{-3}).[14]

The integrated area under the curve (cross-sectional mineral profiles into the enamel; Figure 1a), using mineral concentration values (g_{HAp} cm^{-3}) was calculated and the integrated loss of subsurface mineral (Δ_{gHAp} cm^{-3}) determined.[15] The differential mineral concentration profiles (Figure 1b) for the materials and mineral concentrations of sound enamel (ie, g_{HAp} cm^{-3} value of materials minus 2.40 g_{HAp} cm^{-3}) at each of the material groups were also calculated. These differential profiles were then integrated over two depth zones in the lesion and underlying sound enamel to yield the ΔIML values.[16] Zone boundaries were selected at 33.6 and 89.6 μm to highlight differences in the mineral concentration at different depths.

Statistical Analysis

Analyses were performed using the SigmaPlot software (version 12.0, Systat Software Incorporation, San Jose, CA, USA), and the level of statistical significance was established at 5%. After confirmation of the normal (Shapiro-Wilk) and homogeneous (Cochran) distributions, the data from the SH_1 and Δg_{HAp} cm^{-3} were submitted to one-way analysis of variance, followed by the Student-Newman-Keuls test. The results from the ΔIML were submitted to two-way analysis of variance, followed by the Student-Newman-Keuls test. The Pearson's correlation coefficient was calculated for the SH_1 and Δg_{HAp} cm^{-3}.

RESULTS

The surface hardness data of the enamel before the pH cycling exhibited similar mean values (352.1–356.0 kgf/mm^2; p=0.755).The resin no-F group showed the lowest values of hardness after the pH-cycling (p=0.001) when compared to the other groups (Table 2). The resin F and ionomer groups showed similar values of hardness (p=0.807) and were higher than the other groups ($p<0.001$).

The mean of mineral concentration values for sound bovine enamel of the blocks was 2.40 ± 0.06 (2.28–2.54) g_{HAp} cm^{-3}. The results of the Δg_{HAp} cm^{-3} (Table 2) showed that treatment with ionomer, resin ACP-F, and resin F did not present with mineral loss. Resin ACP reduced the mineral loss compared with the resin no-F ($p<0.001$). The combined fluoride/ACP (resin ACP-F group) had an improved resistance against mineral loss, presenting similar values when compared with the resin F group ($p>0.264$).

The profiles of mineral concentration (Figure 2a) showed a subsurface lesion from the resin no-F and resin ACP. The enamel closer (2.8–5.6 μm) to ionomer (2.82 ± 0.25 g_{HAp} cm^{-3}), resin ACP-F (2.75 ± 0.20 g_{HAp} cm^{-3}), resin F (2.61 ± 0.08 g_{HAp} cm^{-3}) and resin ACP (2.61 ± 0.20 g_{HAp} cm^{-3}) presented hypermineralization at 2.8 μm (p=0.133). These groups did not show the formation of a subsurface lesion, except for the resin ACP group. The resins with fluoride and/or ACP presented the same results (p=0.156) at the outer enamel (zone A). However, in

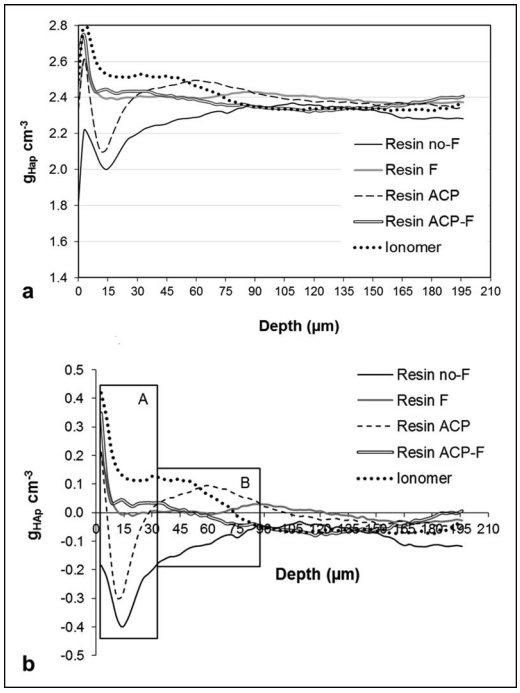

Figure 2. *(a) Depth profiles of mineral concentration (g_{HAp} cm^{-3}) in lesions for each group. (b) Differential mineral concentration profiles (g_{HAp} cm^{-3} vs depth) calculated by subtracting resin profiles from sound enamel value (2.40 g_{HAp} cm^{-3}). Positive values thus indicate a higher mineral concentration at the given depth in the resin groups than the sound enamel, and vice versa. Differential profiles were integrated into each of the marked zones (A–B) to give depth-dependent ΔIML values.*

the depth of the lesion (zone B, 36.4–89.6 μm), the resin ACP showed higher mineral concentration (p=0.038) along with the ionomer (p=0.049) when compared with the resin no-F (Table 2 and Figure 2b).

DISCUSSION

Because ACP is capable of increasing the calcium and phosphate concentrations within a lesion, it has been added to dental products as a remineralizing agent. The addition of ACP in sealant improves the enamel remineralization, as does a fluoride sealant, even presenting different forms of apatite deposition.[7,10] The capacity of ACP deposits on the enamel surface to inhibit the enamel demineralization has not yet been studied. Besides the *in vitro* demineralization produced in the present study, marginal adaptation failed between the material and enamel, a problem that can occur *in vivo*. The present study showed that ACP released by the sealant led to limited effects against demineralization, but combining it with fluoride can improve its effects. For this study, CPPs were not used to stabilize fluoride and ACP.

The calcium and phosphate release from the resin ACP led to supersaturation of the biofilm with respect to the hydroxyapatite as observed in previous research.[8] In the present study, these products were able to reduce the mineral loss, thus increasing the mineral concentration on the enamel surface to the same degree as the fluoride materials. This effect is mainly a product of the ionic activity product of the $CaHPO_4^0$ and the buffering capacity of the ACP.[8,17] These mineral depositions do not contribute to recovery of the prismatic structure and consequent mechanical properties. The mineral deposition produced by the ACP sealant between 2.8 and 5.6 μm was able to attenuate the X-ray photons but it seems this did not lead to the mechanical restructuring of the prismatic structure as the hardness was lower for this group. Moreover, the supersaturation of the biofilm with respect to the hydroxyapatite produced by resin ACP was not able to reduce the mineral loss in the enamel subsurface at a depth between 8.4 μm and 28.0 μm, where the acid activity should be more intense. However, the ACP can produce a great flux of calcium and phosphate in the deeper part of the enamel,[18] leading to a great mineral gain as observed in the present study in the depth of 36.4 μm to 89.6 μm.

Nevertheless, the fluoride materials are able to increase the biofilm saturation with respect to hydroxiapatite and fluorapatite.[8] This can explain the higher mineral concentration of the enamel, mainly at the enamel surface. Greater ionic activity does not occur just for the $CaHPO_4^0$ product but also for the HF^0.[8,17] This leads to the effect being mainly on the surface.[18] The mineral deposited in the enamel surface is harder in the presence of fluoride, but there was not a total recovery of the surface hardness. As the ionomer group was prepared with a powder/liquid ratio (¼:1) that leads to higher fluoride release,[11,12] this can explain the greater anticaries effects.[11]

Adding fluoride to resin with ACP (ACP-F resin group) improved the capacity to inhibit the demineralization to a similar degree as the fluoride materials. Probably, this is because the resin ACP-F led to an increase in saturation with respect to the hydroxyapatite and fluorapatite. Because the ACP in the resin materials is stabilized by hybridization with ions such as Zr^{2+} or SiO^{4-},[5,6] adding sodium fluoride is possible without ACP to react to the fluoride in the composite body. These associations can present a greater ionic activity of the $CaHPO_4^0$ and HF^0 products which is important mainly in the remineralization of the enamel.[8,17] This might have

produced a greater effect on the process of remineralization than demineralization, even with increased cariogenic challenge. The data from the mineral concentration profile (Figure 2) support this observation as there was an increase in the Δg_{HAp} cm^{-3} values. This can explain the hypermineralization at 30 μm from the enamel surface.

CONCLUSION

The present study showed that the ACP inhibits demineralization in the deeper part of enamel, whereas the fluoride products had a greater effect at the outer part of the enamel. The combination of two remineralizing agents (fluoride and ACP) was highly effective in preventing demineralization.

Conflict of Interest

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

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Effect of Ceramic Etching Protocols on Resin Bond Strength to a Feldspar Ceramic

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Clinical Relevance

Acid neutralization following ceramic etching with hydrofluoric acid appears to not impair adhesion of resin cement to hot-pressed leucite-reinforced feldspar ceramic.

SUMMARY

This study sought to evaluate the resin microtensile bond strength (MTBS) stability of a leucite-reinforced ceramic after different ceramic etching protocols. The microtensile test had 40 ceramic blocks (5×5×6 mm) assigned to five groups (n=8), in accordance with the following surface etching protocols: NE non-etched (control); 9HF: hydrofluoric (HF) acid etching (9%HF)+wash/dry; 4HF: 4%HF+wash/dry; 5HF: 5%HF+wash/dry; and 5HF+N: 5%HF+neutralizer+wash/dry+ultrasonic-cleaning. Etched ceramic surfaces were treated with a silane agent. Next, resin cement blocks were built on the prepared ceramic surface and stored for 24 hours in distilled water at 37°C. The specimens were then sectioned to obtain microtensile beams (32/block), which were randomly assigned to the following conditions, nonaged (immediate test) and aged (water storage for 150 days plus 12,000 thermal cycles), before the microtensile test. Bond strength data were submitted to one-way analysis of variance and Tukey test (α=0.05). Additional ceramic samples were subjected to the different ceramic etching protocols and evaluated using a scanning electron microscope (n=2) and atomic force microscopy (n=2). Aging led to a statistically significant decrease in the MTBS for all groups, except the untreated one (NE). Among the groups submitted to the same aging conditions, the untreated (NE) revealed inferior MTBS values compared to the 9HF and 4HF groups. The 5HF and 5HF+N

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groups had intermediate mean values, being statistically similar to the higher values presented by the 9HF and 4HF groups and to the lower value associated with the NE group. The neutralization procedure did not enhance the ceramic/resin cement bond strength. HF acid etching is a crucial step in resin/ceramic bonding.

INTRODUCTION

Feldspar-based ceramic restorations, which can be etched by hydrofluoric (HF) acid, have shown high rates of survival.[1,2] According to the literature, these positive clinical outcomes seem to be associated with achieving a strong and stable bond between the tooth structure/resin cement as well as between the resin cement/ceramic surface.[1,3-7] Briefly, the bond between these restorations and the tooth structure is obtained through the application of phosphoric acid followed by the use of an adhesive system.[1,5] Meanwhile, the bond between resin cement and the ceramic is accomplished by HF acid etching followed by the use of a silane agent, which allows the establishment of both mechanical interlocking and chemical interaction between the materials.[4,5,8-11]

Typically, HF acid etching can appreciably alter the microstructure as well as the surface topography of feldspar-based ceramics, producing different pore sizes and geometries, depending upon the acid concentration and etching time.[12,13] Moreover, after these ceramics have been etched by HF acid and rinsed with water, precipitates are formed that remain on the surface and within its porosities and irregularities making resin bonding more challenging.[4,14-18] The use of neutralizing agents has been suggested[17] to solve this problem and to prevent the continuous etching effect of the acid, as well as the overall acidic environment that could affect resin cement polymerization.[15] Hence, acid precipitations are generated after the reaction between the HF acid and the salt used in the neutralization process, leading to the formation of sodium fluoride and unstable carbonic acid ($NaHCO_3+HF \Leftrightarrow NaF+<H_2CO_3>$). These precipitates remain on the ceramic surface, hindering the penetration of resinous materials into the irregularities to obtain mechanical interlocking.[15] Taken together, the use of neutralizing agents prior to cementation is still debatable, given the lack of consensus in the literature about its real benefits. Therefore, the aims of this study were to 1) evaluate the bond strength between resin cement and a hot-pressed leucite-reinforced feldspar ceramic submitted to different etching protocols, a neutralizing agent, and aging conditions and 2) assess the changes in the ceramic microstructure and surface topography after the different etching protocols. The tested hypotheses were as follows: 1) the surface conditioning protocols and neutralizing agent would not influence the bond strength values; 2) the surface conditioning protocols would not alter the ceramic microstructure and surface topography; and 3) the thermal cycling aging would influence the bond strength values, independently of the etching protocol.

METHODS AND MATERIALS

Ceramic Block Preparation and Etching Protocols

Sixty (5×5×6 mm) blocks were made with vegetal wax (GEO, Renfert, Hilzingen, Germany). Then, using hot-pressed leucite feldspar-reinforced ceramic ingots (VITA PM9, VITA Zahnfabrik, Bad Säckingen, Germany), 60 ceramic blocks were obtained following the manufacturer's instructions. The ceramic bonding surface of each block was wet-finished with 600-1200 grit silica carbide paper (3M, St Paul, MN, USA) for 60 seconds in a polishing machine (EXTEC Labpol 8-12, Extec Corp, Enfield, CT, USA).

The ceramic blocks were allocated into five groups (N=12/group), in accordance with the ceramic etching protocol and aging conditions (Table 1). Forty blocks were used in the microtensile test (n=8/group); 10 blocks (n=2/group) were used for scanning electron microscopy (SEM) evaluation; and 10 blocks (n=2/group) were used for atomic force microscopy (AFM) evaluation. For the 5%HF+neutralizer+wash/dry+ultrasonic-cleaning (5HF+N) group, the neutralizing agent (Kit IPS Ceramic, Ivoclar-Vivadent, Schaan, Liechtenstein) was applied following the manufacturer's instructions. A 3-methacryloxypropyltrimethoxysilane (MPS)–based silane agent was applied onto the surface of all the etched ceramic blocks (Porcelain Primer, Bisco, Schaumburg, IL, USA).

Resin Cement Block Preparation

Upon ceramic surface etching, the blocks were inserted into an addition silicone mold (Elite HD, Zhermack, Badia Polesine, Italy) to a depth of 5 mm, keeping the etched surface up. A resin cement (Panavia F2.0, Kuraray, Okayama, Japan) was manipulated in a 1:1 ratio, applied on the etched surface, occupying all the space created in the silicone material, and photoactivated for 40 seconds with a quartz-tungsten-halogen unit (XL 3000, 3M,

Table 1: *Study Design*[a]

	Ceramic Etching Protocols	Aging	Groups
NE	Nonetched (control)	No	NE-dry
		Yes	NE-aged
9HF	9% Hydrofluoric acid during 1 min + washing[b] + drying[c]	No	9HF-dry
		Yes	9HF-aged
4HF	4% Hydrofluoric acid during 1 min + washing + drying	No	4HF-dry
		Yes	4HF-aged
5HF	5% Hydrofluoric acid during 1 min + washing + drying	No	5HF-dry
		Yes	5HF-aged
5HF+N	5% Hydrofluoric acid during 1 min + neutralizing agent (N) + washing/drying + sonic cleaning for 5 min	No	5HF+N-dry
		Yes	5HF+N-aged

Abbreviations: NE, nonetched; HF, hydrofluoric acid; N, neutralizer.
[a] 9% Hydrofluoric acid: Ultradent Porcelain Etch (Ultradent Products Inc, South Jordan, UT, USA); 4% hydrofluoric acid: Porcelain Etchant (Bisco, Schaumburg, IL, USA); 5% hydrofluoric acid and neutralizer (neutralizing powder): Kit IPS Ceramic (Ivoclar-Vivadent, Schaan, Liechtenstein).
[b] Washing with oil-free air-water spray for 20 seconds.
[c] Drying with air spray for 20 seconds.

St Paul, MN, USA) through the upper surface. Next, the resin cement/ceramic block was removed from the silicone mold and the other bonded surfaces were photoactivated for 40 seconds. Finally, the assemblies were stored at 37°C in distilled water for 24 hours.

Sample Preparation, Aging, and Microtensile Bond Strength Test

The blocks were fixed with cyanoacrylate adhesive gel (Super Bonder Gel, Loctite, Dusseldorf, Germany) to a metallic device that was then attached to a sectioning machine (Labcut 1010, Extec, Enfield, CT, USA). The blocks were positioned perpendicularly to the diamond disc and four cuts of 1-mm thickness were made. The blocks were then rotated 90° and an additional four cuts of similar dimension were done to obtain microtensile beams with an adhesive area of 1 mm^2 and 10 mm in length. The beams located at the outer part of the blocks were discarded. Half of the specimens were submitted immediately to the microtensile bond strength test (without aging groups) in a universal testing machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil) at a crosshead speed of 1 mm/min, while the remaining specimens were submitted to an aging protocol involving storage in distilled water at 37°C for 150 days followed by 12,000 thermal cycles of alternates baths at 5°C and 55°C, for 30 seconds each, with intervals of two seconds between them. After aging, the samples were tested as previously described.

Bond strength was calculated using the formula $\sigma = F/A$, where σ is the bond strength (MPa), F is the load to fracture (N), and A is the adhesive area (mm^2). The adhesive area of each specimen was measured prior to the test with a digital caliper (Starrett, Itu, SP, Brazil).

Failure Analysis

After the microtensile test, all specimens were examined under a stereomicroscope (Discovery V-20, Zeiss, Germany) at 50× magnification to determine the failure mode. The failures were classified as Adhesive (Adhes)—failure in the interface between resin cement and ceramic; Cohesive of resin cement (Cohes-cem)—cohesive failure of the resin cement; Cohesive of ceramic (Cohes-cer)—cohesive failure of the ceramic; and Mixed (Mix)—adhesive failure associated with a cohesive failure.

Statistical Analysis

For statistical analysis, the bond strength means of the samples (repetitions) from each block were calculated, considering each block (n=8) as the experimental unit.[19] The bond strength means of aged groups and not aged groups were compared by one-way analysis of variance (ANOVA) and Tukey tests using the software Statistix 8.0 (Analytical Software, Tallahassee, FL, USA). The comparison between nonaged (immediate) vs aged groups submitted to the same etching protocol was performed by Student t-test. All analyses were done at the 5% significance level. Specimens with cohesive failure were not included in the statistical analysis. Pretest failures received an arbitrary value of 2 MPa, which corresponds to half of the minimal bond strength value observed during the microtensile test.[7,20-22]

Table 2: *Means and Standard Deviations of the Bond Strength Data and Tukey and Student* t*-Test (5% Significance Level)*

Ceramic Etching Protocol	Aging		*p*-Value[c]
	No[a]	Yes[b]	
NE	3.4 ± 1.6 C	2.3 ± 0.5 B	0.0844
9HF	13.7 ± 2.1 A	8 ± 4.8 A	0.0077
4HF	15.2 ± 2.7 A	9 ± 4.1 A	0.0032
5HF	16.8 ± 2.2 A	6 ± 4.9 AB	0.0001
5HF+N	10.6 ± 2.2 B	5.2 ± 2.9 AB	0.0010

Abbreviations: NE, nonetched; HF, hydrofluoric acid; N, neutralizer.
[a] *Comparison for nonaged groups using one-way analysis of variance (ANOVA) and Tukey tests: different letters indicate statistically significant differences.*
[b] *Comparison for aged groups using one-way ANOVA and Tukey tests: different letters indicate statistically significant differences.*
[c] *Comparison for nonaged vs aged groups, for each etching method, using Student* t*-test:* $p < 0.05$ *indicates statistical difference.*

Micromorphological Analysis—SEM and AFM

After ceramic etching, four blocks from each group were analyzed by SEM (n=2) and AFM (n=2) to assess changes in surface topography. For SEM, the samples were mounted onto aluminum stubs, sputter-coated with gold, and evaluated under a SEM (JEOL, JSM-T330A, Jeol Ltd, Tokyo, Japan) at different magnifications. For AFM (Bruker BioScope Catalyst, Santa Barbara, CA, USA), the images (20 μm×20 μm) were collected in peak force tapping mode using RTESPA probes (Bruker, radius nominally 8 nm, k=40 N/m). AFM micrographs were analyzed using a scanning probe microscopy data analysis software (Gwyddion™, version 2.33, GNU, Free Software Foundation, Boston, MA, USA).

RESULTS

One-way ANOVA revealed a significant influence of the ceramic etching protocols for both immediately tested ($p<0.0001$) and aged groups ($p=0.0001$) (Table 2).

Among nonaged groups, all the etching protocols tested promoted higher bond values (10.6-16.8 MPa) than did the control (3.4 MPa). The group etched by 5HF+N (dry condition) presented lower (10.6 MPa) bond values than did its counterpart (without neutralization; ie, 5HF [16.8 MPa]). After aging, the group that was subjected to neutralization (5HF+N) had values (5.2 MPa) similar to that of the nonetched group (2.3 MPa).

When comparing the same etching protocols before and after aging, the Student *t*-test revealed that aging led to a significant decrease in the bond strengths of all the etching protocols, except the untreated (nonetched [NE]) group (Table 2). The failure analysis is depicted in Table 3. All of the pretest failures were adhesive (Table 3). SEM micrographs revealed very similar microstructures and topographical patterns, regardless of the HF acid concentration used (Figure 1). AFM three-dimensional topographical analyses further confirmed the morphological findings provided by SEM.

DISCUSSION

HF acid application followed by a silane coupling agent has been recommended as the main conditioning protocol of the intaglio surface of feldspar-based ceramic restorations.[5,8-10,23] However, different HF acid concentrations can change both the pH and the

Table 3: *Number and Percentage of the Pretest Failures and Failure Types*

Groups	Aging	No. of Pretest Failures	Type of Failure (%)[a]			
			Adhesive	$COHES^{cem}$	$COHES^{Cer}$	Mixed
NE	No	13	13 (40.6)	1 (3.2)	0 (0)	18 (56.2)
9HF		0	0 (0)	0 (0)	0 (0)	32 (100)
4HF		0	0 (0)	0 (0)	0 (0)	32 (100)
5HF		0	0 (0)	2 (6.3)	1 (3.2)	29 (90.5)
5HF+N		0	0 (0)	0 (0)	3 (6.3)	29 (93.7)
NE	Yes	23	23 (71.8)	2 (6.3)	1 (3.2)	6 (18.7)
9HF		5	5 (15)	0 (0)	0 (0)	27 (85)
4HF		5	16 (50)	3 (9.4)	0 (0)	13 (40.6)
5HF		16	5 (15.60)	0 (0)	4 (12.5)	23 (71.8)
5HF+N		11	11 (34.3)	0 (0)	0 (0)	21 (65.6)
Total			73 (22.8)	8 (2.5)	9 (2.8)	230 (71.8)

Abbreviations: NE, nonetched; HF, hydrofluoric acid; N, neutralizer.
[a] *Adhesive: failure at the interface between resin cement and ceramic;* $COHES^{cem}$*: cohesive failure of the resin cement;* $COHES^{Cer}$*: cohesive failure of the ceramic; Mixed: adhesive failure combined with cohesive failure.*

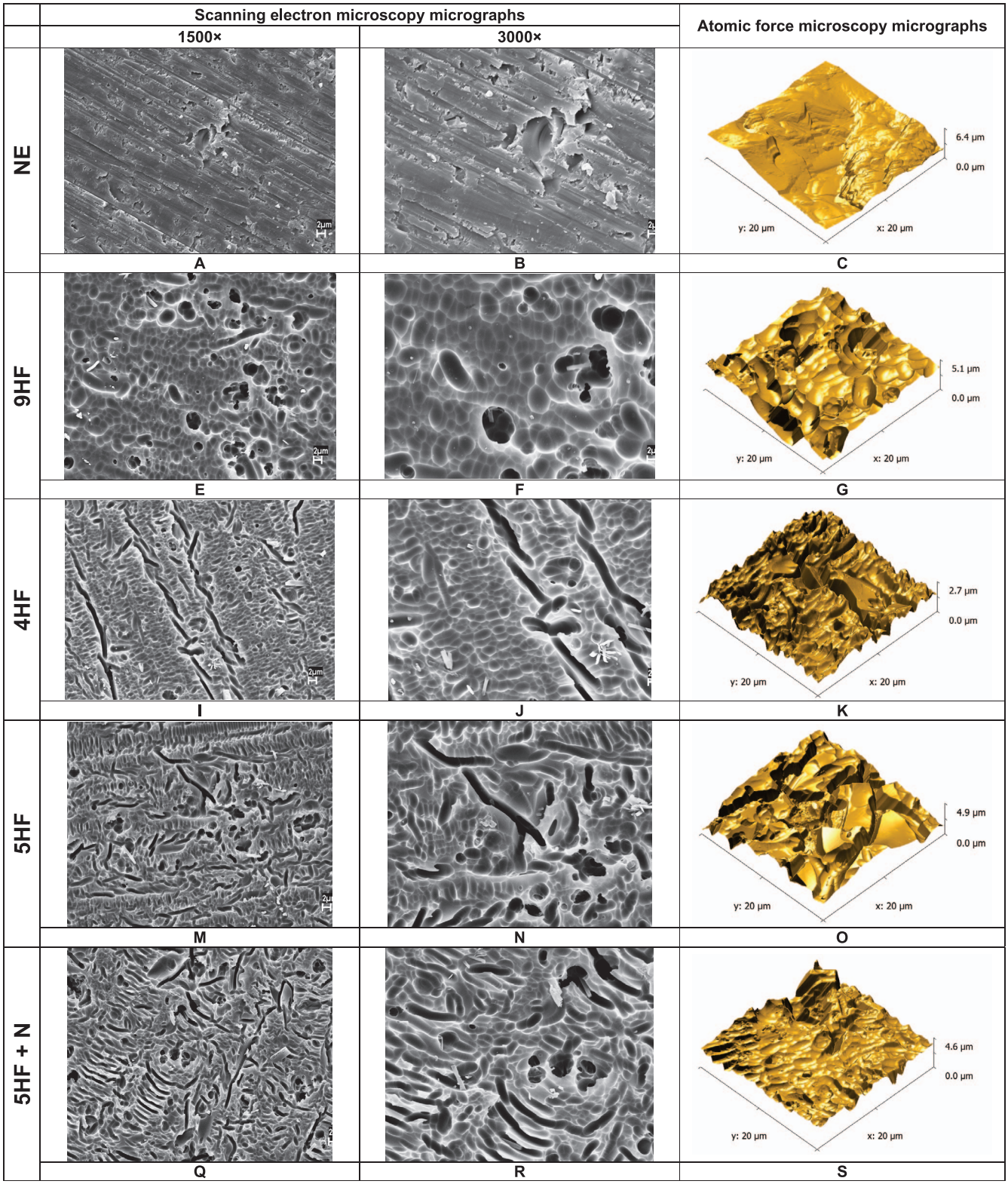

Figure 1. *Representative SEM micrographs and AFM 3D topography images of the ceramic surfaces submitted to the different acid etching protocols. The A, B, and C images show the control group with no surface alteration topography after etching protocol. The other images (E-S), correspond to different acid etching protocols, presenting similar surface topography modification.*

ceramic surface energy, affecting the bonding process.[13,14] In addition, the use of products for neutralizing the pH of the ceramic surface after etching is still a very debatable topic.[14,15]

Our data revealed a significant influence of the different ceramic etching protocols on the resin/ceramic bond strength (Table 2), counter to the first hypothesis of the study. Based on the results, the nonaged groups 4HF, 5HF, and 9HF showed higher bond strength values than did the NE and 5HF+N groups. Meanwhile, after aging, those groups were statistically similar: in other words, the neutralization procedure did not enhance ceramic/resin cement bond strength. The improved results obtained after ceramic etching relate to the fact that the HF acid selectively attacks the glassy phase of the ceramic, changing its surface topography, which in turn provides sites for mechanical interlocking between the resin cement and the ceramic.[5,9-11,23] Furthermore, HF acid etching increases the surface energy of the ceramic, augmenting its adhesive potential.[5,9,10] Additionally, silane agents based on MPS present molecules that react with water, forming silanol groups (–Si–OH) from the methacryloxy groups (–Si–O–CH_3). Silanol groups react to form a siloxane network (–Si–O–Si–O–) with silicon oxide present in the ceramic, forming a chemical bond between the materials.[5,11,24-28] The monomeric ends of the silane molecule react with the methacrylate groups of the resin cement.

The higher bond strengths associated with the etched group compared to the group etched and neutralized are in agreement with the findings of other studies.[14,15] These previous studies showed that the use of neutralizing products decreases the ceramic surface energy and creates precipitates within the etched region, damaging the bonding capability between resin cement and ceramic.[14,15] Taken together, the neutralization process appears not to impair adhesion for the cementation of a hot-pressed leucite-reinforced feldspar ceramic.

The ceramic microstructure imaged through both SEM and AFM (Figure 1) showed no apparent difference in surface topography after the different ceramic etching protocols, in agreement with the second hypothesis of the study. The very similar topographical changes observed among the etched groups may have contributed to the statistical similarity of the bond strength values between groups (Table 2), similar to the findings of Amaral and others.[14] However, when comparing the etched groups vs the nonetched, significant differences were apparent in terms of surface topography. An unappreciable surface modification was seen in the nonetched group, supporting the lower bond strength values.[9,17] Indeed, the failure analysis (Table 3) showed a higher number of pretest failures associated with the aged groups, confirming that the adhesive interface was affected.

CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn: 1) the neutralization process did not improve the bond strength or stability between resin cement and ceramic; and 2) HF acid etching of hot-pressed leucite-reinforced feldspar ceramic is indispensable to enhance resin bonding.

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Conflict of Interest

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

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Evaluation of Genotoxicity and Efficacy of At-home Bleaching in Smokers: A Single-blind Controlled Clinical Trial

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Clinical Relevance

The results of this study indicate that 10% carbamide peroxide gel did not induce DNA damage in gingival tissue during the evaluated period. The bleaching procedure was effective in smokers.

SUMMARY

Objective: This single-blind controlled study evaluated the genotoxicity and efficacy of at-home bleaching in smokers and nonsmokers.

Methods: We selected 60 patients with central incisors A2 or darker: 30 smokers (experimental group) and 30 nonsmokers (control group). The bleaching was carried out with 10% carbamide peroxide for three hours a day for three weeks. The color was evaluated using a shade guide, Vita Bleachedguide 3D-Master, at baseline, during bleaching (first, second, and third weeks), and one week and one month after bleaching. Smears were obtained with a moistened wooden spatula from marginal gingiva. All the cytologic smears were stained with Giemsa solution. From each slide, 1000 cells were examined under 40× magnification and where micronuclei (MN) were located, they were examined under 100× magnification. The change in shade guide units at the different assessment periods and the frequency of MN were subjected to a two-way repeated measures analysis of variance and Tukey test (α=0.05).

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Results: In both groups we detected a whitening of approximately 4 to 5 shade guide units, without color rebound after one month ($p>0.05$). The frequency of MN was significantly higher in the experimental group than in the control group, regardless of the bleaching treatment ($p>0.001$).

Conclusion: The efficacy of bleaching does not appear to be affected by the smoking habit. Additionally, at-home bleaching did not induce DNA damage to the gingival tissue during the bleaching period.

INTRODUCTION

The demand for dental esthetics has increased the number of dental bleaching procedures among dental professionals.[1] As dental bleaching is a relatively simple procedure, dentists usually offer it to their patients[2] to solve discoloration problems in the permanent dentition. This procedure became more popular after the introduction of at-home bleaching in 1989.[3]

Although the effectiveness of this procedure is well reported in the literature,[4-6] the clinical trials on this technique are usually performed on patients with sound teeth and who are nonsmokers. Smoking is usually included as an exclusion criterion in most clinical trials of at-home[4,7-12] and in-office bleaching.[1,13,14]

There are at least two reasons for this exclusion. First, cigarette smoke contains water, air, carbon monoxide, carbon dioxide, and tar. During cigarette burning, components such as tar, sugar, and cocoa are transferred to the smoke hue,[15] which probably stain teeth because of their dark hue and ability to adhere to the dental surface.[16]

Another concern is that there are around 1.2 billion smokers in the world, and it is estimated that this habit causes more than 1 million cancer deaths per year;[17] this is probably the rationale behind excluding smokers from studies of dental bleaching procedures. The prevalence of self-assessed tooth discoloration in smokers is almost twice that reported by nonsmokers,[18] and therefore, they are probably the main candidates for bleaching procedures in a daily practice.

DNA damage in the cells of the oral mucosa of smokers usually signals the genotoxicity potential of the smoking habit.[19,20] This can be indirectly observed by the increase in the frequency of micronuclei (MN) in exfoliated epithelial cells.[21,22] During the division of the cells from the basal layer of the mucosa, the damage to the DNA molecule leads to the formation of MN, which consist of acentric chromosomes, chromatid fragments, or whole chromosomes that failed to be incorporated in the daughter nuclei during mitosis. The formation of MN is, therefore, induced by substances that cause breakage of chromosomes (clastogens) and by agents that affect the spindle apparatus (aneugens). This usually occurs days or weeks after contact with a carcinogenic agent.[21,22] Evaluation of the frequency of MN is a viable method for detecting risk of cancer in humans, because most tumors possess epithelial origin.[23] In regard to smoking, a positive correlation was already reported between a higher frequency of MN and tobacco users.[20,24,25]

Although the effects of bleaching agents on hard tissues have already been extensively investigated,[26] the response of the soft tissue to these agents remains largely unknown in humans.[27] Although it was demonstrated that hydrogen peroxide can induce pathological alterations in soft tissues in animal models[28,29] and in cell research,[30-36] few studies have been conducted on humans.[37,38] This is especially important when it comes to smokers as it is well established that they have an increased risk of developing oral cancer or other forms of epithelial cancer.[39-41] Therefore, the aim of this single-blind controlled clinical trial was to evaluate the efficacy and genotoxicity of at-home bleaching in smokers and nonsmokers.

METHODS AND MATERIALS

This controlled single-blind nonrandomized clinical trial recruited patients by printed advertising at the local universities. During the screening, dental prophylaxis was performed for dental screening and color evaluation at baseline, which usually occurred two weeks before starting the bleaching protocol.

Inclusion and Exclusion Criteria

Participants included in this clinical trial were between 18 and 40 years old and had good general and oral health. Each subject had at least one central incisor with shade 1M2 or darker, assessed by comparison with a value-oriented shade guide (Vita Bleachedguide 3D-Master, Vita Zahnfabrik, Bad Säckingen, Germany). Participants who underwent previous dental bleaching procedures during orthodontic treatment, pregnant or lactating women, and participants with bruxism habits were not included in the trial.

Additionally, participants with restorations on the labial surfaces of anterior teeth and noncarious cervical lesions, teeth veneers or full crowns, gingival recession, spontaneous tooth pain, severe internal tooth discoloration, and teeth with endodontic treatment or stains classified as 3 or higher according to the Thylstrup-Fejerskov Index[42] were also excluded from this trial. A total of 60 volunteers signed the consent form and were enrolled in this study.

Sample Size Calculation

The sample size calculation was based on the frequency of MN per 1000 cells in nonsmokers. In the pilot study it was observed that the normal frequency of MN in nonsmokers is about 1 ± 1.1.[24,43-45] In order for the bleaching procedure to be considered safe, it was expected that we would find a mean difference of not more than 1.0. Thus, we needed a minimum sample size of 27 participants for a study with a predictive power of 90% and an alpha of 5%.

Experimental Groups

Participants who met the inclusion criteria were asked about their daily smoking habits. Those who did not smoke were part of the group of nonsmokers and those who smoked at least 10 cigarettes a day belonged to the group of smokers. Thirty participants were included in each group.

Bleaching Procedure

Alginate impressions were made of each subject's maxillary and mandibular arch, and these were filled with dental stone. To produce study models, no block-out material was applied to the labial surfaces of teeth.[46] A 1-mm soft vinyl material provided by the manufacturer (FGM Dental Products, Joinville, Brazil) was used to fabricate the custom-fitted tray that would hold the bleaching gel. The excess material from the labial and lingual surfaces was trimmed 1 mm from the gingival junction. The tray and 10% carbamide peroxide gel (Whiteness Perfect, FGM) were delivered to each subject, with verbal instructions for use. All subjects were instructed to wear the tray containing the bleaching agent for at least three hours a day for a period of three weeks. After the daily three-hour period, they were instructed to remove the tray, wash it with water, and brush their teeth as usual. With regard to oral hygiene, all participants were instructed to brush their teeth regularly and were asked to not use whitening toothpastes and mouthwashes containing peroxides.

Shade Evaluation

The shade evaluation was performed with a value-oriented shade guide (Vita Bleachedguide 3D-Master). Two calibrated evaluators with a previous agreement of at least 85%, as determined by weighted kappa statistics, recorded the shade of the upper central right incisor at different time assessments: at baseline, during treatment (after the first, second, and third weeks of bleaching), and one week and one month after the end of the bleaching protocol. As evaluators could guess which group the participants were from, usually by the smoking smell, this procedure could not be blinded.

The area of interest for measuring tooth color matching was the middle third of the facial surface of the anterior central incisor, according to the American Dental Association guidelines.[47] Shade changes were calculated from the beginning of the active phase to the individual recall times by calculating the change in the number of shade guide units (ΔSGUs), which occurred toward the lighter end of the value-oriented list of shade tabs. In the event of disagreements between the examiners during shade evaluation, a consensus was reached.

Tooth Sensitivity (TS) Evaluation

Subjects were instructed to keep a daily record of whether they experienced TS, using a visual analog scale.[1,7,11,13] They were asked to place a line perpendicular to a 10-mm long line with zero at one end indicating "no TS" and the 10-mm end indicating "unbearable TS."

Sample Collection for MN in Exfoliated Epithelial Cells

Exfoliated oral mucosa was collected before and immediately after the third week of at-home bleaching. Before cell collection, the participants rinsed their mouths with tap water for one minute. Subsequently, the cells were scraped with wooden spatulas from the marginal gingiva.[24,25,44,48] The scraped cells were placed on clean glass slides, and smears were prepared. The smear was dried with a jet of air from a triple syringe for one minute at a distance of approximately 30 cm, avoiding excessive dehydration of the cells.[48]

Staining Procedures

The staining protocol was prepared immediately after the smear collection. Five to six drops of Giemsa stock solution (Cinética Química, São Paulo, Brazil) was applied directly over the slide

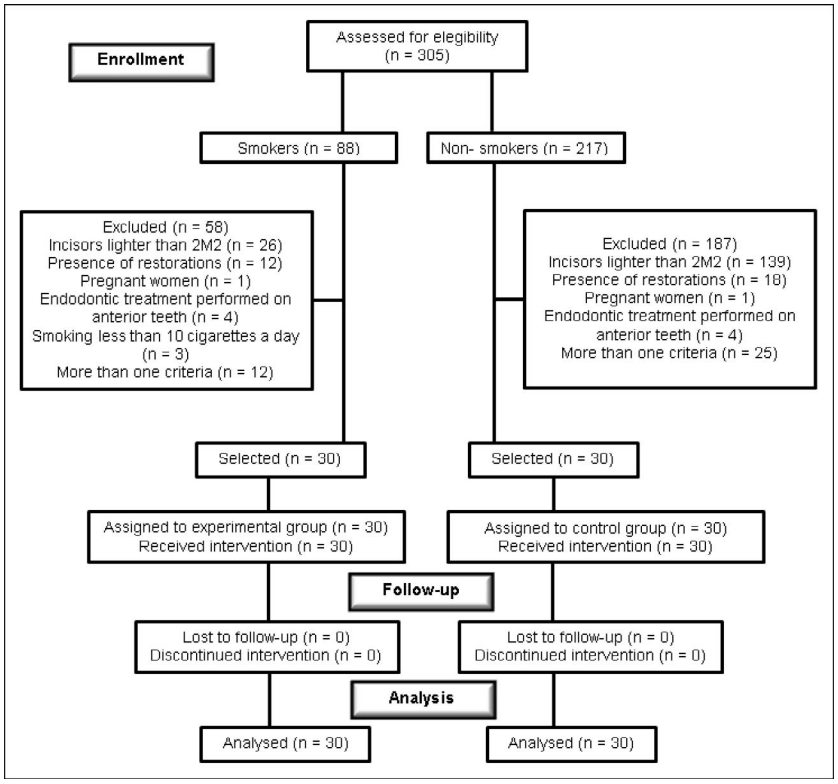

Figure 1. Study Design

for two minutes; then the slides were washed in a container with tap water (container 1 = three to four washes, container 2 = two to three washes). The differentiation of the cells was performed in a third container (1200 mL of tap water and one drop of glacial acetic acid, Vetec Quimica Fina Ltda., Rio de Janeiro, Brazil). After this process, the slide was dried for one minute in the same manner described before. Then, three drops of the adhesive Entellan (Merck KGaA, Darmstadt, Germany) were applied on the visibly dry slide for cover glass positioning.[48]

Evaluation of the Slides

Two blinded examiners were trained and calibrated for the evaluation of the slides by one expert in stomatology. From each participant, at least 1000 cells were evaluated at each period with the staining procedure. Cell counting was performed under an optical microscope with 100× magnification, and when MN were found, the magnification was increased to 400× (Nikon E800, Tokyo, Japan). Criteria for inclusion in the total cell count were the following: 1) cytoplasm intact and lying relatively flat; 2) little or no overlap with adjacent cells; 3) little or no debris; and 4) nucleus normal and intact, nuclear perimeter smooth and distinct.[49]

The parameters for identifying micronuclei were as follows: 1) rounded smooth perimeter suggestive of a membrane, 2) less than a third of the diameter of associated nucleus but large enough to discern shape and color, 3) staining intensity similar to nucleus, 4) texture similar to nucleus, 5) same focal plane as nucleus, and 6) absence of overlap with or bridge to nucleus.[49] Dead or degenerating cells (karyolysis, karyorrhexis, nuclear fragmentation) were excluded from evaluation. Nuclear blebbings (micronucleus-like structure connected with the main nucleus by a bridge) were also not considered.

Statistical Analysis

The ΔSGU at the different assessment periods and the data frequency of MN were tabulated using the software SigmaPlot 5.0 for Windows (Systat Software Inc, San Jose, CA, USA) and subjected to a two-way analysis of variance (ANOVA) (treatment group vs time; α=0.05) for repeated measures (time). The Tukey test was performed for contrast of means (α=0.05). The percentage of participants who experienced TS at least once during the bleaching therapy was determined to be the absolute risk of TS. The absolute risk and intensity of TS of both groups was compared with the χ^2 and Mann-Whitney tests (α=0.05), respectively.

Table 1: *Demographic Characteristics of the Participants*

	Nonsmokers	Smokers
Baseline color (SGU; mean±SD*)	8.2 ± 1.3	7.8 ± 1.1
Age (years; mean±SD)	24.1 ± 6.8	26.3 ± 6.5
Sex (male; %)	53.3	63.3
Cigarettes/day (mean±SD)	--	13.2 ± 4.0
Average smoking years (mean±SD)	--	8.0 ± 5.9

** Abbreviations: SD, standard deviation; SGU, shade guide unit.*

RESULTS

A total of 305 participants in the range of 18 to 40 years old were screened to select 60 participants who met the inclusion criteria (Figure 1). The mean age and baseline tooth color of the participants were similar between groups. Most of the participants were men (Table 1). All participants attended the recall visits during the bleaching protocol. None of the patients continued bleaching as they were satisfied with the outcome reached after three weeks of treatment.

Shade Evaluation

Two-way ANOVA revealed that the interaction of treatment group vs time (p=0.372) and the main factor treatment group (p=0.098) were not significant. Only the main factor time was statistically significant (Table 2; $p<0.001$). A significant color change of approximately 4.5 to 5.0 SGUs was observed after bleaching for both groups, which was stable one month after the procedure (Table 2).

Tooth Sensitivity Evaluation

Table 3 presents data on the prevalence of dental sensitivity for the sample investigated. The risk of TS was similar between the two study groups (χ^2, p=1.0), and approximately 50% of patients had at some point in the procedure, tooth sensitivity. Similarly, the TS intensity (Mann-Whitney test, p=0.83) was not significantly different between groups. None of the patients from this trial gave up the treatment.

Table 2: *Means and Standard Deviations Between Assessment Points for the Characteristics of Nonsmokers and Smokers in Change in Shade Guide Units (ΔSGUs) Assessed by Means of Subjective Using Vita Bleachedguide 3D-Master*[a]

Assessment Time Intervals	ΔSGU	
	Nonsmokers	Smokers
Baseline vs 1 wk	2.1 ± 1.1 C	1.9 ± 1.0 C
Baseline vs 2 wk	3.9 ± 1.3 B	3.4 ± 1.2 B
Baseline vs 3 wk	4.9 ± 1.4 A	4.4 ± 1.0 A
Baseline vs 1 wk postbleaching	5.0 ± 1.4 A	4.4 ± 1.0 A
Baseline vs 1 mo follow-up	4.7 ± 1.4 A	4.1 ± 1.1 A

[a] *Two-way analysis of variance and Tukey test (p<0.001).*

Table 3: *Comparison of the Number of Patients Who Experienced Tooth Sensitivity (TS) at Least Once During the Bleaching Regimen in Both Groups and Intensity of TS for Both Groups Under the Pain Scale*

Treatment	Number of participants with TS		Absolute Risk (95% Confidence Interval)[a]	Visual Analog Scale[b]
	Yes	No		
Nonsmokers	14	16	47 (30–64)	0.5 ± 0.9
Smokers	15	15	50 (33–67)	0.7 ± 1.2

[a] χ^2 *test (p=1.0).*
[b] *Mean ± standard deviation; Mann-Whitney (p=0.83).*

Assessment of Genotoxicity by MN frequency

The two-way ANOVA revealed that the interaction of treatment group vs time (p=0.067) and the main factor time (p=0.248) were not statistically significant (Table 4). Only the main factor treatment group was statistically significant ($p<0.001$), which means the bleaching procedure did not increase the frequency of MN. The amount of MN was significantly higher in smokers than in nonsmokers, regardless of the bleaching procedure ($p<0.001$).

DISCUSSION

Although smokers also require dental bleaching in daily practice, the literature lacks information about the efficacy and safety of the procedure in such patients. Most of the studies in dental bleaching use shade guides for color evaluation.[4-6,12,13] Although these shade guides were primarily designed for shade matching with composite resins, their use is supported in the literature for evaluating bleaching effectiveness.[4-6,12,13] Compared with the spectropho-

Table 4: *Means and Standard Deviations of the Micronuclei Frequency per 1000 Exfoliated Buccal Mucosa Cells of Nonsmokers and Smokers.*[a]

Assessment Periods	MN Frequency	
	Nonsmokers	Smokers
Before bleaching	1.4 ± 2.2 A	3.7 ± 2.0 B
After bleaching	0.5 ± 0.7 A	3.9 ± 1.8 B

[a] *Two-way analysis of variance and Tukey test (p< 0.001). Same letters are statistically similar.*

tometer, the shade guides show better visual correlation and have the potential to allow for more accurate monitoring, and consistent and reliable color of teeth.[49]

Vita Classical (Vita Zahnfabrik)[4,5,11,13] and Trubyte Bioform (Dentsply Intl, York, PA, USA) [50-53] are the most frequently used shade guides in dental bleaching studies. However, they have a nonlinear color arrangement, as they were not primarily designed to evaluate dental bleaching. This is why we used the shade guide Vita Bleachedguide 3D-Master. This new shade guide is already organized from lowest to highest value; it contains lighter shade tabs with subtle color gradation and more uniform color distribution compared to Vita Classical and Trubyte Bioform scales.[50] Additionally, Vita Bleachedguide was scored as the easiest to rearrange and the most preferred for dental bleaching monitoring and other dental procedures that require shade matching.[54]

An effective bleaching was observed after three weeks of treatment, which remained stable one month after bleaching. An overall bleaching of 4 to 5 SGUs herein reported is in agreement with earlier at-home studies that used 10% carbamide peroxide gel.[4,5,9,12,55] Surprisingly, no significant difference was observed between groups. As reported in the introduction, some components of cigarette smoke are responsible for tooth discoloration in smokers; however, it appears to be superficial and easily removed with professional cleaning[56] and dental bleaching.[16] Perhaps, color rebound may occur earlier in smokers than nonsmokers because of the continuous deposition of cigarette smoke components on the enamel surface. This hypothesis was not confirmed with the results of the present study because the bleaching results after one month did not show any effect in terms of color rebound. However, this is a short-term follow-up, and only long-term clinical evaluations can assess this hypothesis.

Several *in situ*[30-36] and *in vivo* studies in animals[28,29] observed different types of DNA damage by various concentrations of bleaching agents. Different *in vivo* studies in animals showed no risk involved in the bleaching procedure.[57-60] However, these results cannot be directly extrapolated to humans, as they do not resemble the clinical scenario.

In light of these considerations, methods that assess the genotoxicity potential of bleaching agents under a realistic condition are essential. An increased frequency of chromosome breaks has been recently demonstrated to be an initial event in carcinogenesis, suggesting that these alterations may play a significant role in assessing oncogenic risk.[61,62] Among biomarkers that can be used for this purpose, the measurement of MN appears to be one of the most suitable. An increased frequency of MN in exfoliated cells from oral mucosa has served traditionally as an index for evaluating the genotoxicity of exposure to various carcinogens.[63,64] MN originates from chromosome fragments or whole chromosomes that are not included in the main daughter nuclei during nuclear division. They reflect chromosome damage and may thus provide a marker of early-stage carcinogenesis.

In the present study, we observed a statistically significant difference between the frequency of MN in smokers and nonsmokers, which has been shown previously in most recent studies.[20,24,25,46] The frequency of MN in normal oral mucosa is between 0.5 and 2.0/1000 cells,[24,44,45] which is within the range we detected for nonsmokers.

On the other hand, an average of 3.8/1000 cells was detected in smokers in this study, which is also within the range reported by some studies.[65-67] This is probably due to the high number of carcinogens in the tobacco smoke that produces related DNA damage.[68]

Although many studies have already assessed the frequency of MN in smokers,[20,24,25,44,45,62,65,66] only one used this method to assess the genotoxicity of in-office bleaching agents.[38] Fortunately, we demonstrated that the frequency of MN was not increased after bleaching with 10% carbamide peroxide in both study groups, suggesting that the low-concentration carbamide peroxide gel did not seem to induce DNA damage to the gingival tissue when used for three hours daily over a three-week period.

These findings, however, are not in agreement with those of Klaric and others,[38] who observed a higher MN frequency 72 hours after in-office bleaching. A more concentrated bleaching gel (35% hydrogen peroxide) was used in the aforementioned study; additionally, the authors isolated the gingival tissue from the participants with a light-curing gingival barrier, which may also have played a role in the results obtained.

Another *in vivo* study in humans,[37] which assessed the effects of bleaching agents on the gingival tissue by biopsy, observed a proliferative activity of the gingival epithelium after bleaching with 10% carbamide peroxide gel (eight hours daily for a five-week period). However, this study should be interpreted with caution as an increase in the prolifera-

tive activity of epithelial cells does not necessarily mean that the agent has a genotoxic potential.

One should also mention the current study's limitations. First, during color evaluation the examiners could not be blinded. Although patients were asked to rinse their mouth with mouth rinses, the smoking smell was impregnated in the participant's clothes, hair, hands, and breath. Second, the frequency of MN was only evaluated soon after the end of the bleaching procedure. It is known that it takes approximately 10-12 days for the regeneration of the cells from the gingival tissue,[69] which is a little bit shorter than the period of the bleaching protocol. The study of DNA damage in exfoliated cells collected from the oral cavity holds great promise as a minimally invasive method for monitoring exposure to genotoxic agents; according to Thomas and others,[70] as the buccal cells turn over every 7 to 21 days, thereafter it is theoretically possible to observe the genotoxic effects of acute exposure within this period. Last, care should be taken during the extrapolation of the study results to women, as most of the participants in the smokers group were men.

Future studies should be conducted with other bleaching agents and protocols. Additionally, samples from the gingival tissue should be collected for longer periods after the end of the bleaching protocol to definitely ensure the safety of this cosmetic procedure in dentistry.

CONCLUSION

It can be concluded that the effectiveness of dental bleaching does not seem to be affected by smoking and at-home bleaching does not induce DNA damage to the gingival tissue.

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Human Subjects Statement

This study was conducted in accordance with all the provisions of the local human subject oversight committee guidelines and policies. The approval code for this study was 208.355 under protocol number 16457/2012. This study was conducted at State University of Ponta Grossa, Paraná, Brazil.

Conflict of Interest

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

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Effect of Beverages on Color and Translucency of New Tooth-Colored Restoratives

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Clinical Relevance

When exposed to dark beverages, nano-filled glass ionomer and Giomers were more susceptible to staining and translucency changes than composites but less susceptible than resin-modified glass ionomer cement. Increased staining was correlated with a decrease in translucency, compromising clinical esthetics.

ABSTRACT

This investigation examined the susceptibility to staining and translucency changes of some new tooth-colored restorative materials after immersion in different beverages. The materials studied were 3M Filtek Z350XT (ZT), 3M Filtek 350XT Flowable Restorative (ZF), Shofu Beautifil Flow Plus (BF), Shofu Beautifil II (B2), 3M Ketac Nano (N100), and 3M Photac Fil (PF). Following the manufacturers' instructions, 42 samples were made from each material and placed in an incubator at 100% humidity and 37°Celsius for 24 hours. Baseline L*, a*, b* readings were taken against white and black backgrounds using a photospectrometer. The samples were then randomly assigned to be immersed in seven beverages, namely cola drink, orange juice, red wine, vodka, black coffee, green tea, and distilled water for a period of seven days. Color readings were taken again by recording the L*, a*, b* values. Data was analyzed using *t*-tests, one-way analysis of variance with Tukey post hoc and Pearson's correlation ($p<0.05$). BF generally performed as well as the conventional composite resin materials (ZT and ZF) but N100 and B2 did not. PF had the largest staining and translucency changes. Coffee, red wine, and tea resulted in the most staining and negative translucency changes. An inverse correlation between ΔE and ΔTP was observed for all materials and beverages with the exception of orange juice.

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INTRODUCTION

Patients desire long-lasting restorations that are functional and esthetically pleasing.[1] Influences

from the mass media[2] and changes in social economics have increased the esthetic demands of patients.[3] Coupled with recent efforts to phase out dental amalgam, there is a pressing need for tooth-colored materials that possesses comparable mechanical properties.

Resin-based composites and glass ionomer cements form the cornerstone of the spectrum of tooth-colored restoratives. The development of these materials has focused on modifying their biphasic compositions. Hybrid materials, with the principal base material incorporating elements and strengths from the other group, have also been developed. Today, tooth-colored restorative materials are routinely used to restore teeth. With adhesive technology and use of micromechanical retention, tooth-colored restorations are not only more conservative but also have less microleakage than amalgam restorations.[4]

Tooth-colored restorations, however, have their shortcomings. These include surface degradation (resulting in roughness), technique-sensitive bonding, fracture, and susceptibility to staining. [5–8] Studies have compared and reported on the susceptibility to staining of tooth-colored restorations in different food-simulating liquids or beverages through color or translucency changes.[9–13] Tian & others state that "most materials are susceptible to staining by 'dark' beverages while distilled water causes no perceptible color change."[10] These dark beverages have constantly shown to be coffee and red wine.[9–13]

Tooth-colored materials discolor via three mechanisms: 1) intrinsic discoloration, caused by the material itself aging; 2) extrinsic discoloration, caused by the accumulation of plaque and surface staining from diet; and 3) surface degradation, with staining agents reacting with the material (absorption).[1,15] The susceptibility to staining of tooth-colored restorations has been attributed to both internal and external factors. Factors include the material itself, the environment, the patient, and the clinician. The composition of the matrix, amount of filler loading and filler size, along with the biphasic nature of resin-based composites, are important intrinsic components to consider. Externally, the type of staining agent, the duration of exposure, and its compatibility with the matrix of the material are factors influencing the susceptibility to staining. The patient's diet and oral hygiene habits, along with the clinician's manipulation of the material, are no doubt important as well.[16]

New hybrid tooth-colored restorative materials that combine resin-based composite and glass ionomer technology are constantly being developed. They include nano-ionomers (Ketac N100) and pre-reacted glass ionomer filled composites (Giomer). Ketac N100 (N100) contains nanofillers and clusters of nano-sized zirconia/silica that result in a highly packed filler composition. Bala & others[17] and Coutinho & others[18] examined the surface roughness after polish and the bonding effectiveness to tooth structure of Ketac N100, respectively. The studies concluded that N100 had the smoothest surface after polishing compared with other glass ionomer cements. The bond strength to tooth structure of N100 was comparable to conventional glass ionomer cement (Fuji IX GP) but lower than resin-modified glass ionomer cement (Fuji II LC).[18] The fluoride release profile was deemed comparable to other resin-modified glass ionomer cements.[19]

Giomers are novel resin-based composites developed from surface-modified pre-reacted glass ionomer cement (SMPRG) technology. Giomers have methacrylate-based resin matrixes similar to resin-based composites, with SMRPG fillers instead of traditional quartz and glass fillers. Studies have shown that Giomers have better polishability and higher flexural strength and fluoride release compared with regular glass ionomer cements.[20–22] Compared with resin-based composites, Giomers were shown to have similar mechanical properties in terms of compressive strength, flexural strength, fracture toughness, micro-hardness, and polymerization shrinkage.[23]

Studies have been conducted to examine the staining susceptibility of Giomers in food- simulating liquids and limited beverages,[10] but there have been limited studies examining staining susceptibility of nano-ionomers and injectable hybrid Giomers and their performance in comparison to other materials.

This study investigated the color and translucency changes of these new restorative materials. Standard and flowable materials' susceptibilities to staining and translucency changes were compared. The staining ability and translucency change caused by the different beverages were also compared, and possible correlations between color and translucency change were investigated.

METHODS AND MATERIALS

Six commercial tooth-colored restorative materials were selected for this experiment: two resin-based composites, 3M Filtek Z350XT (ZT) and 3M Filtek

Table 1: *Technical Profiles and Manufacturers of the Materials Evaluated*

Material/Shade/ Lot number	Category	Composition		Mean filler size (μm)	Manufacturer
Filtek™ Z350XT/ A2/ N454576	Nanocomposite	Silane Treated Ceramic Silane Treated Silica UDMA BISEMA6	BISGMA Silane Treated Zirconia Polyethylene Glycol Dimethacrylate TEGDMA 2,6-Di-Tert-Butyl-P-Cresol	0.6 to 1.4	3M™ ESPE™ St Paul, MN, USA
Filtek™ Z350XT Flowable/A2/ N452481	Nanocomposite	Silane Treated Ceramic BISGMA TEGDMA Silane Treated Silica	Silane Treated Zirconium Oxide BISEMA6 Functionalized Dimethacrylate Polymer	0.6 to 1.4	3M™ ESPE™ St Paul, MN, USA
Beautifil Flow Plus/ A2/121240	Giomer	BISGMA TEGDMA	Aluminofluoro-borosilicate glass $Al_2 O_3$ DL-Camphorquinone	0.01–4.0	Shofu Dental Corporation, Osaka, Japan
Beautifil/A2/111268	Giomer	BISGMA TEGDMA	Aluminofluoro-borosilicate glass $Al_2 O_3$, DL-Camphorquinone	0.01–4.0	Shofu Dental Corporation, Osaka, Japan
Ketac™ Nano/A2/ N432469	Nanofilled RMGIC	Paste A: Silane Treated Glass Silane Treated Zirconia PEGDMA Silane Treated Silica HEMA Glass Powder BISGMA TEGDMA	Paste B: Silane Treated Ceramic Copolymer Of Acrylic And Itaconic Acids Water HEMA	0.0001(nanoparticle)-0.1(nanoclusters)	3M™ ESPE™ St Paul, MN, USA
Photac™ Fil/A2/ 501018	RMGIC	Powder: Silane Treated Glass Powder N,N-Dimethylbenzocaine	Liquid: 2-Hydroxyethyl Methacrylate Copolymer Of Acrylic Acid-Maleic Acid Water Mono- and Di- HEMA Phosphate, Magnesium Salt Diurethane Dimethacrylate	7–40	3M™ ESPE™ St Paul, MN, USA

BISEMA6; bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA, bisphenylglycidyl dimethacrylate; BISGMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; RMGIC, resin-modified glass ionomer cement; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Z350XT Flowable Restorative (ZF); two Giomers, Shofu Beautifil II (B2) and Beautifil Flow Plus (BF); and two glass ionomer cements, 3M Ketac Nano (N100) and 3M Photac Fil (PF). The technical profiles of the materials are described in Table 1.

The materials were activated and mixed according to the manufacturer's instructions, where applicable. They were packed into standardized circular molds measuring 9 mm in diameter and 1 mm in thickness. The molds and materials were then covered with Mylar strips on the top and bottom and placed between two microscope slides. Finger pressure was then applied to extrude excess material and eliminate porosities. The tooth-colored materials were light-cured (COXO LED curing light, Foshan Coxo Medical Instruments Co. Ltd, Guangdong Province, China; wavelength, 440-480 nm; intensity, 1200 mW/cm^2) according to manufacturer's instructions, checked for uniform thickness with a vernier caliper, and left to fully set for 24 hours in an incubator at 100% relative humidity and a temperature of 37°C. Forty-two specimens were fabricated for each material giving a total of 250 samples. Finishing was not done to minimize potential variables.

The samples were then placed on a neutral white background with chroma of <4 Munsell units, under D55 lighting conditions with intensity between 18 and 28 lux. Baseline L*, a*, b* values were determined using a spectrophotometer (Konica Minolta CM-2600d, Tokyo, Japan) placed at the center of the sample. This was then repeated with the samples placed on a black background.

For each material, the samples were randomly assigned to seven different liquids, namely cola drink, orange juice, red wine, vodka, black coffee, green tea, and distilled water, in groups of six. The samples were immersed in the beverages and incubated at standardized conditions of 37°C and 100% humidity for seven days with the container cover fastened to prevent evaporation that could lead to changes in volumes of the beverages. Beverages were changed daily at a standardized time.

After seven days, color readings were taken using the spectrophotometer. Color change was determined with the formula: $\Delta E = [(L^*_a - L^*_b)^2 + (a^*_a - a^*_b)^2 + (b^*_a - b^*_b)^2]^{1/2}$ with the set of L*, a*, b* values taken with respect to the white background. The subscript "a" referred to readings after soaking in the beverage and the subscript "b" referred to baseline readings. The translucency parameter was determined by calculating the difference in L*, a*, b* values measured over the white and black backgrounds: $TP = [(L^*_W - L^*_B)^2 + (a^*_W - a^*_B)^2 + (b^*_W - b^*_B)^2]^{1/2}$, where subscript "W" referred to the color coordinates over the white backing and subscript "B" to those over the black backing.[24] The difference in TP (ΔTP) was then calculated by: $\Delta TP = TP_a - TP_b$.

Statistical testing was performed using SPSS Version 20 (IBM SPSS Statistics for Windows, Version 20.0, Released 2011, IBM Corp. Armonk, NY). *t*-tests; one-way analysis of variance with Tukey post hoc test and Pearson's correlation were done at a significance of $\alpha=0.05$.

RESULTS

Table 2 shows the mean values and standard deviation of the results for ΔE and ΔTP. A $\Delta E \geq 3.3$ was used to stipulate a clinically observable color change based on the work of Ruyter et al.[25] and significant changes in translucency were also noted. Results of statistical analyses of color and translucency changes are reflected in Tables 3/4 and 5/6, respectively. Tables 7 and 8 show the results of Pearson's correlation tests examining the correlations between changes in translucency and color change with specific beverages and materials.

Susceptibility to Staining

Color change (ΔE) depended on both material and beverage and ranged from 0.40 ± 0.15 (B2 in water) to 18.73 ± 1.77 (N100 in coffee). In general, immersion in coffee produced the greatest change in color and immersion in water produced the least.

Clinically perceivable color change was anticipated (ΔE values ≥3.3) when PF was exposed to all seven beverages. For ZT, ZF, and BF, ΔE values were only ≥3.3 when exposed to three beverages (Table 2). For all materials evaluated, exposure to red wine and coffee is expected to result in clinically visible color changes as ΔE values were ≥3.3.

Changes in Translucency

Change in translucency (ΔTP) was material and beverage dependent and ranged from −7.39 ± 1.51 (PF in tea) to 0.88 ± 0.43 (ZF in water). A negative result suggests that the material became more opaque after immersion whereas a positive result suggests increased translucency. In general, coffee produced the greatest change in translucency, causing materials to become more opaque, and water produced the least change in translucency.

Translucency of the materials was affected by the different beverages. A significant increase in opacity was observed when PF was immersed in all seven beverages. In contrast, the translucency of ZF was affected by only two beverages (ie, coffee and water). For all materials evaluated, exposure to coffee resulted in a decrease in translucency. Conditioning in water and vodka resulted in increased translucency for ZF and N100, respectively.

Correlation Between ΔE and ΔTP

There was an inverse correlation between ΔE and ΔTP for all materials and beverages, except for orange juice. The strength of association was found to be moderate to strong.

DISCUSSION

In this study, the change in color and translucency of tooth-colored restorative materials after exposure to beverages for seven days was investigated. The beverages were chosen to represent the spectrum of beverages commonly consumed and tested.[9–12] Distilled water was chosen as it was previously shown to have produced no perceptible change in glass ionomer and resin-based composites. Most of the materials investigated contained nanoparticles.

A Mylar finish was selected as it gave the smoothest finish, eliminated the need for different polishing techniques applied clinically, and minimized operator variability. The samples were soaked for seven days in different beverages as tooth-colored restorative materials were shown to take up significant staining within the first week in a previous study, and any changes beyond this were not

Table 2: *Results of Statistical Analysis*

Material	Beverage	Mean Values (SD) of Materials in Respective Beverage ΔL* (SD)	Δa* (SD)	Δb* (SD)	ΔE (SD)	Clinically Visible Change	ΔT (SD)[a]
ZT	Coke	0.41 (0.96)	–0.05 (0.18)	0.27 (0.33)	0.92 (0.58)	No	–0.54 (1.60)
	Orange juice	0.89 (1.32)	–0.54 (0.40)	2.63 (0.41)	3.09 (0.43)	Yes	0.69 (0.82)
	Red wine	–9.19 (1.28)	2.15 (0.40)	8.79 (0.72)	12.96 (0.63)	Yes	–3.46 (0.91)O
	Vodka	1.57 (1.42)	0.53 (0.17)	0.96 (0.63)	2.29 (0.73)	No	0.55 (0.55)
	Coffee	–10.21 (1.97)	2.54 (0.55)	8.82 (0.29)	13.77 (1.66)	Yes	–3.02 (1.00)O
	Tea	–1.19 (0.55)	–0.31 (0.07)	2.71 (0.22)	3.02 (0.16)	No	–0.83 (0.29)O
	Water	–0.05 (1.07)	0.17 (0.18)	0.08 (0.40)	0.92 (0.62)	No	0.02 (0.26)
ZF	Coke	–0.19 (0.33)	0.28 (0.25)	0.35 (0.41)	0.71 (0.17)	No	0.09 (0.35)
	Orange juice	0.42 (2.19)	−0.27 (0.11)	2.19 (0.32)	2.87 (1.06)	Yes	0.37 (0.45)
	Red wine	0.69 (1.94)	−0.09 (0.19)	5.27 (0.51)	5.59 (0.59)	Yes	0.46 (0.51)
	Vodka	0.66 (1.58)	0.57 (0.10)	0.74 (0.47)	1.69 (0.93)	No	0.66 (0.68)
	Coffee	–9.59 (3.44)	2.14 (0.44)	5.43 (0.88)	10.24 (2.08)	Yes	–2.76 (0.82)O
	Tea	–0.24 (0.50)	0.05 (0.17)	1.74 (0.66)	1.85 (0.58)	No	–0.58 (1.37)
	Water	1.29 (1.12)	0.53 (0.09)	0.92 (0.49)	1.74 (1.10)	No	0.88 (0.43)T
BF	Coke	0.56 (0.97)	0.51 (0.54)	–0.03 (0.80)	1.32 (0.68)	No	–0.44 (1.05)
	Orange juice	–0.40 (0.59)	–0.40 (0.24)	1.71 (0.14)	1.69 (0.26)	No	0.20 (0.37)
	Red wine	–4.02 (2.52)	–0.13 (0.35)	13.23 (1.53)	14.07 (0.87)	Yes	–0.29 (1.87)
	Vodka	–0.87 (1.42)	–0.39 (0.81)	–0.15 (1.65)	2.23 (0.70)	No	–0.25 (1.97)
	Coffee	–11.55 (1.14)	1.71 (0.78)	7.42 (1.75)	13.98 (0.8)	Yes	–2.70 (0.84)O
	Tea	–2.91 (0.40)	–1.15 (0.37)	0.88 (1.00)	3.41 (0.18)	Yes	–1.37 (0.61)O
	Water	0.34 (1.39)	–0.36 (0.33)	–0.68 (0.42)	1.54 (0.49)	No	–0.49 (1.49)
B2	Coke	–1.58 (1.42)	–0.15 (0.34)	–3.19 (1.43)	3.85 (1.29)	Yes	–3.33 (1.05)O
	Orange juice	4.12 (0.34)	–0.65 (0.22)	3.72 (0.41)	5.60 (0.37)	Yes	0.62 (0.67)
	Red wine	–7.94 (2.16)	–0.39 (0.49)	15.28 (0.97)	17.33 (1.30)	Yes	–2.67 (0.90)O
	Vodka	0.18 (0.81)	0.27 (0.32)	–0.63 (0.77)	1.22 (0.43)	No	–0.24 (0.58)
	Coffee	–13.53 (2.09)	1.69 (0.60)	5.72 (2.60)	15.02 (1.75)	Yes	–5.37 (1.51)O
	Tea	–4.17 (0.52)	–1.52 (0.11)	1.09 (0.57)	4.61 (0.36)	Yes	–2.59 (0.49)O
	Water	–0.27 (0.23)	0.20 (0.11)	–0.04 (0.13)	0.40 (0.15)	No	0.38 (0.94)
N100	Coke	0.63 (0.56)	0.16 (0.24)	–0.65 (0.75)	1.24 (0.34)	No	–1.12 (0.74)O
	Orange juice	2.15 (1.14)	–0.43 (0.26)	1.20 (1.15)	2.74 (1.08)	Yes	–0.50 (1.13)
	Red wine	–3.11 (1.22)	–0.75 (0.48)	0.19 (1.20)	3.57 (0.38)	Yes	–1.69 (0.34)O
	Vodka	–0.20 (0.57)	–0.01 (0.08)	0.03 (0.23)	0.56 (0.23)	No	0.81 (0.12) T
	Coffee	–16.51 (1.97)	2.88 (1.34)	8.05 (1.94)	18.73 (1.77)	Yes	–4.57 (1.04)O
	Tea	–4.79 (1.04)	–1.38 (0.34)	–3.13 (0.52)	5.91 (1.03)	Yes	–2.41 (0.58)O
	Water	–0.21 (0.75)	–0.41 (0.19)	–1.17 (0.42)	1.48 (0.22)	No	0.41 (0.61)
PF	Coke	–2.14 (1.94)	1.70 (1.18)	1.39 (5.75)	6.37 (0.84)	Yes	–6.35 (0.99)O
	Orange juice	2.64 (2.31)	–0.80 (0.34)	–2.73 (3.04)	4.06 (3.59)	Yes	–3.71 (1.93)O
	Red wine	–1.13 (1.17)	7.86 (1.46)	–6.72 (2.80)	14.01 (1.97)	Yes	–7.31 (1.20)O
	Vodka	2.25 (1.36)	0.16 (0.06)	–2.45 (0.77)	3.54 (0.82)	Yes	–5.28 (2.12)O
	Coffee	–14.76 (2.44)	2.60 (0.61)	5.07 (1.00)	15.88 (2.25)	Yes	–5.81 (1.72)O
	Tea	–11.07 (2.12)	–2.55 (0.22)	–3.32 (0.54)	11.86 (2.04)	Yes	–7.39 (1.51)O
	Water	1.82 (0.53)	0.08 (0.17)	–2.45 (0.84)	3.13 (0.66)	Yes	–3.62 (1.36)O

B2, Shofu Beautifil II; BF, Beautifil Flow Plus; PF, 3M Photac Fil; N100, 3M Ketac Nano; ZF, 3M Filtek Z350XT Flowable Restorative; ZT, 3M Filtek Z350XT.
[a] *Superscript O: Significant change, more opaque; T: Significant change, more translucent.*

Table 3: *Comparisons of Color Change Induced by Different Beverages for Each Material*

Materials	Beverages
ZT	Coffee, Red wine > Tea, Orange juice, Vodka > Coke, Water
ZF	Coffee > Red wine > Orange juice, Tea, Water, Vodka > Coke;
	Orange juice > Tea, Water, Vodka, Coke
BF	Red wine, Coffee > Tea > Vodka, Orange juice, Water, Coke
B2	Red wine > Coffee > Tea, Orange juice, Coke > Vodka, Water
N100	Coffee > Tea > Red wine, Orange juice > Water, Coke, Vodka;
	Red Wine > Orange juice, Water, Coke > Vodka
PF	Coffee > Red wine, Tea > Coke, Orange juice, Vodka, Water
	Coffee, Red wine > Tea

B2, Shofu Beautifil II; BF, Beautifil Flow Plus; PF, 3M Photac Fil; N100, 3M Ketac Nano; ZF, 3M Filtek Z350XT Flowable Restorative; ZT, 3M Filtek Z350XT.

Table 4: *Comparisons of Color Change Between Different Materials When Immersed in the Same Beverage*

Beverage	Materials
Coke	PF > B2 > BF, N100, ZT, ZF
Orange juice	B2 > PF, ZT, ZF, N100, BF;
	B2, PF, ZT, ZF > N100
Red wine	B2 > PF, BF, ZT > ZF, N100
Vodka	PF > ZT, BF, ZF, B2 > N100;
	BF > ZF, B2, N100
Coffee	N100 > PF, B2, BF, ZT > ZF;
	N100, PF > B2
Tea	PF > N100, B2 > BF, ZT, ZF
	N100 > B2, BF, ZT > ZF
Water	PF > ZF, BF, N100, ZT > B2
	N100 > ZT, B2

B2, Shofu Beautifil II; BF, Beautifil Flow Plus; PF, 3M Photac Fil; N100, 3M Ketac Nano; ZF, 3M Filtek Z350XT Flowable Restorative; ZT, 3M Filtek Z350XT.

significant.[26] An A2 shade was selected as it was often used in similar studies. [9,11,12,24] A lighter shade is also more imperative as staining from beverages will generally be more visible clinically when compared with darker shades.

Color

The CIELAB colorimetric system was used in the study to evaluate color differences. In a study by Seghi & others, the authors concluded that it is "a valuable tool for material selection and restoration design, particularly in the area of aesthetic restorative dentistry." [27] There are 3 parameters to consider in the CIELAB system: 1) The L* coordinate is related to the lightness of the material; 2) the a* coordinate is related to red (more positive) and green (more negative); and 3) the b* coordinate is related to yellow (more positive) and blue (more negative).[27]

Conventional resin-based composite materials such as ZT and ZF generally fared better than novel materials such as B2 and N100 (Table 3-2). The conventional resin-modified glass ionomer cement, PF, had the greatest susceptibility to staining, corroborating a previous study.[9] No significant trend was noted for the least affected material.

An exception to the aforementioned trends was BF, which exhibited staining properties similar to that of ZF and ZT. It performed better than the other novel materials, B2 and N100, both of which demonstrated greater color changes when immersed in darker beverages. This suggested that BF had a higher resistance to staining compared with the other novel materials and might be a potential alternative to conventional materials. When comparing packable and flowable materials, ZT and ZF did not exhibit any significant differences, whereas BF performed better than B2. This was in agreement with existing literature[10] and may be attributed to the lower filler content in BF. The lower filler content in BF resulted in reduced surface roughness following erosion as fewer fillers are exposed, allowing the material to resist staining. Another possible reason could be the higher TEGDMA (triethylene glycol dimethacrylate) content in BF, which promoted greater conversion of the resin matrix,[20] decreasing its water sorption and hence susceptibility to staining.

N100, a nano-filled resin-modified glass ionomer cement, performed better than its conventional counterpart PF. This might be because N100 contains a smaller percentage of HEMA (2-hydroxyethyl methacrylate) by weight. HEMA increases water sorption and, hence, the potential for staining.[28] Another reason could be related to the maturity and the water sorption potential of the glass ionomer cement. Despite leaving the cement to set for a day, it might not have matured fully, resulting in significant water sorption and staining.[28] A third reason could be the smaller filler particle size in N100. This could have resulted in an even wearing of the surface, resulting in a smaller increase in surface roughness and staining susceptibility.

Table 5:
Comparison of translucency change induced by different beverages for each material

Materials	Beverages (Opaque → Translucent)	
ZT	Red Wine > Coffee > Tea, Coke, Red Wine, Coffee, Tea > Coke	Water, Vodka, Orange juice
ZF	Coffee > Tea, Tea >	Coke, Orange juice, Red wine, Vodka > Water; Coke, Orange juice, Red wine, Vodka, Water
BF	Coffee > Tea, Water, Coke, Red wine, Vodka, Coffee, Tea, Water, Coke > Red wine	Orange juice
B2	Coffee > Coke, Red wine, Tea > Vodka,	Water, Orange Juice
N100	Coffee > Tea, Red wine > Coke > Orange juice, Tea > Red wine, Coke, Orange juice >	Water , Vodka Water
PF	Tea, Red wine > coke, Black Coffee, Vodka, Orange, Water	

When examining color change in terms of the individual L* a* b* components, it was noted that ΔL* (decrease) and Δb* (increase) were often the affected values, and there were smaller changes in Δa*. This suggested that beverages often resulted in the materials becoming darker and yellower. The trend was consistent for all materials except PF, which exhibited large changes in all 3 components, and no clear trends were observed in the increase or decrease of color parameters. This might be attributed to its susceptibility to water sorption,[29] leading to greater surface adsorption and absorption of colorants.

Comparing the effects of different beverages, it was observed that coffee generally produced the worst stains, followed by red wine and tea. These beverages exhibited negative ΔL* and positive Δb* values, indicating that the materials became darker and yellower respectively. These three beverages had varied effects on Δa*. This might be due to the variation in colorants present in the three beverages. Although previous studies suggested that tea produced more staining than red wine, this study had a different result. This might be due to the use of green tea, which had a lower amount of colorants compared with black tea. Otherwise the results corroborated with prior literature.[9–12]

Vodka and water are colorless beverages and were expected to produce the least amount of color change based on previous studies on water.[12] Vodka with an alcohol content of 40% might produce color changes by degrading the resin matrix of the materials.[30] Results pertaining to cola drink, which produced minimal staining in comparison with other dark beverages, were consistent with a study done by Tian & others in 2012.[10]

The acids in coffee, red wine, and tea might have influenced their staining ability. For glass ionomer restorative materials, acid attack from the beverages on the glass ionomer matrix released metal cations, extracting more metal cations from the glass particles, which eventually dissolved. This resulted in increased surface roughness and, over time, caused food pigments to be trapped on the surface of the material. [9] Although acidity is measured as pH, the total amount of acid present (titratable acidity) may be a better gauge. This is determined by titration against a standardized sodium hydroxide solution.[10] Correlation between pH and titratable acidity is not apparent, meaning that a beverage can have both a high pH and titratable acidity. Although

Table 6:
Comparisons of translucency change of different materials when immersed in the same beverage

Beverages	Material (Opaque → Translucent)	
Coke	PF > B2 > N100, ZT, BF,	ZF
Orange juice	PF > N100,	BF, ZF, B2, ZT
Red wine	PF > ZT, B2, N100 > BF, B2 > N100, BF >	ZF ZF
Vodka	PF > BF, B2,	ZT, ZF, N100
Coffee	PF > B2 > N100, ZT, ZF, BF PF, B2, N100 > ZT	
Tea	PF > B2 > N100, BF, ZT > ZF PF > B2, N100, BF > ZT N100 > BF, ZT, ZF	
Water	PF > BF,	ZT, B2, N100, ZF

Table 7: *Correlation Between Color Change and Change in Translucency for a Specific Beverage*

Beverage	Correlation (P Value)	Strength
Coke	Yes ($p<0.001$)	–0.87 (strong)
Orange juice	No ($p=0.081$)	–0.27
Red wine	Yes ($p=0.002$)	–0.46 (moderate)
Vodka	Yes ($p=0.001$)	–0.50 (moderate)
Coffee	Yes ($p<0.001$)	–0.79 (strong)
Tea	Yes ($p<0.001$)	–0.84 (strong)
Water	Yes ($p=0.001$)	–0.51 (moderate)

Table 8: *Correlation Between Color Change and Change in Translucency for a Specific Material*

Material	Correlation (*p* Value)	Strength
ZT	Yes ($p<0.001$)	−0.80 (strong)
ZF	Yes ($p<0.001$)	−0.64 (moderate)
BF	Yes ($p=0.045$)	−0.31 (moderate)
B2	Yes ($p<0.001$)	−0.61 (moderate)
N100	Yes ($p<0.001$)	−0.83 (strong)
PF	Yes ($p<0.001$)	−0.56 (moderate)

B2, Shofu Beautifil II; BF, Beautifil Flow Plus; PF, 3M Photac Fil; N100, 3M Ketac Nano; ZF, 3M Filtek Z350XT Flowable Restorative; ZT, 3M Filtek Z350XT.

red wine exhibited lower pH, coffee and tea had a greater number of acids present in their composition and may have exhibited a larger total acidity. This might have increased the chemical erosion of material surfaces and worsened staining.

The presence of visible color change was not always accompanied by a ΔE of large magnitude. The interplay between the intensity of the color and how readily the colorants are taken up could be the reason for this observation. Coffee and red wine, which are dark beverages, exhibited both visible color change and a large ΔE.[9–12] In the case of orange juice, although it produced clinically significant changes ($\Delta E \geq 3.3$) in five materials, its magnitude of change was comparably lower than that of coffee, red wine, and tea. Its colorants might have been readily taken up to produce color change, but the colorants were comparatively lighter in color than the darker colorants of red wine, coffee, and tea. Although tea's colorants were also readily taken up, producing changes in four materials, the colorants were not as intense compared with coffee and red wine, leading to a smaller ΔE.

Translucency

Translucency of tooth-colored restorations depends on the passage of light through the material. It can be altered by changes on the external surface or body of the material. Translucency was quantified with the translucency parameter, which is the color difference when a specimen of uniform thickness is placed over a white and a black background and corresponds directly to common visual assessments of translucency.[24] Currently, no thresholds have been determined when considering visible clinical changes in translucency parameter.

Comparing between materials, PF was the most severely affected and had the highest ΔTP among all materials. The least affected in general was ZF. The flowable materials performed better, exhibiting a smaller increase in opacity compared with the packable materials.

An explanation for the observed trends could be that the changes in surface roughness after being exposed to the various beverages resulted in differential scattering of light and thus difference in opacity. This was probably especially true for PF because of its larger filler particle size (Table 1). Surface profilometry to compare the changes in surface roughness is needed to confirm this hypothesis.

Another potential cause for the change in opacity in resin-based composites and Giomers was the weakening of the resin/filler bond, resulting in migration of colored pigments into the resin material.[30,31] Filler particles and resin had different refractive indexes; thus visible light passing through the composite was scattered differently by the fillers and pigments, decreasing translucency.

Flowable materials such as ZF had fewer filler particles and, as such, would be least likely to be affected by changes to the resin/filler interface and subsequent incorporation of color pigments. This resulted in lower ΔE of ZF, compared with ZT, for all beverages.

Water and vodka caused minute increases in translucency in ZF and N100, respectively. Selective removal of larger filler particles and nanoclusters from ZF and N100, respectively, from the surface could have occurred, leading to a lower refractive index on the surface. The decrease in the difference in refractive index between filler particles and the matrix resulted in less surface scattering of light, allowing greater light penetration into the bulk of the material, hence increasing translucency.

Color pigments in the beverages had different refractive indexes compared with the filler particles and resin. Hence, these pigments could scatter and

absorb light, resulting in an increase in opacity in the materials. Darker drinks such as coffee, tea, and red wine contained more color pigments compared with the lighter beverages, vodka, orange juice, and water. This accounts for the observed differences in the effects of different beverages on translucency of the materials.

Correlation

The correlation between susceptibility to staining and changes in translucency was explored. There was an inverse relationship of moderate to strong strength between ΔE and ΔTP for all materials and beverages ($p<0.05$) except for orange juice ($p>0.05$). Hence, when there was increased color change, there was increased opacity for all materials and beverages with the exception of orange juice.

The varying amount of absorption of color pigments from beverages due to water sorption or changes in resin/filler matrix resulted in varying intensity of staining. The absorbed pigments not only absorbed light, causing a change in color, but also scattered light, resulting in a change in opacity, giving rise to the correlation between ΔE and ΔTP. No correlation between ΔE and ΔTP was observed for orange juice. Despite a significant change in ΔE, the quantity of pigments deposited by orange juice may not be sufficient to cause changes in translucency. The actual mechanism is not known and warrants further investigation.

CONCLUSION

The study sought to investigate the effects of common beverages on the color and translucency of tooth-colored restorative materials. All materials were affected to varying degrees. PF fared the worst, followed by B2. The performance of the novel materials, in terms of both color and translucency changes, depended on their composition and which end of the spectrum of tooth-colored materials they were closer to. Materials closer to the resin-based composites end of the spectrum in terms of chemistry and structure fared better than those closer to the glass ionomer cement restoratives.

For most materials, coffee, followed by red wine and tea, was the most prominent staining beverage and produced the largest decreases in translucency as well. Clinically perceivable color change ($\Delta E \geq 3.3$) was observed in all materials for coffee and red wine. With the exception of orange juice, the change in color was inversely proportional to changes in the translucency, suggesting a negative correlation.

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Conflict of Interest

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

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Air Abrasion Before and/or After Zirconia Sintering: Surface Characterization, Flexural Strength, and Resin Cement Bond Strength

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Clinical Relevance

The air abrasion performed before and after zirconia sintering can provide stronger bond strength at the zirconia–resin cement interface as well as an increase in the short-term flexural strength.

SUMMARY

The purpose of this *in vitro* study was to evaluate the effect of air-abrasion/zirconia sintering order on the yttria partially stabilized tetragonal zirconia polycrystal (Y-TZP) surface characterization (roughness, morphology, and phase transformation), flexural strength (FS), and shear bond strength (SBS) to a resin cement. Y-TZP specimens were air abraded with 50-μm Al_2O_3 particles after (AS), before (BS), or before and after zirconia sintering (BAS). For roughness (Ra), 30 block specimens (12×12×3.0 mm; n=10) had their surfaces analyzed by a profilometer. Next, on the air-abraded surfaces of these specimens, composite resin discs (n=30) were bonded with RelyX ARC. The bonded specimens were stored for 24 hours in distilled water at 37°C before shear testing. Failure mode was determined

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with a stereomicroscope (20×). The surface morphology (n=2) was evaluated by SEM (500×). For the four-point flexural strength test (EMIC DL2000), 39 bar-shaped specimens (20×4.0×1.2 mm; n=13) were air abraded according to the three conditions proposed, and an additional group (nonabraded) was evaluated (n=13). The quantitative analysis of phase transformation (n=1) was completed with Rietveld refinement with X-ray diffraction data. Ra (μm) and SBS (MPa) data were analyzed by one-way analysis of variance (ANOVA) and the Tukey test (α=0.05). Pearson correlation analysis was used to determine if there was a correlation between roughness and SBS. For FS (MPa) data, one-way ANOVA and the Dunnett C-test (α=0.05) were used. The air-abrasion/zirconia sintering order influenced significantly ($p<0.001$) Ra, SBS, and FS. The BS and AS groups presented the highest (1.3 μm) and the lowest (0.7 μm) Ra. The highest SBS (7.0 MPa) was exhibited by the BAS group, followed by the AS group (5.4 MPa) and finally by the BS group (2.6 MPa). All groups presented 100% adhesive failure. A weak correlation ($r=-0.45$, $p<0.05$) was found between roughness and SBS. The air-abrasion/zirconia sintering order provided differences in the surface morphology. The nonabraded (926.8 MPa) and BS (816.3 MPa) groups exhibited statistically similar FS values but lower values than the AS (1249.1 MPa) and BAS (1181.4 MPa) groups, with no significant difference between them. The nonabraded, AS, BS, and BAS groups exhibited, respectively, percentages of monoclinic phase of 0.0 wt%, 12.2 wt%, 0.0 wt%, and 8.6 wt%. The rougher surface provided by the air-abrasion before zirconia sintering may have impaired the bonding with the resin cement. The morphological patterns were consistent with the surface roughness. Considering the short-term SBS and FS, the BAS group exhibited the best performance. Air abrasion, regardless of its performance order, provides tetragonal to monoclinic transformation, while sintering tends to zero the monoclinic phase content.

INTRODUCTION

Yttria partially stabilized tetragonal zirconia polycrystal (Y-TZP) has been widely used to manufacture metal-free fixed partial dentures or implant-supported prostheses due to its optical properties,[1] biocompatibility,[2] low thermal conductivity,[3] chemical stability,[4] as well as its high fracture toughness and mechanical performance when compared to the other dental ceramics.[5] In minimally retentive situations, the resin cements might be a good option[6] because of their improved mechanical properties when compared to zinc phosphate and glass ionomer cements[7] and also because of the possible chemical interactions between zirconia surface and the resin cement components (adhesive cementation).[8,9] With regard to micromechanical retention, which contributes significantly to improve the bonding between zirconia and resin cements,[10-13] although there are different methods to roughen zirconia surface, such as nanostructured alumina coating,[14] laser,[15,16] selective infiltration-etching (SIE),[17,18] and hot-etching solution,[18] among others, air abrasion with alumina (Al_2O_3) particles is still an effective and one of the most applicable methods.[10,15,16,18]

Air abrasion can be performed with Al_2O_3 particles of different sizes and is usually carried out after zirconia sintering and prior to cementation. However, since zirconia is a densely sintered material and consequently exhibits high hardness,[5] it is difficult to roughen its surface,[19] requiring higher air pressure and/or coarser Al_2O_3 particles capable of promoting a desirable surface roughness. On the other hand, if this procedure is severe, it may create surface flaws that can propagate into the bulk of the zirconia, compromising its mechanical properties.[20,21] Another way to solve this question would be performing air abrasion before the zirconia sintering, that is, when this material does not exhibit such high hardness. This simple modification may allow the use of smaller particles to provide a surface whose roughness and morphology are favorable to the adhesive bonding at the zirconia–cement interface without jeopardizing the mechanical strength of the zirconia. Monaco and others[22] observed that regardless of the particle size evaluated (30, 50, and 110 μm), the air abrasion performed before zirconia sintering provided higher roughness in comparison with that performed after sintering. However, Monaco and others[23] and Moon and others[24] reported no significant differences in the shear bond strength between the groups abraded before and after zirconia sintering. Besides the increase in zirconia roughness reported by Monaco and others,[22] another very important aspect observed by these authors, as well as by Moon and others,[24] was the decrease of the monoclinic phase when abrasion was performed before zirconia sintering.

Besides the few studies[22,24] that have investigated the effect of the air abrasion performed before and after zirconia sintering on its roughness and adhesive bonding, there is no consensus with respect to the influence of the air-abrasion/zirconia sintering order on roughness. Moreover, the association between the air abrasion performed before and after zirconia sintering is another viable option to be investigated. In addition, it would be important to evaluate the influence of the air-abrasion/zirconia sintering order not only on phase transformation but also on the mechanical strength of the zirconia.

Thus, the purpose of this *in vitro* study was to evaluate the effect of the air-abrasion/zirconia sintering order (air abrasion performed after, before, or before and after zirconia sintering) on the Y-TZP ceramic surface characterization (roughness, morphology, and phase transformation) and flexural strength (FS) and also its efficacy on the shear bond strength (SBS) at the zirconia–resin cement interface. The null hypothesis was that the air-abrasion/zirconia sintering order does not modify zirconia roughness, its flexural strength, or its bond strength with a resin cement.

METHODS AND MATERIALS

Preparation of Zirconia Specimens

Thirty block specimens (15×15×3.5 mm) were prepared for roughness analysis and SBS test, while 52 bar-shaped specimens (25×5.0×1.5 mm) were prepared for four-point flexural strength testing (ISO 6872).[25] The specimens were obtained by cutting presintered zirconia frames (Lava, 3M ESPE AG, Seefeld, Germany) with a sectioning machine (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA) using a diamond-coated disc saw (Diamond Wafering Blade, Series 15LC no. 11-4276, Buehler) under water irrigation. The specimens were washed in tap water to remove the cutting debris, and their ends were finished manually using a ceramic polisher (Exa Cerapol 0361HP, Edenta AG, Au, SG, Switzerland) in a low-speed hand piece.

The specimens were air abraded with 50-μm Al_2O_3 particles (Bio-Art Equip. Odontol. Ltda, São Carlos, SP, Brazil) after (AS), before (BS), or before and after zirconia sintering (BAS). For the four-point flexural strength test, 39 specimens were obtained according to the three air-abrasion conditions proposed, and an additional group (nonabraded) was included (n=13).

The sintering process was performed in a specific oven (Lava Furnace 200, Dekema Dental-Keramiköfen GmbH, Freilassing, Germany) according to the manufacturer's instructions (heating rate=20°C/min: 0°C-1000°C; 10°C/min: 1000°C-1500 °C; holding time=2 h and cooling rate=15°C/min: 1500°C-800°C; 20°C/min: 800°C-250°C; the oven was opened at 250°C). The dimensions of the specimens after sintering were 12×12×3.0 mm for roughness and SBS and 20×4.0×1.2 mm for flexural strength. For the air-abrasion procedure, the specimens were mounted on a holder (developed for each specimen shape) at a 90-degree angle and a distance of 10 mm from the tip of the air-abrasion unit (Basic Classic, Renfert GmbH, Hilzingen, Germany).[26,27] The specimens were air abraded for 20 seconds and 15 seconds at a pressure of 0.05 and 0.28 MPa for abrasion before and after sintering, respectively. The parameters (pressure and time) used for air abrasion before zirconia sintering were determined after some preliminary experiments. After sintering, all specimens were cleaned in 99% isopropanol using an ultrasonic cleaner for 10 minutes and left to dry at room temperature for 24 hours. Both analyses (surface roughness and SBS) were performed on the same specimens of each group.

Surface Roughness Measurements

The surface roughness of all specimens was determined after their sintering, using a profilometer (Surftest SJ-400, Mitutoyo Corporation, Kawasaki-shi, Japan) with a cutoff value (λ_c) of 0.8 mm.[16] A diamond stylus with a 5-μm tip radius at 0.5 mm/s and resolution of 0.01 mm examined a surface length of 4.0 mm. Three equidistant parallel measurements were made perpendicularly to the direction of the air abrasion with the stylus at a 90-degree angle on different areas of the specimen. The average reading was designated as the Ra (μm) value of each specimen evaluated. A single calibrated operator (intraclass correlation coefficient=0.89) recorded all measurements.

Bonding Procedure and SBS Test

Thirty composite resin discs (Z100, 3M ESPE, St Paul, MN, USA) were produced using a custom-made metal split matrix (4.0-mm internal diameter and 2.0-mm thickness) positioned between two glass slabs covered with transparent polyester films. The light curing (Radii-Cal light-curing unit, SDI Ltd, Bayswater, Australia) was performed for 40 seconds on the top surface and two diametrically opposed sides of the resin discs (total of 120 s) at a light intensity of 800 mW/cm^2. After the metal matrix was removed, the sides were light cured, taking care not to polymerize the bottom surface of the resin discs.

Table 1: *Scheme of XRD Measurements*

Nonabraded	AS	BS	BAS
Nonabraded/nonsintered	Nonabraded/sintered	Air abraded/nonsintered	Air abraded 1/nonsintered
First measurement	First measurement	First measurement	First measurement
Sintering process	Air-abrasion procedure	sintering process	Sintering process
Second measurement	Second measurement	Second measurement	Second measurement
—	—	—	Air-abrasion procedure (air abraded 2)
—	—	—	Third measurement

RelyX ARC resin cement (Bis-GMA, TEGDMA, silanated zirconia/silica filler, 3M ESPE) was proportioned by weight (0.010 g of each paste) and mixed for 10 seconds, and the composite resin discs were immediately bonded to the air-abraded zirconia surfaces. Next, a load of 1000 *g* was applied on top of the composite resin disc for five minutes.[28] After excess removal, the cement was light cured in two different positions (equidistant sides) for 40 seconds each.

The composite resin disc was inserted in a metal matrix (25-mm diameter) with a circular opening (4.2-mm diameter) with the zirconia block upward. Polyvinyl chloride tubes (20 mm in diameter and 20 mm high) were centrally positioned over the matrix and filled with polymethyl methacrylate (PMMA) autopolymerizing acrylic resin (Jet, Classico Odontological Goods Ltd, São Paulo, SP, Brazil), assembling the air-abraded zirconia surface to remain exactly at the same level of PMMA resin. All specimens were stored for 24 hours in distilled water at 37°C.

Each specimen was mounted on a metal holder in a mechanical testing machine (model DL2000, EMIC Equipment and Systems Testing Ltd, São José dos Pinhais, PR, Brazil), and a uniaxial compressive force was applied at the cement–zirconia interface by means of a knife-edged blade running at a crosshead speed of 0.5 mm/min until failure. SBS values were recorded in MPa.

Failure Analysis

Debonded specimens were examined under a stereomicroscope (model M80, Leica Microsystems Ltd, Heerbrugg, Switzerland) at 20× magnification by a single trained observer, and the failure mode was classified as adhesive (complete zirconia surface was visible), cohesive within the cement layer or within the composite resin (almost all of the fracture surface was covered with cement or with composite resin), or mixed (a combination of adhesive and cohesive), according to the dominant mode of failure in each quadrant of the zirconia surface.[29]

Surface Morphology Analysis

For the surface morphology analysis, two additional specimens from each experimental group were mounted on metallic stubs and analyzed under a field emission scanning electron microscope (model JSM-7500F, JEOL Ltd, Peabody, MA, USA), which operated at 500× magnification with an accelerating voltage of 2.0 kV.

Four-Point Flexural Strength Test

For the four-point flexural strength test (ISO Standard 6872)[25], the specimens were positioned over two 0.8-mm-radius rounded bearers with a span distance of 16 mm. Two rounded loading pistons (0.8-mm radius, distance of 8 mm) running at a crosshead speed of 1.0 mm/min applied a uniaxial compressive force to the nonabraded surface, while for the air-abraded groups, the treated surface was submitted to the tensile load until failure. The test was performed at room temperature in a mechanical testing machine (model DL2000, EMIC Equipment and Systems Testing). The flexural strength values (MPa) were calculated according to the equation recommended by the ISO standard.

X-ray Diffraction Analysis

The X-ray diffraction (XRD) analysis assessed the effect of the air-abrasion/zirconia sintering order on the phase transformation of zirconia. Table 1 presents the scheme of XRD measurements according to the experimental groups.

The XRD data (n=1) were collected using a RIGAKU RINT2000 rotating anode diffractometer (40 kV, 70 mA) with Cu kα radiation (λkα1=1.5405 Å, λkα2=1.5443 Å, Ikα1/Ikα2=0.5) monochromatized by a curved graphite crystal. An interval from 20° to 120° (2θ) with a step size of 0.02° (2θ), 4 seconds per step, divergence 0.5, and open receiving slits, were the

Table 2: *Mean (±SD) of Ra (μm), SBS (MPa), and FS (MPa) Values*[a]

	Ra	SBS	FS
non-abraded	-	-	926.8 ± 95.4[b]
AS	0.7 ± 0.1[c]	5.4 ± 0.6[b]	1249.1 ± 303.9[a]
BS	1.3 ± 0.1[a]	2.6 ± 0. 9[c]	816.3 ± 112.4[b]
BAS	1.0 ± 0.1[b]	7.0 ± 1.1[a]	1181.4 ± 262.7[a]

[a] *Different letters indicate significant differences in columns (p<0.05).*

selected conditions for Rietveld refinement.[30] The Rietveld refinements were performed using the General Structure Analysis System program[31] suite with EXPGUI interface.[32] The peak profile function was modeled using a convolution of the Thompson-Cox-Hastings pseudo-Voigt function (pV-TCH),[33] using the asymmetry function described by Finger and others,[34] which accounts for the asymmetry resulting from axial divergence. The bidimensional model for crystallite size described by Larson and Von Dreele[31] was used to account for the anisotropy in the half width of the reflections, and the model described by Stephens.[35] The following parameters were refined: atomic coordinates, occupancies, unit cell, scale factor, sample displacement, atomic displacement, and full width at half maximum. The crystal structure parameter used as basis of the Inorganic Crystal Structure Database code was 66781 (ZrO_2, tetragonal), 18190 (ZrO_2, monoclinic), and 53998 (ZrO_2, cubic).

Statistical Analysis

The Shapiro-Wilk test indicated that the normality assumption for all data was satisfied, while the homogeneity by Levene test proved to be violated ($p=0.001$) only for FS (MPa) data. Surface roughness (μm) and SBS (MPa) data were analyzed by one-way analysis of variance (ANOVA) followed by the Tukey honestly significant difference (HSD) *post hoc* test ($\alpha=0.05$) to determine differences among the means. In addition, to test for a possible correlation between roughness and SBS, a linear correlation r was calculated by Pearson correlation analysis. The analysis of FS (MPa) data was performed by one-way ANOVA and the Dunnett C-test ($\alpha=0.05$). Statistical analysis was performed using IBM SPSS Statistics (version 20, IBM Corp, Armonk, NY, USA).

RESULTS

According to the results of the one-way ANOVA, the air-abrasion/zirconia sintering order significantly influenced surface roughness ($F=70.1$, $p<0.001$), SBS ($F=65.4$, $p<0.001$), and FS ($F=12.0$, $p<0.001$). Table 2 shows the mean Ra (μm) and SBS (MPa) values, standard deviations, and statistical analysis results identified with the Tukey HSD test and the FS (MPa) mean values, standard deviations, and statistical results obtained by the Dunnett C-test. The BS group presented the highest Ra value (μm), while the AS group yielded the lowest. The highest SBS value was exhibited by the BAS group, followed by the AS group and finally by the BS group. The failure mode observed was 100% adhesive in all groups. A weak correlation ($r=-0.45$, $p<0.05$) was found between roughness and SBS. The nonabraded and BS groups exhibited statistically similar FS values but lower values than the AS and BAS groups, with no significant differences between them.

The representative SEM images (Figure 1) indicated that the groups abraded before zirconia sintering (BS and BAS groups) exhibited more prominent microretentive grooves when compared to the smoother surface of the AS group. The BAS group exhibited a surface texture similar to that presented by the BS group but with more rounded edges.

Table 3 lists the results of quantitative phase analysis, and Figure 2 presents the representative diffraction patterns of the experimental groups according to each step performed to obtain them. Air abrasion provided an increase in the monoclinic phase for the BS and BAS/air-abraded 1 groups and a "decomposition" of t-ZrO_2 and c-ZrO_2 phases in others (t-ZrO_2 and m-ZrO_2) for the AS and BAS/air-abraded 2 groups. The sintering process promoted the total incorporation of monoclinic phase into tetragonal and/or cubic phases.

DISCUSSION

The null hypothesis of the present study was rejected since the air-abrasion/zirconia sintering order influenced roughness, shear bond strength, and flexural strength. The air abrasion performed before zirconia sintering (BS group) provided the roughest surface, followed by the BAS and AS groups. The higher roughness provided by the air abrasion with 50-μm Al_2O_3 particles before sintering in comparison with that performed after sintering was also observed by

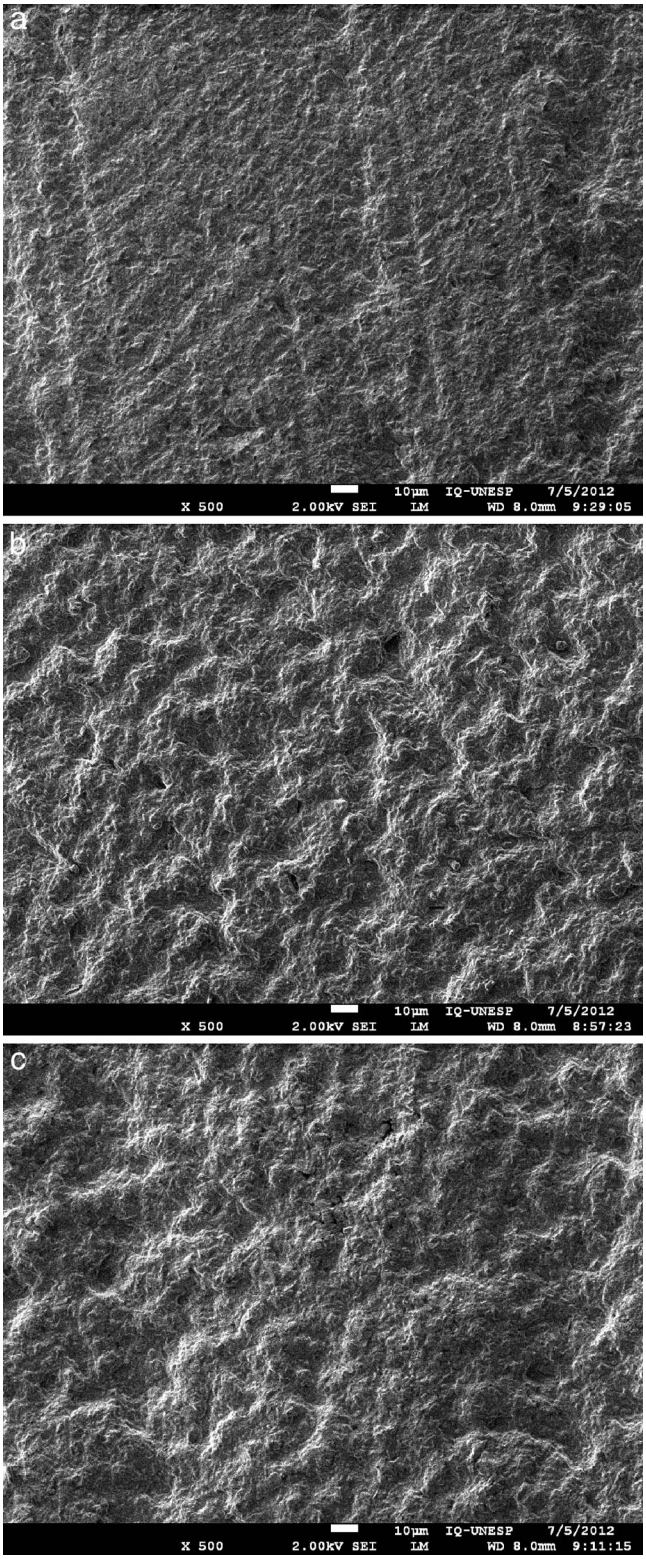

Figure 1. *SEM images (500×) of the experimental groups according to the air-abrasion/zirconia sintering order. (a): After zirconia sintering (AS). (b): Before zirconia sintering (BS). (c): Before and after zirconia sintering (BAS).*

Monaco and others[22] and may be due to the lower hardness of the zirconia in its green stage, which favors the imprint of its surface by the harder Al_2O_3 particles. Regarding the morphological pattern, which was in line with roughness, we observed that the BS group exhibited more prominent microretentive grooves when compared to the smoother surface of the AS group, as reported by Monaco and others.[22] The BAS group, which was not evaluated by these authors,[22] exhibited a surface texture similar to that presented by the BS group but with more rounded edges, probably resulting from the air abrasion performed after sintering. Considering that the effect of the air-abrasion/zirconia sintering order has been poorly investigated, no additional information was found to further discuss our results.

Regarding the shear bond strength, the lowest mean value observed for the BS group probably is related to its higher roughness accompanied by the prominent edges observed by SEM, which probably impaired the wettability of the zirconia by the resin cement, considering that in this study a bonding agent was not used. On the other hand, the lowest roughness and a "flatter" morphology exhibited by the AS group may have been unfavorable to the micromechanical retention at the zirconia–resin cement interface, explaining its intermediate SBS value. The highest SBS mean value of the BAS group probably resulted from its intermediate roughness and morphology, which allowed a higher wettability of the zirconia by the resin cement when compared to the BS group and a higher micromechanical retention when compared with the AS group. Contrary to our SBS results, some studies[23,24,26] reported statistical similarity between the groups abraded before (BS group) and after (AS group) zirconia sintering. The difference in behavior between those studies[23,24,26] and ours may be related to the use or nonuse of a bonding agent.

The weak correlation between roughness and bond strength observed in this study can be corroborated by the findings of Winkler and Moore.[36] These authors evaluated the correlation between these two properties, varying the direction of the roughness measurements, that is, parallel or perpendicular to the scratches, and they concluded that when the reading was parallel, a correlation was observed. On the other hand, when the reading was perpendicular, as performed in the present study, the correlation was significantly lower. Also, in the study by Subaşı and Inan,[16] no significant correlation was observed when the relationships between roughness and bond strength values were compared

Table 3: *Phase Content (wt%) of the Experimental Groups*

Phases		Nonabraded		AS		BS		BAS		
		Nonsintered	Sintered	Nonabraded	Air abraded	Air abraded	Sintered	Air abraded 1	Sintered	Air abraded 2
t-ZrO_2	wt%	85.5(1)	89.2(1)	89.2(1)	59.2(5)	83.7(7)	74.6(2)	83.5(7)	73.2(3)	51.0(4)
m-ZrO_2		14.4(1)	—	—	12.2(6)	16.3(3)	—	16.5(3)	—	8.6(2)
t-ZrO_2		—	—	—	28.6(1)	—	—	—	—	40.3(5)
c-ZrO_2		—	10.7(1)	10.7(1)	—	—	25.4(7)	—	26.8(8)	—

Abbreviations: t, tetragonal ; m, monoclinic ; c, cubic.

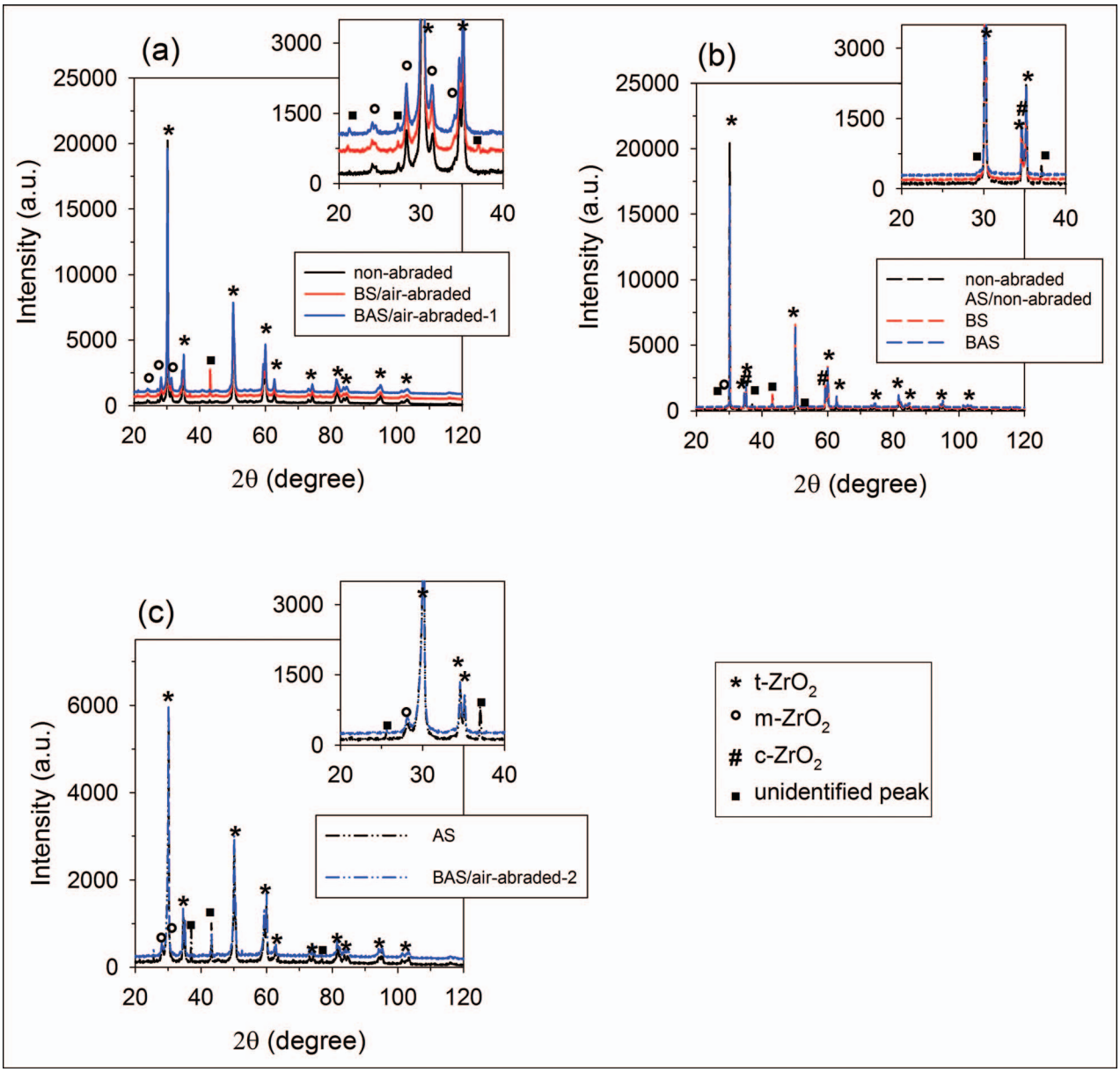

Figure 2. *Diffraction patterns of the experimental groups according to each step performed to obtain them. (a): Nonsintered (nonabraded; BS/air abraded; BAS/air abraded 1). (b): Sintered (nonabraded; AS/nonabraded; BS; BAS). (c): Abraded after sintering (AS; BAS/air abraded 2).*

for each surface treatment and resin cement. Similarly, in the study of Oyagüe and others,[28] although a correlation analysis was not performed, by observing the results of the zirconia roughness and microtensile bond strength, it seems that there is no correlation between these two variables.

Concerning flexural strength, the AS (1249.1 MPa) and BAS (1181.4 MPa) groups presented higher FS (with no significant difference between them) than the nonabraded (926.8 MPa) and BS (816.3 MPa) groups (with no significant difference between them). Although the BS group exhibited the lowest SBS value, it did not exhibit a decrease in the FS in comparison with the nonabraded group. It is possible that if a bonding agent (silane or adhesive monomers) had been applied after sintering, the wettability of the zirconia by the cement would be improved, resulting in higher SBS values.[11,15,37]

The XRD analysis performed after abrasion for the BS group revealed a monoclinic phase content of 16.3 wt%; however, after sintering, this percentage was zero. For this same condition, Moon and others[24] reported a monoclinic phase content of 16.9 wt% after abrasion, which dramatically decreased to almost zero after sintering. According to these authors,[24] this behavior may be explained by the fact that air abrasion itself induced tetragonal to monoclinic transformation, but a reverse transformation (monoclinic to tetragonal) occurred during the sintering process. Regarding the statistical FS superiority of the AS and BAS groups, it was probably due to the air-abrasion step. These two groups presented higher percentage values of monoclinic phase (AS=12.2 wt% and BAS=8.6 wt%) in comparison with the nonabraded (0.0 wt%) and BS (0.0 wt%) groups. Using 50-μm Al_2O_3 particles, Monaco and others[22] and Moon and others[24] observed 10.0 wt% and 11.4 wt% of monoclinic phase for the group abraded after sintering. It is known that air abrasion creates surface microcracks around which the grains exhibit a volumetric increase resulting from the tetragonal to monoclinic phase transformation. This outward expansion due to a plastic deformation of the surrounding zirconia provides compressive stresses that counteract the crack propagation.[38] This process, known as transformation toughening, may increase the bulk strength of zirconia,[6,21,27,39,40] as indicated by this study. Although this study did not evaluate the existence of a possible correlation between phase transformation (tetragonal to monoclinic) and flexural strength, it seems that there is some relation between them. Some studies[21,27,40] concluded that the increase in the mechanical performance of the zirconia seems to be related to the phase transformation (toughening mechanism), given that a higher amount of monoclinic ZrO_2 content resulted in higher flexural strength values.

Besides the lack of studies that compared the air abrasion routinely performed (AS group) with that performed before sintering (BS group), the novelty of the current research is that the combination of both was tested and yielded the best results with regard to the short-term shear bond strength and flexural strength. However, a concern that should be taken into account is the behavior of the nondesirable microcracks in the three air-abrasion conditions. In the BS group, microcracks are created by air abrasion, resulting in a phase transformation (tetragonal to monoclinic) that contains their propagation. After sintering, an inverse phase transformation occurred (monoclinic to tetragonal),[24] releasing the compressive stresses,[22,40] which is not so damaging given that the zirconia has a sintering shrinkage of about 20%-25%, which could promote a partial or total sealing of the cracks.[41] This fact may explain the similar behavior concerning the FS of the BS group in comparison with the nonabraded one. Although the BS group exhibited lower FS than the AS and BAS groups, in the long term it may behave more favorably under cyclic load and moisture. On the other hand, in the AS (the air abrasion routinely performed) and BAS groups, the microcracks created by the air abrasion after sintering were probably contained by the compressive stresses resulting from the phase transformation (tetragonal to monoclinic).[21,27,39,40] This fact may explain the higher FS of these groups in comparison with the BS one. However, we wonder whether the condition of the AS and BAS groups is maintained for a sufficiently long period of time under the adverse effects of the oral environment.

Regardless of choosing the BAS method, which revealed the highest SBS and FS values, or the BS one, which could be more interesting if we consider its supposed long-term mechanical performance, the air-abrasion step performed before zirconia sintering is clinically viable regarding the micromechanical retention. However, in this study, instead of zirconia frameworks, geometrical specimens were used and no attention was given to the potential damage that the air abrasion performed previous to zirconia sintering may cause mainly to the margins of the restorations. Another concern is when this procedure is performed with silica-coated Al_2O_3 particles, the chemical bond, which was not the focus of this study,

could be impaired by the surface contamination during the clinical/laboratory steps. Therefore, further research must be carried out investigating other aspects related to the subject of the current study and how these conditions resulting from the air-abrasion/zirconia sintering order behave in a long-term moisture environment, which favors the propagation of microcracks due to the low temperature degradation phenomenon, and under cyclic loading in order to simulate the adverse conditions of the oral cavity.

CONCLUSIONS

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

1. The rougher surface provided by the air abrasion before zirconia sintering may have impaired the bonding with the resin cement.
2. The morphological patterns resulting from the air-abrasion/zirconia sintering order were consistent with the surface roughness.
3. Considering the short-term adhesive bonding and flexural strength, the air abrasion before and after zirconia sintering, when used in combination, exhibited the best performance.
4. Air abrasion, regardless of the order in which it is performed, provides tetragonal to monoclinic transformation, while sintering tends to zero the monoclinic phase content.

Conflict of Interest

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

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Effects of the Concentration and Composition of In-office Bleaching Gels on Hydrogen Peroxide Penetration into the Pulp Chamber

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Clinical Relevance

The amount of hydrogen peroxide that reaches the pulp chamber of premolars after in-office bleaching depends on the bleaching protocol and the composition of the product.

SUMMARY

In tooth whitening, the hydrogen peroxide (HP) diffuses in the enamel and dentin, reaching the pulp. This *in vitro* study aimed to quantify the penetration of HP in the pulp chamber in teeth submitted to bleaching agents of different concentrations of HP without calcium (HP 20% [20CF], HP 35% [35CF]) and with calcium (HP 20% [20CC], HP 35% [35CC]).

Method: Fifty human premolars were sectioned 3 mm from the cemento-enamel junction and the pulp tissue was removed. The teeth were divided into five groups according to treatment and with a control group (n=10). An acetate buffer solution was placed in the pulp chamber of all teeth. The control group was exposed only to distilled water, while the other groups were treated with a bleaching procedure, according to the manufacturer's recommendations. After treatment, the acetate

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buffer solution was transferred to a glass tube in which leuco-crystal violet and peroxidase solutions were added, resulting in a blue solution. The optical density of this blue solution was determined spectrophotometrically and converted into micrograms equivalent to the HP. Data were analyzed using analysis of variance and Tukey tests (α=0.05).

Results: The HP concentration did not affect the HP inside the pulp chamber, but the presence of calcium significantly reduced it ($p<0.0001$).

Conclusion: The amount of HP that reaches the pulp chamber depends on the bleaching protocol and the product employed, and it seems to be less affected by HP concentration.

INTRODUCTION

Vital tooth whitening is a noninvasive treatment commonly used in dental practice to achieve a harmonic smile in terms of shade.[1] Among the techniques used for vital tooth bleaching, the in-office protocol offers quicker whitening results than the at-home procedure, with fewer applications.[1,2]

In both at-home and in-office bleaching techniques, hydrogen peroxide (HP) is the active molecule. It forms free radicals, reactive oxygen molecules, and HP anions,[3] which oxidize the organic dentin matrix,[4,5] leading to a whitening effect. Two clinical sessions of in-office bleaching allow a whitening effect of approximately five to eight classical shade guide units,[6,7] and the bleaching has been shown to be stable after periods ranging from nine to 24 months.[7,8]

However, the HP does not only whiten teeth. As a result of its low molecular weight, HP penetrates into the dental structure to the pulp chamber,[9-11] causing pulp reactions. This is reflected in minor histological changes,[12-14] including pulp degeneration at some sites.[15]

High concentrations of HP and its by-products exceed the antioxidant capacity of the pulp tissue, causing oxidative stress[16,17] and pulp inflammation,[18] which trigger the most prevalent bleaching-induced side effect, tooth sensitivity.[7,19]

Previous studies reported that the intensity of bleaching-induced tooth sensitivity varies from mild[20,21] to severe.[22] In some cases, the tooth sensitivity is so painful that it leads the patient to abandon the treatment.[23] In an attempt to reduce the bleaching-induced discomfort, some manufacturers have released in-office bleaching gels with lower HP concentrations. This was based on the assumption that the amount of HP that reaches the pulp is proportional to its original concentration in the bleaching agent.[11,24]

However, the results of a recent study[25] challenge the concept that tooth sensitivity is directly correlated to the initial HP concentration. This study compared the tooth sensitivity of two 35% HP gels. The alkaline calcium-containing gel presented lower absolute risk of tooth sensitivity than did the slightly acid calcium-free product.[25] The amount of HP that reaches the pulp chamber may be different as a result of the presence of other additives. To the extent of the authors' knowledge, this issue has not yet been investigated.

Therefore, the aim of the present study was to compare the amount of HP that reaches the pulp chamber using a calcium-free and a calcium-containing bleaching agent with different HP concentrations.

METHODS AND MATERIALS

We selected 50 extracted sound premolar teeth with only one root for this study. The roots of all teeth were cut approximately 3 mm apical to the cemento-enamel junction, and the pulp tissue was removed and washed with distilled water. The entrance to the pulp cavities was widened carefully with a round bur (#1014; KG Sorensen, Barueri, SP, Brazil) to allow the introduction of a micropipette (LABMATE Soft, HTL Lab Solutions, Warsaw, Poland) inside the pulp chamber.

With the aim of measuring the thickness of the dental structure on the buccal surface of premolars, X-ray radiographs (Timex 70C, Gnatus, Ribeirão Preto, SP, Brazil) were taken with an exposure time of 0.5 seconds and a 30-cm focus-object distance (70 kVp and 7 mA). The central X-ray beam focused at a 90° angle to the buccal surface of the teeth. The images were digitalized, and we measured the buccal tooth thickness with the UTHSCSA ImageTool 3.0 software (University of Texas Health Science Center, San Antonio, TX, USA).

Four different bleaching gels were evaluated (Table 1) according to the combination of the main factors of HP concentration (20% to 35%) and composition (calcium-free and calcium-containing products). An additional control group, in which no bleaching treatment was performed, was added to the experimental design. Ten teeth were used in each group.

Table 1: *Batch Number, Composition, pH, and Mode of Application of the Bleaching Products*

Bleaching Agent[a]	Composition[b]/Batch No.	pH[c]	Mode of Application[b]
35% Whiteness HP Maxx (35CF)	35% HP, thickeners, dye mixture, glycol, inorganic load, and deionized water/ 191111	6.5	3 Applications of 15 min each
35% Whiteness HP BLUE (35CC)	35% HP, thickeners, inert violet pigment, neutralizing agent, calcium gluconate, glycol, and deionized water/ 250712	9.0	A single 40-min application
20% Whiteness HP Maxx (20CF)	20% HP, thickeners, dye mixture, glycol, inorganic load, and deionized water	6.6	3 Applications of 15 min
20% Whiteness HP BLUE (20CC)	20% HP, thickeners, inert blue pigment, neutralizing agent, calcium gluconate, glycol, and deionized water/270511	9.2	A single 50-min application

Abbreviation: HP, hydrogen peroxide.
[a] *All products are manufactured by FGM Dental Products (Joinville, Santa Catarina, Brazil).*
[b] *According to the manufacturer's recommendations.*
[c] *Measured with a pH meter (pHmetro Nova Técnica NT-PHM, Piracicaba, São Paulo, Brazil) in triplicate.*

Throughout this study, analytical-grade chemicals without previous purification were used; they were prepared with deionized water from a Millipore Milli-Q system (MS2000, Gehaka, São Paulo, SP, Brazil). HP was purchased from LABSYNTH (34%-36%, Diadema, SP, Brazil). A 5000-μg/mL stock solution was prepared in acetate buffer solution (pH 4) and standardized by conventional methods. The solution was titrated with potassium permanganate standard solution.[26] Aliquots of the stock solution of HP were diluted volumetrically to obtain working standard solutions of 0.032-0.397 μg/mL (Table 2; Figure 1).

All teeth were fixed vertically to a wax plaque, and the labial surface of each tooth was isolated by applying a light-cured resin dam (Top Dam, FGM Dental Products, Joinville, SC, Brazil). A 25-μL aliquot of acetate buffer (pH 4.5) was placed into the pulp chamber of each tooth to absorb and stabilize any peroxide that might penetrate into the pulp chamber.

The bleaching gels were applied over the enamel surface, as recommended by the manufacturer (Table 1). After the exposure period, the acetate buffer solutions in the pulp chamber of each tooth were removed by means of a mechanical micropipette (LABMATE Soft; HTL Lab Solutions) and transferred to a glass tube. The pulp chamber of each tooth was rinsed four times with 25 μL of acetate buffer, and this solution was removed from the pulp chamber and placed into the same glass tube. Next, more deionized water (2.725 μL) was added to the glass tube along with 100 μL of 0.5 mg/mL of leuco-crystal violet (Aldrich; Sigma-Aldrich Chemie GmbH, Steinheim, Germany) and 50 μL of 1 mg/mL enzyme horseradish peroxidase (Peroxidase Type VI-A; Sigma Chemical Co, St Louis, MO, USA). When the absorbance value of this sample was higher than 1500 μL, the solution was diluted even further with 3000 μL of deionized water and measured again. This procedure was repeated separately for each tooth.

Table 2: *Data Points for the Calibration Curve*

H_2O_2 Data for Each Point		Solutions Required to Obtain 3000 μL for Each Point for the Calibration Line				
H_2O_2 Weight, μg	H_2O_2 Concentration, μg/mL	Acetate Buffer Solution, μL	47.67 μg/mL H_2O_2 Solution, μL	Peroxidase, μL	Leuco-crystal Violet, μL	Deionized Water, μL
1.192	0.397	75	25	50	100	2750
0.953	0.318	80	20	50	100	2750
0.715	0.238	85	15	50	100	2750
0.477	0.159	90	10	50	100	2750
0.381	0.127	92	8	50	100	2750
0.191	0.064	96	4	50	100	2750
0.095	0.032	98	2	50	100	2750
0.000	0.000	100	0	50	100	2750

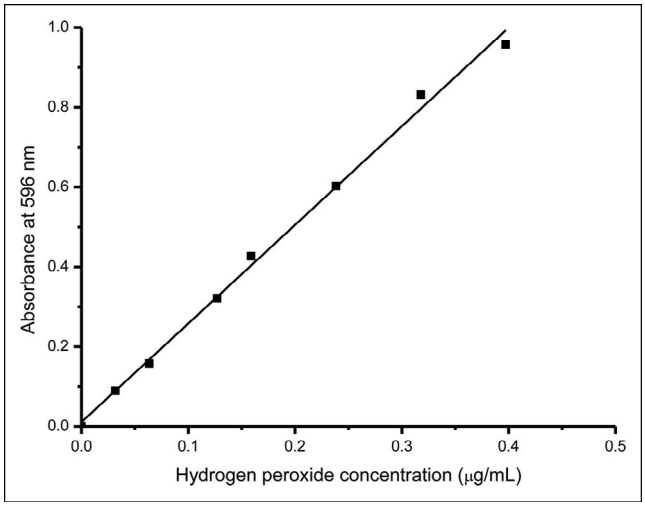

Figure 1. *Spectrophotometric calibration curve used in this study.* R = *0.99524.*

The absorbance at 596 nm of the resultant violet color in the tubes was measured in a Cary 50 UV-Vis spectrophotometer (Varian, Palo Alto, CA, USA). According to Beer's Law, absorbance is directly proportional to the concentration. Therefore, the concentration of HP (μg/mL) was determined by comparing it to the calibration curve previously obtained (Figure 1).[27] By knowing the concentration (μg/mL) and volume of the solution, the HP mass (μg) was calculated by the following equation: $m = C \times MM \times V$, where m represents mass, C is the concentration, MM is the HP molar mass (34,158), and V is the volume (3×10^{-3} L).

The data related to HP concentration and mass were subjected to one-way analysis of variance (ANOVA) and Tukey tests for pairwise comparisons (α=0.05).

RESULTS

The mean buccal tooth thickness of the teeth employed in this study was 2.5 ± 0.5 mm. One-way ANOVA revealed statistically significant differences among groups (p=0.001 and p=0.00001 for HP concentration and HP mass, respectively).

As can be seen in Table 3, an insignificant amount of HP was detected in the pulp chamber of the control groups ($p<0.05$). When the bleaching gels were compared, a significantly lower amount of HP was found in the pulp chamber after application of the calcium-containing gel, regardless of the HP concentration.

Table 3: *Means and Standard Deviations of the H_2O_2 Concentration (μg/mL) and the H_2O_2 Weight (μg) Detected Inside the Pulp Chamber for the Treatment Groups*[a]

Groups	HP Concentration, μg/mL	HP Weight, μg
Control	0.004 ± 0.002 C	0.012 ± 0.005 c
35% Calcium-free	1.156 ± 0.338 A	3.469 ± 1.014 a
35% Calcium-containing	0.201 ± 0.185 B	0.640 ± 0.554 b
20% Calcium-free	0.943 ± 0.487 A	3.251 ± 1.179 a
20% Calcium-containing	0.115 ± 0.082 B	0.664 ± 0.982 b

Abbreviation: HP, hydrogen peroxide.
[a] *Identical uppercase and lowercase letters within a column indicate statistically similar means (one-way analysis of variance and Tukey test, α=0.05).*

DISCUSSION

The results of the present study confirm the ability of the HP to penetrate the tooth structure and to reach the pulp chamber immediately after an in-office bleaching session. This finding had already been demonstrated by several researchers.[9-11]

However, the present study showed that the amount of HP that reached the pulp chamber was not proportional to the HP concentration of the bleaching gel applied on the enamel surface. This is contrary to the findings of other published studies.[9,11] For instance, Gokay and others[9] demonstrated that the amount of HP detected in the pulp chamber was three times higher for a 30% HP than for a 35% carbamide peroxide product, which delivers approximately one-third less HP. In the same study, an even greater difference was reported when the 30% HP product was compared to 10% and 15% carbamide peroxide products.[9] It is worth mentioning that none of these studies compared in-office bleaching gels: high HP concentration was compared to carbamide peroxide gels, which release one-third less HP.

Additionally, carbamide peroxide usually takes longer to deliver the HP[1] than do HP-based gels.[28] This may explain why a huge difference in the amount of HP in the pulp is detected when a high concentration of HP gel is compared with carbamide peroxide products.

The 15% difference in the concentration of HP between the in-office bleaching gels employed in this study does not yield a significant difference in the amount of HP that reaches the pulp, at least when measured immediately after bleaching. The findings of a recent study[29] strengthen this hypothesis. The

authors did not detect differences in the tooth sensitivity prevalence using in-office bleaching gels with 20% and 35% HP. Further studies comparing different concentrations of in-office bleaching gels should be encouraged to increase the generalizability of the findings of this study to other products available in the market.

However, the most surprising finding was the fact that the amount of HP that reached the pulp chamber was statistically lower for the calcium-containing agent than for the calcium-free product, regardless of the initial HP concentration. This finding correlates well with the finding of a recent clinical trial[25] that reported that the prevalence of tooth sensitivity with a calcium-containing agent was lower than that associated with the equivalent calcium-free product.

The fact that HP is detected within the pulp chamber means that not all HP molecules decompose into free radicals within the dental structure (ie, there is an exceedingly high amount of HP within dentin independent of the composition and concentration of the product). For the calcium-containing gel, this HP surplus may react with calcium gluconate present in its composition, leading to the formation of calcium hydroxide, reducing even further the surplus of HP that travels to the pulp chamber.

Additionally, the calcium-containing agent is delivered in an alkaline pH, which is different from the situation with the calcium-free product. In an alkaline media, the dissociation of HP into free radicals is the greatest, as the dissociation constant (pKa) of the HP is around 11.5. It has already been reported[30] that HP in a pH of 9 dissociated 2.7 times more than it did in a pH of 4.4. Thus, if more HP dissociates into free radicals within dental structure, less surplus of HP is available to travel within dentin and reach the pulp chamber. This may explain the lower amount of diffused HP for the high-pH, calcium-containing agent.

The pH of the media affects not only the decomposition kinetics but also the type of by-products produced. While in an acidic solution, free oxygen radicals and hydroxyl anions are produced, in an alkaline media there is a higher concentration of perhydroxyl ions.[31] Although these variations in the bleaching gel composition did not produce differences in the degree of clinical whitening,[25] little is known about the deleterious effects of these different oxidizing agents on the dental-pulp complex, a subject that deserves further evaluation.

This study design did not allow us to measure the decomposition by-products of the HP in the pulp chamber. Therefore, the fact that low HP was detected in the pulp chamber of the calcium-containing products does not mean that the HP by-products are in a reduced concentration in the pulp chamber. The use of electron spin resonance could offer a method with which to evaluate the presence of active oxygen or free radicals produced by the bleaching products in the pulp chamber.[32]

We cannot rule out the role that the difference in bleaching protocols between the calcium-free and calcium-containing gels may have played with regard to the results presented herein. The calcium-containing gel was applied in a single 40-50-minute application (Table 1), per the manufacturer's directions, while the calcium-free gel was applied in three consecutive 15-minute applications. Considering that it took some minutes to remove and reapply the calcium-free gel, this protocol usually took 46 to 47 minutes to complete, meaning that the 35% calcium-free HP gel remained in contact with the buccal surfaces for approximately six to seven minutes longer than did the calcium-containing product with the same concentration. Previous studies[33-35] have already demonstrated that the application time influences the amount of HP that reaches the pulp: the longer the period the HP is in contact with the buccal surface of the teeth, the higher the amount of HP detected in the pulp chamber.

Additionally, the calcium-free gel was replenished three times, while the calcium-containing product was only applied once. This means that we might have delivered more HP in the case of the calcium-free product. While the standardization of the bleaching protocols would allow us to eliminate these protocol variables, it would reduce the clinical significance of the study, as clinicians usually apply the material according to the manufacturer's instructions.

Finally, it is worth mentioning that besides the concentration, composition, and pH of the in-office products evaluated in this study, the literature reports that there are other important factors that can influence HP penetration into the pulp chamber, such as the presence of restorations,[11] enamel craze lines,[36] association with light sources,[10] thickness and type of tooth,[15,37] and chemical activation.[38] All of these factors might explain the high variability within the data when measuring the amount of HP

in the pulp chamber using the UV-VIS spectrophotometer.

CONCLUSIONS

The amount of HP that reached the pulp chamber after in-office bleaching was dependent on the bleaching protocol and gel composition, regardless of the product concentration (20% or 35%).

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Human Subjects Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies approved by the local Ethics Committee of the State University of Ponta Grossa. The approval code for this study was 11005/11. This study was conducted at the State University of Ponta Grossa.

Conflict of Interest

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

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