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Clinical Tech/Case Report

Amelogenesis Imperfecta: A Conservative and Progressive Adhesive Treatment Concept

S Ardu • O Duc • I Krejci
R Perroud

Clinical Relevance

This article presents management of a patient with amelogenesis imperfecta through an adhesive and progressive treatment.

SUMMARY

Objective: The aim of this study was to present a case report of a patient affected by amelogenesis imperfecta showing a possible minimal and conservative adhesive treatment approach.

Clinical Procedure: A treatment philosophy of amelogenesis imperfecta is illustrated by means of a case report of a 14-year-old boy who consulted us for a full mouth rehabilitation.

Discussion: This clinical report describes step by step how to manage a case of amelogenesis imperfecta from childhood over time.

Significance: This kind of minimally invasive, progressive approach allows the conservation of maximum tooth substance together with an acceptable esthetic outcome.

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INTRODUCTION

Resin composite materials are widely used due to their good mechanical and esthetic properties and relatively low cost, especially if compared with full ceramic or ceramo-metallic crowns. Their clinical success is related to the materials' capacity to mimic esthetic tooth appearance. Furthermore, it requires common skills in order to reach satisfying results. Due to their potential to re-create the appearance of sound teeth[1] and to their capacity to mask imperfect substrate,[2] their use has been proposed in order to correct congenital illnesses such as amelogenesis imperfecta.[3,4] This is a hereditary disease that causes structural anomalies in dental enamel of the

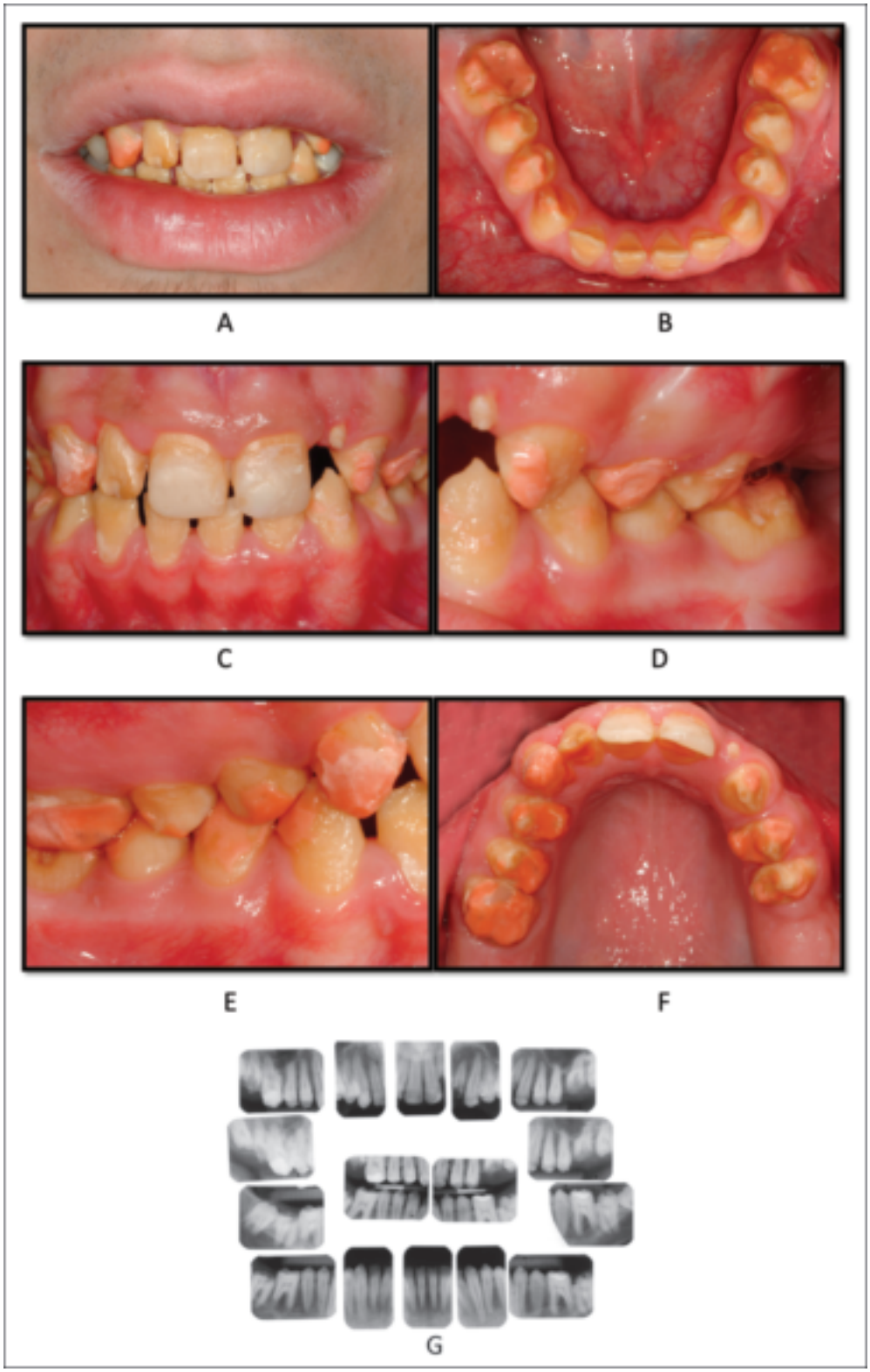

Figure 1. *(*a-g*): Clinical views at the patient's first appointment. Two composite restorations on the upper central incisors as well as glass ionomer cements in posterior areas can be seen in these photos. The last image represents the corresponding treatment status.*

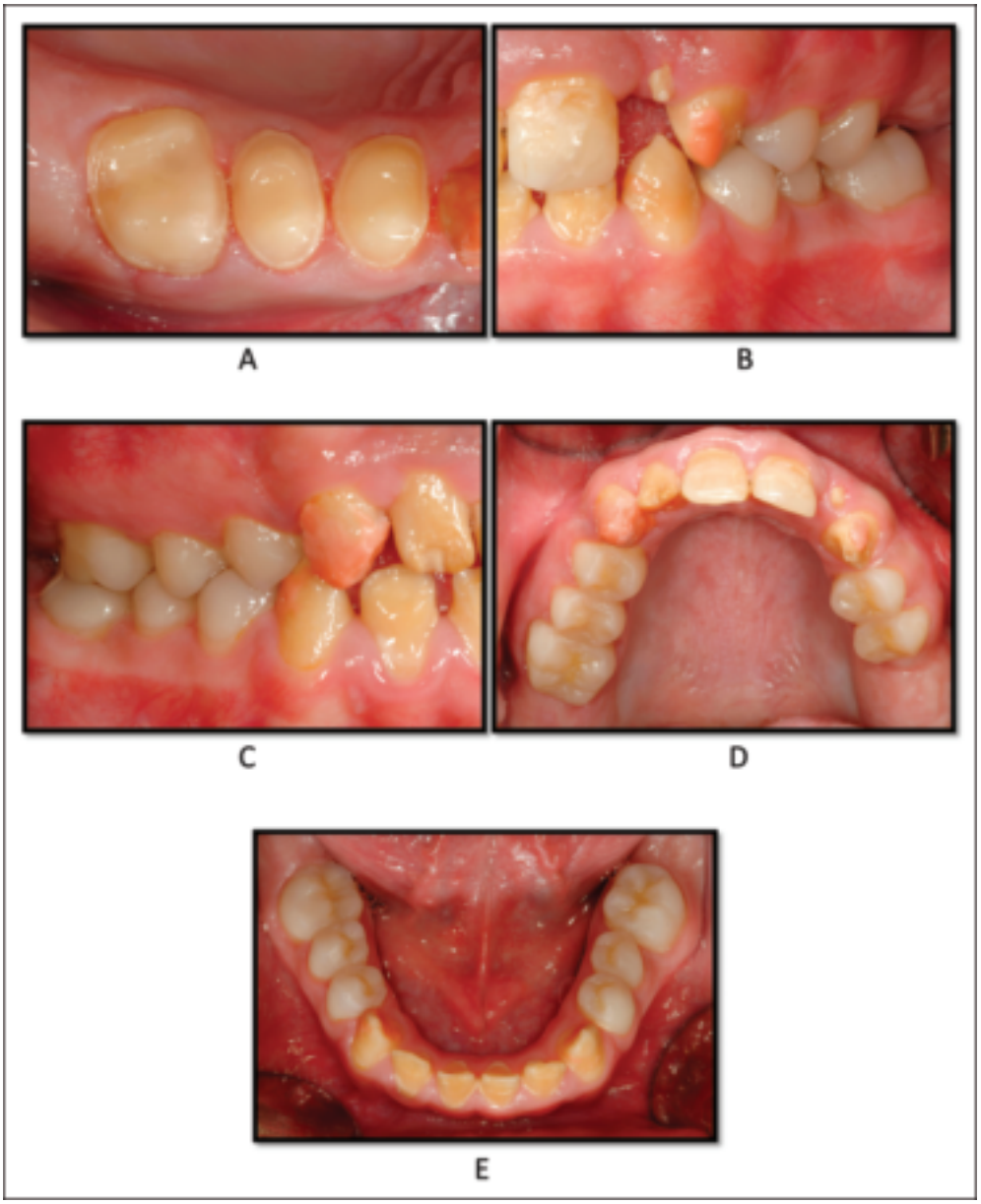

Figure 2. *(a-e): Intraoral view of the conservative preparations for resin composite onlays and clinical view after posterior onlays placement.*

primary and permanent dentition. The anomaly may present a variety of clinical forms and appearances, with its main characteristics being the loss of tooth structure, compromised esthetic appearance, and dental sensitivity.[5]

The aim of this article is to describe, step by step, a rehabilitation concept based on conservative and progressive adhesive treatments by means of free-hand bonded restorations, onlays, and, once complete soft and hard tissue maturation is achieved, adhesive ceramic crowns.

CLINICAL PROCEDURE

Once the diagnosis of amelogenesis imperfecta is confirmed, a progressive, comprehensive treatment plan is proposed to the patient. From childhood and adolescence, patients may complain of teeth sensitivity and an evident lack of esthetics (Figure 1a-g).

Due to the fact that soft and hard tissues have not been completely developed yet, a fixed prosthetic approach cannot be proposed until at least 18–20 years of age.[6,7] In order to answer the patient's demand of not feeling pain during common eating habits and to give him an acceptable esthetic

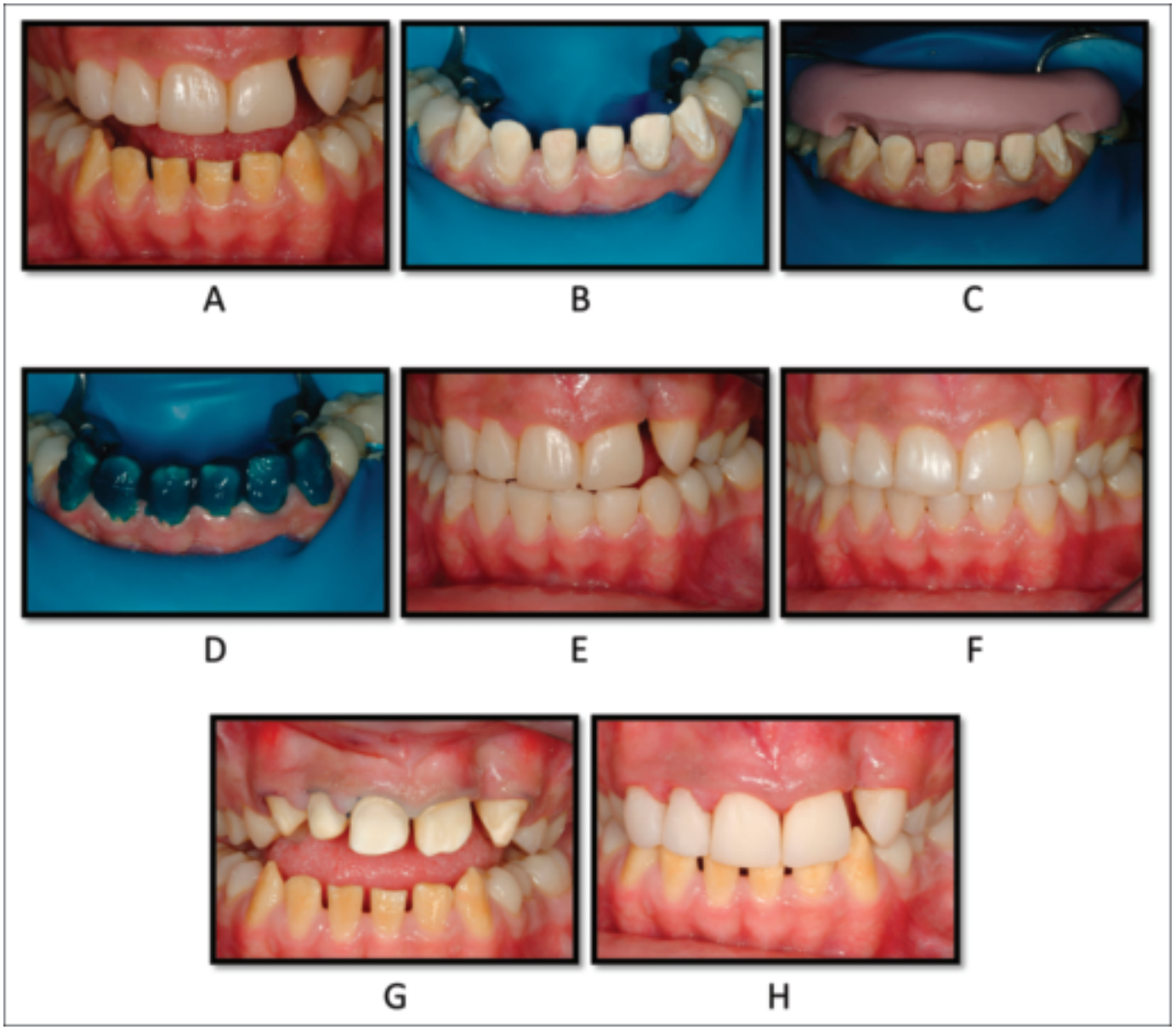

Figure 3. *(a-h): Intraoral view of the preparations for freehand bonded composite restorations and clinical view after teeth rehydration. Last image shows the clinical view after the placement of a cantilever bonded bridge 23-22.*

appearance, a full mouth rehabilitation based on an adhesive approach was planned. In the posterior area conservative resin composite onlays were realized (Figure 2a-e), whereas in the anterior area freehand bonded composite restorations (Miris 2, Coltène/Whaledent, Altstätten, Switzerland) were performed according to the natural layering technique proposed by Dietschi[8] (Figure 3a-h). No orthodontic treatment was realized due to the fact that the patient refused to wear brackets. Finally an adhesive cantilever bridge[9] was seated in order to replace the missing upper left lateral incisor. Having then reestablished function and esthetics, as well as having solved the problem of sensitivity, the patient was kept with these restorations and under recall every 4 months up to the point of complete soft and hard tissue maturation. Four years later, at 20 years of age, a clinical and radiographic evaluation was done, and a crown lengthening procedure of the upper two posterior quadrants was performed. Two months later preparations for lithium disilicate crowns (E Max, Ivoclar Vivadent SAS, Saint-Jorioz, France) were performed on all the teeth, which consequently received their crowns bonded with a

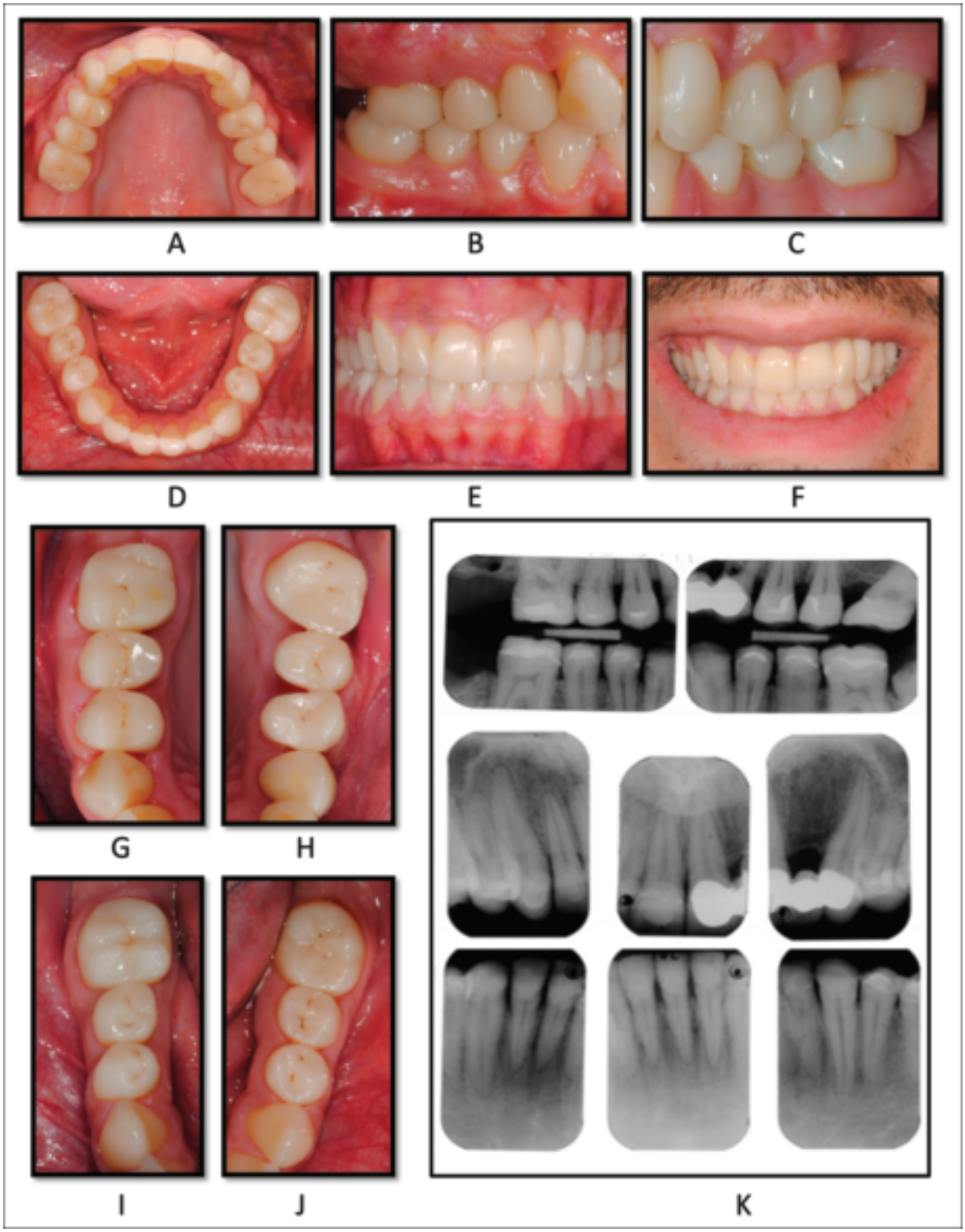

Figure 4. *(a-k): Clinical views at the end of the treatment, together with the final treatment status at the 3-month recall. Except for the lithium disilicate bridge from 21–23, all other teeth are single-unit lithium disilicate bonded crowns. The last image represents the final radiographic status.*

dual resin cement (RelyX Adhesive Resin Cement, 3M ESPE, St Paul, MN, USA).

At the end of the treatment (Figure 4a-k) a vacuum-formed soft resin splint (Erkoloc, Erkodent Erich Kopp GmbH, Pfalzgrafenweiler, Germany) was given to the patient, who was instructed to wear it during the night in order to prevent possible crown chipping and fractures.[10]

DISCUSSION

Adhesive treatment is modern dentistry's answer to the traditional and more invasive prosthetic approach. Bonding procedures have been demonstrated to be a solid tool on which dentists can stand and believe. Resin composite allowed for minimal intervention in this severely compromised case where teeth were affected by amelogenesis imperfecta. The patient's complaints were related to extreme sensitivity that was due to widely exposed zones of dentin and to his unpleasant smile. The two-stage restorative approach was based on a first phase where composite onlays in posterior regions and freehand composites in the anterior sector were realized. During this first restorative phase, minimally invasive dentistry was performed; only the most external, porous enamel layer was removed before resin composite placement. Five years later, at the end of the hard and soft tissue development, an adhesive fixed prosthetic approach was chosen in order to finalize the restoration, always keeping in mind to sacrifice only a minimum of tooth substance. The main advantage of the presented two-stage adhesive approach is that resin composite restorations require almost no maintenance except common polishing procedures. This is a very favorable intermediate solution while waiting for completion of the patient's soft and hard tissue maturation. Furthermore, the first stage can be used as a testing period to check oral hygiene and to determine the definitive form and shade of teeth to be restored. Composite material has some advantages when compared with ceramic restorations: It is less expensive; it allows a direct approach, thus avoiding costs of a dental technician; and it is easily reparable. On the other hand, ceramics may be superior to resin composites in terms of aesthetic, gloss durability,[11] and plaque accumulation.[12] That is why as soon as the patient reached complete soft and hard tissue development, a fixed-adhesive prosthetic approach was used.

With respect to the choice of the ceramic material, among all different products on the market, lithium disilicate crowns were preferred due to their clinical and mechanical advantages. They exhibit high durability, do not appreciably wear the opposing natural dentition,[13] and have already proven that they may be used in clinical situations with promising results.[14] Furthermore, the use of bonding procedures for disilicate crowns have already shown to increase clinical success,[14] offering stable, esthetic, and natural-looking restored teeth to patients.

CONCLUSIONS

This case report describes a possible application of a minimally invasive approach for treating amelogenesis imperfecta in a young adolescent. This conservative adhesive treatment allows a two-stage intervention that accompanies the patient during his adolescence until the complete formation of hard and soft tissues. This will allow acceptable esthetics and lack of pain during eating and will allow the patient to have a more normal social life.

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Conflict of Interest Declaration

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

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Fiber-reinforced Resin Coating for Endocrown Preparations: A Technical Report

GT Rocca • N Rizcalla • I Krejci

Clinical Relevance

The presented clinical technique using fiber-reinforced composite as a resin-coating layer was developed for adhesive endocrown restorations. This may reduce the risk of catastrophic fractures and thus improve the success rate of this type of restoration on nonvital teeth.

SUMMARY

Coronal rehabilitation of endodontically treated posterior teeth is still a controversial issue. Although the use of classical crowns supported by radicular metal posts remains widespread in dentistry, their invasiveness has been largely criticized. New materials and therapeutic options based entirely on adhesion are available nowadays, from direct composite resins to indirect endocrowns. They allow for a more conservative, faster, and less expensive dental treatment. However, the absence of a metal or high-strength ceramic substructure as in full-crown restorations can expose this kind of restoration to a higher risk of irreversible fracture in case of crack propagation. The aim of this case report is to present a technique to reinforce the cavity of an endodontically treated tooth by incorporating a fiber-reinforced composite (FRC) layer into the resin coating of the tooth preparation, before the final impressions of the cavity. This technique allows the use of FRCs in combination with any kind of restorative material for an adhesive overlay/endocrown.

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INTRODUCTION

The tendency of endodontically treated teeth (ETT) to fracture is still a highly debated issue.[1] The biomechanics of an ETT are principally altered by the tissue loss due to prior pathologies (caries, fracture, cavity excavation), endodontic treatment (access cavity, root canal shaping), and invasive

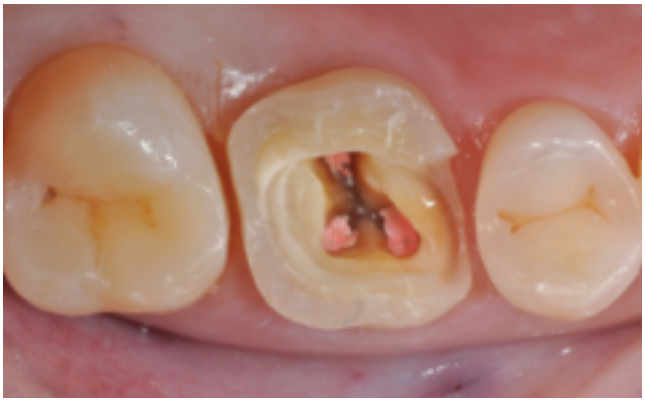

Figure 1. *Initial view of the endodontically treated first maxillary molar after the removing of the provisional restoration.*

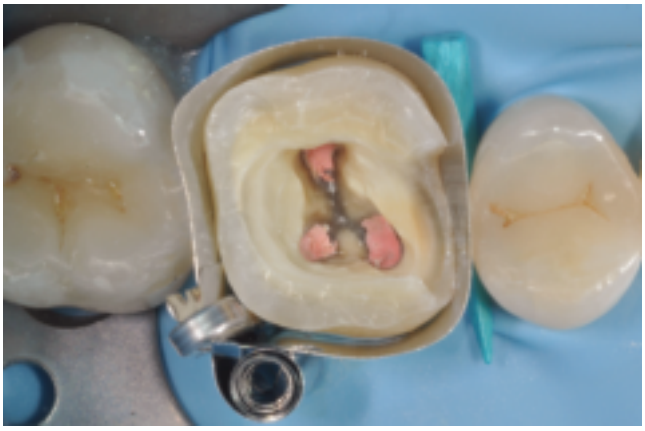

Figure 2. *Isolation of the cavity.*

restorative procedures (post placement, crown fabrication).[2] All of these factors may contribute to a consistent elimination of coronal and radicular tissues, which increases the fragility and thus the fracture risk of an ETT.[3] Recently, the restoration of ETT with adhesive techniques has been advocated both in the root and in the crown to prevent further loss of sound tissues as adhesion ensures sufficient material retention without the need for aggressive macroretentive preparation.[4–6] In particular, the use of bonded overlays, such as endocrowns, for the coronal restoration of an ETT is becoming more common than classic full-crown restorations. The reason for this change of paradigm is to achieve a more conservative approach, which preserves tooth tissues and allows reintervention in case of failure. Furthermore, endocrowns eliminate many technical steps during the fabrication, such as post cementation, core buildup, temporary crown, and potential crown lengthening, which increase treatment time and costs. Several *in vitro* studies and some *in vivo* trials have confirmed the validity of this adhesive approach, especially for molars.[4,7–12]

However, even with conservative overlays/endocrowns, drastic failures—below the cementoenamel junction (CEJ)—are possible, and they have been reported.[12–14] In case of crack propagation, the absence of a metal or high-strength ceramic substructure as in full crowns can expose this type of restoration to higher risk. To improve toughness, leucite and lithium-disilicate reinforced ceramics have been proposed.[15,16] As an alternative to ceramics, composite resins have been suggested because of their superior stress-absorbing properties and high degree of toughness.[8,10] In some *in vitro* studies, fiber-reinforced composites (FRCs) have been also employed to reinforce this kind of cusp-replacing restoration.[15,17,18] Beside improving the strength of the restoration, results of these studies demonstrate that the incorporation of glass fibers into composite resin materials usually leads to more favorable fracture patterns—above the CEJ—because the fiber layer acts as a stress breaker and stops the crack propagation. For classic lab-made indirect composite restorations, FRCs are commonly incorporated during the laboratory fabrication into the base of the work piece.[15,19] Unfortunately, this technique is not possible when the composite restoration is milled from a CAD/CAM block or with any kind of ceramic material. The aim of this case report is to present a technique that will allow the reinforcement of the cavity of an ETT, as opposed to the restoration.

Before taking the final impressions of the cavity, the FRC layer is incorporated on the surface of the tooth preparation. This technique allows for the use of FRCs in combination with any kind of restorative material for an adhesive overlay/endocrown.

METHODS AND MATERIALS

The case reported is an endodontically treated maxillary first molar in need of a restoration (Figure 1). A conventional indirect technique to fabricate an endocrown is accomplished by programming two appointments. During the first appointment,[20] the cavity is cut under local anesthesia. Once the cavity is properly isolated (Figure 2), an adhesive system is applied to the entire dentin and to the mesial thin subgingival portions of enamel margins and then light cured.[21] Then, an adequate amount of composite resin is applied on the dentin and into the mesial box and light cured. The goal is to fill the pulp chamber eliminating the undercuts, cover all the dentin, and relocate the cervical margins 1 mm

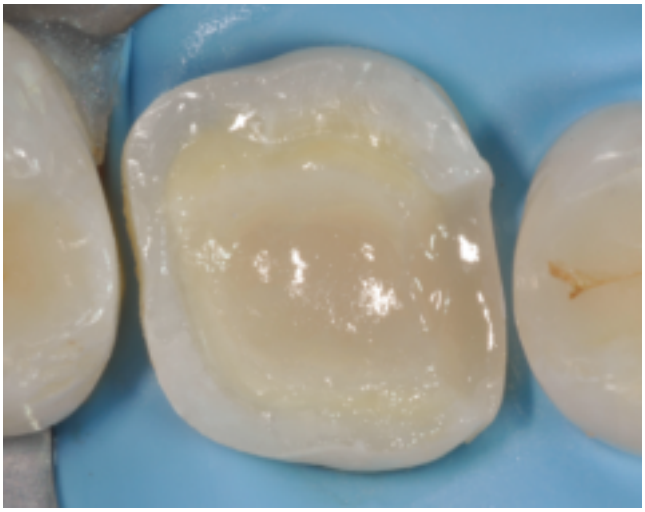

Figure 3. *The composite resin coating.*

supragingivally. For that purpose, a low shrinking nano-hybrid composite is applied (Tetric EvoCeram, Ivoclar-Vivadent AG, Schaan, Liechtenstein). Considering the thickness of the future restoration, at least 1.5 mm is recommended.[9,] Although adhesive luting does not require any particular taper of the cavity or a macro-retentive geometry, the fabrication of a concavity in the middle of the pulpal chamber will help with the positioning of the restoration during insertion (Figure 3). The next step is the insertion of the frame of resin preimpregnated bidirectional glass fibers (Dentapreg UFM, ADM A.S., Brno, Czech Republic) on top of the cavity preparation. The suitable mesiodistal length of the fiber network can be measured in the oral cavity with a periodontal probe (Figure 4). Fibers are then cut and left under light protection outside the mouth. Then, a transparent silicon key (Elite

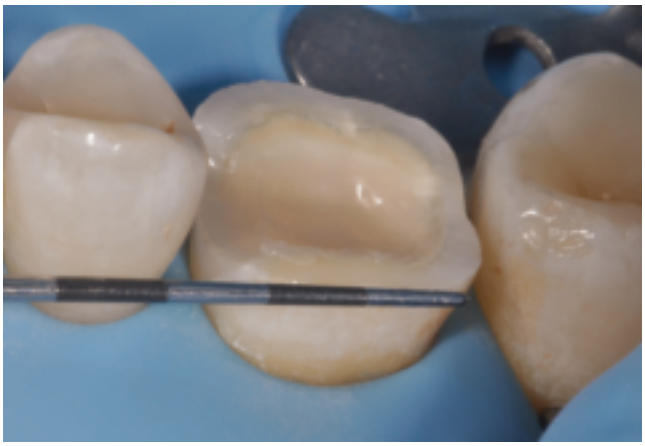

Figure 4. *The suitable length of the fiber-reinforced composite sheet is measured in the mouth with a periodontal probe.*

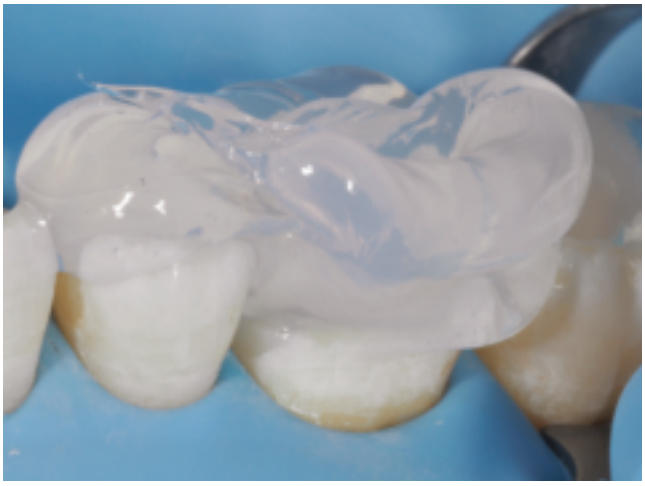

Figure 5. *The customized transparent silicon key.*

transparent, Zhermack SpA, Badia Polesine, Italy) is made to replicate the molar cavity and the occlusal part of adjacent teeth (Figure 5). A layer of about 0.5 mm of flowable composite (Tetric EvoFlow, Ivoclar-Vivadent AG), just enough to accommodate the FRCs, is spread into the cavity and left uncured. The fiber network is then inserted into the cavity over the flowable composite film, and its mesh is slightly opened (Figure 6). Thereafter, the FRC is completely adapted to the cavity with the customized silicon key and light cured (Figure 7). A second layer of flowable composite is applied over the FRC and light cured to cover all the exposed fibers (Figure 8). The enamel margins are finished with fine diamond burs (Composhape, Intensiv SA, Grancia, Switzerland) to obtain well-defined and sharp margins before the impression of the cavity (Figure 9). The

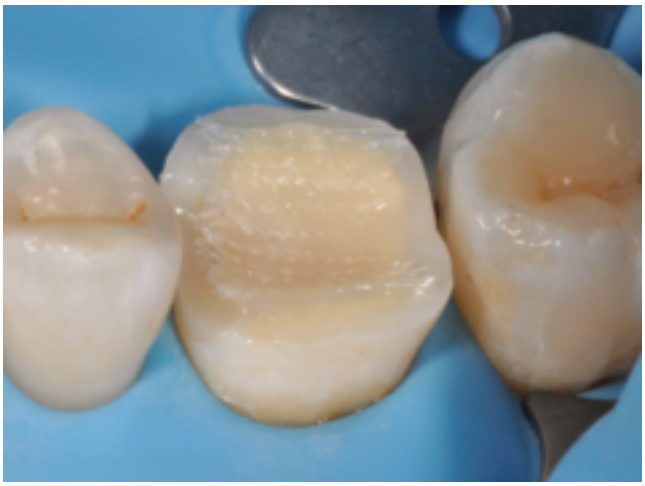

Figure 6. *The fiber frame embedded in a flowable resin is inserted into the cavity.*

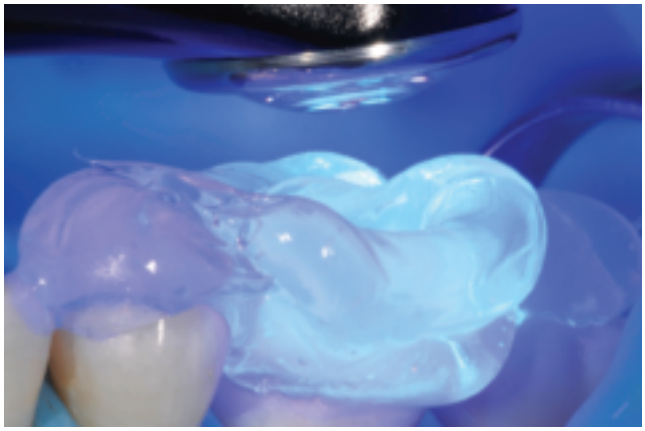

Figure 7. *The fiber-reinforced composite layer is polymerized through the transparent key with a powerful LED lamp.*

indirect restoration is then fabricated. In the case mentioned, the endocrown was milled from a CAD/CAM composite resin block (LAVA Ultimate, 3M ESPE AG, Seefeld, Germany; Figure 10). During the following appointment, the intaglio surface of the restoration and the cavity are adhesively treated, and the restoration is luted with a conventional light-cured micro-hybrid resin composite.[22]

DISCUSSION

In the past 30 years, the optimal performances achieved by modern adhesive systems and the growing emphasis on minimal invasive principles in all fields of dentistry have finally promoted adhesive strategies for ETT. Although metallic restorations and classic PFM crowns supported by radicular metal posts remain widespread, their invasiveness in the root as well as in the crown has been largely criticized, and new therapeutic options

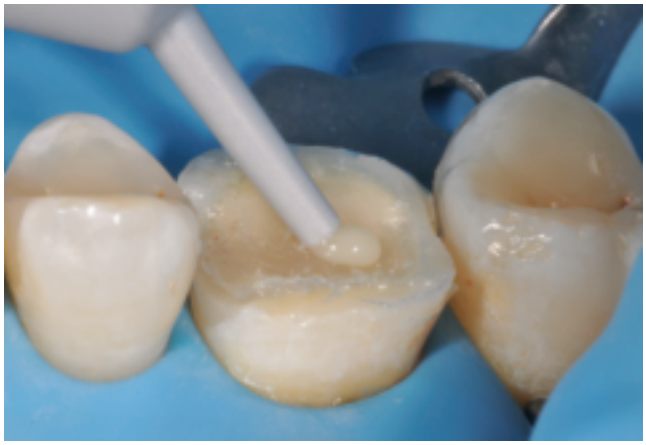

Figure 8. *A further thin layer of flowable resin is applied on fibers to isolate and protect them.*

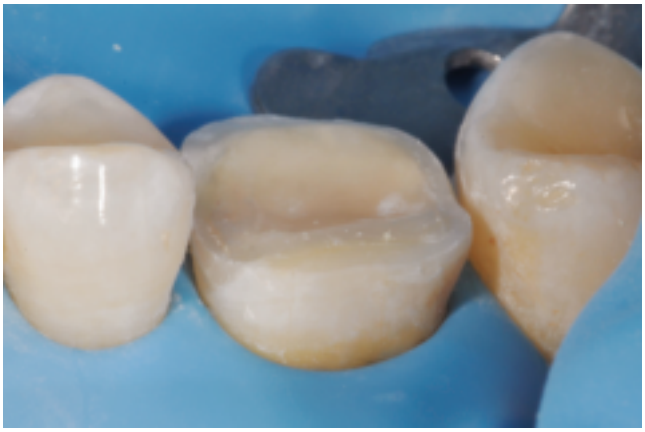

Figure 9. *Enamel is refurbished before the impressions.*

based on adhesion are available nowadays.[3,23–25] In the case of small to medium cavities, direct composite resins and indirect inlay/onlay restorations have almost replaced metallic restorations.[26–29] In the case of large cavities, or whenever a cuspal coverage is needed, bonded endocrowns made of ceramic or composite currently represent a valid alternative to classical full crowns to restore the esthetics and function of ETT.[4,8,10–12,15]

In the clinical case presented, the large amount of tissue lost due to pathology and to the endodontic treatment supports the use of a minimally invasive adhesive endocrown restoration instead of a full crown. This technique allows for the conservation of sound dentin and, above all, peripheral enamel, maintaining the possibility of bonding margins of the future restorations to it, which is known to have a beneficial effect on marginal stability.[30] The adhesive procedure also eliminates the need for the use of a post and a core, which would be otherwise necessary

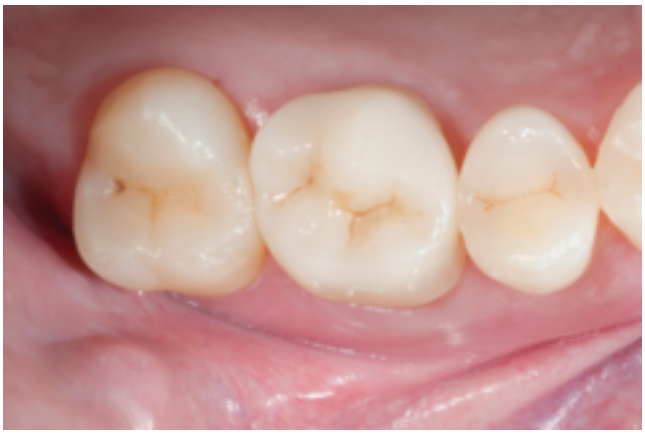

Figure 10. *The CAD/CAM composite restoration one month after the luting.*

in a typical crown preparation. Moreover, the adhesive cavity configuration keeps all margins of the restoration away from the periodontium, which is beneficial for hygiene and periodontal health.[1,31]

Once the tooth is isolated by a rubber dam, a micro-hybrid composite resin is applied to the cavity. Regardless of its composition, resin coating aids elevating cavity margins in slightly subgingival areas as well as eliminating cavity undercuts, thus saving sound tooth structure. Besides these structural functions, the placement of this composite layer on dentin immediately after cavity preparation provides optimal cavity sealing and protection of the endodontic treatment during the temporization period.[32–34] Potential exposure to oral fluids and consequent water sorption of bonding resin are minimized as well.[35] In addition, a composite base leads to the fabrication of thinner inlays and onlays. This implies a better light penetration through the definitive restoration during light polymerization, introducing the use of light-cured luting composites above chemical or dual-cured resins for cementation. Furthermore, and especially for ETT, this composite base reinforces cavity walls during the temporary phase.[12]

Thereafter, a frame of resin preimpregnated bidirectional glass fibers is applied to the cavity (Dentapreg UFM, ADM A.S.). FRCs have been largely tested as materials above all in fixed partial dentures, and they have proved to have superior mechanical properties compared with conventional restorative particulate filler composite resins.[18,36,37] Their use is growing in cusp-replacing single-tooth restoration to overcome limitations in terms of fracture toughness of conventional composite restorative materials in high–load-bearing posterior areas.[15,17–19,38] The FRC layer is positioned between the tooth cavity and the restoration, in a more tensile zone.[37] In case of a classical indirect technique, this configuration is achieved by incorporating the fibers at the base of the composite overlay during the in-lab fabrication. During function, in case of a vertical crack inside the restoration, the FRC layer has the ability to slow or stop the crack propagation through underlying tissues, thus avoiding irreversible fractures. Considering the fibers' orientation, the choice of bidirectional or woven fibers seems more appropriate than unidirectional ones, as in the mouth the restoration is submitted to multidirectional chewing loads.[15,18] Although some authors consider that in single-tooth restorations, the ability of fibers to yield better failure modes is the most beneficial effect of FRC incorporation,[17] Dere and others[15] have recently found that the presence of a multidirectional FRC layer under cusp-replacing composite restorations also led to an improvement of fracture strength for endodontically treated molars.

In the specific case presented, the fiber layer is applied to the cavity before an impression is taken. The incorporation of the fiber layer is accomplished with the help of a layer of flowable composite. The same highly filled micro-hybrid composite used to seal the cavity may be the best choice from different points of view, as flowable composites exhibit high contraction stress during polymerization and may not be sufficiently resistant to deformation under load.[39,40] On the other hand, highly filled micro-hybrid composites are quite difficult to spread in a thin layer because of their high viscosity. The low viscosity of the flowable composite guarantees the diffusion of this resin into the preimpregnated fiber network and decreases the risk of void incorporation. The use of a customized transparent silicon key to push the fiber layer in place during polymerization improves the adaptation of the FRC sheet to the geometry of the cavity and limits the thickness of this intermediate layer. This aspect is of prime importance when a thin restoration is indicated.[41] Moreover, the customized key simplifies the application and the polymerization of the FRC layer compared with the use of specific metallic instruments as suggested by the manufacturer (Dentapreg Fork, ADM A.S.). Once the FRC layer is cured, further application of flowable composite over the fibers protects them from an accidental exposure during the temporary phase.[22,37]

The incorporation of the FRC layer into the tooth cavity before the impression gives the operator the choice between different restorative options. Several materials can be used to fabricate endocrowns, such as feldsphatic porcelain or reinforced glass-ceramic, hybrid composite, or CAD/CAM ceramic and composite blocks. The scientific literature is still not clear about which material is best indicated for this kind of restoration. The authors prefer hybrid composite resins, citing their stress-absorbing properties and their practical benefits such as the possibility to modify and repair the surface easily.[42] In particular, CAD/CAM resin blocks (LAVA Ultimate, 3M ESPE AG) may be used instead of classical lab-made restorations in order to avoid defects inherent in a free-hand laboratory technique and thus improving mechanical properties. The in-lab insertion of an FRC layer at the base of a milled CAD/CAM composite restoration, even if theoretically possible, would mean cutting the restoration, thus compromising its homogeneity.

CONCLUSIONS

Adhesive overlays, often called endocrowns, are increasingly used as a restorative alternative to full crowns for nonvital teeth. Their advantages are minimal invasiveness, simpler preparation, and optimal coronal seal. The risk associated with these restorations is rare but may result in a catastrophic vertical fracture of the tooth-restoration complex, often leading to the extraction of the tooth. The presented clinical technique with FRC reinforcement of the resin-coating layer was developed for use with CAD/CAM composite or ceramic restorations. It may reduce this risk of extensive fractures and thus improve the success rate of this type of restoration on nonvital teeth.

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Conflict of Interest

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

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Clinical Research

Clinical Effectiveness of a Hydrophobic Coating Used in Conjunction With a One-step Self-etch Adhesive: An 18-month Evaluation

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Clinical Relevance

This 18-month clinical evaluation showed that the use of a hydrophobic resin coat over a one-step self-etch adhesive does not improve clinical performance in noncarious cervical lesions.

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SUMMARY

The purpose of this randomized clinical trial was to evaluate the clinical performance of a one-step self-etch adhesive in noncarious cervical lesions with inclusion of a hydrophobic bonding layer not included in the original bonding system as a test of potentially improved bonding. Patients with noncarious cervical lesions received two or four restorations after being randomly assigned to two adhesive technique protocols (n=32): EB, application of Adper Easy Bond (3M ESPE) following manufacturer's instructions; and EB+B, application of Adper Easy Bond, immediately followed by the application of a hydrophobic resin coat (Scotchbond Multi-Purpose Bonding Agent, 3M ESPE). All restorations were restored with a microhybrid composite (Filtek Z250, 3M ESPE). Clinical effectiveness was recorded in terms of retention, marginal discoloration, marginal integrity, postoperative sensitivity,

recurrent caries, periodontal health, and pulpal vitality, according to the modified USPHS criteria, for 18 months. Data were analyzed using chi-square, Fisher exact, and McNemar tests at α=0.05. Two restorations of each group were debonded after six months, leading to an overall clinical success rate of 93.8% for both groups. At the 18-month evaluation period, no new restoration was debonded. However, one restoration of the EB group displayed recurrent caries at the dentin margin, decreasing the overall success rate to 90.6% in comparison to 93.8% of EB+B. The success rate between EB and EB+B was not statistically significant (p=0.5). The application of a hydrophobic resin coat over EB did not increase bonding effectiveness in noncarious cervical lesions after 18 months.

INTRODUCTION

One-step self-etch adhesives are produced from a complex blend of hydrophilic and hydrophobic monomers, solvents, and water in order to combine etching, priming, and bonding application steps into a one-bottle solution.[1] One-step self-etch adhesives are hydrophilic in nature, allowing water to flow from dentin into the adhesive up to the adhesive/composite interface even after polymerization.[2,3] By evaporative, osmotic, and convective processes, water is attracted into the adhesive during gelation phase and travels through interconnecting channels to bind to polar groups of hydrophilic and ionic monomers through hydrogen bonding and van der Waal's forces.[4] The water affinity of this category of dentin adhesive can result in negative consequences to marginal sealing and bond strengths to dentin. Water tends to accumulate on the top surface of the hybrid layer, inhibiting copolymerization within the adhesive, modifying the polymers and accelerating degradation of the adhesive.[5–7] The adverse effect of one-step self-etch adhesive hydrophilicity on bonding to dentin has been reported.[8–12] *In vitro* dentin bond strength tests indicate that the hydrophilicity of one-step self-etch adhesives is a contributive cause for reduced short-term[13] and medium-term values.[14]

As much as *in vitro* evidences are used to predict general clinical outcomes of dental materials through mechanical testing and aging simulations,[15] only well-controlled randomized clinical trials can supply definitive statements on the effectiveness of a given material. The clinical performance of one-step self-etch adhesive systems has been shown to have inferior clinical success rates when compared with two-step or three-step self-etch and etch-and-rinse systems.[16] *In vivo* evidence of water blistering within and protruding from the one-step self-etch adhesive layer has also been documented,[8,17] especially for HEMA-containing formulations. The water diffusion channels are made visible in cross-sectional images by ammonical silver nitrate tracing,[18] revealing geometries that imply the outward fluid flow from a moist substrate and dentinal tubules. When dehydrated dentin was used as substrate, one-step self-etch adhesives failed to show such water blister formation.[19]

The application of an additional layer of hydrophobic resin over unpolymerized one-step self-etch adhesives has been suggested as an alternative procedure to prevent such thoroughgoing water sorption.[20] An additional supply of hydrophobic cross-linking monomers (eg, Bis-GMA) would increase its concentration within the hydrophilic layer, reduce its affinity to water, and enhance its physical properties.[21] The beneficial effect of converting one-step self-etch adhesives into two-step self-etch adhesives by applying an additional coat of a hydrophobic resin has been proven under laboratory conditions,[21–23] and only scarce clinical evidence has been made available.[24]

Therefore, the purpose of this study was to evaluate the clinical performance of a novel one-step self-etch adhesive in noncarious cervical lesions with and without the application of an additional layer of hydrophobic resin.

MATERIALS AND METHODS

A total of 64 restorations were placed in noncarious cervical lesions of vital teeth (assessed through the sensitivity test) of 17 patients, 6 male and 11 female, with a mean age of 42 years (range 22–68). All patients signed an informed consent under a protocol approved by the Federal University of Santa Catarina Ethics Committee. The selection criteria excluded participants with compromised medical history, moderate or chronic periodontitis, lesions with associated caries, absence of antagonist teeth, severe bruxism, and active orthodontic treatment, teeth with cracks, premature contact, or previously placed restorations. The noncarious cervical lesions to be restored were typical V- or U-shaped abrasion/erosion/abfraction lesions in the buccal surface of the maxillary and mandibular incisors, canines, and premolars. Lesions less than 1.0 mm in depth were also excluded from the study. All lesions had incisal/occlusal margins in enamel and gingival margins in dentin. Carious lesions were not included due to a

possible retentive geometry after decayed tissue removal. The lesions were preoperatively categorized according to sensitivity, shape, angle, cervicoincisal height and depth, presence of wear facets, and degree of dentinal sclerosis (Table 1). "No sclerosis" referred to lesions with normal dentin color and spontaneous or provoked sensitivity reported by the patient after the application of a high-pressure air-blow for 3 seconds at a distance of 3.0 cm. "Slightly sclerotic" was used to classify lesions with more opaque or yellow discoloration with spontaneous or provoked sensitivity; these lesions usually presented less severe sensitivity. Lesions classified as "moderate sclerotic" were those that had an opaque or yellow dentin and no sensitivity at all, spontaneous or provoked. The lesions classified as having "severe sclerosis" presented transparent dentin without sensitivity. Noncarious cervical lesions with severe sclerosis (according to the classification above) were also excluded from the study since this condition would theoretically prevent outward dentinal fluid flow.

For a direct comparison of the different bonding approaches, a split-mouth design was selected, in which the same patient received both restorative groups according to the adhesive technique used. The selection of the bonding technique of the first lesions to be restored was determined by flipping a coin. The same calibrated operator, familiar with the adhesive procedures, restored 64 lesions, 32 restorations per group. Six patients received two restorations, and 13 patients received four restorations per group. The distribution of the restorations is shown in Table 2.

The teeth were cleaned with a pumice-water slurry and rubber cup to remove salivary pellicle and any bacterial plaque in a gentle way to prevent gingival dilacerations, which could provoke bleeding and bias during baseline sensitivity evaluation. No local anesthesia was given in order to maintain normal pulpal pressure during the bonding procedure. The field isolation was accomplished by using a labial retractor, cotton rolls, and saliva aspirator. Retraction cords (Ultrapack #00 or #000, Ultradent, South Jordan, UT, USA) were used to retract the gingiva, expose the dentin margins, and prevent gingival fluid from contaminating the lesion. No enamel bevel or dentin roughening was performed. The bonding procedures followed the two experimental adhesive techniques: a group restored with the all-in-one adhesive Adper Easy Bond (3M ESPE, St Paul, MN, USA) according to the manufacturer's instructions (EB) and a group restored with the all-in-one adhesive Adper Easy Bond (3M ESPE) followed by the application of an additional hydrophobic adhesive layer, without rubbing movements (Scotchbond Multi- Purpose Bonding Agent, 3M ESPE) over the unpolymerized all-in-one adhesive (EB+B). The adhesive was then light-polymerized for 40 seconds using a LED light-curing unit (Blue-Phase, Ivoclar Vivadent, Schaan, Liechtenstein) with 1200 mW/cm^2 output intensity, checked using a radiometer (Bluephase meter, Ivoclar Vivadent). Details of the bonding procedures are shown in Table 3.

After the bonding procedures the lesions were restored with a microhybrid composite (Z250, 3M ESPE) in two or three incremental layers, beginning with the gingival margin increment. Each composite increment was light-polymerized for 40 seconds using the same LED light-curing unit at 1200 mW/cm^2. Final contouring and polishing of the restorations were performed at the same appointment, using a fine-grit diamond bur (Komet, Lemgo, Germany), silicon carbide polisher (Jiffy polishers cups, Ultradent, Salt Lake City, UT, USA), flexible discs (Sof-Lex, 3M ESPE), and polishing pastes (Diamond polish 1 μm and 0.5 μm, Ultradent).

Two previously calibrated evaluators (κ=0.893), fully blinded to the adhesive techniques used, evaluated the restorations at baseline (one day after placement of restorations), after six and 18 months using modified USPHS criteria.[25] The criteria evaluated were: retention, postoperative sensitivity, marginal discoloration, marginal integrity, recurrent caries, periodontal health, and pulpal vitality (measured through the sensitivity test). Retention, marginal integrity, marginal discoloration, and occurrence of caries were used as key parameters determining the overall clinical success. Severe marginal defects and marginal discoloration that needed repair or replacement of the restorations were considered as failure.

The chi-square and Fisher exact tests were used to evaluate the association between groups, and McNemar test was used to verify the alpha ratings variation time within the same group ($p \leq 0.05$). For all statistical analyses a 5% significant level was adopted (α=0.05).

RESULTS

Recall results are summarized in Table 4. All patients and restorations were examined in the first and second recalls (recall rate 100%). After six months, the overall retention rate was 93.8% for

Table 1: *Evaluation Methods and Number of Lesions According to Their Characteristics*

Characteristics of the Treated Lesions	Evaluation Method	Number of Lesions
Sensitivity	Anamnesis and high-pressure air-blow for 3 sec at a distance of 3 cm	
Provoked (nonspontaneous)		8
Spontaneous (nonprovoked)		17
Spontaneous and provoked		10
No sensitivity		29
Lesion shape	Visually and tactilely (probe)	
U-shaped		24
V-shaped		40
Lesion angle, degrees	Visually and tactilely (probe)	
<45		4
45–90		24
90-135		21
>135		15
Cervicoincisal height, mm	Periodontal probe	
<1.5		6
1.5-2.5		28
>2.5		30
Lesion depth, mm	Periodontal probe	
≥ 1 < 1.5		44
≥ 1.5		20
Degree of sclerosis	Visually and high-pressure air-blow	
No sclerosis	Normal dentin with spontaneous or provoked sensitivity	21
Slightly sclerotic	Opaque (or yellow) dentin with spontaneous or provoked sensitivity	14
Moderately sclerotic	Yellow (or opaque) dentin without provoked sensitivity	29
Severely sclerotic	Transparent dentin without provoked sensitivity	0
Presence of wear facets	Visually (after air drying)	
Wear facets		21
No wear facets		43
Tooth distribution		
Upper incisor		5
Lower incisor		0
Upper canine		13
Lower canine		2
Upper premolar		15
Lower premolar		29
Upper molar		0
Lower molar		0

both groups due to full debonding of four restorations in total, being two restorations lost per group. Between six and 18 months no full debonding was observed, but one of the restorations of EB presented with a carious lesion at the dentin margin, decreasing the overall success rate for this group to 90.6%. However, statistical analysis revealed no significant differences between the 18-month and baseline evaluations for both groups for the retention criteria. The four debonded restorations were from different patients, which indicate a low influence of patient

Table 2: *Distribution of Noncarious Cervical Lesions*

Adhesive Technique	Arch	Right Quadrants (34)			Left Quadrants (30)		
		Incisor	Canine	Premolar	Incisor	Canine	Premolar
EB	Maxilla	0	2	4	1	5	2
	Mandible	0	1	6	0	2	9
EB+B	Maxilla	3	3	5	1	2	4
	Mandible	0	0	10	0	0	4

factor. Moreover, these lost restorations were bonded to shallow lesions (<1.5 mm), presenting with slight or moderate sclerosis levels, all in lower premolars presenting wear facets. Two of them belonged to U-shaped lesions and the other two to V-shaped lesions.

For marginal integrity criteria, two restorations from both groups showed bravo scores in enamel. In dentin, one restoration from EB+B and four restorations from EB were assigned bravo, but no significant differences were found (p=0.177). For all other criteria, no statistical differences could be detected between groups and evaluation periods, including overall clinical success (p=0.500).

DISCUSSION

The present clinical trial showed that the application of a hydrophobic resin coat over a one-step self-etch hydrophilic adhesive did not increase bonding effectiveness in noncarious cervical lesions after 18 months. The degradation of the bonded interfaces with one-step self-etch adhesives is linked to its high hydrophilic nature. Thus, in order to maximize the water supply at the interface during and after the restorative procedures, the study design involved the exclusion of lesions presenting severely sclerotic dentin and the maintenance of normal pulpal pressure by not using anesthesia of any kind. Severe

Table 3: *Application Procedures of the Adhesive Technique Protocols Used in This Study*

Adhesive Technique	Code	Manufacturer	Composition	Batch Number	Application Procedure
Adper Easy Bond	EB	3M ESPE, St Paul, MN, USA	HEMA, Bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, methacrylate functionalized polyalkenoic acid, silica fillers (7 nm), ethanol, water, initiators based on CQ, stabilizers	301394	1. Apply adhesive for 20 sec in both enamel and dentin. 2. Air-dry with high-pressure for 10 sec at a distance of 10 cm. 3. Light-cure for 10 sec
Adper Easy Bond + Adper Scotchbond Multi-Purpose Bonding Agent (SBMP)	EB+B	3M ESPE, St Paul, MN, USA	Easy Bond: see above. SBMP adhesive: Bis-GMA, HEMA, tertiary amines, photoinitiator	301394 (Adper Easy Bond) 8RF (Adper SBMP)	1. Apply Easy Bond for 20 sec in both enamel and dentin. 2. Air-dry with high-pressure for 10 sec at a distance of 10 cm. 3. Apply SBMP in enamel and dentin. 4. Apply air spray until no more adhesive movement on the surface. 3. Light-cure for 20 sec

Abbreviations: Bis-GMA, bisphenol A diglycidyl ether dimethacryalate; HEMA, 2-hydroxyethyl methacrylate.

Table 4: *Evaluation Results in Percentage of Alpha Score at Each Evaluation Period*[a]

	Recall Period						*p* Value at 18 Months
	Baseline		6 Months		18 Months		
	EB	EB+B	EB	EB+B	EB	EB+B	
Recall rate	100	100	100	100	100	100	0.999
Retention rate	100	100	93.8	93.8	93.8	93.8	0.999
Spontaneous sensitivity	100	100	100	100	100	100	0.999
Provoked sensitivity	100	100	100	100	93.3	96.7	0.500
Marginal discoloration in enamel	100	100	100	100	86.7	86.7	0.999
Marginal discoloration in dentin	100	100	100	100	86.7	86.7	0.999
Marginal integrity in enamel	100	100	96.7	96.7	93.3	93.3	0.999
Marginal integrity in dentin	100	100	93.3	96.7	86.7	96.7	0.177
Absence of caries occurrence	100	100	100	100	96.7	100	0.500
Periodontal health	100	100	100	100	100	96.7	0.500
Pulpal vitality	100	100	100	100	100	100	0.999
Overall clinical success rate	100	100	93.8	93.8	90.6	93.8	0.500

Abbreviations: EB, Adper Easy Bond; EB+B, Adper Easy Bond plus Scotchbond Multi-Purpose Bonding Agent.
[a] *Percentages of all parameters evaluated refer to retained restorations, except for recall rate, retention rate, and overall clinical success rate. Baseline percentages for sensitivity refer to evaluation after the restorations were placed. Number of lesions with sensitivity prior to restoration is shown in Table 1.*

dentin sclerosis is known to result in tubule occlusion by physiologic mineralization preventing the outward flow of fluid. Indeed, water absorption and water tree formation in one-step self-etch adhesives seemed to be minimized when they were applied to transparent sclerotic carious dentin.[26]

To assess the bonding efficacy of both bonding techniques used (EB and EB+B), no bevel or selective etching of enamel was performed. The no-preparation approach allowed assessment of whether the low etching capacity of self-etch systems to unground enamel compromises the establishment of micromechanical retention and marginal sealing.[27] Retention of such poorly bonded noncarious cervical composite restorations to the enamel margin would then rely mainly on dentin bond quality. At 18 months, only two restorations were not rated alpha for enamel marginal integrity, but marginal discoloration was evident in four restorations for both groups. Between six and 18 months, marginal discoloration in enamel increased from 0% to 13.3% for both groups. In a randomized clinical trial evaluating the effect of selective enamel etching prior to the application of a mild self-etch system, unfavorable results for marginal integrity and discoloration were already obtained for the non-etched group at two years.[28] Abdalla and Garcia-Godoy[29] found more marginal discrepancies and discoloration when a self-etching adhesive was applied to mandibular premolars without selective enamel etching following a period of one and two years in cervical lesions.

Studies have demonstrated that all-in-one adhesives form structures that can act as semipermeable

membranes after polymerization, permitting bidirectional water movement across the adhesive layer.[3,4,12,17] However, the hydrophobic bonding applied over the noncured one-step self-etch adhesive creates a thicker and more uniform layer with lower concentrations of retained water and solvent.[21] The additional resin coating applied over a noncured all-in-one adhesive system provides additional free radicals to enhance the rate and extent of polymerization of the self-etching primers with an expected increase in the bond strength to dentin.[20] Furthermore, thickening the adhesive layer has also shown to bring advantages to dentin bonding, once the interface permeability is reduced.[30,31] Clinically, the encouraging results of adding a hydrophobic layer over a one-step self-etch adhesive showed improved retention rates when Clearfil S3 Bond and iBond Gluma were sealed with a hydrophobic resin coat.[24]

Belli and others[32] reported an increased resistance to bond degradation for Adper Easy Bond after one year of storage under simulated pulpal pressure in comparison to other simplified adhesives. Adper Easy Bond showed to be less permeable to dentin humidity.[33] From the present 18 month study of retention rate results, it was not possible to affirm whether Adper Easy Bond is resistant to water sorption or whether the application of an additional coat of hydrophobic resin improved the bond effectiveness. Therefore, hydrolytic degradation of one-step self-etch adhesive is still a concern.

The only restorations to debond were placed in mandibular premolars presenting with wear facets and moderate sclerosis. Although it is early at this stage of the present investigation to correlate these clinical covariables to retention loss, one cannot overlook the potential effect of excessive occlusal loading on cervical stress concentration,[34,35] which can challenge the bond between tooth and restoration.[36,37] Early restoration debonding in allegedly higher loaded teeth may be a sign of bond fragility of the tested adhesive, irrespective of the application method. Teeth with wear facets usually are not excluded from Class V clinical trials, and even so, many adhesives have shown excellent retention rates after six months.[38–40] The fact that all lost restorations were bonded to moderate sclerotic dentin, and therefore, theoretically less subjected to water degradation, may indicate occlusal factors as the main cause of retention loss. Since none of the four lost restorations were from different patients, patient-related factors may be considered minimal in this case.

The use of a hydrophobic resin coat over one-step self-etch adhesive makes bonding procedures more complex, increasing clinical chairside time. Moreover, this clinical approach did not improve clinical performance of the composite resin restorations in noncarious cervical lesions over 18 months of clinical trial. Longer periods of observation and additional studies will be indispensable to further evaluate the clinical performance of one-step self-etch adhesive.

CONCLUSION

From the results of this clinical study, the application of a hydrophobic resin coat over an uncured one-step self-etch adhesive did not statistically improve bonding effectiveness over the 18-month trial period.

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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Two-year Clinical Performance of Self-etching Adhesive Systems in Composite Restorations of Anterior Teeth

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Clinical Relevance

Clinical studies evaluating the clinical performance of one-step self-etching adhesives are scarce. In this study, one-step self-etching adhesives showed good clinical performance at the end of 24 months compared with a two-step etch-and-rinse adhesive.

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SUMMARY

Objective: **The aim of this study was to evaluate the two-year clinical performance of Class III, IV, and V composite restorations using a two-step etch-and-rinse adhesive system (2-ERA) and three one-step self-etching adhesive systems (1-SEAs).**

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Material and Methods: **Two hundred Class III, IV, and V composite restorations were placed into 50 patients. Each patient received four composite restorations (Amaris, Voco), and these restorations were bonded with one of three 1-SEAs (Futurabond M, Voco; Clearfil S3 Bond, Kuraray; and Optibond All-in-One, Kerr) or one 2-ERA (Adper Single Bond 2/3M ESPE). The four adhesive systems were evaluated at baseline and after 24 months using the following criteria: restoration retention, marginal integrity, marginal discoloration, caries occurrence, postoperative sensitivity and preservation of tooth vitality. After two years, 162 restorations were evaluated in 41 patients. Data were analyzed using the χ^2 test ($p<0.05$).**

Results: **There were no statistically significant differences between the 2-ERA and the 1-SEAs regarding the evaluated parameters ($p>0.05$).**

Conclusion: **The 1-SEAs showed good clinical performance at the end of 24 months.**

INTRODUCTION

The advantages of self-etch adhesives are less postoperative sensitivity, fewer operative steps, faster technique, less technique sensitivity, and clinical time savings.[1] In the self-etching systems, acid monomers are combined with water, and hydrophilic solvents can condition the dental structure while simultaneously promoting the infiltration of resinous monomers, diminishing the risk of postoperative sensibility and reducing the chances of having demineralized dentin that has not being impregnated.[1,2] Additionally, there are no concerns about the total removal of the acid and overdrying of dentin in these systems.[2,4].

Although self-etching adhesives have demonstrated good performance in dentin,[5,6] some studies have suggested that their performance in enamel is lower and that their use may lead to low bond-strength values.[7,8] Another shortcoming of self-etching adhesives is that *in vitro* studies have shown that they are more susceptible to microleakage in enamel because of the lower bond strength.[9,10] Self-etching adhesive systems are also subject to hydrolysis caused by the high permeability of the adhesive layer (osmosis), which generates nanoleakage at the adhesive interface.[11-13]

Some clinical studies have showed good clinical performance of two-step self-etching systems.[14-18] Recently, one-step self-etching adhesive systems (1-SEAs) were introduced as the seventh generation of etching systems, which combine acid conditioning, primer, and adhesive in one bottle. Some *in vitro* studies showed that these systems demonstrated a similar impregnation in dentin compared with two-step self-etching systems[19] or total-etch systems,[20] although these experiments were conducted *in vitro*. However, clinical studies evaluating the clinical performance of the 1-SEAs are scarce.[1,2,21-24]

Because of those concerns, the objective of this study was to evaluate the clinical behavior of anterior composite restorations bonded with three 1-SEAs compared with their bonding behavior in a two-step etch-and-rinse adhesive system (2-ERA).

MATERIALS AND METHODS

The São Paulo State University Committee on Investigations Involving Human Subjects reviewed and approved the protocol and consent form used for this study.

Two clinical investigators selected 50 patients according to the following inclusion criteria: presence of four Class III, IV, and/or V carious lesions or unsatisfactory restorations; teeth with pulp vitality; a good general state of health; age between 18 and 65 years; absence of periodontal disease; appropriate oral hygiene; nonsmoker; and absence of parafunctional habits.

Before participating in the study, all patients signed an informed consent. According to the treatment rules from the São José dos Campos School of Dentistry, São Paulo State University, São Paulo, Brazil, all subjects received oral hygiene instructions before treatment.

Restoration Procedures

The restorations were performed by three master's degree students under the supervision of two professors, according to a predetermined procedure that included prophylaxis using rubber cup with pumice and water, shade selection before isolation, rubber dam isolation, and cavity preparation by removing preexisting restorations or excavating carious tissue with carbide spherical burs in a slow-speed headpiece. A bevel was made at the labial cavosurface angle of the anterior teeth using a diamond bur.[25] The cavities were cleaned with pumice and water in a rubber cup, rinsed, and dried.

Each subject received at least four restorations in which the adhesive systems were allocated randomly using a coin toss. The adhesive systems used in this

Table 1: *Materials, pH, Compositions, and Application Mode*

Material	pH	Composition	Application Mode
37% Phosphoric acid/ Adper Single Bond 2 (3M ESPE)	0.6	BIS-GMA, HEMA, diurethane dimethacrylate, polyalcenoic acid copolymer, camphorquinone, water, ethanol, glycerol, nanoparticles of silica	Acid etch (15 seconds); rinse (15 seconds); air dry (30 seconds); remove excess moisture with absorbent paper; apply two coats of adhesive systems (15 seconds each); air dry (5 seconds at 20 centimeters); light cure (10 seconds at 600 mW/cm^2)
Futura Bond M (Voco)	2.0	Organic acids, UDMA, HEMA, camphorquinone, and BHT	Apply one coat actively (20 seconds); air dry (5 seconds at 20 cm); light cure (10 seconds at 600 mW/cm^2)
Clearfil S3 Bond (Kuraray, Tokyo)	2.5	MDP, BIS-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, ethanol, water, silanized colloidal silica	Apply one coat actively (20 seconds); air dry (5 seconds at 20 cm); light cure (10 seconds at 600 mW/cm^2)
Optibond All-in-One (Kerr Corporation)	2.7	Glycerol phosphate dimethacrylate, mono- and dimethacrylate, water, acetone, ethanol, camphorquinone, nanoparticles	Apply two coats actively (20 seconds); air dry (5 seconds at 20 cm); light cure (10 seconds at 600 mW/cm^2)

Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; UDMA, urethane dimethacrylate; MDP, 10-Methacryloyloxydecyl dihydrogen phosphate; BHT, Butylated hydroxy toluene.

study were one 2-ERA, Adper Single Bond 2 (3M ESPE, St Paul, MN, USA), and three 1-SEAs, Futurabond M (Voco, Cuxhaven, Germany), Optibond All-in-One (Kerr Corporation, Orange, CA, USA), and Clearfil S3 Bond (Kuraray, Tokyo, Japan). The adhesive systems were used according to manufacturer's instructions and are described in detail in Table 1.

After the adhesive procedures, resin restorations were completed using a Mylar matrix band and wood wedges. The teeth were restored incrementally with the microhybrid composite, Amaris (Voco). After seven days, the restorations were finished with a sequential protocol using fine grit diamond burs and polishing discs (Soflex, 3M ESPE) under water cooling (Figures 1A,B and 2A,B).

Clinical Evaluation

Evaluations were performed by the operator and at regular time intervals by other evaluators with a kappa agreement of 80%. The restorations were evaluated at baseline, 12 months, and 24 months with regard to (1) restoration retention, (2) enamel and dentin marginal integrity, (3) marginal discoloration, (4) caries occurrence, (5) postoperative sensitivity, and (6) preservation of tooth vitality. The restorations were evaluated according to the criteria introduced by Vanherle and others,[26] as shown in Table 2.

Statistical Analysis

Descriptive statistics were used to describe the frequency distributions of the evaluated US Public Health Service criteria. Clinical success between the adhesive systems was determined using the χ^2 test at a significance level of 5% ($p<0.05$).

RESULTS

Baseline Data

Fifty subjects were enrolled. The mean age of the patients was 38.5 ($\pm$11.1) years. Two hundred composite restorations were placed, 50 with Futura-Bond M, 50 with Optibond All-in-One, 50 with Clearfil S3 Bond, and 50 with Single Bond 2. The distribution of the adhesive systems according to Black's classification is listed in Table 3.

Recall Rate

At the one-year and two-year recalls, the recall rates were 86.0% (43 patients with 172 restorations) and 80.0% (41 patients with 162 restorations), respectively (Table 4). Table 5 summarizes the number of restorations evaluated at each recall period. A cumulative number of two restorations failed during the two years; two restorations with Single Bond 2 were lost.

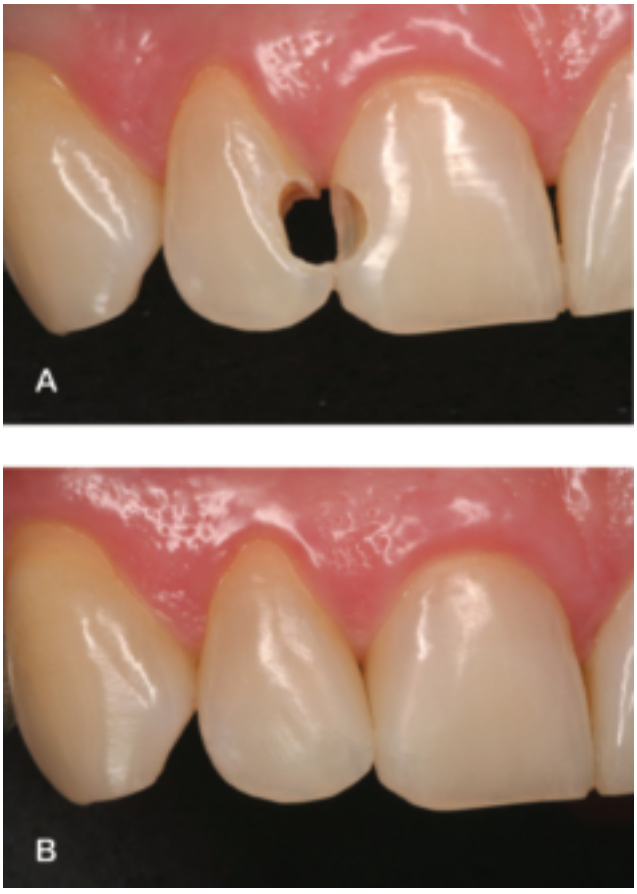

Figure 1. *A. Some examples of Class III cavities; B. Class III restorations bonded with Single Bond (central incisor) and Clearfil S3 Bond (lateral incisor).*

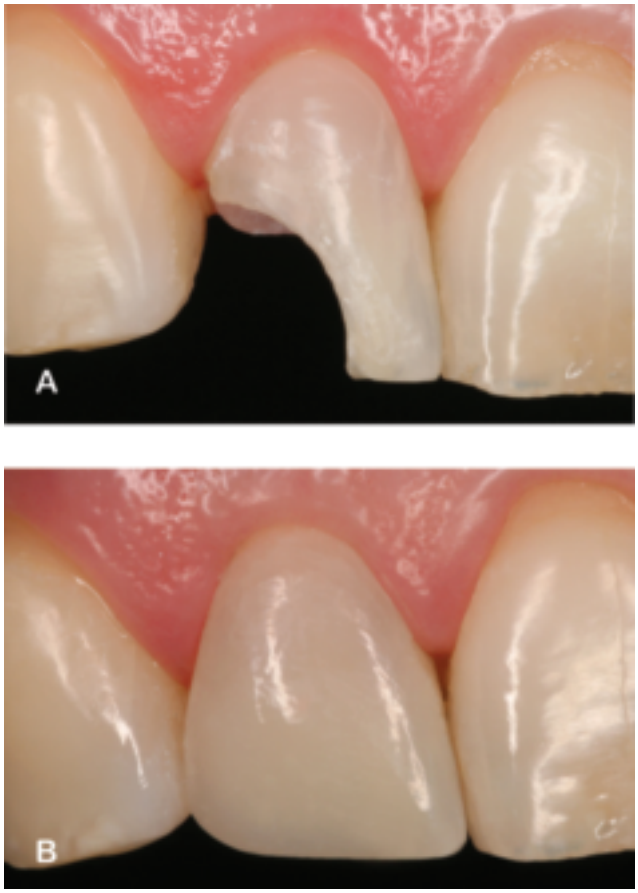

Figure 2. *A. An example of Class IV cavity; B. Class IV restoration bonded with Futurabond M (central incisor).*

Restoration Retention

At the two-year recall, the restoration alpha retention rate was 95.0% because of the loss of two restorations bonded with Single Bond 2. No statistical differences between the 2-ERA and the 1-SEAs were observed for retention rate ($p>0.05$).

Marginal Integrity

At the one-year recall, the marginal integrity rates for Single Bond 2 were 97.0%. At the two-year recall, the marginal integrity rates for Single Bond 2 were 97.5%, Clearfil S3 Bond 97.5% and Optibond All-in-One 97.5%. No significant differences in marginal integrity rates were observed between the systems ($p>0.05$).

Marginal Discoloration

At the one-year recall, the marginal discoloration rates for Single Bond 2 were 93.0%, Clearfil S3 Bond 90.0%, Futurabond M 95.0% and Optibond All-in-One 90.0%. At the two-year recall, Single Bond 2 was 90.0%, Clearfil S3 Bond 90.0%, Futurabond M 97.5% and Optibond All-in-One 92.5%. No statistical differences were observed between the total etch and the self-etching adhesives systems for marginal discoloration rates ($p>0.05$).

Caries Occurrence

Caries occurrence was observed for only one restoration bonded with Clearfil S3 Bond after the two-year recall (2.5%)**.** No statistical differences between the total etch and the 1-SEAs for caries occurrence rate were observed ($p>0.05$).

Postoperative Sensitivity

At the one-year recall, one restoration bonded with Clearfil S3 Bond, one with Single Bond 2 and one with Optibond All-in-One showed postoperative sensitivity (2.5%). At the two-year recall, the postoperative sensitivity rates were 2.5% for Clearfil S3 Bond and 2.5% for Optibond All-in-One. No

Table 2: *Criteria for Assessing Composite Restorations*

Criteria	Score and Definition	Evaluation Method
Marginal discoloration	Alpha: There is no visual evidence of marginal discoloration. There is no difference between restorative material color and the adjacent structure color.	Visual inspection with a mirror at 18 inches
	Bravo: There is visual evidence of marginal discoloration between the tooth structure and restoration, but the discoloration does not penetrate the interface in a pulpal direction.	
	Charlie: There is visual evidence of marginal discoloration between the tooth structure and restoration, and the discoloration penetrates along the restoration in a pulpal direction.	
Caries occurrence	Alpha: There is no visual evidence of dark and deep discoloration adjacent to the restoration.	Visual inspection
	Charlie: There is visual evidence of dark and deep discoloration adjacent to the restoration, but it is not directly related to the cavosurface margin.	
Marginal integrity	Alpha: The explorer does not stick when it is passed from the restoration surface to the tooth, or, if the explorer sticks, there is no visible fracture along the restoration margin.	Visual inspection with an explorer
	Bravo: The explorer sticks and there is no clear and visible fracture where the explorer enters, indicating that the margin of the restoration is not adapted closely with the structure of the tooth. The dentin and/or the base are not exposed, and the restoration has no mobility.	
	Charlie: The explorer enters a mass defect of the fracture that extends to the dentoenamal junction.	
	Delta: The restoration is totally or partially fractured, mobile, or missing.	
Postoperative sensitivity	Alpha: No postoperative sensitivity.	Blowing a stream of compressed air for 5 s
	Bravo: Postoperative sensitivity is present.	
Retention	Alpha: The restoration is completely retained.	Visual inspection with an explorer
	Bravo: The restoration is partially retained.	
	Charlie: The restoration is completely lost.	
Tooth vitality	Alpha: Vital.	Thermal sensitivity test
	Bravo: Non-vital with retracted pulp.	
	Charlie: Non-vital; endodontic treatment is needed.	
	Delta: Non-vital due to restoration.	

Table 3: *Distribution of the Adhesive Systems According to Black's Classification*

Adhesive System	Black's Classification		
	Class III	Class IV	Class V
Single Bond 2	26	8	16
Futurabond M	24	9	17
Clearfil S3 Bond	22	8	20
Optibond All in One	24	7	19

significant differences in postoperative sensitivity rates were observed between the systems ($p>0.05$).

Preservation of Tooth Vitality

One hundred percent of teeth with retained restorations preserved tooth vitality at the two-year recall.

Overall Clinical Success Rate

Because two restorations with Single Bond 2 were lost, the overall clinical success rate was 95.0% after the two-year recall. There was no significant difference between the five adhesives at the two-year recall ($p>0.05$).

DISCUSSION

The 1-SEAs were developed following the recent trend of simplifying the clinical steps and saving operator time. Yet it is important to determine the longevity of these restorations and provide evidence of the safety and efficacy of the new 1-SEAs. Therefore, to that end, the current clinical study evaluated the short-term clinical effectiveness of three of the newest generation of 1-SEAs and compared them to a two-step etch-and-rinse adhesive (2-ERA).

The clinical efficacy of the systems tested was determined by evaluating the restorative retention, marginal integrity, marginal discoloration, caries occurrence, and tooth vitality.[23,26] In most of the restorations evaluated, few changes were noted from baseline to the two-year evaluation visit. No statistically significant differences were observed between the 2-ERA and the 1-SEAs in terms of retention or any of the other evaluation criteria.

The American Dental Association requires a retention rate of at least 90% of the restorations

Table 4: *Number of Restorations Evaluated After Each Recall*

Adhesive Systems	No. of Restorations Evaluated		
	Baseline	12 months	24 months
Single Bond 2	50	43	40
Futurabond M	50	43	41
Clearfil S3 Bond	50	43	41
Optibond All in One	50	43	40
Total	200	172	162

placed after 18 months to obtain full acceptance.[27] Because of the retention loss of two Class IV restorations belonging to the Single Bond 2, the overall clinical success rate was 95.0% for Single Bond 2 after two years. Therefore, all systems evaluated in this study demonstrated good clinical performance and full acceptance. It is possible that the Single Bond 2 restorations failed because large resin composite restorations have a higher failure rate.[28] Additionally, Moura and others[29] observed that Class IV restorations had a high prevalence of failures in a three-year clinical performance of composites. With regards to bonding performance and longevity, *in vitro* investigations found that 1-SEAs presented similar tensile bond strengths to dentin when compared with two-step self-etching systems[19] or total-etch systems.[20]

Corroborating the current results, Zhou and others[24] and Brackett and others,[21] at one-year and two-year recalls, respectively, evaluated the clinical performance of two-bottle and one-bottle self-etching adhesives and observed no statistically significant differences between the systems. Van Landuyt and others[23] and Ermis and others,[22] after one-year and two-year clinical evaluations, respectively, found that a one-step self-etch adhesive and a three-step etch-and-rinse adhesive were equally clinically successful. Fron and others[1] observed that the effectiveness of a one-step self-etch adhesive was very good after two years of clinical service.

Slightly more restorations exhibited marginal discoloration at the two-year recall when bonded with Single Bond 2 (10%) than when bonded with Optibond All-in-One (7.5%) and Futurabond M (2.5%), but no statistically significant differences

Table 5: *Summary of Restorations Evaluated*

Adhesive System and Criteria	Restorations with an Alpha Score/Recalled Restorations (% with Alpha Score) per Evaluation Period		
Single Bond 2	Baseline	1 year	2 years
Restorative retention	50/50 (100%)	43/43 (100%)	38/40 (95%)
Marginal integrity	50/50 (100%)	42/43 (97%)	39/40 (97.5%)
Marginal discoloration	50/50 (100%)	40/43 (93%)	36/40 (90%)
Caries occurrence	50/50 (100%)	43/43 (100%)	40/40 (100%)
Postoperative sensitivity	50/50 (100%)	42/43 (97%)	40/40 (100%)
Tooth vitality	50/50 (100%)	43/43 (100%)	40/40 (100%)
Futurabond M	Baseline	1 year	2 years
Restorative retention	50/50 (100%)	43/43 (100%)	41/41 (100%)
Marginal integrity	50/50 (100%)	43/43 (100%)	41/41 (100%)
Marginal discoloration	50/50 (100%)	41/43 (95%)	40/41 (97.5%)
Caries occurrence	50/50 (100%)	43/43 (100%)	41/41 (100%)
Postoperative sensitivity	50/50 (100%)	43/43 (100%)	41/41 (100%)
Tooth vitality	50/50 (100%)	43/43 (100%)	41/41 (100%)
Clearfil S3 Bond	Baseline	1 year	2 years
Restorative retention	50/50 (100%)	43/43 (100%)	41/41 (100%)
Marginal integrity	50/50 (100%)	42/43 (97%)	40/41 (97.5%)
Marginal discoloration	50/50 (100%)	39/3 (90%)	37/41 (90%)
Caries occurrence	50/50 (100%)	43/43 (100%)	40/41 (97.5%)
Postoperative sensitivity	50/50 (100%)	42/43 (97%)	40/41 (97.5%)
Tooth vitality	50/50 (100%)	43/43 (100%)	41/41 (100%)
Optibond All-in-One	Baseline	1 year	2 years
Restorative retention	50/50 (100%)	42/43 (97%)	40/40 (100%)
Marginal integrity	50/50 (100%)	43/43 (100%)	40/40 (100%)
Marginal discoloration	50/50 (100%)	39/43 (90%)	37/40 (92.5%)
Caries occurrence	50/50 (100%)	43/43 (100%)	40/40 (100%)
Postoperative sensitivity	50/50 (100%)	42/43 (97%)	39/40 (97.5%)
Tooth vitality	50/50 (100%)	43/43 (100%)	40/40 (100%)

were observed. The differences among all of the adhesive systems were not statistically significant. All restorations that exhibited marginal discoloration were classified with a bravo score, "the visual evidence of marginal discoloration between tooth structure and restoration, but the discoloration does not penetrate in the interface in pulp direction" (Table 2). Rates were high for this short period of time. These results could be due to the fact that the patients enrolled have great difficulty in finding access to dental services because they have low incomes and probably have worse oral health than the general population.

Marginal discoloration usually results from defects present between the tooth-colored restoration and the cavity margins and walls, and the etiology can be inadequate restoration placement, finishing procedures, and unsatisfactory bonding.[30] According

to some studies, 1-SEAs are more susceptible to microleakage in enamel tissue because of their lower bond strength.[9,10] However, the current results showed good clinical performance of 1-SEAs compared with a 2-ERA, as also observed by Van Landuyt and others[23] and Ermis and others.[22]

At the one-year recall, alpha scores for marginal integrity were 97.0% for restorations bonded with Single Bond 2. At the two-year recall, alpha scores for marginal integrity (marginal adaptation) were 97.5% for restorations bonded with Single Bond 2, Clearfil S3 Bond, and Optibond All-in-One. Statistical differences between the adhesive systems were not significant. These results demonstrate that the tested adhesive systems showed good marginal integrity during the evaluated period, which is desirable, because restorations with deteriorating margins are more likely to fail than restorations with ideal margins.[31] However, *in vitro* studies have shown that 1-SEAs are subject to hydrolysis, which is caused by the high water content of the dentin surface, which can generate a phenomenon called water treeing.[32] Water trees are water canals at the adhesive interface created by the highly osmolarity of the adhesive solution generating nanoleakage.[11-13] *In vivo* studies are in agreement with the current results, showing good results of 1-SEAs for marginal integrity.[1,21,22,24]

With regards to postoperative sensitivity, one restoration bonded with Clearfil S3 Bond, one with Single Bond 2. and one with Optibond All-in-One showed postoperative sensitivity at the one-year recall. At the two-year recall, one restoration bonded with Clearfil S3 Bond and one with Optibond All-in-One showed sensitivity. One of the advantages of the self-etching adhesives is a reduction of postoperative sensitivity, as they are less technique sensitive, which reduces possible failures caused by imperfections in the adhesion that result from overetching or drying of the dentin.[1,22,33] However, the current results showed no significant differences in postoperative sensitivity between the systems, corroborating the findings of Van Landuyt and others[23] and Ermis and others.[22] According to Van Landuyt and others,[23] the reduction of postoperative sensitivity can be explained by the protective effect of the restoration combined with the passage of time.

The excellent short-term clinical performance of 1-SEAs in the present study can be attributable to (1) the experience of the operators in performing adhesive dentistry; (2) the presence of a bevel at the labial cavosurface angle, which can improve retention and reduce marginal discoloration of restorations;[3,34] and (3) the low pH of the systems and functional monomers in their composition (ie, manufacturers have developed products with lower pH, which increases their acidity, and consequently, they produce an acceptable enamel etching pattern.)[6]

In conclusion, after a two-year recall, the 1-SEAs Clearfil S3 Bond, Futurabond M, and Optibond All-in-One performed similarly to a 2-ERA (Single Bond 2) in terms of restorative retention, marginal integrity, marginal discoloration, caries occurrence, and tooth vitality. However, further studies are necessary to evaluate the long-term clinical performance of these systems.

CONCLUSIONS

There were no statistically significant differences between the 2-ERA and the 1-SEAs with regards to the evaluated parameters. The 1-SEAs showed good clinical performance at the end of 24 months.

Conflict of Interest

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

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Laboratory Research

Effect of Substrate Age and Adhesive Composition on Dentin Bonding

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Clinical Relevance

The bonding efficacy of current dentin adhesives is not affected by the dentin substrate age. Chemical bonding may play a role in the bonding effectiveness of specific adhesives.

SUMMARY

Purpose: **To study the effect of dentin age and adhesive composition on the microtensile dentin bond strengths (μTBS) of five dentin adhesives.**

Materials and Methods: **Sixty extracted caries-free human teeth were assigned to the appropriate age group: less than 21 years of age (<21), 21–40 years of age (21–40), and greater than 40 years of age (>40). For each age group, specimens were randomly divided into five dentin adhesives: (1) Adper Easy Bond (EB, 3M ESPE), a one-step self-etch adhesive; (2) Experimental Adper Easy Bond without the Vitrebond Co-polymer (CP) (EBnoCP, 3M ESPE); (3) Adper Single Bond Plus (SB, 3M ESPE), a two-step etch&rinse adhesive; (4) Experimental Adper Single Bond Plus without CP (SBnoCP, 3M ESPE); and (5) Adper Scotchbond Multi-Purpose (MP, 3M ESPE), a three-step etch&rinse adhesive, as the control group. Specimens were sectioned in X and Y directions and the resulting beams were tested to failure in tension mode at a crosshead speed of 1 mm/min. Statistical analysis was computed using *t*-test and two-way analysis of variance followed by Fisher least significant difference multiple comparison *post hoc* test at $p < 0.05$.**

Results: **The highest mean μTBS values were obtained in the control group (MP) for all age groups. EB resulted in statistically similar mean μTBS compared to EBnoCP for all age groups: $p = 0.538$ for (<21); $p = 0.974$ for (21–40); and $p = 0.909$ for (>40). SB resulted in statistically higher mean μTBS than SBnoCP for all age groups [$p<0.009$ for (<21); $p<0.028$ for (21–40); and $p<0.041$ for (>40)]. MP, the control group, resulted in statistically lower mean μTBS when applied to the oldest age group (>40) compared to the youngest age group (<21), at $p < 0.04$. When means were pooled for the variable 'age group,' SB resulted in**

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significantly higher mean μTBS than SBnoCP at $p < 0.009$, while EB resulted in statistically similar mean μTBS compared to EBnoCP ($p=0.9$). MP resulted in statistically higher mean μTBS than all other adhesives—SB ($p<0.0001$), SBnoCP ($p<0.0001$), EBnoCP ($p<0.022$), and EB ($p<0.046$).

Conclusions: **The substrate age influenced the bonding ability of the three-step etch&rinse adhesive. The presence of a carboxylic-based polymer (CP) enhanced the bonding ability of the two-step etch&rinse adhesive.**

INTRODUCTION

Dentin adhesion has not yet achieved the ideal characteristics as a result of dentin tubular structure, organic content, and intrinsic moisture.[1] Physiological changes resulting from dentin aging or changes in response to caries and other aggressive stimuli increase the degree of mineralization of dentin, with a consequent increase in dentin thickness and reduction of dentin permeability.[2–4] Since dentin permeability is an important factor in the adhesion process, reduction of permeability with age may have a direct effect on dentin bond strengths.[1,5] In spite of increased dentin calcification with age, adhesion studies[6–9] have not shown an obvious correlation between dentin age and bonding ability of dentin adhesives.

The interaction of acids or acidic monomers with hydroxyapatite is a fundamental factor in the adhesion process. Acids demineralize dental hard tissues,[10] opening a pathway for the infiltration of resin monomers into the microporosities previously occupied by hydroxyapatite crystals.[4] For dentin, these monomers polymerize *in situ* within the collagen network spaces to create a hybrid layer of collagen and resin.[4,11] While micromechanical dentin-resin entanglement is essential for immediate dentin bond strengths, chemical adhesion would be desirable to improve bonding stability.[4]

Polycarboxylic or polyalkenoic acids promote a stable chemical bonding between the carboxylate groups (COO^-) and calcium in hydroxyapatite.[12–14] Glass ionomer cements (GICs) are the only direct restorative material to bond chemically to hard dental tissues as a result of the formation of ionic bonds between carboxylate groups and calcium, enhanced by a shallow interfacial absorption layer into the dentin for the newest resin-modified GIC.[12,14,15] The chemical adhesion provided by GIC has led some manufacturers to introduce carboxylate-based polymers in the composition of dental adhesives. The polyalkenoic acid copolymer first used in Vitrebond (3M ESPE, St Paul, MN, USA) is now known as the Vitrebond copolymer.[16] Mitra and others[16] reported that this specific copolymer bonds chemically to calcium in hydroxyapatite. As several adhesive formulations from the same manufacturer (3M ESPE) now contain this molecule, it is relevant to study whether the copolymer improves the bonding ability of dentin adhesives.

The null hypotheses tested in this study were (1) dentin age does not influence the bonding ability of dentin adhesives; and (2) the inclusion of a polyalkenoic acid copolymer in the composition of two adhesives does not influence dentin microtensile bond strengths (μTBS).

MATERIALS AND METHODS

Sixty extracted sound third molars and maxillary premolars were collected in function of the patient's age (n=20, 15 molars and five premolars per age group): Less than 21 years of age (<21, only teeth with complete root formation); between 21 and 40 years of age (21–40); and greater than 40 years of age (>40). Premolars were used as a result of the difficulty in obtaining extracted molars categorized by age. Teeth were then stored in 0.5% chloramine solution for up to one month and left in distilled water for 24 hours at 4°C prior to use. Middle dentin was exposed by sectioning the crowns parallel to the occlusal surface in a slow-speed diamond saw (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA) under water-cooling. Dentin was polished with wet 600-grit silica-carbide abrasive paper for 60 seconds to create a standardized smear layer.[17] For each age group, specimens were randomly and equally assigned to five dentin adhesives (three molars and one premolar per subgroup), as follows: Adper Easy Bond (EB, 3M ESPE); Adper Easy Bond without the Vitrebond Co-polymer (CP) (EBnoCP, 3M ESPE); Adper Single Bond Plus (SB, 3M ESPE); Adper Single Bond Plus without CP (SBnoCP, 3M ESPE); and Adper Scotchbond Multi-Purpose (SMP, 3M ESPE), a three-step etch&rinse adhesive, as the control group (Table 1). The adhesives were applied with a microbrush (Microbrush International, Grafton, WI, USA) and polymerized according to the manufacturer's instructions (Table 1). The crowns were restored with Filtek Z250, shade A2 (3M ESPE) in three increments of 2 mm each using an Elipar S10 (3M ESPE) curing light with an output of >800 mW/cm^2. Specimens were then automatically sectioned with a slow-speed diamond saw (Accutom 50,

Table 1: *Materials, Batch Numbers, Compositions, and Instructions for Use*

Material	Composition	Instructions for Use
Adper Easy Bond (EB), Lot 380005	Bis-GMA; HEMA; water (10–15 Wt%); ethanol (10–15 Wt%); phosphoric acid-6-methacryloxy-hexylesters; silane-treated silica; 1,6-hehadeniol dimethacrylate; copolymer of acrylic and itaconic acid (Vitrebond copolymer) (1–5 wt%); (dimethylamino)ethyl methacrylate, camphorquinone; 2,4,6-trimethylbenzoyldiphenylphosphine oxide	Dry the cavity with gentle stream of air free of water and oil or by blotting with cotton pellets. Do not overdry. Apply the adhesive with the disposable applicator for 20 s to all surfaces of the cavity. Rewet the disposable applicator as needed during application. Subsequently, air-thin the liquid for approximately 5 s until the film no longer moves, indicating complete vaporization of the solvent. Cure the adhesive with a commonly used curing light for 10 s.
Experimental Adper Easy Bond (EBnoCP), Lot HB2-primer-0427	Same as for Adper Easy Bond, but without the Vitrebond copolymer	Same as for Adper Easy Bond
Adper Single Bond Plus (SB), etchant: Lot N103490; adhesive: Lot 153567-41-3	Etchant: amorphous silica-thickened 35% phosphoric acid gel	Apply Scotchbond Etchant to tooth surface for 15 s. Rinse thoroughly for 10 s. Blot excess water using a cotton pellet or minisponge. Do not air dry! Apply two to three consecutive coats of adhesive for 15 s with gentle agitation using a fully saturated applicator. Gently air thin for 5 s to evaporate solvent. Light cure for 10 s.
	Adhesive: ethyl alcohol (25–35 Wt%); silane-treated silica (nanofiller); Bis-GMA; HEMA glycerol 1,3-dimethacrylate; copolymer of acrylic and itaconic acid (Vitrebond copolymer) (5-10 wt%); diurethane dimethacrylate; water (<5%)	
Experimental Adper Single Bond Plus (SBnoCP), etchant: Lot N103490; adhesive: Lot 153567-41-2	Same as for Adper Single Bond Plus, but without the Vitrebond copolymer	Same as for Adper Single Bond Plus
Adper Scotchbond Multi-Purpose (MP), etchant: Lot N103490; primer: Lot 120768; adhesive: Lot 120161	Etchant: amorphous silica-thickened 35% phosphoric acid gel	Apply Scotchbond etchant to enamel and dentin. Wait 15 s. Rinse for 15 s. Dry for 5 s. Apply Adper Scotchbond Multi-Purpose primer to etched enamel and dentin. Dry gently for 5 s. Apply Adper Scotchbond Multi-Purpose adhesive to the primed enamel and dentin. Light cure for 10 s.
	Primer: water (40–50 Wt%); HEMA (35–45 Wt%); copolymer of acrylic and itaconic acids (Vitrebond copolymer) (10–20 wt%)	
	Adhesive: Bis-GMA (60–70 Wt%); HEMA (30–40 Wt%)	
Filtek Z250, Lot N117387	Bis-EMA, TEGDMA, UDMA, zirconium, silica	

Bis-EMA: bisphenol A-polyethylene glycol dieter dimethacrylate; Bis-GMA: bisphenol A diglycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate, TEGDMA: triethyleneglycol-dimethacrylate; UDMA: urethane dimethacrylate.

Struers A/S, Ballerup, Denmark) into two perpendicular directions to obtain beams with a cross section of 0.7 ± 0.2 mm^2. The beams from the periphery were discarded. The remaining beams were individually attached to a stainless-steel grooved jig with cyanoacrylate glue (Zapit, Dental Ventures of America Inc, Corona, CA, USA) and tested to failure in tension mode (the jig was in line relative to the axis of the applied load) using a universal testing machine (Shimadzu Autograph AG-IS, Tokyo, Japan) at a crosshead speed of 1 mm/min. Failures were analyzed under a stereomicroscope (Leica MZ6, Leica Microsystems AG, Heerbrugg, Switzerland) at 20×. The mode of failure was classified as adhesive (A), mixed (M), and cohesive. Failures were considered A when they occurred at the dentin-adhesive interface; they were of cohesive nature when the failure occurred in dentin (CD) or in composite (CC); and they were of M nature when there was composite and dentin at the interface.

For each tooth, the μTBS values of all beams were averaged for statistical purposes, each tooth serving

Table 2: *Number of Beams per Group, Each Tooth as a Statistical Unit; Mean Microtensile Dentin Bond Strength (μTBS) ± Standard Deviation (SD) (MPa)*[a]

	<21		21–40		>40		All Ages
	No. of beams[b]	μTBS, Mean ± SD	No. of Beams[b]	μTBS, Mean ± SD	No. of Beams[b]	Mean μTBS, Mean ± SD	μTBS Pooled for Age Group, Mean ± SD
EB	64	60.7 ± 4.4^{A,a}	58	56.0 ± 4.0^{A,e,f}	68	59.0 ± 9.3^{A,g}	58.6 ± 6.1^{k}
EBnoCP	58	57.2 ± 5.8^{B,a}	64	56.1 ± 7.7^{B,e,f}	65	58.3 ± 7.7^{B,g}	57.2 ± 6.5^{k}
SB	76	44.0 ± 14.4^{C,b}	98	47.0 ± 12.6^{C,e}	98	42.9 ± 7.3^{C,h}	44.7 ± 10.8^{l}
SBnoCP	70	27.4 ± 5.3^{D,c}	89	32.5 ± 4.7^{D,g}	54	30.9 ± 4.4^{D,i}	30.3 ± 4.9^{m}
MP	89	71.4 ± 4.6^{E,d}	113	65.3 ± 10.1^{E,F,f}	95	59.6 ± 10.2^{F,g}	65.4 ± 9.5^{j}

Abbreviations: EB, Adper Easy Bond; EBnoCP, EB without Vitrebond copolymer; MP, Adper Scotchbond Multi-Purpose; SB, Adper Single Bond Plus; SBnoCP, SB without Vitrebond copolymer; <21, less than 21 years of age; 21–40, between 21 and 40 years of age; >40, more than 40 years of age.
[a] Within each row, means with the same superscript uppercase letter are not significantly different at $p > 0.05$; within each column, means with the same superscript lowercase letter are not significantly different at $p > 0.05$.
[b] The statistical software automatically compensates for the different number of beams.

as a statistical unit. Once a normal distribution and equality of the variances were confirmed, statistical analyses included two-way analysis of variance to determine whether a significant difference existed between dentin ages using different adhesives, followed by Fisher least significant difference multiple comparison *post hoc* tests at $p < 0.05$. The fracture types were compared using Spearman correlation coefficient (SPSS 14.0, SPSS Inc, Chicago, IL, USA).

RESULTS

The mean bond strengths, respective standard deviations, and the number of beams tested per group are displayed in Table 2. EB resulted in statistically similar mean μTBS compared to EBnoCP for all age groups: $p = 0.538$ for (<21); $p = 0.974$ for (21–40); and $p = 0.909$ for (>40). SB resulted in statistically higher mean μTBS than SBnoCP for all age groups [$p<0.009$ for (<21); $p<0.028$ for (21–40); and $p<0.041$ for (>40)]. MP, the control group, resulted in statistically lower mean μTBS when applied to the oldest age group (>40) compared to the youngest age group (<21) at $p < 0.04$. All the other pairwise comparisons within the same adhesive resulted in no statistical significance. There were no significant interactions between the independent variables ($p>0.123$).

When means were pooled for the variable 'age group' (Table 2, far right column), MP resulted in statistically higher mean μTBS compared to all other adhesives—SB ($p<0.0001$), SBnoCP ($p<0.0001$), EBnoCP ($p<0.022$), and EB ($p<0.046$). SB resulted in significantly higher mean μTBS than SBnoCP at $p < 0.009$, while EB resulted in statistically similar mean μTBS compared to EBnoCP ($p=0.9$).

There were no pretesting failures in any of the groups. The distribution of the type of failures per group is displayed in Table 3. Most failures (72%) were of the A type. There was a significant correlation between fracture type and adhesive/age group (Spearman rho, $p<0.01$), with MP resulting in a greater number of cohesive failures in dentin.

DISCUSSION

The amount of secondary dentin is directly correlated with dentin age.[18] As teeth age, dentin continues to be secreted, resulting in the dentinal tubules becoming narrower.[2] This increased calcification is part of physiological aging (or physiological sclerosis), as well as a response to external stimuli such as attrition and caries.[2,3] Adhesion studies have not shown a direct correlation between dentin age and dentin bonding. A study compared the second-generation adhesive Scotchbond DC with the third-generation adhesive Scotchbond 2, both of which were applied in teeth with a mean age of 22.5 vs 65.6 years.[9] Scotchbond 2 contains maleic acid and 2-hydroxyethyl methacrylate (HEMA) in the primer; therefore, it may be considered a predecessor of self-

Table 3: *Type of Fractures per Group (%)*

Fracture Type (Total %)	Adhesive/Age														
	EB			EBnoCP			SB			SBnoCP			MP		
	<21	21–40	>40	<21	21–40	>40	<21	21–40	>40	<21	21–40	>40	<21	21–40	>40
A (72.1)	70.3	70.7	85.3	65.5	98.1	83.1	69.7	62.2	79.6	92.9	82.0	85.2	56.2	55.6	56.8
M (7.8)	0	3.4	0	3.4	0	0	21.0	22.4	4.1	2.8	7.9	6	16.8	11.3	0
CD (17.9)	21.9	24.1	13.2	27.6	1.9	15.4	5.3	12.2	14.3	4.3	9.0	2	22.5	30.8	43.2
CC (2.2)	7.8	1.4	1.5	3.4	0	1.5	3.9	3.1	2.0	0	1.1	0	4.5	2.3	0

Abbreviations: A, adhesive; CC, cohesive in composite; CD, cohesive in dentin; EB, Adper Easy Bond; EBnoCP, EB without Vitrebond copolymer; M, mixed; MP, Adper Scotchbond Multi-Purpose; SB, Adper Single Bond Plus; SBnoCP, SB without Vitrebond copolymer; <21, less than 21 years of age; 21–40, bewteen 21 and 40 years of age; >40, more than 40 years of age.

etch adhesives. Authors reported shorter marginal gaps when Scotchbond 2 was used in older teeth. Another study,[7] which included a copolymer-containing dentin adhesive (Scotchbond Multi-Purpose), did not find any correlation between dentin bond strengths and dentin age for teeth younger than 30 years vs. those older than 50 years of age. A different study[6] used an acetone-based etch&rinse two-step adhesive and a resin-modified GIC in young subjects vs. subjects older than 60 years of age. No differences in bond strengths were found for any of the materials for different ages. A more recent study[8] used Single Bond (the unfilled version of SB used in the present study) in dentin from 18 to 22–year-old or 55- to 60–year-old patients. Bonding to older dentin with 30 seconds of etching time resulted in higher bond strength than was achieved when dentin was etched for 15 seconds. However, no statistical differences were found between young and older dentin for the same etching time.[8]

The relatively high bond strengths obtained with the self-etch adhesive EB were somewhat unexpected. A recent research project[19] using the same metholodogy found lower mean bond strengths for EB than were obtained in the present study. The operator-related variability may have, therefore, played an important role. According to the respective manufacturer's literature, the composition of EB is similar to that of SB, except for the presence of methacrylated phosphoric esters in the former.[20] This similarity between the chemical composition of both adhesives may indicate that the bond strengths depend not only on the type of surface treatment prior to applying each material (ie, etching with phosphoric acid with a pH of 0.5 vs the application of an acidic monomer with a pH of 2.4[21]) but also on the way in which the material is applied on the dentin surface. Using transmission electron microscopy (TEM), Mine and others[21] showed a thin dentin hybrid layer as a result of the application of EB, resembling other 'ultra-mild' self-etch adhesives. The same authors suggested that the chemical interaction between the carboxylate groups in EB and the hydroxyapatite crystals available on the mildly decalcified dentin surface might be responsible for the bonding ability of EB.

In the present study, the presence of the copolymer (CP) in EB did not influence mean dentin bond strengths independent of the age group. On the other hand, SB (the commercial version with CP) resulted in significantly higher dentin bond strengths than SBnoCP for all of the three age groups. This apparent paradox may be explained by the concentration of the CP in the two adhesives. While EB contains 1–5%wt of CP, SB contains twice as much CP (Table 1). Therefore, the removal of the CP from SB, as in the SBnoCP group, may be more detrimental to the behavior of this particular adhesive than the removal of CP from the composition of EB, as in the EBnoCP group.

Polycarboxylates used in classical GIC-based materials do not demineralize dentin as deeply as phosphoric acid; however, they result in stable chemical adhesion between COO^- groups and Ca^{2+} groups in hydroxyapatite.[12,13,16] Carboxylate groups replace phosphate ions of the substrate and bond ionically with calcium of hydroxyapatite.[13] This chemical bonding mechanism is explained by the

adhesion-decalcification concept.[10] A different reaction occurs with resin-modified GIC. The self-adhesive bonding mechanism of these materials is twofold: (1) the ionic bonding to hydroxyapatite around collagen, as in conventional GIC, and (2) the micromechanical interlocking for those resin-modified GIC that additionally hybridize dentin.[22] For Vitrebond, the first material to contain the copolymer used in EB and SB, there is no evidence of the second mechanism (ie, hybridization or gel phase deposition).[22] Since the bonding associated with Vitrebond is stable and there is an tight contact between this material and dentin, chemical interaction is the primary bonding mechanism for this resin-modified GIC.[22] A five-year clinical study[23] with a CP-containing resin-modified GIC material reported excellent retention rates. As Mitra and others[16] reported, the CP in Vitrebond bonds chemically to calcium in hydroxyapatite, which supports the idea that the CP in dentin adhesives may also bond chemically to hydroxyapatite. The percentage threshold of CP for effective bonding effectiveness remains to be determined, as it may vary depending on the solvents and other components of each adhesive system. Nevertheless, this chemical adhesion may have somehow contributed to the good clinical performance of etch&rinse CP-containing materials.[24–26] On the other hand, there are not many published clinical studies with EB. A recent study[27] in noncarious cervical lesions reported a 100% retention rate at 12 months, which was not statistically different from that of SB (92.86%).

Taking into account that acid etching removes calcium from the superficial 3–4 μm of intertubular dentin, the interaction of the CP with calcium after etching (as in SB and MP) is somewhat difficult to understand. Using TEM, Van Meerbeek and others[28] showed a deposition of a dark electron-dense amorphous phase on the dentin surface, extending into the dentin tubule orifices and lining the tubule walls when dentin was bonded with Scotchbond Multi-Purpose. As the tubules walls are not completely demineralized, it is quite possible that the chemical bonding to calcium on the tubule walls is partially responsible for the bonding efficacy of both SB and MP. Additionally, the deposition of calcium-carboxylate salts on dentin may be responsible for the moist resistance of bonded interfaces.[29]

The decrease in bond strengths for MP in the >40 age group indicates that this particular material may not bond as well to older dentin. One study[30] compared the bond strengths to caries-affected dentin of MP with and without the polyalkenoate component (CP) in the primer. Removal of CP from the primer lowered the bond strength of MP to caries-affected dentin, which indicates that the amount of residual calcium in caries-affected dentin may be crucial to establishing ionic bonding with the polyalkenoate in the primer. In our study, we used ideal dentin without preexisting caries lesions or old restorations, which does not allow for a direct comparison between the results of both studies.

One of the limitations of the present study is that when data are pooled, as in μTBS testing, the variance is averaged out and information about the variation may be lost. Additionally, the number of beams varied among groups, as a function of the dimensions of the teeth used. Regarding the high number of cohesive fractures in dentin for MP, the ultimate bond strength of the bonding resin (the hydrophobic resin in MP) may have played a role in the dentin bonding ability of the respective adhesive, as shown in previous studies,[31,32] leading to a greater number of cohesive failures.

We have to reject both null hypotheses. Although dentin age was not a significant factor when means were pooled for dentin adhesive, age influenced the bonding ability of MP for the older age group, and the presence of a polyalkenoic acid copolymer (CP) in the composition of SB increased the respective dentin μTBS.

CONCLUSIONS

Within the limitations of an *in vitro* study, the substrate age influenced the bonding ability of the three-step etch&rinse adhesive. The presence of a carboxylic-based polymer (CP) enhanced the bonding ability of the two-step etch&rinse adhesive Adper Single Bond Plus (as in the commercial version), resulting in statistically higher mean μTBS compared to the same formulation without the CP.

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Conflict of Interest

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

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Effect of Chlorhexidine Application on the Long-term Shear Bond Strength to Dentin of a Resin-modified Glass Ionomer

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Clinical Relevance

The application of 0.05% chlorhexidine digluconate after polyalkenoic acid conditioning (Cavity Conditioner®) of dentin and prior to application of resin-modified glass ionomer cement (Fuji II LC®) should be avoided.

SUMMARY

Purpose: **The aim of this study was to investigate the effect of chlorhexidine digluconate (CHX) application on the shear bond strength (SBS) of a resin-modified glass ionomer cement (RMGIC) to polyalkenoic acid–preconditioned dentin after 24 hours, six months, and 12 months of water storage at 37°C.**

Materials and Methods: **Cylindrical molds, placed on flat, polyalkenoic acid (Cavity Conditioner® [*GC*]) preconditioned dentin surfaces of 90 human teeth embedded in resin, were filled with Fuji II LC® (*GC*), a RMGIC, with (n=45) or without (n=45) the prior application of a 0.05% CHX solution. Within each group, SBS was determined after 24 hours (n=15), six months (n=15), and 12 months (n=15) of storage in water at 37°C. The results were analyzed with two-way analysis of variance followed by Tukey multiple means comparisons ($p<0.05$). The type of bond failure (adhesive/cohesive/mixed) was noted and the results were analyzed with chi-square test ($p<0.05$).**

Results: **After 24 hours, the SBS of RMGIC was not significantly different with (9.0 ± 2.8 MPa) or without (8.3±0.6 MPa) the application**

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of CHX. After six months, however, SBS increased significantly in the group without CHX (12.7±3.4 MPa) but remained unchanged in the CHX group (9.4±4.0 MPa). Similar results without CHX (12.6±3.8 MPa) and with CHX (9.5±3.2 MPa) were obtained after 12 months. No significant differences in the type of debonding were found between the various groups tested.

Conclusion: **The application of 0.05% CHX after dentin preconditioning did not seem to have affected the 24-hour SBS of RMGIC. However, the six- and 12-month SBS was significantly lower for CHX-treated samples, possibly as a result of CHX interference with both the bonding mechanism and the maturation reaction of RMGIC.**

INTRODUCTION

The loss of bond strength is the major shortcoming that affects adhesive restorations[1] and decreases their longevity.[2-5] To prolong the clinical lifetime of adhesive restorations, it would be interesting to improve the stability of both the tooth tissue and of the involved adhesive interfaces.

Several authors have shown the hydrolytic degradation of collagen matrices in aged dentin-resin bonds,[6,7] even in the absence of bacterial enzymes,[8] *via* host-derived matrix metalloproteinases (MMPs), a class of zinc- and calcium-dependent endopeptidases,[8,9] responsible for degrading practically all extracellular matrix components of connective tissues. Human dentin contains at least collagenase (MMP-8), gelatinases MMP-2 and MMP-9, and enamelysin MMP-20.[10-13]

Studies[8,14,15] have revealed that chlorhexidine (CHX) could function as a potent MMP inhibitor. With etch-and-rinse adhesive systems, pretreating the cavity with CHX after phosphoric acid-etching may prevent or delay the interfacial degradation of the dentin-resin bond, preserving the bond strength of *in vitro* aged specimens.[15-19] With self-etching adhesives, some studies[17,20-22] showed that CHX was able to diminish the loss of microtensile bond strength over time. However, with a self-adhesive resin cement, CHX seemed to have no effect on bonding durability.[20]

Glass ionomer cements (GICs) and resin-modified glass ionomer cements (RMGICs) are materials that self-adhere to hard tooth tissues.[23] A short polyalkenoic-acid pretreatment is recommended to clean the tooth surface, to remove the smear layer, and to expose collagen fibrils up to approximately 0.5-1-μm depth[24] into which cement components could interdiffuse and establish a micromechanical bond, following the principle of hybridization,[25-28] even if the chemical adhesion mechanism through ionic attraction is preponderant.

Previous studies[29,30] have reported that CHX did not significantly affect the bond strength of RMGIC when CHX was applied before the dentin conditioner as a cavity disinfectant. To our knowledge, no studies have evaluated the effect of CHX applied after dentin conditioning on the bond strength of RMGIC to dentin. The aim of this study was to investigate the *in vitro* effect of 0.05% CHX on the shear bond strength (SBS) of a RMGIC applied to dentin preconditioned with polyacrylic acid after 24 hours, six months, and 12 months of storage in water at 37°C.

The null hypothesis tested was that CHX application after dentin conditioning has no effect on SBS over 24 hours, six months, and 12 months of ageing.

MATERIALS AND METHODS

Ninety freshly extracted human molars were collected, cleaned of soft tissue, stored in a solution of 1% chloramine-T at 4°C, and used within one month. The criteria for tooth selection included absence of cracks caused by extraction forceps as well as absence of decay. The teeth had been extracted for reasons unrelated to the objectives of this study and with the patients' informed consent. The project was approved by the scientific council of the Faculty of Dental Surgery, University of Paris–Descartes. These selected teeth had the greater portion of the roots removed with use of sandpaper (80 grit). The occlusal surface of the crowns was then abraded on water-cooled sandpaper (800 grit) using a polishing machine (Planopol 3, Struers, Kobenhavn, Denmark) to expose a flat dentin surface (>7 mm^2), onto which a cylinder of RMGIC could be formed and bonded. Finally, the residual crowns were embedded in self-cured acrylic resin in plastic cylinders with the flat dentin surface exposed. The flat surfaces were inspected under 40× magnification to ensure that the enamel had been completely removed and the dentin cleared of debris.

For all of the samples (n=90), polyalkenoic acid (Cavity Conditioner® [*GC*]) was applied onto the dentin surface, was left undisturbed for 10 seconds, was rinsed with water for 10 seconds, and was then gentle air-dried for five seconds to leave a moist surface.

Table 1: *Materials, Manufacturers, Batch Numbers, Chemical Composition, and Application Directions*

Materials	Manufacturer	Batch Number	Composition[a]	Application
Cavity Conditioner®	GC, Tokyo, Japan	080581	20% Polyalkenoic acid, 3% aluminum chloride (pH 1.2)	Apply to dentin surfaces and leave undisturbed for 10 s; rinse with water for 10 s; gently air-dry for 5 s, to leave a moist surface.
Fuji II LC®	GC, Tokyo, Japan	0804141	Powder: fluoro-alumino-silicate glass. Liquid: polyalkenoic acid, hydroxyethyl methacrylate (HEMA), dimethacrylate, camphorquinone, water (pH 1.3)	Automatic mixing of capsules for 10 s; application to dentin surfaces; light-curing for 20 s
Chlorhexidine digluconate solution, 0.05%	Gilbert, Hérouville Saint-Clair, France	1001001	Chlorhexidine digluconate	Apply with a microbrush; blot-dry after a dwell time of 60 s

[a] *Manufacturers' data.*

Randomly, half of the samples (n=45) were treated with 0.05% CHX (applied with a microbrush and blot-dried after a dwell time of 60 seconds) prior to bonding, while the other half (n=45) were not. The 0.05% CHX concentration is lower than the 0.2% or 2% typically used, but it is a concentration commonly used in the clinic and sufficient to prevent interface degradation for up to six months.[31]

A cylindrical Teflon mold that allowed us to build cylinders of 2-mm height and a plane base of 3-mm diameter was placed onto the prepared dentine surface. The mold was bulk-filled with Fuji II LC® (GC), which was then light-cured for 30 seconds with a Demetron LC curing light (Kerr Corporation, Orange, CA, USA) with a minimum output of 600 mW/cm^2. After light-curing, the mold was removed and the excess cement, if present, was gently removed from around the base of the RMGIC cylinder with a scalpel. The samples were stored in 37°C water until tested. The materials, their composition, manufacturers, batch numbers, and application details are presented in Table 1.

Subgroups (n=15) of each group were tested for SBS after 24 hours, six months, and 12 months of storage using a universal testing machine (LRX, Lloyd Instruments, Fareham, Hants, UK). For testing, each sample was immobilized in a device provided with a sliding blade acting like a guillotine, thus loading the dentin-RMGIC interface in shear. A cross-head speed of 0.5 mm/min was used. The fractured specimens were observed under a binocular microscope (Olympus Europe SZH10, Hamburg, Germany) at 40× magnification and the fractures were classified as adhesive (failure at the interface between dentin and RMGIC), cohesive (failure in RMGIC), or mixed (involving both adhesive and cohesive failures).

The results of SBS were analyzed by two-way analysis of variance for the factors "dentin treatment" (with CHX vs without CHX) and "ageing period," followed by Tukey post hoc pairwise comparison tests. A chi-square test was used for the analysis of the modes of failure. Statistical significance for all tests was set at $p < 0.05$. Statistical calculations were performed using StatView® Version 5.0 software for Windows (SAS® Institute Inc, Cary, NC, USA).

RESULTS

The results of SBS along with the results of the statistical analysis are summarized in Table 2. The statistical analysis revealed that SBS was significantly influenced by CHX application and time. At 24 hours, the SBS was not significantly different with or without the application of CHX. After six months and 12 months, however, significant differences were identified: in the subgroups without CHX, SBS increased significantly, as compared to

Table 2: *Means and Standard Deviations of Shear Bond Strengths (SBS) (in MPa) for the Various Groups Tested*[a]

Groups of RMGIC	SBS Without CHX	SBS With CHX
24 h	8.3 ± 0.6 A	9.0 ± 2.8 A
6 mo	12.7 ± 3.4 B	9.4 ± 4.0 A
12 mo	12.6 ± 3.8 B	9.5 ± 3.2 A

Abbreviations: CHX, chlorhexidine; RMGIC, resin-modified glass ionomer cement Fuji II LC®.
[a] Values with the same online small capital letter are not significantly different at p=*0.05.*

24 hours, and it was higher than in the subgroups with CHX; within the CHX subgroups, SBS after six months and 12 months was not significantly different from that after 24 hours.

The results of the failure mode along with the results of the statistical analysis are summarized in Table 3. Fracture mode analysis did not demonstrate any statistically significant differences between or within the groups at any time point.

Table 3: *Mode of Fractures for the Various Groups Tested*[a]

Groups of RMGIC	Number of Samples	Adhesive, No. (%)	Mixed, No. (%)	Cohesive, No. (%)
Without CHX	45	26 (58)	17 (38)	2 (4)
24 h	15 A	9 (60)	5 (33)	1 (7)
6 mo	15 A	9 (60)	6 (40)	0 (0)
12 mo	15 A	8 (53)	6 (40)	1 (7)
With CHX	45	28 (62)	15 (34)	2 (4)
24 h	15 A	9 (60)	5 (33)	1 (7)
6 mo	15 A	9 (60)	5 (33)	1 (7)
12 mo	15 A	10 (67)	5 (33)	0 (0)

Abbreviations: CHX, chlorhexidine; RMGIC, resin-modified glass ionomer cement Fuji II LC®.
[a] Values with the same online small capital letter are not significantly different at p=*0.05.*

DISCUSSION

In the first part of this *in vitro* work we investigated whether the use of 0.05% CHX interfered with the SBS of a RMGIC to dentin after 24 hours of storage (early/short term) in water at 37°C. Under the conditions of the study, the magnitude of early SBS values without CHX (8.3 MPa) was close to those obtained in the literature with similar adhesion tests[32-34] and with adhesive failures being predominant. The presence of CHX did not affect the early SBS (9.0 MPa). This finding has also been observed with etch-and-rinse and self-etch adhesives as well as with self-adhesive resin cements[18,20,35-39] used for direct or indirect adhesive restorations. However, adverse effects of CHX on early bond strength have also been reported,[22] and they were attributed to the water content of the 0.05% CHX solution. The authors speculated that after the application of this solution, residual moisture might diminish the adhesive properties of some adhesive systems. In the current study, the air-drying of the dentin surface after CHX application and the hydrophilicity of the RMGIC may explain why such adverse effects were not observed.

In the second part of this *in vitro* work we investigated whether the use of 0.05% CHX interfered with the long-term (six months and 12 months of storage in water at 37°C) SBS of a RMGIC. In the samples with no CHX, after six months a significant increase in SBS, from 8.3 MPa to 12.7 MPa, was determined. After 12 months, SBS was maintained at 12.7 MPa, with predominant adhesive failures as well. These results confirm those of previous studies that observed an increase in bond strength with time, concomitant with the RMGIC maturation.[40] The results are also consistent with studies[41-44] that have indicated that the cross-link density in RMGIC increases as a result of the slow diffusion of calcium and aluminum ions and that this phenomenon is responsible for the increase in compressive strength with ageing.

Mount[45] described an ion-enriched layer, formed by the displacement of calcium and phosphate ions from apatite by the carboxyl group of glass ionomer liquid, and re-precipitation of ions at the cement-tooth interface with the setting of glass ionomer material. Mitra and others[46] also reported an amorphous zone, which resembles a hybrid layer and is thought to be a reaction product of the RMGIC with inorganic material from dentin, described as a diffusion-based adhesion. Moreover, it may be noted that RMGICs involve a chemical bonding by ionic

interactions of the carboxyl groups of the polyalkenoic acid with calcium ions of hydroxyapatite attached to collagen fibrils.[23,46] It has been shown that this chemical bonding contributes to the excellent long-term adhesion, microleakage resistance, and dentin sealing ability of these materials. Furthermore, the combination of chemical adhesion and micro-mechanical retention may be beneficial to the long-term resistance to hydrolytic degradation of this hybrid layer.

The CHX-treated group, however, showed no increase in SBS after six months or 12 months of ageing. The results obtained indicate that the presence of CHX impeded the increase in SBS observed in the non-CHX group, and, therefore, the null hypothesis was rejected. This phenomenon has not been reported before, and it may be explained as follows: CHX, which has strong cationic properties,[47] could react with the anionic carboxyl groups of the RMGIC, thus impeding the formation of carboxyl-calcium linkages and therefore reducing the dentin bonding capability of RMGIC. The interference of CHX with the chemical adhesion mechanism of RMGIC could decrease the resistance to hydrolytic degradation of the hybrid layer. Furthermore, CHX may also interfere with the second step of the setting reaction of RMGIC by competing with aluminum ions for the carboxyl groups, thus perturbing the maturation reaction of RMGIC.

The long-term negative effect of CHX on the SBS of the RMGIC investigated does not follow the results obtained by numerous studies for resin composite and etch-and-rinse adhesive systems, which showed that CHX has the potential to minimize the reductions in the resin-dentin bond strengths commonly observed after long-term water storage.

Finally, the current results are in line with the findings of a study[48] that reported a decrease in the physical properties of RMGIC combined with CHX. Future studies with alternative MMP inhibitors that cannot interfere with the bonding or maturation mechanisms of RMGIC should be conducted to investigate if their presence can affect the long-term performance of RMGIC.

CONCLUSIONS

Under the conditions of this *in vitro* study, the results have shown that the 24-hour SBS of the tested RMGIC to dentin was not affected by CHX; at six and 12 months, however, the CHX-treated samples exhibited lower SBS, which might have been caused by an interference of CHX with both the bonding mechanism and the maturation reaction of the cement.

Finally, the present findings should be interpreted with caution, as the results were obtained under laboratory conditions, and it remains to be seen whether or not CHX could inhibit the *in vivo* degradation of the hybrid layer at the dentine-RMGIC interface by endogenous MMPs.

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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Cuspal Deflection in Premolar Teeth Restored Using Current Composite Resins With and Without Resin-modified Glass Ionomer Liner

E Karaman • G Ozgunaltay

Clinical relevance

Using a recently introduced silorane-based composite resin, Filtek Silorane, and the placement of resin modified glass ionomer cement liner under the composite resin restorations resulted in reduced cuspal deflection.

SUMMARY

Aim: To evaluate the effects of four different types of composite resins and a resin modified glass ionomer cement (RMGIC) liner on the cuspal deflection of large MOD cavities in vitro. Materials & Methods: One hundred twenty-eight extracted human upper premolar teeth were used. After the teeth were divided into eight groups (n=16), standardized large MOD cavities were prepared. The distance between cusp tips was measured before and after the cavity preparations with a digital micrometer. Then the teeth were restored with different resin composites (Filtek Supreme XT, Filtek P60, Filtek Z250, Filtek Silorane - 3M ESPE, St Paul, MN, USA) with and without a RMGIC liner (Vitrebond, 3M ESPE, St Paul, MN, USA). Cuspal deflection was measured 5 min, 24 h, and 48 h after the completion of restorations. The data were statistically analyzed with Friedman and Kruskal Wallis tests. Results: A significant reduction in cuspal deflection was observed in Filtek Silorane restorations with and without RMGIC liner ($p<0.05$). In all restored teeth, the distance between cusps was reduced but they did not return to their original positions during the 48 h period. All teeth showed cuspal deflection,

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but placement of RMGIC liner reduced it. Conclusion: The use of silorane-based composites and the placement of RMGIC liner under the composite resin restorations resulted in significantly reduced cuspal deflection.

INTRODUCTION

The increasing demands for esthetic restorations and public concerns related to mercury in dental amalgams have produced increased interest in composite resin as an alternative posterior restorative. The mechanical performance, wear resistance, and esthetic potential of composite resins have significantly improved over the last few years. However, polymerization shrinkage of composite resins remains a challenge and still imposes limitations in the application of direct techniques.[1]

Polymerization shrinkage can produce two types of problems. When the filling material is weakly adhered to the dental tissues, detachment of the enamel margins can occur and/or gaps can form, resulting in marginal microleakage that allows the passage of bacteria, fluids, molecules, or ions between the cavity surface and the composite resin. In contrast, if the adhesive strength exceeds the contraction stress, there is no detachment but the restoration maintains an internal tension that pulls the cavity walls together, reducing the intercuspal distance (cuspal deflection). Cuspal deflection may cause changes in occlusion points, postoperative pain, and, in some cases, tooth fractures.[2]

To avoid these shortcomings and to make tooth-colored restorations not only esthetic but also functional, many techniques and new materials have been introduced. Recently, as the result of increasing demand for a universal restorative material indicated for all types of direct restorations, including posterior teeth, a new category of resin composite was developed, nanofilled composites. Nanocomposites show high translucency, high polish, and polish retention similar to those of microfilled composites while maintaining physical properties and wear resistance equivalent to those of several hybrid composites.[3]

During the late 1990s manufacturers introduced packable composites with high inorganic filler loading into the market as alternatives to amalgam. Packable composites use amalgam techniques for placement and produce acceptable interproximal contact. These allow these composites to be safely and successfully used in Class II restorations.[4]

To overcome the polymerization shrinkage problem, extensive efforts have been made over the years to develop low-shrinkage composite resins. Some modern developments in dental composite research have focused on the use of ring-opening systems like oxirane-based resins cured under visible light conditions. Weinmann and others[5] described the synthesis of a new monomer system, named silorane, obtained from the reaction of oxirane and siloxane molecules. The novel silorane-based resin was claimed to have combined the two key advantages of the individual components: low polymerization shrinkage due to the ring-opening oxirane monomer and increased hydrophobicity due to the presence of the siloxane species.

The use of resin-modified glass ionomer (RMGIC) liners to decrease the amount of polymerization shrinkage of large composite restorations and cuspal deflection is a controversial issue in dentistry. Cara and others[6] suggested that using RMGIC liners reduces cuspal deflection, while Taha and others[7] reported similar cuspal deflection regardless of whether a RMGIC liner was placed. As a result, the current study aimed to assess cuspal deflection with and without a RMGIC liner for the restoration of large mesio-occlusal-distal MOD cavities with four different composite resins. The research hypothesis of the study was that using silorane-based composites and the placement of RMGIC liner under the composite resin restorations would result in reduced cuspal deflection.

MATERIALS AND METHODS

Selection of Teeth

One hundred twenty-eight upper premolar teeth, extracted for orthodontic purposes, that on visual examination were free from caries, hypoplastic defects, and cracks were selected. Calculus deposits were carefully removed using a hand scaler. The teeth had been stored in distilled water for a maximum of three months prior to use. The maximum bucco-palatal width (BPW) of each tooth was measured with a digital micrometer gauge (Series 480–505, resolution 1 μm, SHAN™; Precision Measuring Instruments, Guilin, China). The BPW dimensions were used to divide the teeth into eight groups of 16 teeth, and the mean BPW of the teeth between groups varied by no more than 5% according to one-way analysis of variance and a paired Tukey test comparison (Table 1). The teeth were stored in water at room temperature (23°C±1°C) except when aspects of the experimental procedure required isolation from moisture.

Table 1: *Bucco-palatal Width (BPW) Dimensions of Teeth (μm) Highlighting No Statistical Differences Between Groups (n=16)*

Groups	Mean	Standard Deviation
1 (Filtek Supreme XT)	9.85	0.41
2 (Filtek P60)	9.88	0.37
3 (Filtek Z250)	9.90	0.35
4 (Filtek Silorane)	9.87	0.42
5 (Vitrebond + Filtek Supreme XT)	9.82	0.35
6 (Vitrebond + Filtek P60)	9.92	0.35
7(Vitrebond + Filtek Z250)	9.85	0.35
8 (Vitrebond + Filtek Silorane)	9.94	0.35

Cavity Preparation

The teeth were embedded with crown uppermost and long axis vertical so that the resin extended to within 2 mm of the cementoenamel junction (CEJ) in a plastic ring with acrylic resin (Ortocryl EQ, Dentautum, Germany). Buccal and palatal cusp tips of each tooth were acid etched for 30 seconds, washed for 20 seconds, and dried. Adper Single Bond 2 was applied according to the manufacturer's recommendations and light-cured with light-emitting diode (Radi Plus, SDI, Victoria, Australia) for 10 seconds. The curing light intensity was measured with a radiometer (Curing Radiometer Model 100; Demetron Research Corp, Danbury, CT, USA). Then flowable composite (Filtek Flow, 3M ESPE, St Paul, MN, USA) was applied to the buccal and palatal cusp tips as reference balls for intercuspal distance measurements, followed by light-curing for 20 seconds (Figure 1). After one week, the distance between reference balls of each tooth was measured using a digital micrometer and recorded as "initial distance" (Figure 2).

Standardized large MOD cavities were prepared with boxes two-thirds the BPW of the tooth, and the occlusal isthmus was prepared to half of the BPW. The cavity depth at the occlusal isthmus was also standardized to 3.5 mm from the tip of the palatal cusp and 1 mm above the CEJ at the cervical aspect of the proximal boxes (Figure 3). The cavosurface

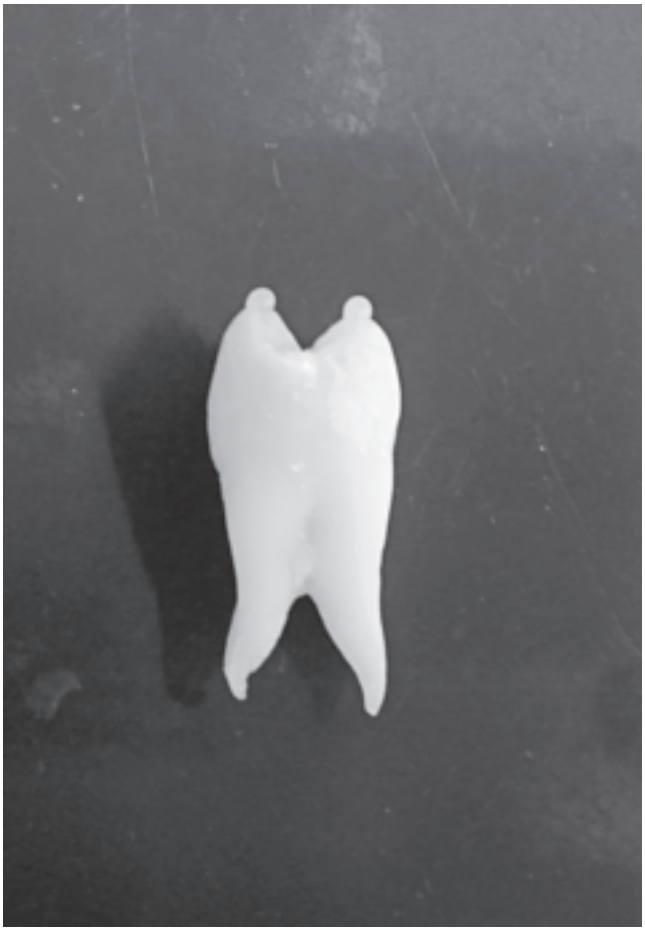

Figure 1. *Specimen with reference points for intercuspal distance measurements.*

margins were prepared at 90°, and all internal line angles were rounded. The facial and lingual walls of the cavity were also prepared parallel to each other in accordance with a previously reported procedure.[6,8] Diamond fissure burs (DIATECH, Swiss Dental, Heerbrugg, Switzerland) were used in a high-speed handpiece with water coolant and changed for every five cavity preparations.

Restorative Procedures

The materials used in this study are listed in Table 2. All of the teeth were restored with the same manufacturer's composite resin and its associated bonding system in accordance with the manufacturer's instructions.

Group 1—Etching of enamel and dentin was performed with 35% phosphoric acid for 30 seconds, followed by rinsing with air-water spray for 20 seconds. Enamel surfaces of the cavity were dried with compressed air and dentin surfaces were dried with cotton pledgets. Two consecutive coats of Adper

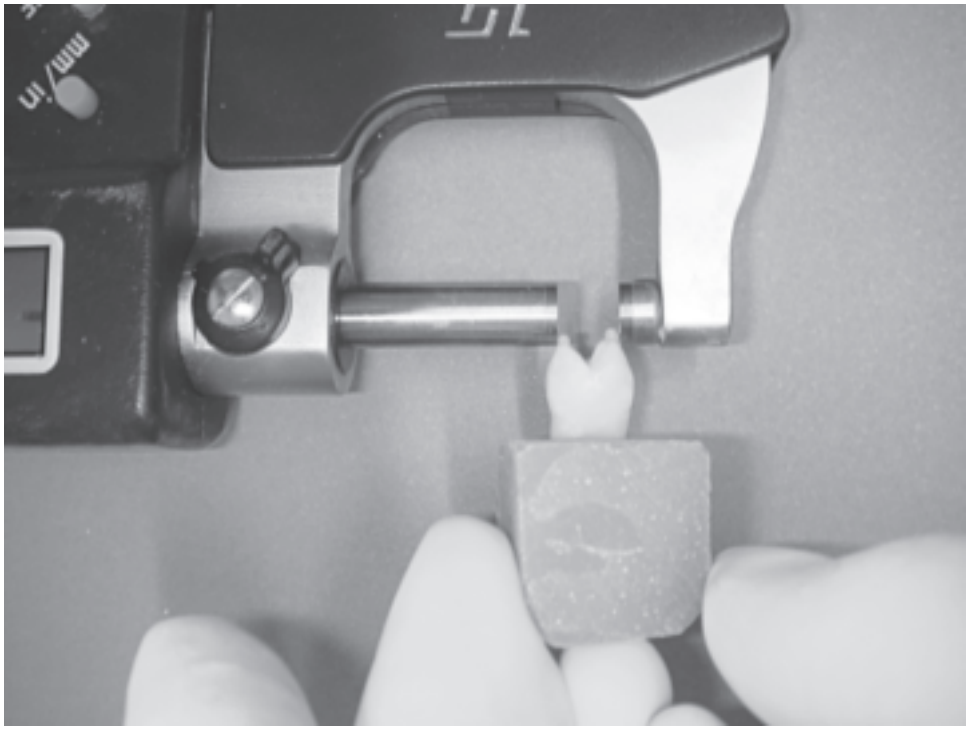

Figure 2. *Measuring the distance between cusp tips.*

Single Bond 2 were applied using a microbrush for 15 seconds, followed by gentle air-drying and then light-curing for 10 seconds. Filtek Supreme XT (Shade A3B) was placed and light-cured for 20 seconds.

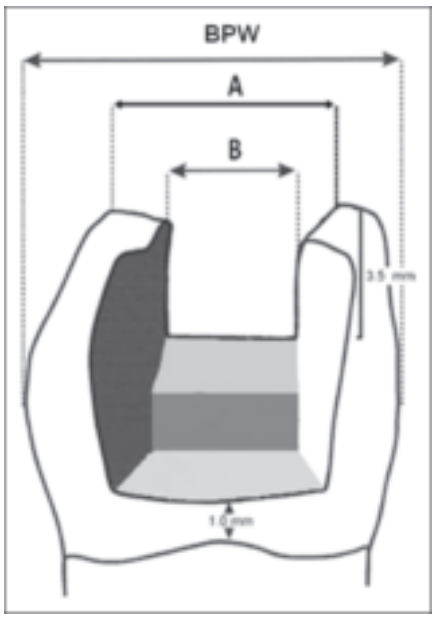

Figure 3. *Schematic view of MOD cavities.*

Group 2—Teeth were restored with Filtek P60 (Shade A3) as previously described.

Group 3—Teeth were restored with Filtek Z250 (Shade A3) as previously described.

Group 4—Silorane Adhesive System primer was applied using a microbrush for 15 seconds, followed by gentle air-drying and then light-curing for 10 seconds. After that the Silorane Adhesive System bond was applied, followed by a gentle stream of air, and light-cured for 10 seconds. Filtek Silorane (Shade A3) was placed and light-cured for 20 seconds.

Group 5—Teeth were lined with a thin layer of Vitrebond on the pulpal and axial walls with approximately 1-mm thickness and light-cured for 30 seconds. Then the teeth were restored with Filtek Supreme XT (Shade A3B) using the same method as for group 1.

Group 6—Teeth were restored with Filtek P60 (Shade A3) using the same method as for group 5.

Group 7—Teeth were restored with Filtek Z250 (Shade A3) using the same method as for group 5.

Group 8—Vitrebond was applied as previously described and teeth were restored with Filtek Silorane (Shade A3) using the same method as for group 4.

Table 2: *Materials Used in the Current Study(3M ESPE is manufacturer for all materials)*

Product (Batch No.)	Material	Ingredient
Filtek Supreme XT (20080117)	Nanofilled composite resin	Inorganic fillers (59.5%), Bis-GMA, UDMA, Bis-EMA, TEGDMA, silica nanofillers (5–7 nm) zirconia/silica nanoclusters (0.6-1.4 μm)
Filtek P60 (20081004)	Packable composite resin	Inorganic fillers (61%), Bis-GMA, UDMA, Bis-EMA, zirconia/silica nanofillers (0.01–3.5 μm)
Filtek Z250 (20090406)	Universal hybrid composite resin	Inorganic fillers (60%), Bis-GMA, UDMA, Bis-EMA, zirconia/silica nanofillers (0.01–3.5 μm)
Filtek Silorane (N105399)	Low shrink composite resin	Inorganic fillers (55%), hydrophobic resin matrix
Scotchbond (20071207)	Acid	Aqueous solution of 35% phosphoric acid
Adper Single Bond 2 (7MX)	Etch & rinse adhesive	HEMA, Bis-GMA, dimethacrylate, polyacrylic and polyitaconic acids, water, ethanol
Silorane System Adhesive (20081117)	Primer	HEMA, Bis-GMA, water, ethanol, phosphoric acid–methacryloxy-hexyl ester, 1,6-hexanediol dimethacrylate, acrylic and itaconic acid copolymer, (dimethylamino) ethyl methacrylate, DL-camphorquinone, phosphine oxide
Silorane System Adhesive (20081117)	Bonding agent	Substitute dimethacrylate, TEGDMA, phosphoric acid–methacryloxy-hexyl ester, DL-camphorquinone, hexanediol dimethacrylate
Vitrebond (20090521)	Resin-modified glass ionomer	Fluoramino silicate glass, polyalkenoic acid

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

Eight nominally triangular increments of approximately 2-mm thickness were used to restore the teeth, three for each proximal box and two for the occlusal surface.[6,8,9] Each increment was cured for 20 seconds, per the manufacturer's instructions. The occlusal aspect of the restorations was carved to approximate the normal occlusal anatomy of an upper premolar tooth. Each tooth was restored by placing a transparent matrix (Auto matrix II, combination matrix intro-kit, Dentsply, Petrópolis, Brazil), which was removed after the restorations were completed. Between measurements on subsequent days teeth were stored under the same wet conditions (distilled water) at room temperature (23°C±1°C).

The distance between reference points was measured with a digital micrometer[2,10–12] five minutes, 24 hours, and 48 hours after restorations were completed to determine the stress relaxation in the cusps.[11,13] All measurements were made by the same operator and three measurements were recorded for each tooth, and the mean was used for the subsequent statistical analysis. The cuspal deflection was obtained by calculating the difference between "initial" and the other measurements at five minutes, 24 hours, and 48 hours.

Statistical Analysis

Since the data did not show a homogeneous distribution, the global comparison among the study groups for the different measurements was carried out using the Kruskal-Wallis test. The data were subjected to Friedman test to determine significant changes in cuspal deflection of each group with time. Statistical significance was set in advance at the 0.05 confidence level. All data were analyzed using SPSS 11.5 for Windows software (SPSS Inc, Chicago, IL, USA).

Table 3: *Mean Cuspal Deflection Measurements and Standard Deviations (SDs) for Each Group Examined in the Current Study (n=16)*[a]

Groups	5 Minutes	24 Hours	48 Hours
1 (Filtek Supreme XT)	21.7 ± 12.6 $_{A}^{1}$	17.5 ± 17 $_{A}^{1}$	9.8 ± 8.2 $_{A}^{2}$
2 (Filtek P60)	30.5 ± 22.8 $_{A}^{1}$	20.5 ± 23.3 $_{A}^{2}$	17.3 ± 14 $_{A}^{2}$
3 (Filtek Z250)	14.9 ± 3.6 $_{B}^{1}$	11 ± 7.6 $_{B}^{1}$	9 ± 4.1 $_{B}^{1}$
4 (Filtek Silorane)	0.2 ± 0.5 $_{C}^{1}$	0.3 ± 0.6 $_{C}^{1}$	0.3 ± 0.6 $_{C}^{1}$
5 (Vitrebond + Filtek Supreme XT)	14.2 ± 5.9 $_{B,D}^{1}$	11 ± 6.9 $_{B,D}^{1}$	8.8 ± 7.4 $_{B,D}^{1}$
6 (Vitrebond + Filtek P60)	13.3 ± 6.5 $_{B,D}^{1}$	11.3 ± 7.1 $_{B,D}^{1}$	10 ± 15.4 $_{B,D}^{1}$
7 (Vitrebond + Filtek Z250)	9.5 ± 5.5 $_{D}^{1}$	9.3 ± 5.3 $_{D}^{1}$	8.4 ± 7.3 $_{D}^{1}$
8 (Vitrebond + Filtek Silorane)	0.1 ± 0.4 $_{C}^{1}$	0.1 ± 0.4 $_{C}^{1}$	0.1 ± 0.4 $_{C}^{1}$

[a] *Mean values exhibiting different letters (within columns) and different superscripted numbers (within rows) are significantly different.*

RESULTS

No significant differences were identified between the groups when the mean cuspal deflection after cavity preparation of the teeth was compared ($p=0.807$). Cavity preparation produced approximately 9.68 μm of cuspal deflection in all groups.

The mean cuspal deflection and standard deviations for each group are shown in Table 3. The cuspal deflection was greatest in the group restored with Filtek P60 without Vitrebond (group 2) and least in the group restored with Filtek Silorane and Vitrebond (group 8) at all measurements after restorations were completed.

At five-minute measurements, differences between cuspal deflection were statistically significant for all groups except groups 1 and 2; groups 3, 5, and 6; groups 4 and 8; and group 6 and 3, 5, and 7 ($p<0.05$). RMGIC (Vitrebond) liner usage produced a statistically significant reduction in cuspal deflection for the groups restored with the same composite resins (groups 1–5, groups 2–6, groups 3–7), except for the groups restored with Filtek Silorane (groups 4–8) ($p<0.001$).

At 24-hour and 48-hour measurements, groups restored with Filtek Silorane showed less cuspal deflection than did the other groups, and the differences were statistically significant ($p<0.001$). Using Vitrebond with composite resins influenced cuspal deflection; greater mean cuspal deflection was detected for all groups restored without Vitrebond, although no significant difference was revealed except for the 48-hour measurement of groups restored with Filtek P60 (groups 2–6).

The intercuspal distance increased in all experimental groups during the 48 hours, but the cusps did not fully achieve their original dimensions in any of the groups.

DISCUSSION

The magnitude of cuspal deflection depends on many factors, including the size and configuration of the cavity and the properties of the restorative material and the bonding system.[8,14] Consequently, in addition to the standardization of cavity sizes, the incremental packing of the composite resins and the application of the associated adhesive systems were carefully performed by one operator in each cavity in the current study.

The preparations in the current study were large MOD cavities, and the geometry of the cavity preparations resulted in a high C-factor. The preparations were designed to weaken the remaining tooth structure, in order to maximize possible cuspal movement during restoration, and to provide a realistic *in vitro* simulation of the clinical situation. However, these cavities could be considered typical of large amalgam replacement cavities, and the number of such restorations currently placed in

clinical practice is increasing since improved matrix and bonding systems have made the use of composite resin restorations more viable.[15]

Many methods have been used to measure cuspal deflection, including microscopy,[13] strain gauges,[16] direct current differential transformers,[9] linear variable differential transformers,[17] and digital micrometers.[2,11,12] In the current study a digital micrometer was used for cuspal deflection measurements.

Polymerization shrinkage of composite resins resulted in an inward deflection of cusps for all experimental groups evaluated, in agreement with the findings of previous reports.[9,13,18] Nevertheless, values of cuspal deflection in the present study were different than other reported values, probably because of differences in experimental design. The methods utilized in the current study during placement of the composite resins replicated those commonly used in clinical practice.

The results of our study showed that cuspal deflection was lower in the teeth restored with silorane-based composite resin than in the teeth restored with methacrylate-based composite resins. In agreement with this, Laughlin and Sakaguchi[19] found significantly lower cuspal deflection when the teeth were restored with experimental silorane-based composite resin compared to those restored with Filtek Z250 (methacrylate-based composite resin). The results of our study also agree with those reported by Palin and others,[8] who found that the use of oxirane- and silorane-based composite resins reduced cuspal deflection. The increase in cuspal deflection of cavities restored with methacrylate-based composite resins may have been expected as a result of the differences in polymerization reaction between the free-radical and cationic species, respectively.

The difference between cuspal deflection measurements of methacrylate-based composite resins applied without RMGIC liner were statistically similar, except for the measurement five minutes after the restorations were finished. This result can be explained by the similar filler loading and resin constituents of the composites used in the study (Table 2).

The cuspal deflection generated by Filtek P60 was greatest at all measurement times. Fleming and others[20] and Cara and others[6] have also reported that Filtek P60 caused higher cuspal deflection than Filtek Z250 and Filtek Supreme, respectively.

In this study the placement of RMGIC liner reduced the amount of cuspal deflection, which is in accordance with the findings reported by Alomari and others[13] and McCullock and Smith.[21] The RMGIC used in this study (Vitrebond) had a modulus of elasticity of 1.1 GPa and a volumetric polymerization shrinkage of 2.3 vol%.[22] The volumetric polymerization shrinkage and the elastic modulus have opposite effects on the total stress on the tooth structure and eventually on cusp deflection. A material with low elastic modulus, particularly when placed in posterior regions, will result in a higher deformation under masticatory stresses, potentially resulting in more catastrophic failures as a consequence.[23] Low cuspal deflection with RMGIC usage can be explained in this way.

Comparisons among the measurements of cuspal deflection at five minutes, 24 hours, and 48 hours showed that all restored teeth tended to recover their original position, although none of them fully recovered during the 48-hour period. It has been reported that cusps recover their original position after inward deflection because of shrinkage of composite restorations and that this recovery is strongly influenced by tooth hydration conditions and cavity size. However, some studies[18,24] have shown that the total and near-total recovery of the initial intercuspal distance is a slow process that may last up to two weeks and is never complete in medium-sized and large restorations. Teeth restored with methacrylate-based composites had greater recovery than those restored with silorane-based composites. This may be due to the differences between the water sorption properties, saturation time, and capacity of the composite resins used in this study. Palin and others[25] reported that water sorption of silorane-based composite resins is lower than that of methacrylate-based composite resins.

CONCLUSION

Within the limitations of this *in vitro* study the research hypothesis was accepted: the use of silorane-based composites and the placement of RMGIC liner under the composite resin restorations resulted in significantly reduced cuspal deflection.

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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The Use of Bur and Laser for Root Caries Treatment: A Comparative Study

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Clinical Relevance

The marginal seal of composite restorations placed in cavities prepared using an Er,Cr:YSGG laser is not satisfactory as a result of the presence of irregularities on the edge of the cavities and the difficulties associated with leaving a substrate free of caries.

Summary

This research analyzed the influence of bur and erbium, chromium:yttrium-scandium-gallium-garnet (Er,Cr:YSGG) laser caries removal on cavity characteristics and marginal seal of composite resin restorations. One hundred and forty human dental root samples were used. After *in vitro* root caries induction using *Streptococcus mutans*, the carious lesions were removed either by a conventional technique using burs (G1=control) or by using an Er,Cr:YSGG laser (λ=2.78 μm, 20 Hz, pulse duration≅140 μs, noncontact mode using a 600-μm tip) with the following power outputs: G2: 1.0 W; G3: 1.25 W; G4: 1.5 W; G5: 1.75 W; G6: 2.0 W; G7: 2.25 W; G8: 2.5 W; G9: 2.75 W; G10: 3.0 W; G11: 3.25 W; G12: 3.5 W; G13: 3.75 W; and G14: 4.0 W. Samples in the 14 groups (n=10) were conditioned with Clearfil SE Bond and restored with a flowable composite. They were then thermocycled (1000 cycles) and immersed into a 2% methylene blue solution for microleakage analysis. The data were statistically compared (analysis of variance or Spearman correlation tests; $p \leq 0.05$). The lased groups showed significantly greater microleakage indexes, cavity depths, and presence of residual caries than did those of the control group. There was a strong positive correlation between residual caries and microleakage. The results indicate that Er,Cr:YSGG laser irradiation is not a good alternative to the use of burs for root caries removal since it may cause a significant loss of marginal sealing in composite resin restorations.

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INTRODUCTION

Treatment of dental root caries is challenging, especially nowadays, when the elderly population is

significantly increased worldwide.[1] To improve root caries removal procedures and the longevity of restorations in the elderly population, research is required to develop more comfortable and efficacious clinical procedures.

Root caries may occur when root surfaces become exposed to the oral environment. This exposure can be due to periodontal diseases, mechanical injury, surgical treatment, or a combination of these factors.[2]

The elderly population is at high risk for root caries as a result of health and motor deficiencies that can lead to xerostomia and poor capacity for mouth cleansing. Some clinical situations, such as the presence of deep carious lesions and/or pulpal sensitivity, demand restorative procedures. Research in the elderly population is focused on finding a method with which to remove diseased dental hard tissue without the negative stimuli associated with conventional caries removal techniques.[3]

Currently, the most common method for removing deep root caries lesions is the use of spherical burs in low-speed handpieces.[4] This conventional technique for caries treatment is invasive and has some disadvantages, such as the necessity for local anesthesia during clinical procedures and production of noise and vibration that can be inconvenient for patients. For these reasons, new methods of caries management are being developed, such as laser treatment, particularly using erbium lasers.[5,6]

Since the introduction of the first ruby laser in 1965, many wavelengths have been investigated with regard to their clinical application in dentistry.[7,8] In the past, use of CO_2, Nd:YAG, and ruby lasers for cavity preparation and caries removal produced unsatisfactory results, which included destruction of enamel and dentin as well as increases in pulpal temperature to critical levels.[9] In the late 1980s, desirable results were obtained by using different wavelengths. A study[10] showed that tooth structure could be removed by the Er:YAG wavelength without causing any measurable degree of thermal damage. Furthermore, this study showed that thermal damage to enamel and dentin was minimal when proper settings and an adequate water cooling spray were used.[10]

In the last 10 years, two wavelengths have been developed for clinical use on hard tissues. These include the Er:YAG (2.94 μm) and the erbium, chromium:yttrium-scandium-gallium-garnet (Er,Cr:YSGG) (2.78 μm), which have very similar properties according to many scientific accounts. These two wavelengths constitute the erbium family of lasers. Preliminary studies[5,11] investigating the safety and efficacy of using the Er,Cr:YSGG wavelength found it to be a precise tool for dental hard tissues. Furthermore, it was considered more comfortable for patients than the conventional bur method for caries removal, because less anesthesia or no anesthesia was required during clinical procedures and because the technique presented low noise and no pressure or vibrations on the tooth structure.[5,12] The mechanism of dentin removal by this laser involves a thermomechanical process in which the emission laser light is absorbed by the water within the hydroxyapatite of the dental hard tissue.[13] The water is then heated and evaporated, resulting in high-pressure steam that causes a microexplosion of tooth tissue below the melting point of tooth tissue (approximately 1200°C).[14] The surface irregularities of ablated dentin are comparable to those of the dentin surface after acid etching. This may promote micromechanical interlocking between dental restorative materials and the tooth surface. For this reason, several studies[6,15-19] have aimed to evaluate the adhesion of composites to lased dentin, but the data obtained in these studies are conflicting and inconclusive.

Considering that the Er,Cr:YSGG laser has been advocated especially for preparation of microcavities in light of minimal invasive dentistry[12,20] and given that laser treatment is more comfortable for patients, the efficacy of this caries removal method remains to be clarified. Therefore, the aim of this study was to analyze the efficiency of Er,Cr:YSGG laser irradiation for root caries removal by observation of the macromorphological characteristics of the cavity and microleakage of composite resin restorations.

MATERIALS AND METHODS

Selection of Teeth

Seventy human teeth (molars and premolars) extracted as a result of periodontal disease were used. After cleansing and root planing using a Gracey curette, the teeth were stored in distilled water under refrigeration (4°C). This study was approved by the University of São Paulo School of Dentistry Ethical Committee.

Tooth Preparation

The dental roots were separated from the crowns at the cement-enamel junction using a sectioning machine (Labcut Model 1010; Extec, Enfield, CT,

Table 1: *Laser Conditions for Experimental Groups*

Groups	Repetition Rate, Hz	Power Output, W	Fluence, J/cm^2
2	20	1	17.85
3	20	1.25	22.31
4	20	1.5	26.78
5	20	1.75	31.24
6	20	2	35.7
7	20	2.25	40.16
8	20	2.5	44.63
9	20	2.75	49.09
10	20	3	53.55
11	20	3.25	58.01
12	20	3.5	62.48
13	20	3.75	66.94
14	20	4	71.4

USA) with a diamond disk (Buehler Ltd, Lake Bluff, IL, USA) at low speed. One hundred and forty dentin blocks (5 × 5 mm) were obtained from the dental roots using the same equipment. The dentin blocks were embedded into acrylic resin (Jet; Classico, São Paulo-SP, Brazil), leaving a 3 × 3-mm root surface–exposed window. Samples were individually placed into 24-well cell culture plates and sterilized by gamma radiation (25 KGY).[21]

For the cariogenic challenge, *Streptococcus mutans* (ATCC 25175) grown repeatedly in sucrose medium was used. The samples in the sterile cell culture plates were coated with 1.5 mL of Brain Heart Infusion broth supplemented with 5% sucrose (BHI-S; Difco, Sparks, MD, USA), inoculated overnight with standardized cultures (≅8.8×10^7 colony-forming units [CFU]/mL) in the same medium, and incubated for 24 hours at 37°C in an atmosphere containing 10% CO_2. The sterile BHI-S medium was changed at 24-hour intervals during the first four experimental days and then every other day during the next 20 days. All incubations were carried out as previously described.[22,23] After incubation, the root dentin fragments showed macroscopic alterations similar to those of root caries. These samples were then used for experiments.

Experimental Groups

The samples (n=140) were randomly divided into 14 groups (n=10 per group) according to the technique used for caries removal, as follows: group 1 (control): caries removal carried out with a conventional technique using spherical carbide burs in a low-speed handpiece. Groups 2 to 14: caries removal carried out with Er,Cr:YSGG laser irradiation. An Er,Cr:YSGG laser (Waterlase; BioLase Technology Inc, San Clemente, CA, USA) was used at a wavelength of 2.78 μm with a pulse duration of ≅140 μs and a repetition rate of 20 Hz. The average power output of this laser could be varied from 0.0 to 6.0 W. The laser energy was delivered through a fiber-optic system to a sapphire tip (terminal diameter, 600 μm) that was bathed in an adjustable air/water spray. In this study, the power output was set between 1.0 W and 4.0 W at 0.25-W intervals, yielding an energy density ranging from 17.85 to 71.4 J/cm^2, and the air/water spray was adjusted to 55%/65%. Groups were treated as shown in Table 1.

In all groups, caries removal was considered complete when no carious tissue was noted in the cavity walls and at the bottom of the cavity on visual and probe examination. The removal of carious lesions determined the form of the cavity.

Cavity Restoration

The cavities were treated with a self-etch adhesive system (Clearfil SE Bond; batch numbers: primer, 005438; bond, 007234; Kuraray, Osaka, Japan) according to the manufacturer's instructions. The cavities were restored with a flowable composite resin (Palfique Estelite LV; color A3; batch number J244J; Morita Inc, Irvine, CA, USA) in two increments. Each increment of composite resin was light-cured for 30 seconds using an XL 3000 halogen curing light (3M ESPE, St Paul, MN, USA). After 24 hours, the restorations were finished with Sof-Lex disc systems (3M ESPE).

Thermocycling and Microleakage Test

The specimens were thermally cycled using 1000 cycles between water baths at 5°C ± 1°C and 55°C ± 1°C with a one-minute dwell time. The teeth were immersed into 2% methylene blue for four hours and

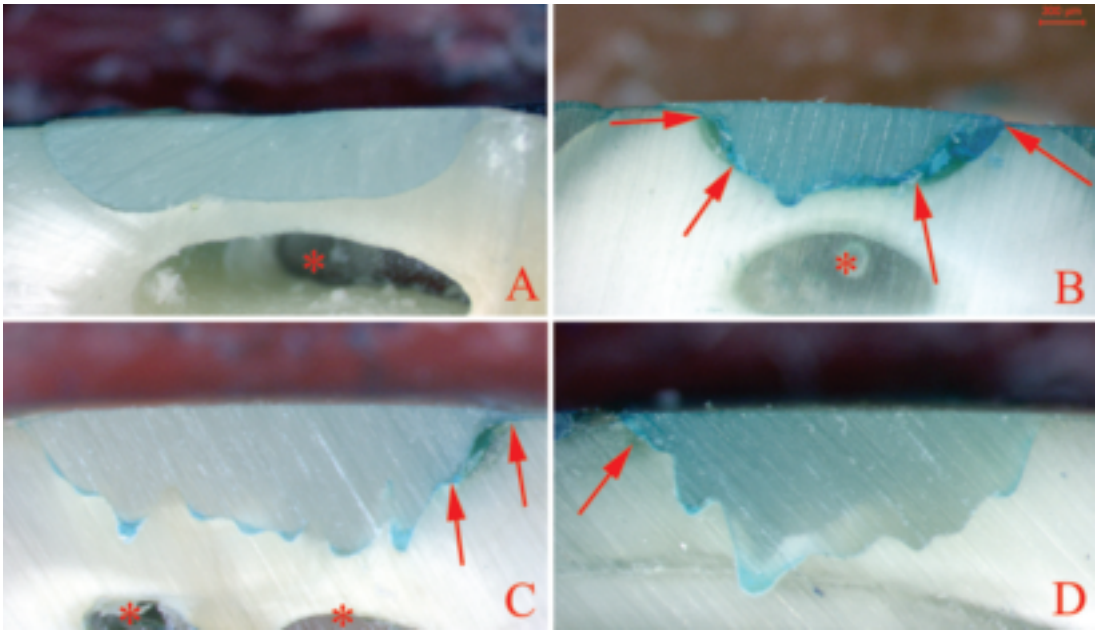

Figure 1. *Representative images of cavities restored after caries removal with a bur (A) and with a laser, using power outputs of 2.0 W (B), 3.0 W (C), and 4.0 W (D). The arrows indicate the presence of residual caries lesions on the cavity walls. It is possible to note that the cavities become more irregular and deeper as the laser power output increases. The symbol (*) identifies the pulp chamber.*

then rinsed with distilled water and air-dried for 10 minutes. Next, the samples were sectioned with a diamond disc (Buehler Ltd) mounted in a sectioning machine (Extec).

The sectioned surfaces were observed using a stereoscope (Stemi SV11; Zeiss, Thornwood, NY, USA) at 40× magnification and photographed with a digital camera (Cybershot 3.3 MPEG Movie EX; model number DSC-S75; Sony, Japan). The digital images obtained were transmitted to a personal computer and analyzed using Axion Vision 3.1 software (Carl Zeiss Vision, Peabody, MA, USA), which performed a standardized assessment of the extent of the tracer agent along the dentin/composite interface and provided quantitative measurements in millimeters. To calculate the percentage of microleakage, the total length of the tooth/restoration interface was measured. Then the length of the interface infiltrated by dye was calculated. These data were used to calculate the infiltration index for each section as the percentage of the interface length showing infiltration, by multiplying the infiltrated interface length by 100 and dividing that value by the total interface length. As three sections per sample were analyzed, it was possible to calculate an average for each sample. The mean dye penetration in the tooth/restoration interface was then calculated for each group. The maximum depth of the cavities, the area of the tooth/restoration interface, and the dye-infiltrated interface area were also measured.

To analyze for the presence of residual caries, the specimens were observed under a stereomicroscope at 40× magnification. The presence of residual caries under the restoration was classified using three scores, as follows: 1, no residual carious lesions; 2, residual lesion in at least one of the lateral walls of the cavity; and 3, residual lesion in the lateral and in the pulpal walls of the cavity.

Statistical Analysis

The data from 10 samples per group were compared by analysis of variance followed by Tukey test. The Spearman correlation test was performed to compare time vs Er,Cr:YSGG laser power outputs and residual caries scores vs infiltration indexes. The level of significance was determined as 5% ($p \leq 0.05$).

RESULTS

Representative photomicrographs of sections of control and experimental samples are presented in Figure 1. The tooth/restoration interfaces of cavities in the control group were regular (Figure 1A). No dye infiltration or residual caries were observed in these samples. When the caries lesions were removed with the Er,Cr:YSGG laser (Figure 1C,D), the tooth/restoration interface was irregular. Different degrees of dye infiltration as well as the presence

Table 2: *Mean (±Standard Error of the Mean) Values of Maximum Cavity Depths and Mean Areas of Tooth-Restoration Interface of All Experimental Groups (Different Online Small Capital Letters Indicate Statistical Differences Between the Groups)*

Groups	Depth, mm	Interface, mm^2
1	0.84 ± 0.049 A	3.51 ± 0.15 A
2	0.92 ± 0.086 A	4.36 ± 0.14 A
3	0.79 ± 0.031 A	4.17 ± 0.12 A
4	0.81 ± 0.037 A	4.27 ± 0.16 A
5	0.97 ± 0.047 A	5.03 ± 0.14 B
6	0.85 ± 0.034 A	4.58 ± 0.18 B
7	0.92 ± 0.032 A	4.84 ± 0.17 B
8	1.07 ± 0.051 A	4.52 ± 0.38 B
9	1.00 ± 0.033 A	4.64 ± 0.18 B
10	1.27 ± 0.036 B	5.43 ± 0.17 B
11	1.36 ± 0.080 B	5.28 ± 0.19 B
12	1.20 ± 0.053 B	5.17 ± 0.20 B
13	1.29 ± 0.058 B	4.64 ± 0.32 B
14	1.12 ± 0.053 B	4.74 ± 0.18 B

Table 3: *Distribution of the Residual Caries Scores Among the Experimental Groups (1: No Residual Carious Lesions; 2: Residual Lesion in at Least One of the Lateral Walls of the Cavity, and 3: Residual Lesion in the Pulpal Wall of the Cavity)*

Groups	Scores, %			Mode
	1	2	3	
1	100	0	0	1
2	0	50	50	3
3	10	30	60	3
4	0	70	30	2
5	10	80	10	2
6	30	50	20	2
7	30	70	0	2
8	30	60	10	2
9	30	70	0	2
10	20	80	0	2
11	10	70	20	2
12	20	80	0	2
13	10	90	0	2
14	0	100	0	2

of residual caries were observed. In the groups treated with the lowest power, most of the samples showed residual caries in all cavity walls (Figure 1B), whereas in the groups treated with the highest power, residual caries were mainly present in the lateral walls (Figure 1C,D).

The mean values of the maximum depth of the restorations and the area of the tooth/restoration interface of each experimental group are presented in Table 2. The cavities of the control group and of those irradiated with power outputs up to 2.75 W had similar depths. These depths were significantly smaller than those of samples in which higher parameters were used ($p<0.05$). The depths of cavities from samples irradiated from 3 W to 4 W were similar. The tooth/restoration interface areas of samples from groups 1, 2, 3, and 4 (control, 1 W, 1.25 W and 1.5 W, respectively) were similar. These areas were significantly smaller than those of all other groups ($p<0.05$). Samples irradiated with 1.75 W to 4 W presented tooth/restoration interfaces with similar sizes.

Table 3 shows the results of the residual caries examination. All samples in the control group exhibited a score of 1 (ie, no residual caries). All lased groups exhibited residual caries; most of the carious tissue was observed in the lateral wall of the cavities. In groups 2 and 3 (1.0 W and 1.25 W, respectively), residual caries was also found in the

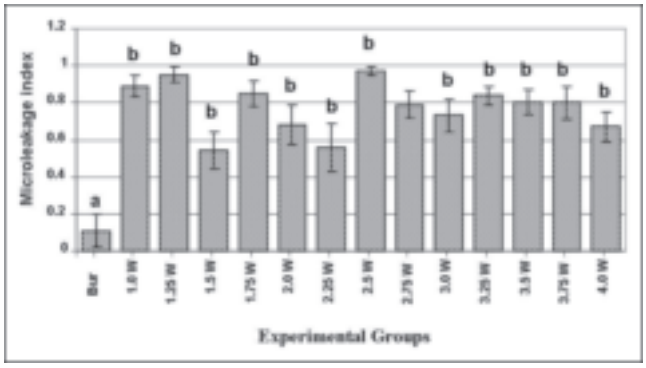

Figure 2. *Mean (±standard error) of the microleakage index in the different experimental groups. Different letters represent statistically significant differences.*

pulpal wall. For microleakage analysis of the restorations, an infiltration index was used. This index was calculated by dividing the area of the dye-infiltrated interface by the total tooth/restoration interface. The mean microleakage index of each group is represented in Figure 2. The infiltration indexes of all lased groups were similar and significantly higher than that of the control group ($p<0.05$).

To verify whether the presence of residual caries had an influence on the microleakage index, the Spearman correlation test was performed. There was a strong positive correlation between residual caries and the degree of infiltration (r=0.35; $p=0.0001$).

DISCUSSION

The methods used for dental caries removal can create dental substrates that have different interactions with restorative materials. The longevity of dental restoration directly depends on these interactions. The presence of a smear layer, irregularities on the cavity walls and margins, the orientation and diameter of the dentinal tubules, or even the presence of remaining caries can affect the success of the adhesive restoration that is performed after caries treatment. For this reason, a comparison between the conventional dental caries removal method using burs at low speed and laser treatment was performed in the current study. Characteristics related to caries removal and tooth restoration were analyzed, such as the depth of the cavities, the adhesive interface, the presence of residual caries, and the marginal seal (microleakage index). The marginal seal of composite resin restorations after root caries removal using the Er,Cr:YSGG laser method was worse than that obtained after caries lesion removal facilitated using a bur. In fact, microleakage indexes, as well as the presence of residual caries, were significantly higher in the lased groups.

The microbiologic artificial caries system was used in this study to produce lesions as similar as possible to those that develop naturally. This model utilizes a specific bacterial culture to induce demineralization as well as protein denaturation of the tooth tissues. Moreover, although the *in vitro* chemical model for caries induction allows better experiment control, this method does not reproduce the *in vivo* situation in such a refined manner as the bacterial model.[24] *Mutans* streptococci were chosen for the cariogenic challenge because they are the major microorganisms involved in development of caries lesions.[25]

The induction of *in vitro* caries using the biological method created dentin lesions that were very similar to caries lesions in both clinical and microscopic aspects. This carious substrate is appropriate for studying caries removal because it offers a substrate similar to that present under *in vivo* conditions.[24]

The main purpose for developing the use of lasers in dentistry was to find a new method for caries removal and cavity preparation that did not result in the typical discomforts associated with the conventional method of caries treatment, such as noise, pressure, vibrations, heating of the tooth structure, and the requirement for local anesthesia. Despite the advantages of the laser treatment previously stated, one factor that could be considered a disadvantage of laser treatment is the time required for caries removal. According to previous research,[26] the conventional method for caries removal is faster than Er,Cr:YSGG treatment. However, in our opinion, considering the patient's comfort (absence of noise and vibration and, sometimes, no requirement for local anesthesia), the longer clinical time required for laser use becomes irrelevant.

In the present study, the removal of carious lesions determined the cavity form. A previous study[26] has shown that the cavity morphology as well as the dentin substrate topography obtained with burs and the Er,Cr:YSGG laser are different. With burs, the margins and depth of the cavities obtained are smoother and smaller, respectively, than those of laser-obtained cavities, especially when parameters higher than 2.75 W are used. The results of that study indicated that the dentin was coated with smear layer when caries lesions were removed with burs and the Er,Cr:YSGG laser with parameters lower than 2.0 W, whereas laser treatment with higher parameters was able to remove carious tissue,

leaving the dentinal tubules open.[26] Hypothetically, this morphological characteristic makes a substrate more favorable for adhesion. However, in the present study, the substrate obtained by laser treatment led to poorer adhesion, as revealed by a significant increase in the microleakage index.

This research used extracted teeth. It is known that the dentin of a vital tooth has some different characteristics from the dentin samples used here; for example, the presence of odontoblastic processes and fluids. There are no reports comparing the effectiveness of the laser ablation of dental hard tissues of vital and nonvital teeth. However, we believe that there is no significant difference in that case, since all the irradiation was carried out using water cooling spray, and, thus, the presence of the dentin fluid becomes insignificant if we take into consideration the quantity of water used during irradiation. Furthermore, to avoid carbonization and other side effects, laser cavity preparation must be done with proper water cooling.[27]

The higher microleakage indexes observed in the laser groups could be due to the presence of residual caries as well as the inability of the Er,Cr:YSGG laser to remove hydroxyapatite crystals without causing damage to the collagen network. Both situations could harm hybrid layer formation. Although these aspects have not yet been described for the Er,Cr:YSGG laser, it has been reported[28] that the Er:YAG laser severely alters the dentin subsurface and causes collagen fibrils to lose their cross-banding and fuse together, thereby eliminating interfibrillar spaces and impairing hybridization.

The Er,Cr:YSGG laser and the Er:YAG laser have almost the same wavelength; therefore, they have similar interactions with hard dental tissue. Therefore, the effect of Er,Cr:YSGG on the dental substrate may damage collagen. In fact, a previous study[17] showed gaps between dentin irradiated with an Er,Cr:YSGG laser and the adhesive interface, indicating alteration in collagen. Morphological alterations produced by Er,Cr:YSGG laser irradiation adversely influence the bonding effectiveness of adhesives to dentin.[19]

One factor closely related to failures in marginal sealing of the laser-treated samples was the fact that the equipment used was not able to completely remove the carious tissue in the lateral walls of the cavity. According to our data, there was a strong positive correlation between the infiltration index and the amount of residual caries under the restorations. These findings concur with those found by other authors,[29] who have reported that the Er:YAG laser was not able to remove all infected carious tissue, with these residual caries noted only at the microscopic level. The current results also show the importance of working with carious dentin substrates rather than healthy dentin for *in vitro* analysis of the marginal seal quality of restorations.

In the present study the cavities were treated with a self-etch adhesive system prior to the placement of the composite resin. According to clinical results obtained in the past,[30-32] we believe that the two-step self-adhesive systems are effective, which justifies their use in the present study. Unlike etch-and-rinse adhesives, self-etch adhesives do not involve a separate etching step, as they contain acidic monomers that simultaneously 'condition' and 'prime' the dental substrate, rendering the technique less sensitive and faster. The reduction in clinical steps is especially important in the case of laser preparations, which require more time to produce than is required of the conventional method. Thus, if the clinical time during laser treatment is reduced, it will make this treatment more comfortable to the patients. Another clinical advantage of self-etch adhesives is the lower incidence of postoperative sensitivity experienced by patients when compared to the cases in which etch-and-rinse adhesives were used.[33] This is attributed to their less aggressive and more superficial interaction with dentin, leaving tubules largely obstructed with smear.[34] Additionally, studies[34,35] have shown that both two-step self-etch and etch-and-rinse techniques have performed successfully in laboratory as well as in clinical research.

The material used to fill the cavities was a flowable resin composite. In fact, previous studies[23,36] have shown that cavity restoration with resin-modified glass ionomer cement (GIC) leads to an efficient adhesion to the dentinal surface for cavities prepared after root caries removal with erbium lasers. These studies indicate that this laser promoted a modification of the dentin substrate by increasing the quantity of calcium, which in turn improved adhesion to the GIC.[23,36] However, although GICs have good adhesion in cavities prepared with erbium, it is important to know if other restorative materials, such as flowable composites, have the same success with GICs. According to past studies,[37-39] flowable composites present mechanical properties similar to those of microhybrid composites: low shrinkage stress, adequate marginal sealing, and high bond strength. Nevertheless, those characteristics were not able to prevent the micro-

leakage of the restorations placed in cavities prepared by the Er,Cr:YSGG laser.

CONCLUSIONS

In conclusion, the removal of root carious tissue using an Er,Cr:YSGG laser with power outputs between 1 W and 4 W creates a dentin substrate unfavorable for promoting marginal sealing. To take advantage of the positive characteristics of Er,Cr:YSGG laser irradiation, especially the added comfort for dentists and their patients, new studies should be performed with equipment allowing more flexibility in the determination of parameters, as well as the use of other restorative materials; this would confirm that irradiation is an alternative method for removal of dental root caries. These studies would help to achieve restorations with both good esthetic results and longevity, improving the life quality of patients through the maintenance of oral health, particularly in the elderly population.

Conflict of Interest

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

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Radiopacity of Flowable Composite by a Digital Technique

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Clinical Relevance

Most of the tested flowable composite materials fulfill the minimal required radiopacity conditions, with slight deviations at different exposure values.

SUMMARY

The aim of this *in vitro* study was to evaluate the radiopacity of 19 current dental flowable composite materials by a digital technique. Digital radiographs were obtained with a CCD sensor using an aluminum step wedge, a 1-mm-thick tooth slice, and a 1-mm-thick flowable composite specimen using five different combinations of exposure and voltage. The radiopacity in pixels was determined using Digora 2.6. software. The equivalent thickness of aluminum for each material was then calculated based on the calibration curve. All of the tested flowable composite materials had higher radiopacities than that of dentin, but in almost every combination of exposure and voltage, there were some composite materials that exhibited radiopacities equal to or slightly greater than enamel $p>\alpha$; $\alpha=0.01$). Of the flowable composite materials tested, 37% showed lower radiopacities than enamel, and 21% of the tested materials had higher radiopacities than the 3-mm aluminum equivalent. The highest radiopacity at all exposure values was produced by the Majesty Flow and Charisma Opal Flow materials, which had radiopacities almost twice that of enamel. Flowable composite materials should have radiopacities greater than that of enamel (ISO 4049), an important consideration for the introduction of new materials to the market. The digital radiopacity analysis techniques used in this study provide an easy, reliable, rapid, and precise method to characterize radiopacity of dental flowable composite materials.

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Introduction

The first generation of flowable resin composites was introduced in late 1996. They were created mainly by

retaining the small particle size of traditional hybrid composites but reducing the filler content and, consequently, the viscosity of the mixture.[1] These materials can be manipulated using a syringe with a loading tip and injected where access using traditional instruments is difficult or impossible because of the low viscosity of these materials.[2] Flowable composite materials are purported to offer higher flow, better adaptation to the internal cavity wall, easier insertion, and greater elasticity than previously available products.[3] They have been recommended for use as liners beneath composite resins because of their low viscosity, increased elasticity, and wettability. These handling characteristics and the syringe delivery system make flowable composite a good choice for sandwich techniques. They are placed at the cementum margins of the proximal box as a liner in Class II resin composite restorations to improve the final marginal integrity, resulting in reduced leakage and postoperative sensitivity.[4–6] Employing an intermediate layer of low-modulus composite can also relieve some of the contraction stress during polymerization. Some *in vitro* studies have shown that the use of flowable composites reduces restoration microleakage and the occurrence of voids and that their use as liners improves the marginal seal of a restoration.[7–9]

Dental materials should be sufficiently radiopaque to be detected against a background of enamel and dentin to facilitate correct evaluation of restorations in every region and detection of secondary caries, marginal defects, contour of restoration, contact with adjacent teeth, cement overhangs, and interfacial gaps.[10–15] The advantages of radiopaque materials over radiolucent ones include easier detection of recurrent dental caries and easier visualization of the radiographic interface between the materials and tooth substrates.[11]

The International Organization for Standardization (ISO) requires that a resinous dental material be at least as radiopaque as the same thickness of pure Al, and the American Dental Association (ADA) recommends a radiopacity equivalent to 1 mm of Al or 1 mm of dentin.[16,17] The radiopacity of dental materials can be analyzed by a digital technique using x-ray digital sensors and computer software. In the digital imaging technique, the gray scale is inverted relative to the optical density, such that white is assigned a value of 255 (for an eight-bit image) and black is assigned a value of 0. Although flowable composite materials have remained popular and have been widely used for the past 15 years, only a few reports are available on their radiopacity using either digital or analog techniques.[18–22] The main purpose of this study was to evaluate the radiopacity of common flowable composite dental materials at five different exposure times by a digital analysis technique.

MATERIALS AND METHODS

Specimen Preparation

Commercially available and commonly used flowable composite materials were evaluated in this study, as listed in Table 1. Three specimens of each material were prepared according to manufacturer instructions and injected into 1-mm-thick stainless-steel cylinders with an internal diameter of 4.1 mm. After filling each cylinder to capacity, the material's surface was covered with a glass slide, and pressure was applied to force out any excess material. Specimens were light-cured using a light-emitting diode polymerization lamp (Elipar Freelight 2, 3M ESPE, St Paul, MN, USA) with a power of 1000 mW/cm^2 and a wavelength of 430–450 nm for 40 seconds on each side. After removal from the cylinders, the specimens were polished using 400-, 600-, and 1000-grit sandpaper; cleansed with 70% ethyl alcohol; and measured with a digital micrometer to verify that the thickness remained at the critical tolerance of 1.0 $\pm$ 0.01 mm. Specimens with macroscopic defects (eg, voids, cracks) were excluded from the study, and new samples were prepared as previously described.

The tooth material for the enamel/dentin specimens was extracted for orthodontic reasons, as approved by the ethics committee of the School of Dental Medicine. A 1-mm enamel/dentin specimen was prepared by longitudinal sectioning of a freshly extracted third molar using a slow-speed Isomet 1000 (Buehler, IL, USA) diamond saw with a constant speed of 250–300 rpm. The tooth specimen was then stored in tap water until use. The step wedge was fabricated by riveting together ten 1-mm-thick plates of aluminum alloy (1100 purity of 99.5% Al). The chemical composition of the aluminum used for fabricating the step wedge was as follows: 0.0014% Cu, 0.0019% Mn, 0.0017% Mg, 0.06% Si, 0.37% Fe, 0.0089% Zn, and 0.025% Ti. The plates were 10 mm wide, and the aluminum wedges ranged from 1- to 10-mm thick.

The Prostyle Intra 50–70 kV digital x-ray machine (Planmeca Oy, Helsinki, Finland) with a DiXi3 B1 digital CCD sensor (Planmeca Oy) was used in this study. Three specimens of each test material, the aluminum step wedge, and a tooth specimen were positioned over the sensor on each of the radiographs

Table 1: *List of Flowable Composite Materials*

Product	Shade	Filler % (wt/vol)	Type (Manufacturer Data)	Manufacturer
Admira Flow	A4	63/50.5	Ormocer-based flowable composite	Voco GmbH, Cuxhaven, Germany
Amaris Flow	HT	NA	Highly esthetic composite-high translucence flowable composite	Voco GmbH, Cuxhaven, Germany
Amaris Flow	HO	NA	Highly esthetic composite-high opaque flowable composite	Voco GmbH, Cuxhaven, Germany
Arabesk Flow	A2	64/NA	Light-curing glass ceramic microhybrid flowable composite with BCS* filler	Voco GmbH, Cuxhaven, Germany
Charisma Opal Flow	A2	62/38	Microparticle hybrid flowable composite	Heraeus Kulzer GmbH, Hanau, Germany
Charisma Flow Baseliner	BS	NA/NA	Microparticle hybrid flowable composite	Heraeus Kulzer GmbH, Hanau, Germany
Filtek Flow	A2	68/47	Flowable composite	3M/Espe, St Paul, MN, USA
Filtek Supreme XT Flow	A3	65/55	Flowable composite	3M/Espe, St Paul, MN, USA
Gradia Direct flo	A3	75/NA	Micro-filled hybrid flowable composite resin	GC Europe NV, Leuven, Belgium
Gradia Direct LoFlo	A3	40/NA	Micro-filled hybrid flowable composite resin	GC Europe NV, Leuven, Belgium
Grandio Flow	A3	80.2/65.7	Nano-hybrid flowable composite	Voco GmbH, Cuxhaven, Germany
Majesty Flow	A3	81/62	Superfilled flowable composite	Kuraray Medical INC, Okayama, Japan
Premise Flow	A3	72.5/54.6	Medium-viscosity flowable composite	Kerr Corporation, Orange, CA, USA
Permaflo	A2	68/NA	Flowable composite	Ultradent, South, South Jordan, USA
Revolution Formula2	A3	51/43	Hybrid flowable composite	Kerr Corporation, Orange, CA, USA
Tetric EvoFlow	A3	62.4/30.7	Nanotechnology flowable composite	Ivoclar Vivadent, Schaan, Liechtenstein
Tetric Econom Flow	A3	64.6/40	Flowable composite	Ivoclar Vivadent, Schaan, Liechtenstein
Tetric Flow	T	NA/NA	Flowable composite	Ivoclar Vivadent, Schaan, Liechtenstein
EsthetX Flow	A3	62/53	Micro hybrid flowable composite	Dentsply DeTrey Gmbh, Konstanz, Germany

(Figure 1). Each specimen was radiographed three times using five different combinations of exposure time and voltage, with a constant source-to-sample distance of 30 cm. These combinations were considered for properly exposed digital images, and they are in accordance with manufacturer instructions. With these combinations of voltage and exposure, we can analyze possible differences in radiopacity between specimens of flowable composite materials. The combinations of voltages and exposures used

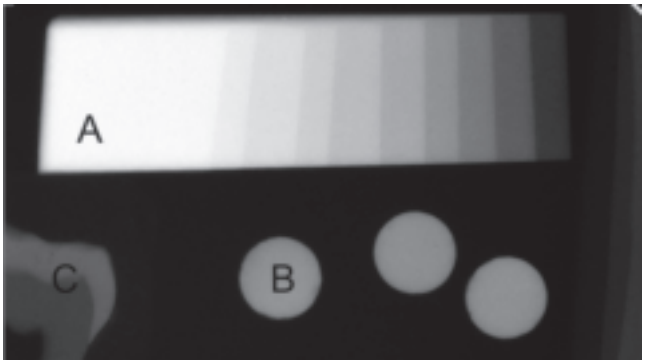

Figure 1. *Digital image obtained from CCD sensor containing tooth structure and tested flowable composite materials. (A): Tooth structure. (B): Tested composite materials. (C): Aluminum step wedge.*

were as follows: (1) 60 kV and 0.06 seconds, (2) 60 kV and 0.08 seconds, (3) 63 kV and 0.06 seconds, (4) 63 kV and 0.08 seconds, and (5) 63 kV and 0.1 seconds.

Digital Imaging

The images, free of any enhancement to contrast or picture quality, were imported into the Dimaxis Pro 4.0 software (Planmeca Oy) and exported in eight-bit TIFF format for subsequent radiopacity analysis (Figure 2). The radiopacity of the specimen, in pixels, was determined using a different type of software, Digora for Windows 2.6 (Soredex, Tuusula, Finland). Digora is Windows-based software capable of measuring density curves of digital radiographs obtained by digital x-ray impregnation on the CCD sensor. The density measurement tool automatically measures the gray shade values in the picture. With the point of the mouse arrow (area 1 pixel $\times$ 1 pixel), five different positions were measured in each of the three material specimens. It was important to analyze only those regions that were free of air voids, gaps, cracks, or other similar defects. Using a similar procedure, a tooth slice with enamel and dentin was also measured in five different regions. This procedure was repeated using five different exposures.

The aluminum step wedge (99.5% Al) was used as an internal standard for measuring the comparative equivalent radiopacity of different materials. In 30 random radiographs, each of the 10 steps of the aluminum step wedge was measured for density, and a graph of density versus the thickness of the aluminum alloy at each step was constructed.[23–25] Subsequently, a calibration curve was plotted for selected data using a best-fit logarithmic regression analysis. The equivalent in thickness of aluminum for each material was calculated based on the

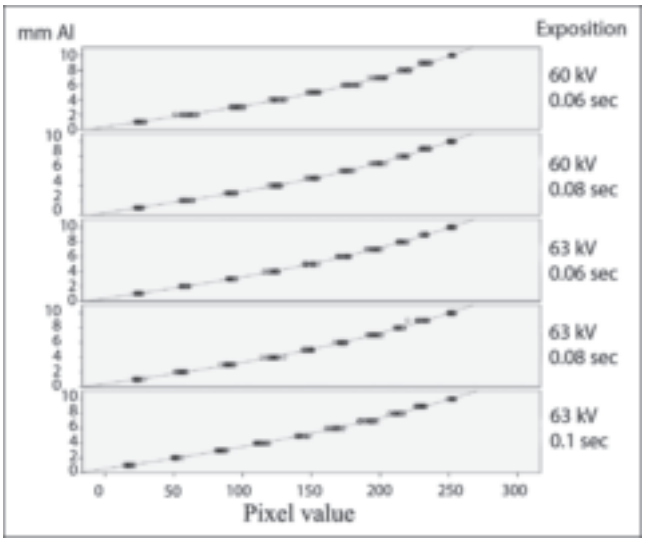

Figure 2. *The curves of pixel values versus mm Al for each exposition.*

calibration curve (Figure 2). The measured gray-scale value for each dental material and aluminum corresponded to the extent of attenuation of x-ray transmission through the materials, producing a value converted into absorbance using the following formula:

$$A = -\log 10(T) = -\log 10\left(\frac{1 - G}{255}\right),$$

where A is the absorbance, T is the transmittance, and G is the gray-scale value of the material.[26] The same procedure was repeated for five different exposures. Since radiopacity was not a normaly distributed variable, nonparametric tests—Mann-Whitney U-test and Kruskal-Wallis—were used to compare mean values of radiopacity accross different groups and different types of flowable composite materials. The results were statistically analyzed using PASW Statistics 18 software (SPSS Inc, Chicago, IL, USA).

RESULTS

The results of the Kolmogorov-Smirnov test indicated that radiopacity (in mm Al) was not a normally distributed variable ($p<0.05$); therefore, the mean values for the different types of flowable composite materials were compared with the values for dentin and enamel using the Mann-Whitney U-test ($\alpha=0.01$). The equations of the best-fit curves from third-degree polynomial function $f(x) = ax^3 + bx^2 + cx + d$ and their associated errors for all combinations of voltage and exposure are listed in Table 2. The

Table 2: *Regressions and Regressions Errors (Radiopacity/Thickness Correlation)*[a]

Exposition	Regression Parameters (SE)				R^2	Mean Residuals
	a	b	c	d		
60 kV 0.06 s	0.332 (0.059)	0.028 (0.002)	−2.36E-05 (0.000)	2.59E-07 (0.000)	0.998	0.018
60 kV 0.08 s	0.310 (0.045)	0.029 (0.001)	−2.96E-05 (0.000)	2.62E-07 (0.000)	0.999	0.011
63 kV 0.06 s	0.362 (0.044)	0.027 (0.001)	−1.231E-06 (0.000)	1.787E-07 (0.000)	0.999	0.011
63 kV 0.08 s	0.418 (0.056)	0.026 (0.002)	8.753E-06 (0.000)	1.537E-07 (0.000)	0.998	0.018
63 kV 0.1 s	0.522 (0.043)	0.028 (0.001)	1.382E-05 (0.000)	1.050E-07 (0.000)	0.998	0.015

[a] *a, b, c, d = coefficients of third-degree polynomial function.*

calibration curve for aluminum thickness versus pixel value was plotted using the best-fit logarithmic regression method. The third-degree polynomial is a mathematical curve that best represents the given data, as shown in Figure 2 for each combination of voltage and exposure. The absorbance of the aluminum step wedge at different combinations of exposure and voltage is reported in Table 3. The radiopacities of flowable composite materials at different exposures are presented in Table 4.

All of the tested flowable composite materials had higher radiopacities than dentin *($p<0.001$)*, but at almost every combination of exposure and voltage, there were some composite materials with radiopacities equal to or slightly greater than enamel ($p>0.01$). Filtek Flow, Grandio Flow, Amaris Flow HO, and Amaris Flow HT showed no statistically significant differences in radiopacity as compared with enamel ($p>0.01$), at an exposure value of 60 kV and 0.06 seconds. The highest radiopacities for all exposure values were produced by the Majesty Flow and Charisma Opal Flow materials, with almost twice the radiopacity of enamel.

DISCUSSION

In this study, the radiopacities of all 19 tested materials were higher than that for dentin, and 12 of the flowable composite materials produced higher radiopacities than enamel. According to the ISO standards, the minimum radiopacity requirement for these materials is equivalent to 1 mm of aluminum alloy 1100.[16] The radiopacity values of tooth samples observed in this study were equivalent to 2.02–2.08 mm of Al for enamel and 1.09–1.13 mm of Al for dentin, according to the different exposure values. It should be noted that differences in radiopacity values for the same material from different studies may be a result of many factors, such as variations in exposure parameters, purity of the Al standard, thickness of the test materials, and differences between analog and digital assay techniques.[24–28] The purity of the aluminum step wedge is very important because 4% copper in an aluminum alloy would result in radiopacity measurements a full 50% lower than those of 99.5% aluminum, creating a systematic error of 1.25%.[29] Therefore, an aluminum step wedge of 99.5% purity was used in this study, containing no more than 0.37% iron or 0.0014% copper.

Digital radiology does not involve film development, a process that introduces variation in the final radiograph.[16,30,31] Digital image analysis is considered to have the same accuracy as transmission densitometry and can produce measurements equivalent to those obtained with film with reduced noise, providing precise and trustworthy numerical values for comparative radiodensity studies.[19,22,31–34] Transmission densitometry measures optical density, a logarithmic measure of the ratio of transmitted to incident light through the film image. In digital image analysis, we measure radiographic density directly using the gray scale of the pixels, measuring the values on a scale of 0 to 255 using the computer software.[26,31] Furthermore, it is not necessary to perform any subtraction (as with conventional x-ray film) when calculating the radiopacity.[22]

The application of a flowable composite between the adhesive and the conventional composite to create an elastic intermediate layer has been proposed.[34–37] The elasticity of this layer may absorb

Table 3: *Absorbance Values for Aluminum Step Wedge*

		60 kV 0.06 s		60 kV 0.08 s		63 kV 0.06 s		63 kV 0.08 s		63 kV 0.1 s	
Aluminum, mm		**Pix**	**Abs**	**Pix**	**Abs**	**Pix**	**Abs**	**Pix**	**Abs**	**Pix**	**Abs**
A1	Mean value	24.90	0.04	24.30	0.04	24.10	0.04	23.60	0.04	17.65	0.03
	SD	1.98	0.00	1.52	0.00	1.33	0.00	1.76	0.00	1.72	0.00
A2	Mean value	59.9	0.12	58.90	0.11	57.60	0.11	55.30	0.11	51.40	0.09
	SD	4.59	0.01	2.79	0.01	2.11	0.01	2.39	0.01	1.54	0.00
A3	Mean value	95.75	0.21	92.00	0.19	91.45	0.19	89.65	0.19	84.15	0.17
	SD	3.19	0.01	2.41	0.01	1.67	0.00	2.43	0.01	2.06	0.01
A4	Mean value	125.20	0.29	124.40	0.29	122.30	0.28	121.90	0.28	112.90	0.25
	SD	3.44	0.01	2.30	0.01	2.98	0.01	3.42	0.01	2.86	0.01
A5	Mean value	152.35	0.39	151.45	0.39	149.75	0.38	148.10	0.38	142.80	0.36
	SD	2.49	0.01	2.39	0.01	3.13	0.01	2.40	0.01	3.02	0.01
A6	Mean value	178.65	0.52	175.85	0.51	173.70	0.50	172.65	0.49	167.80	0.47
	SD	3.68	0.02	2.64	0.01	3.03	0.02	2.23	0.01	3.55	0.02
A7	Mean value	199.70	0.67	198.65	0.66	196.30	0.64	195.55	0.63	191.00	0.60
	SD	3.33	0.03	2.43	0.02	2.98	0.02	2.96	0.02	4.28	0.03
A8	Mean value	218.35	0.84	217.20	0.83	215.15	0.81	213.90	0.79	212.65	0.78
	SD	2.52	0.03	2.09	0.02	1.93	0.02	1.74	0.02	2.82	0.03
A9	Mean value	232.65	1.06	233.20	1.07	232.65	1.06	229.60	1.01	229.60	1.00
	SD	2.39	0.05	2.29	0.05	1.23	0.02	3.49	0.06	2.26	0.04
A10	Mean value	252.40	2.01	251.85	1.97	252.35	2.06	252.00	2.00	252.60	2.07
	SD	0.68	0.13	1.49	0.26	1.49	0.28	1.56	0.27	1.23	0.24

Abbrevations: Abs, absorbance; Pix, pixels.

the contraction stress generated by the conventional composite, reducing tooth/restoration interfacial stress[34] and cuspal deflection occurring during polymerization shrinkage.[38] The flowable composite liner recommended for deep class II cavities may act as a flexible intermediate layer, relieving stresses during polymerization shrinkage of the restorative resin.[39–41] It can be concluded that usage of flowable

Table 4: *Radiopacity of Flowable Composite Materials at Different Exposures*

Composite Materials/Radiopacity Al (SD)	60 kV 0.06 s	60 kV 0.08 s	63 kV 0.06 s	63 kV 0.08 s	63 kV 0.1 s
Majesty Flow A3	3.91 (0.11)	3.91 (0.11)	3.94 (0.17)	4.02 (0.21)	3.91 (0.15)
Charisma Opal Flow A2	3.81 (0.12)	3.8 (0.15)	3.88 (0.12)	3.81 (0.12)	3.84 (0.17)
Tetric EvoFlow A3	3.23 (0.1)	3.2 (0.07)	3.27 (0.11)	3.05 (0.09)	3.3 (0.15)
Tetric Flow T	3.13 (0.13)	3.1 (0.16)	3.12 (0.11)	3.09 (0.1)	3.22 (0.19)
Premise Flow A3	2.91 (0.07)	2.86 (0.11)	2.94 (0.16)	2.95 (0.12)	2.87 (0.2)
Permaflo A2	2.88 (0.1)	2.89 (0.14)	3.12 (0.14)	3.01 (0.17)	3.13 (0.14)
Esthet X Flow A3	2.35 (0.1)	2.35 (0.1)	2.44 (0.1)	2.31 (0.09)	2.36 (0.1)
Charisma Flow BS	2.25 (0.09)	2.25 (0.12)	2.28 (0.1)	2.3 (0.12)	2.22 (0.08)
Gradia Direct flo A3	2.24 (0.11)	2.19 (0.09)	2.12 (0.09)	2.19 (0.07)	2.11 (0.11)[d]
Filtek Supreme XT Flow A3	2.2 (0.13)	2.34 (0.09)	2.2 (0.14)	2.28 (0.12)	1.98 (0.09)d
Filtek Flow A2	2.15 (0.1)[a]	2.12 (0.08)	2.05 (0.11)[c]	2.07 (0.11)[d]	2.14 (0.09)[d]
Grandio Flow A3	2.13 (0.06)[a]	2.14 (0.09)	2.11 (0.11)[c]	2.11 (0.12)[d]	2.19 (0.11)
Enamel	2.07 (0.05)	2.02 (0.08)	2.03 (0.1)	2.04 (0.09)	2.08 (0.1)
Amaris Flow HO	2.05 (0.1)[a]	1.86 (0.07)	1.96 (0.08)[c]	1.92 (0.09)	1.98 (0.12)[d]
Amaris Flow HT	2.02 (0.07)[a]	1.95 (0.06)[b]	1.97 (0.09)[c]	2 (0.07)[d]	2.05 (0.07)[d]
Arabesk Flow A2	1.87 (0.08)	1.83 (0.08)	1.81 (0.11)	1.8 (0.1)	1.87 (0.07)
Tetric Econom Flow A3	1.87 (0.07)	1.83 (0.08)	1.86 (0.1)	1.85 (0.07)	1.92 (0.08)
Admira Flow A4	1.85 (0.09)	1.84 (0.07)	1.87 (0.11)	1.89 (0.12)	1.91 (0.1)
Revolution Formula2 A3	1.58 (0.08)	1.55 (0.08)	1.58 (0.08)	1.59 (0.08)	1.46 (0.1)
Gradia Direct LoFlo A3	1.56 (0.06)	1.49 (0.06)	1.53 (0.09)	1.42 (0.12)	1.41 (0.1)
Dentin	1.12 (0.08)	1.1 (0.06)	1.09 (0.08)	1.11 (0.08)	1.13 (0.08)

[a,b,c,d] *Same letters show no statistical significance in comparison with enamel (*$p > 0.01$*).*

composite materials as a liner below the packable composite materials is recommended.[4,41–45]

Some authors have suggested that composite materials with higher radiopacities than that of the tooth structure should be used for posterior restorations to enhance detection of the interface between the restoration and the tooth.[19,21,46] Materials with greater radiopacities, higher than that of enamel,

were favorable for a true-negative diagnosis.[47] The observed radiopacity for some flowable composite resin materials in this study was lower than is desirable for use under posterior restorations; therefore, they are not recommended as liners. Radiopacity equal to or slightly greater than that of enamel is preferable to facilitate detection of secondary caries and marginal defects in posterior teeth.[7,18–22,28,48] If the initial increment of a posterior restoration has a radiopacity equal to or slightly greater than that of dentin, it may not be possible to detect the extent of the restoration, a small defect, or an overhang.[21] In contrast, some authors have suggested that highly radiopaque restorative materials deteriorate visual acuity and complicate the perception of details such as caries, lesions, and marginal defects, suggesting that moderate radiopacity might be favorable for easier caries detection.[11,15,29] All of the tested flowable materials showed significant differences in radiopacity as compared with dentin, but not all differed significantly from enamel. Introduction of chemical elements with high atomic numbers, such as zinc, strontium, zirconium, barium, and lanthanum, produce more radiopaque materials.[7,12–14] Tetric composite materials contain yttrium (Y, atomic number = 39) and ytterbium (Yb, atomic number = 70), which can contribute a high level of radiopacity, probably the source of high radiopacity in the Tetric Flow group of materials.[19] Further, barium (Ba, atomic number = 56) is the element most commonly incorporated into composite restorative materials to increase their radiopacities. The flowable composites with the highest radiopacities were Majesty Flow and Charisma Opal Flow, with almost four times the radiopacity of dentin and twice that of enamel. According to manufacturer data, Majesty Flow has very high filler loading, similar to that of many universal composite resins, possibly explaining its high radiopacity. The manufacturer of the Charisma Opal composite materials claims that its filler consists of x-ray opaque microglass and silicon dioxide, both of which increase radiopacity. Ergücü and others[18] reported that Clearfil Majesty Flow and Tetric Flow had the highest radiopacities among six tested flowable composite materials observed and that the lowest radiopacity observed in their study was produced by the Gradia Direct LoFlo material. The highest radiopacity values obtained in that study may be attributed to silanated barium glass fillers in the Clearfil Majesty Flow and ytterbium trifluoride particles in Tetric Flow. The lowest observed radiopacity, produced by Gradia Direct LoFlo, may be attributed to its silicon dioxide filler content, which has a radiopacity value similar to that of dentin.[18] Moreover, the Gradia Direct LoFlo material also produced the lowest radiopacity in our study. The Tetric Flow material, as observed in other studies, showed the highest radiopacity, almost twice that of enamel.[19,21] The Tetric Flow material also produced the highest radiopacity (twice that of enamel) among flowable materials when measured using film radiographs and phosphor storage plates (Digora).[22] However, the radiopacity of the Tetric Econom Flow material was lower than the value for enamel in all exposure combinations. This could result from the fact that Tetric Econom Flow is an economy product system as opposed to premium composite systems from the Tetric Group, such as Tetric Flow and Tetric Evo Flow, which have greater amounts of radiopaque elements in their compositions. It is important to note that results from different studies may vary as a result of different techniques and materials used (eg, digital or analog, composition of aluminum step wedge, material thickness, etc). There may also be substantial variation in the composition and content of a filler from what is claimed by the manufacturer; thus, a detailed chemical analysis of each composite material is needed to determine its exact composition.

CONCLUSION

Most of the flowable composite materials tested produced radiopacities similar to or greater than enamel, with slight deviations at different exposure values. The Majesty Flow, Charisma Flow, and Tetric Flow materials showed the highest radiopacities at all exposure values. The digital techniques used for measurement of radiopacity in this study provided an easy, reliable, rapid, and precise approach to evaluate the different dental flowable composite materials.

Conflict of Interest

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

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Dimensional Accuracy of Optical Bite Registration in Single and Multiple Unit Restorations

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Clinical Relevance

Based on the dimensional accuracy measured *in vitro,* the optical bite registration was shown to be more effective in single posterior restorations in comparison with the conventional physical method using silicone material.

SUMMARY

The dimensional accuracy of optical bite registration in the CEREC system was compared to that of the conventional physical method *in vitro* using a bite registration material. Maxillary and mandibular full-arch dentate epoxy models mounted on an articulator were used to measure the interarch distance and the angles created by the occlusal planes. The preparations for a single restoration on the maxillary first molar or for multiple restorations on the maxillary posterior quadrant were made on the model. Optical impression and bite registration data were collected to construct virtual models using computer-aided design software. A silicone material was used for the physical method, and the dimensional accuracy was measured by means of the coordinate measuring machine. The discrepancy relative to the baseline before preparation was analyzed in each registration record. For the single restoration, the optical method created a mean discrepancy of 243.2 μm relative to baseline at the prepared tooth, which was insignificantly but slightly lower than the mean discrepancy of 311.1 μm obtained with the physical method. The mean rotational deviation in the horizontal plane was significantly lower for the optical method. For the multiple preparations, the optical method showed significantly larger discrepancy on the right molar and on the left premolar and molar sites. In the frontal view, the optical method created significantly larger rotational deviation than the physical method. The result indicates that the optical bite registration was effective in terms of dimensional accuracy for single posterior restorations.

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INTRODUCTION

Computer-aided design/computer-aided manufacturing (CAD/CAM)–generated restorations have gained popularity as the technique has evolved and improved over time. Long-term prospective studies[1,2] on CAD/CAM ceramic restorations reported survival probabilities of over 85% at 10+ years. More recently, advanced CAD/CAM systems have been introduced with an optical bite registration method in which the occlusal relationship between the prepared tooth and its antagonist is determined by the CAD software. The data are used to fabricate a restoration at chairside and to place the completed restoration on the same day.[3] The system employs intraoral cameras for optical impression and subsequent bite registration, and the occlusal relationship is digitally determined without the use of a bite registration material.[4] Conventional bite registration materials, such as wax and silicones, expand or shrink[5-9] more or less under varying temperature and moisture conditions after hardening[10] and gradually deform over time during storage.[9,11-13] Since recent clinical case series[4,14] indicated that the optical bite registration method was fairly predictable, the system looks promising and may be potentially effective for extended restorative and prosthodontic reconstruction. However, the accuracy of the optical method has not been compared with that achieved by the conventional physical bite registration. Furthermore, only limited information is available regarding the accuracy of the system when it is applied to multiple restorative units.

The objective of this study was to assess the three-dimensional accuracy of the optical system in comparison with that shown by the conventional bite registration method. The accuracy was evaluated *in vitro* using maxillary and mandibular casts with preparation for single or multiple restorative units. We hypothesized that the optical method increases accuracy of the reproducibility of the interarch relation in comparison with the conventional method using silicone material and that the accuracy of the optical system is not affected by extension of the restorative units.

MATERIALS AND METHODS

Maxillary and mandibular dentate models (D18FE-500A-QF, Nissin, Tokyo, Japan) were duplicated to create epoxy resin casts for the simulation of a patient (Diemet-e, Erkodent, Pfalzgrafenweiler, Germany). The casts were mounted on a semiadjustable articulator (Denar Mark II, Whip Mix Corp, Louisville, KY, USA), with the maximum intercuspal position determined manually by the examiner. The maxillary cast was mounted using a split-cast technique. The occlusal contacts were marked with articulating paper and adjusted in the conventional manner using diamond burs until the baseline occlusal relationship was established. As a result, all teeth had equal contacts with their antagonists, and the molars and premolars had at least two contacting points. To mark the reference points (RPs) for measurement, stainless-steel pins were fixed with light-curing composite material (Premise, Kerr, CA, USA) to the center of the buccal maximum convexity of teeth 14, 24, 34, 44 (premolar RPs), 17, 27, 37, and 47 (molar RPs) and at proximal contact points between teeth 11 and 21 and between teeth 31 and 41 (anterior RPs) (Figure 1a,b).

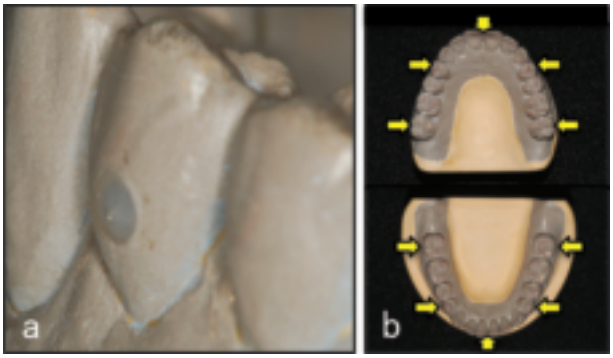

Figure 1. *The RPs for the measurements. Stainless-steel pins (black arrow) were fixed with light-curing composite on the center of the buccal surface (a) of teeth 14, 24, 34, 44 (premolar RPs), 17, 27, 37, and 47 (molar RPs) and at proximal contact points between teeth 11 and 21 and between teeth 31 and 41 (anterior RPs) (b).*

The calibration of the measurement was conducted using a flat plastic plate, whereby five RPs were linearly placed at 3-mm intervals. The distances between the RP on one edge and the others were measured by the coordinate measuring machine (QM–Measure 353, Mitutoyo, Kawasaki, Japan). After this process was completed, each plate was sprayed with titanium oxide powder (CEREC Optispray, Sirona, Bensheim, Germany). The optical impression was taken for each distance using an intraoral camera (CEREC Blue-CAM, Sirona) and recorded on the CAD software. Linear regression analysis indicated that the measured and the optically recorded distances correlated well (r^2=0.9996).

The location of each RP on the articulated casts was recorded by means of the coordinate measuring machine. The measurements were repeated five times by a single examiner (Y.I.), and the mean distances between the RPs on the upper and lower

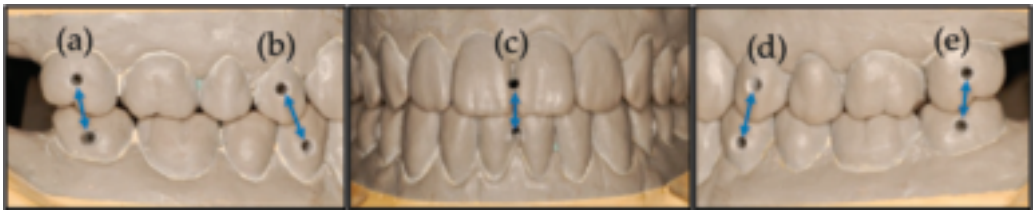

Figure 2. *The measurement sites for the interarch distance. The distances between the reference points of the upper and lower corresponding teeth (arrows) were measured at the right molar (a), premolar (b), anterior (c), left premolar (d), and molar sites (e).*

corresponding teeth (Figure 2) were calculated. The maxillary and mandibular planes were defined so that the right and left molar RPs and the anterior RP were included on each plane (Figure 3). The angle created by the maxillary plane relative to the mandibular plane was calculated on the horizontal and frontal planes for baseline records. A positive angle was recorded when the maxillary plane was deviated to a counterclockwise direction in the horizontal plane when viewed from above (Figure 3a) and when the maxillary plane on the left side was upright in the frontal plane when viewed from the front (Figure 3b).

For the first experiment, the maxillary right first molar in the cast was prepared for an all-ceramic, full-coverage restoration, with tooth reduction of 2.0-mm thickness on the buccal and lingual cusps, 1.5-mm thickness on the occlusal surface, and 1.0-mm thickness on the axial surfaces (Figure 4a). The upper and lower models were sprayed as indicated earlier, and a full-arch optical impression and scanning from the buccal side for the optical bite registration were carried out. Using the CAD software, distances between the maxillary and mandibular RPs of corresponding teeth (Figure 2) were measured 10 times. The mean horizontal and frontal angles of the maxillary plane relative to the mandibular plane were calculated based on the recorded distances between the RPs. The discrepancies of the mean distances and plane angles with the baseline records before preparation were then computed to determine the accuracy of the method.

For physical bite registration, a silicone bite registration material (Blu-Mousse Super-Fast, Parkell, Edgewood, NY, USA) was mixed and placed on the entire lower arch of the casts in the baseline interarch relation, and the casts were kept in occlusion until the material had hardened completely. This process was repeated to create five occlusal registration records. The materials were thereafter stored dry at room temperature for 24 hours before testing and trimmed with a sharp scalpel to eliminate excess material extending into undercuts. The upper split-cast was detached from the articulator. Each record was placed back on the lower model, and the upper model was returned on the top of the lower cast. After ensuring that the models fit together accurately, the relative positions were marked with line patterns on the cast surface using pencil, and the material was removed. The upper model was remounted on the articulator with mounting stone (Elite Arch, Zhermack, Polesine, Italy). The coordinates of the RPs were measured using the coordinate measuring machine. All of the above procedures were repeated for each registration. The discrepancies between the mean distances and the plane angles in comparison with the baseline before preparation were then computed.

The second experiment was conducted using the same cast. The maxillary right first premolar and

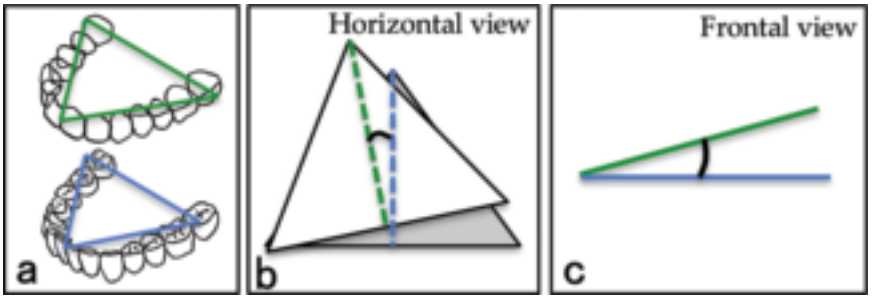

Figure 3. *Occlusal planes and the angles. (a) The maxillary and mandibular planes were created by inclusion of the right and left molar RPs and the anterior RP on each plane. The angle of the maxillary plane relative to the mandibular plane was calculated in the horizontal (b) and frontal (c) views. The discrepancy between the angle of the baseline record and the angle after bite registration was calculated for the physical and the optical methods.*

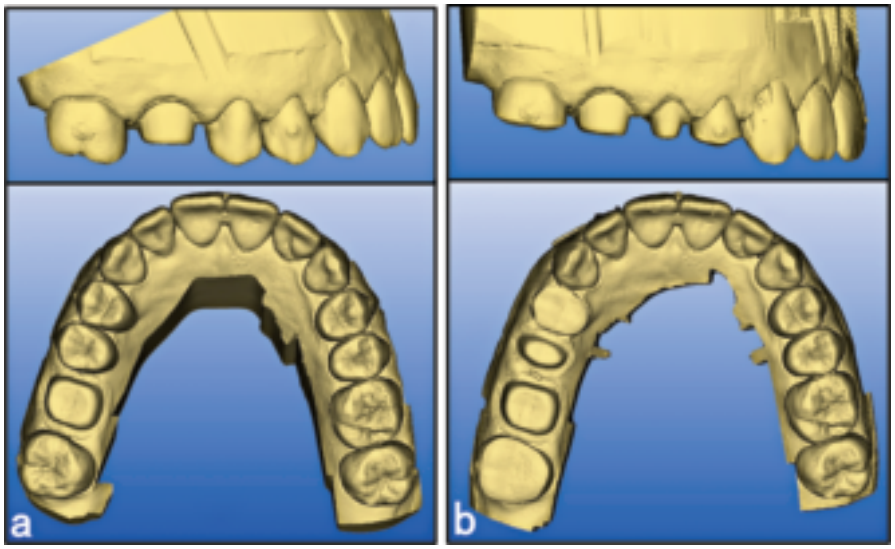

Figure 4. *CAD models. Preparation was made for the single restoration on the maxillary right molar tooth (a) and for the multiple restorations on the premolars and molars in the maxillary right quadrant of the master cast (b).*

second molar were prepared for occlusal onlays without damaging the RPs (Figure 4b). The second premolar was prepared for an all-ceramic restoration with the same regimen as used for the first molar. For the onlay preparation, a tooth reduction of 2.0-mm thickness on the buccal and lingual cusps and of 1.5-mm thickness on the occlusal surface was prescribed. The optical and physical bite registrations were performed with the same procedure as used in the first experiment.

The difference in the discrepancy between the two bite registration methods was statistically analyzed using the Student *t*-test (SPSS 11.5, SPSS Inc, Chicago, IL, USA). The significance level was set at 5%.

RESULTS

For the single preparation, the discrepancy in the distance was not significantly different between the methods on the right restorative side and the anterior site ($p>0.05$) (Table 1). However, on the left intact dentition, the optical method showed a significantly larger discrepancy than the physical method ($p<0.05$). The largest mean discrepancy of 522.6 μm was indicated at the left molar site with the optical method, whereas the physical method resulted in a discrepancy of 77.5 μm. For multiple preparations, the optical method showed significantly larger discrepancy on the right molar and on the left premolar and molars. The largest mean discrepancy of 833.2 μm was indicated at the left molar site by the optical method. For the physical method, the largest discrepancy of 116.8 μm was recorded at the right molar site.

The discrepancy in the horizontal and frontal plane angles between before and after bite registration is shown in Table 2. For the single preparation, the optical method made the upper plane horizontally deviate by only 2° on average, while the physical method made the upper plane deviate approximately 13°, and the difference in the discrepancy was statistically significant ($p<0.05$). For the multiple preparations, the optical method created significantly larger horizontal deviation than did the physical method, but the mean angles obtained by both methods were no more than 3°. From the frontal view, the occlusal planes revealed a rotational deviation; the left side of the maxillary plane was recorded more upwardly than the right side. The optical method created a significantly larger rotational deviation than the physical method for the single and multiple preparations.

DISCUSSION

As shown by the distances between the maxillary and mandibular RPs of the corresponding teeth, the interarch distance increased as a result of the optical and the physical bite registration methods. At the site of the abutment for the single preparation, the optical method created a mean discrepancy of 243.2 μm relative to the baseline, which was insignificantly but slightly lower than the mean discrepancy of 311.1 μm caused by the conventional physical

Table 1: *Mean Discrepancy in Distance Between the Reference Points (RPs) on the Maxillary and Mandibular Teeth*

Preparation	Bite Registration	Distance Discrepancy Relative to Baseline, μm				
		Right Molar	Right Premolar	Anterior	Left Premolar	Left Molar
Single	Optical					
	Mean	243.2	40.1	132	353.1[a]	522.6[a]
	(SD)	31.2	12.8	17.4	94.8	259.2
	Physical					
	Mean	311.1	42.8	137.4	79.9[a]	77.5[a]
	(SD)	33.2	11.8	23.9	7.6	31.3
Quadrant	Optical					
	Mean	554.0[a]	70.7	74.7	441.5[a]	833.2[a]
	(SD)	39.1	7.6	26.4	22.6	90.9
	Physical					
	Mean	116.8[a]	51.9	85.6	54.4[a]	60.4[a]
	(SD)	62	14.4	19.9	10.2	11.4

Abbreviation: SD, standard deviation.
[a] *Significant difference between optical and physical within the same preparation group ($p<0.05$).*

method ($p>0.05$). The difference was significant when the statistical test was based on the significance level of 0.10.

The discrepancy in the optical method at the nonoperational left molar site was significantly larger than that created by the physical method, which was consistent with the larger deviation of the plane angle of the optical method in the frontal view. However, this discrepancy may not cause a critical error in the completed restoration because it was away from the prepared tooth. In contrast, the mean rotational deviation in the horizontal plane was significantly lower for the optical method. In this context, within the scope of the single posterior restoration, it is suggested that the optical bite registration method provides better dimensional accuracy in the interarch relationship in comparison with the conventional physical method.

The increase in the interarch distance via bite registration was largest at the molar site on the nonoperational left side in both the single and multiple preparations. For optical bite registration, matching of the upper and lower occlusal surfaces was digitally carried out based on the images that were scanned from the buccal side of the right posterior dentition. A previous study[15] indicated that the precision of the intraoral camera of this system was 19 μm and that the optical impressions of the full arch obtained with this system are highly accurate when compared with conventional impressions obtained using silicone. However, when reconstructing the full dentition from the partially segmented morphometry, marginal discrepancy in the interarch reproduction was likely to be induced, even though the previous studies[15,16] concluded that the system was still usable. The findings of the current study indicate that accuracy is compromised when matching of the full arch dentition is

Table 2: *Mean Angle Discrepancy of the Maxillary and Mandibular Occlusal Plane Relation Relative to the Baseline Relation (°)*

Preparation	Bite Registration	Horizontal	Frontal
Single	Optical		
	Mean	−2.05[a]	1.76[a]
	(SD)	0.57	0.002
	Physical		
	Mean	13.19[a]	0.18[a]
	(SD)	2.64	0.12
Quadrant	Optical		
	Mean	−2.95[a]	1.77[a]
	(SD)	0.58	0.02
	Physical		
	Mean	−2.33[a]	0.27[a]
	(SD)	5.82	0.08

Abbreviation: SD, standard deviation.
[a] Significant difference between optical and physical within the same preparation group ($p<0.05$).

implemented based on the partial optical bite registration.

For multiple unit preparations, the optical method was significantly less precise in the dimensional accuracy of the interarch relation of the prepared teeth in comparison with the conventional method. Therefore, the result did not support the hypothesis of this study. As a result of the full coverage or the occlusal coverage in onlay preparations, the morphometry of the abutments was considerably simplified from that of the intact teeth. With multiple preparations, the optical impression was less informative for a good match when merging with the antagonist. The results of the current study support recent literature[14] that did not recommend preparation and optical impression of all molars and premolars at the same time when the posterior occlusal support was lost. Instead, the restorations should individually be constructed, with retention of occlusal support by the neighborhood intact teeth.

The optical impression and bite registration method can preclude the use of various materials for fabrication of restorations. The use of an impression material and working cast potentially introduces dimensional errors into the completed restorations. When mounting the casts on the articulator with the physical method, expansion of the plaster[17] as well as technical errors[18] may deteriorate the accuracy of the interarch relationship. Although environmental factors such as optical interference by saliva may potentially cause dimensional errors with the optical system,[19] it is suggested that the method was effective, especially for a single posterior restoration, and further development of this new and promising technology is strongly encouraged.

CONCLUSIONS

The hypothesis that the optical method increases the accuracy of interarch registration was not supported by the results of this study. However, in a single posterior restoration, the optical method provides better dimensional accuracy in the interarch relationship in comparison with the conventional physical registration.

Conflict of Interest

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

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The Effect of a 10% Carbamide Peroxide Bleaching Agent on the Microhardness of Four Types of Direct Resin-based Restorative Materials

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Clinical Relevance

Ten percent carbamide peroxide bleaching agents may minimally reduce the microhardness of microhybrid type of resin-based composite materials compared with a significant microhardness reduction of three other types: nanofilled, silorane-based low shrink, and hybrid.

SUMMARY

Purpose: **This *in vitro* study was undertaken to evaluate the effect of a 10% carbamide peroxide bleaching agent on the microhardness of four types of direct resin-based restorative materials.**

Materials and Methods: **Thirty disk-shaped specimens (10.0 mm diameter × 2.0 mm depth) of each material, including a microhybrid resin composite (Z250), a nanofilled resin composite (Z350), a silorane-based low-shrink resin composite (P90), and a hybrid resin composite (Valux Plus), were fabricated and then polished with medium, fine, and superfine polishing discs. After being polished, specimens were cleaned with distilled water for 2 min in an**

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ultrasonic bath to remove any surface debris and then stored in distilled water at 37°C for 24 hours. Specimens from each material were divided into three groups (n=10). One group was selected as a control group (nontreated with bleaching agent). The other two groups were treated with bleaching agent for 14 days (group A) and for 14 days followed by immersion in artificial saliva for 14 days (group B). The top surfaces of the specimens in the different groups were also subjected to the Vickers hardness test with a load of 300 g and 15-second dwell time. Data were analyzed with a one-way analysis of variance and Tukey's HSD test ($\alpha = 0.05$).

Results: **There was a general reduction of Vickers hardness numbers (VHN) values of treated groups compared with the control group for each material used, but this reduction was minimal, with no significant difference between groups in Z250, whereas the other three materials (Z350, P90, and Valux Plus) showed a significant reduction of VHN of treated groups compared with the control group. Conversely, the findings showed no significant difference between treated groups A and B in all materials used except P90.**

Conclusion: **A 10% carbamide peroxide bleaching agent had an adverse effect on the microhardness of nanofilled, silorane-based low-shrink, and hybrid types of resin-based composite materials compared with the microhybrid type.**

INTRODUCTION

Several esthetic procedures have been described in the literature to alter the appearance of smiles, including alterations in the form, texture, position, and color of teeth. The most conservative and noninvasive of these is vital bleaching.[1-3]

All tooth-whitening procedures use either hydrogen peroxide or carbamide peroxide. Currently available home-bleaching agents often contain up to 10% hydrogen peroxide or 22% carbamide peroxide as active ingredients, applied to the teeth via a ready-made or custom-fabricated tray.[4] The most common at-home bleaching agent is 10% carbamide peroxide because of its favorable clinical results, effectiveness, and safety.[3-5]

Patients seeking bleaching agents may have metal- or resin-based or other kinds of restorations in anterior and/or posterior teeth. It is possible that the clinical durability of tooth-colored restorations might be affected by the chemical processes of bleaching agents.[6,7]

The effects of bleaching agents include alterations in the surface morphology and chemical and physical properties of dental restorative materials.[8] One of the most important physical properties of a restorative material is its surface hardness.[9] Surface microhardness is defined as resistance of a material to indentation or penetration.

Several studies that evaluated the effects of bleaching agents on the microhardness of resin-based restorative materials have reported conflicting results. Some found decreased surface microhardness after the bleaching process[10-12] and others did not.[7,13,14]

The conflicting results of the data published about the effects of dental bleaching agents on the microhardness of resin-based composite materials and the resin-based composite restorative materials newly introduced to the market are further reasons for more research to be conducted.

Therefore, this *in vitro* study was undertaken to evaluate the effect of a 10% carbamide peroxide bleaching agent on the microhardness of four types of direct resin-based restorative materials.

The hypothesis tested was that there would be no effect of a 10% carbamide peroxide bleaching agent on the microhardness of four types of direct resin-based restorative materials.

MATERIALS AND METHODS

Specimen Fabrication

The resin-based composite materials evaluated in the current study and their compositions, with the home bleaching agent, are shown in Table 1: (microhybrid) Filtek Z250 (3M ESPE, St Paul, MN, USA), (nanofilled) Filtek Z350 (3M ESPE), (silorane-based low-shrink) Filtek P90 (3M ESPE), and (hybrid) Valux Plus (3M ESPE). For each resin-based composite, 30 disk-shaped specimens (10.0 mm diameter × 2.0 mm depth) were fabricated in shade A3. Cylindrical rubber molds were positioned on a transparent plastic matrix strip lying on a glass plate. The resin-based composite materials were placed in 2.0-mm increments. After the materials were inserted into the mold, a transparent plastic matrix strip was put over them, and a glass plate was secured to flatten the surface. Every specimen was light polymerized for 40 seconds, by means of a halogen light (Elipar 2500, 3M ESPE) within the

Table 1: *Materials Used in This Study*

Material	Composition	Material	Manufacturer
Filtek Z250	Matrix: Bis-GMA, UDMA, and Bis-EMA	Micro-hybrid resin composite	3M ESPE Dental Products, St Paul, MN, USA
	Filler: zirconia/silica (0.01–3.5 μm)		
	Filler by volume: 60%		
Filtek Z350	Matrix: Bis-GMA, UDMA, TEGDMA, and Bis-EMA	Nanofilled resin composite	3M ESPE Dental Products, St Paul, MN, USA
	Filler: Combination of aggregated zirconia/silica cluster filler (0.6-1.4 μm) and nonaggregated 20-nm silica filler		
	Filler volume: 59.5%		
Filtek P90	Matrix: New ring-opening silorane	Low-shrink resin composite	3M ESPE Dental Products, Seefeld, Germany
	Filler: Epoxy functional silane-treated SiO_2 and ytterbium fluoride (0.1–2 μm)		
	Filler volume: 55%		
Valux Plus	Matrix: Bis-GMA and TEGDMA	Hybrid resin composite	3M ESPE Dental Products, St Paul, MN, USA
	Filler: Single filler 100% zirconia/silica (0.01–3.5 μm)		
	Filler volume: 66%		
Opalescence PF	10% carbamide peroxide, potassium nitrate, carbopol, glycerine, 0.11% fluoride ion, flavoring; pH = 6.7	At-home Bleaching	Ultradent Products Inc, South Jordan, UT, USA

Abbreviations: Bis-EMA, bisphenol-A polyethylene glycol dietherdimethacrylate; Bis-GMA, bisphenol-glycidylmethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

range of 480 to 520 mW/cm^2 and verified with a curing radiometer (Optilux Model 100, SDS Kerr, Danbury, CT, USA). The specimens were polished with medium, fine, and superfine polishing discs (Sof-Lex, 3M ESPE) on a slow-speed hand piece rotating in one direction. The final thickness of the polished specimens was verified by a micrometer (Ultra-Cal Mark III, Fowler Tools and Instruments, Sylvac, Newton, MA, USA). After being polished, specimens were subjected to ultrasonic cleaning with distilled water for 2 minutes to remove any surface debris. All specimens were stored in distilled water at 37°C for 24 hours. Specimens from each material were divided randomly into three groups (10/group):

- Control group: Immersed for 14 days in artificial saliva, with no bleaching treatment
- Group A: Treated with a 10% carbamide peroxide bleaching agent for 14 days
- Group B: Treated with a 10% carbamide peroxide bleaching agent for 14 days and then immersed in artificial saliva for 14 days

Control Group

The specimens in the control group were stored in artificial saliva for 14 days at 37°C and no bleaching, followed by immersion in distilled water for 24 hours at 37°C in preparation for the microhardness test. The artificial saliva was replaced daily**.** Artificial saliva was composed of sodium chloride (NaCl) 0.4 g, potassium chloride (KCl) 0.4 g, calcium chloride ($CaCl_2.H_2O$) 0.795 g, sodium-dihydrogen phosphate ($NaH_2PO_4.H_2O$) 0.69 g, sodium sulfide ($Na_2S.9H_2O$)

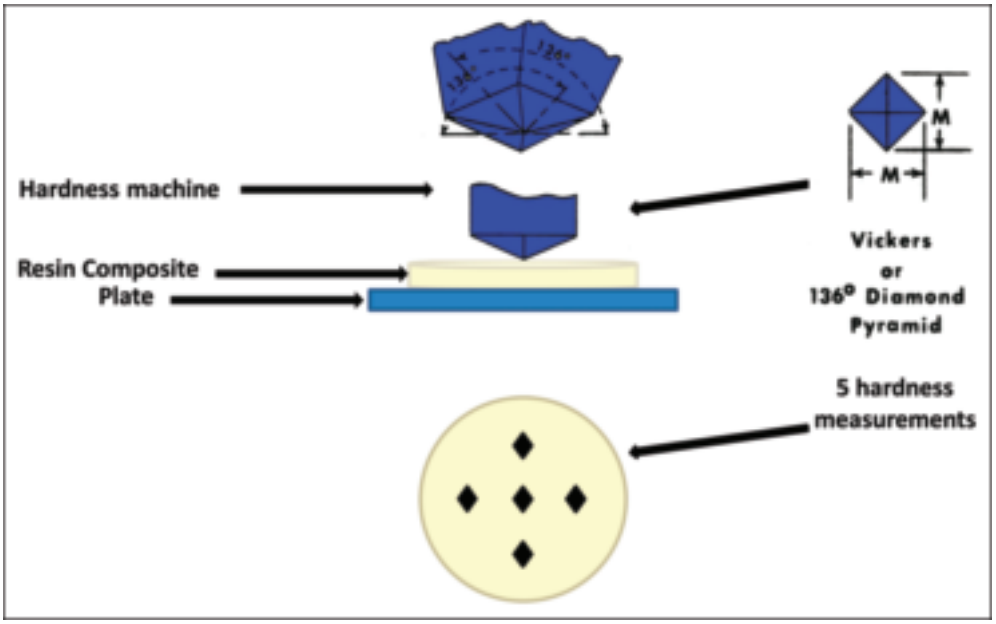

Figure 1. *Schematic showing how specimens were placed under testing facing the Vickers surface hardness diamond indenter.*

0.005 g, and distilled water 1000 mL, and the pH was adjusted to 7.[15]

Bleaching Procedure

The bleaching procedure was performed on the top surfaces of the specimens in groups A and B, with an at-home bleaching material (Opalescence PF 10% Carbamide Peroxide, Ultradent Products Inc, South Jordan, UT, USA). The bleaching agent covered the top surfaces of the specimens at a thickness of 1.0 mm. At the end of every bleaching application, the treated specimens were washed, first with a soft toothbrush under flowing distilled water and then in an ultrasonic cleaner for 5 minutes. They were then placed in fresh artificial saliva for 16 hours at 37°C until the next application. The artificial saliva was replaced daily. In groups A and B, bleaching gel was applied for 8 hours daily for 14 consecutive days at 37°C, to simulate at-home bleaching. In group A, after the 14-day bleaching procedure, all specimens were stored in distilled water for 24 hours at 37°C in preparation for the microhardness test. In group B, after the 14-day bleaching procedure, all specimens were stored in artificial saliva for 14 days at 37°C, followed by immersion in distilled water for 24 hours at 37°C, in preparation for the microhardness test.

Vickers Hardness Test

After the specimens were dried, the Vickers surface hardness test was administered in a universal testing machine (Micromet 2100 series microhardness tester, Buehler, Lake Bluff, IL, USA). The specimens were placed on the platform, with the surface being tested facing the diamond indenter. A load of 300 g was applied to the surface for a 15-second dwell time. Five indentations were made on the top surface and in the middle of each specimen, not closer than 1 mm to the adjacent indentations (Figure 1).

Statistical Analysis

Statistical calculations were performed with SPSS version 16.0 software (SPSS Inc, Chicago, IL, USA). Data were subjected to a one-way analysis of variance (ANOVA) and Tukey's HSD multiple-comparisons test, with the probability for statistical significance set at $\alpha = 0.05$.

RESULTS

Table 2 and Figure 2 show the means and standard deviations of the Vickers Hardness Numbers (VHN) of the specimens in the control group and after the bleaching procedure in the other two groups. The different statistical analyses (one-way ANOVA and Tukey's HSD multiple-comparisons test) are summarized in Table 2.

One-way ANOVA showed a significant difference between the behaviors of the different resin-based composite materials under the different conditions of at-home bleaching in groups A and B compared with the control group ($p<0.0001$).

Table 2: *Vickers Hardness Numbers Means and Standard Deviations of Different Resin-Based Composite Materials Under Different Conditions of Bleaching Compared With Control Group and the Statistical Analysis Summary*[a]

Resin-Based Composite Materials	Resin-Based Composite Types	Vickers Hardness ± SD			One-Way ANOVA	Tukey's HSD Test		
		Group	n	Value	*p* Value	Group	Group	*p* Value
Filtek Z-250	Microhybrid	Control	10	88.22 ± 5.37	<0.0001	Control	A	0.872
		A	10	87.36 ± 2.67		Control	B	0.855
		B	10	87.30 ± 2.92		A	B	0.999
Filtek Z-350	Nanofilled	Control	10	84.64 ± 3.16		Control	A	<0.0001
		A	10	73.98 ± 2.51		Control	B	<0.0001
		B	10	73.74 ± 3.33		A	B	0.983
Filtek P-90	Low-shrink	Control	10	75.68 ± 0.75		Control	A	<0.0001
		A	10	61.76 ± 4.23		Control	B	<0.0001
		B	10	57.46 ± 2.09		A	B	0.005
Valux Plus	Hybrid	Control	10	125.80 ± 5.22		Control	A	<0.0001
		A	10	110.60 ± 4.60		Control	B	<0.0001
		B	10	110.40 ± 3.69		A	B	0.995

[a] *Control = no bleaching; group A = 14-day bleaching; group B = 14-day bleaching followed by 14-day immersion in artificial saliva. Significant difference at* $p \leq 0.05$.

VHN mean values of Filtek Z250 were minimally reduced in the different bleaching groups (A and B) compared with that of the control group, but with no significant difference ($p=0.872$ and $p=0.855$, respectively).

VHN mean values of Filtek Z350 in groups A and B were significantly reduced compared with that of the control group ($p<0.0001$), whereas there was no significant difference between the VHN mean values of groups A and B ($p=0.983$).

VHN mean values of Filtek P90 in the different bleaching groups, A and B, were significantly reduced compared with that of the control group ($p<0.0001$), and the VHN mean value of bleaching group B was significantly reduced compared with that of group A ($p=0.005$).

VHN mean values of Valux Plus in the different bleaching groups, A and B, were significantly reduced compared with that of the control group ($p<0.0001$), whereas there was no significant difference between the VHN mean values of groups A and B ($p=0.995$).

DISCUSSION

Hardness of a material is defined as its resistance to permanent surface indentation or penetration, and this property is related to a material's strength, ductility, elastic stiffness, plasticity, strain, toughness, viscoelasticity, and viscosity. The ability of a material to abrade or to be abraded by opposing dental structures, materials, or any chemical softening has implications for the clinical durability of dental restorations.[16]

The temperature in our study was fixed at 37°C for both bleached and unbleached groups. This was in accordance with the findings of Yu and others[17] who reported that bleaching at 37°C produced a reduction

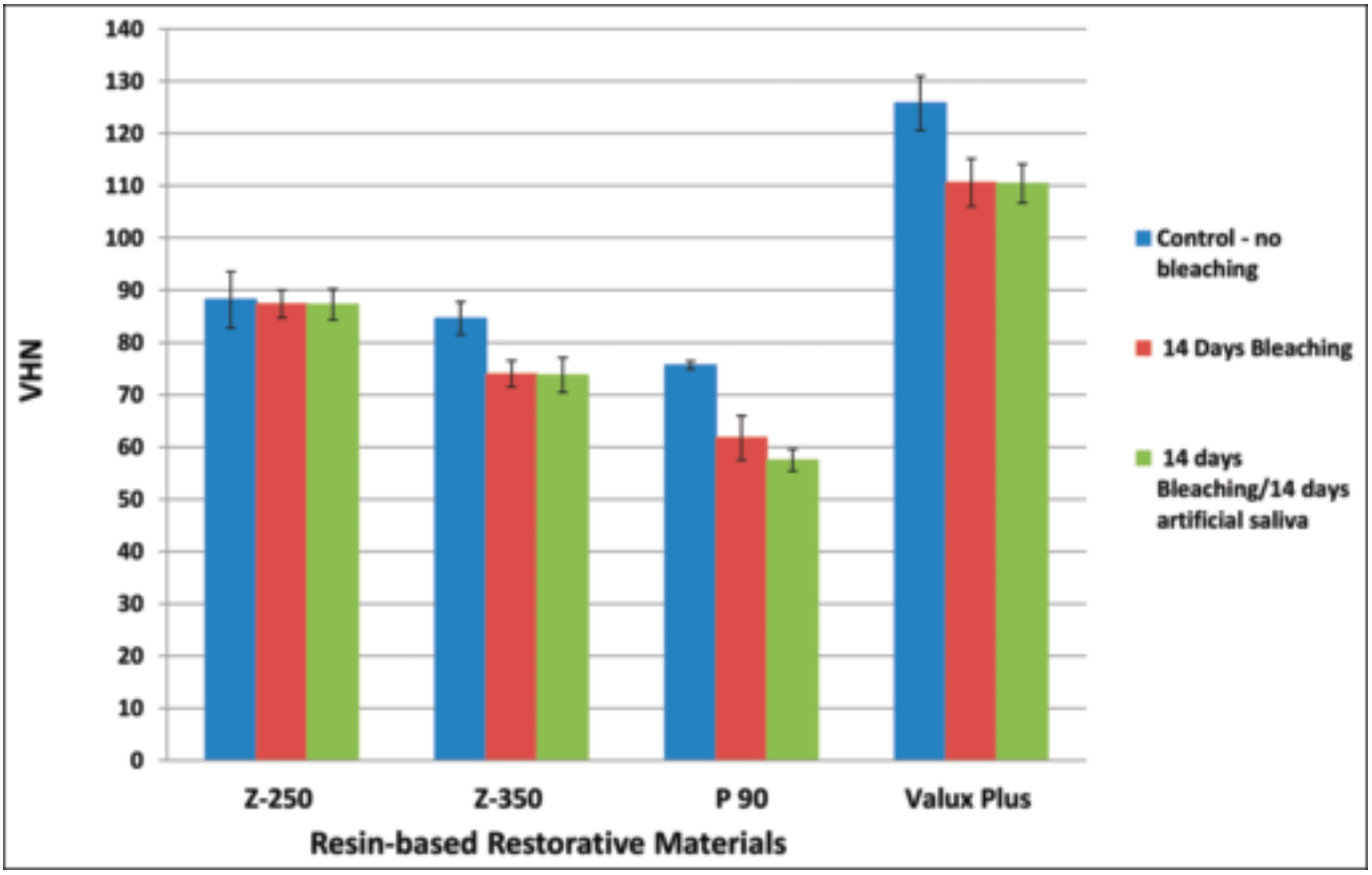

Figure 2. *Vickers hardness numbers means and standard deviations of tested resin-based composite materials under different conditions of bleaching compared with the control group.*

in surface microhardness of composite resin, compomer, and glass-ionomer cement compared with bleaching at 25°C, and that no significant differences were found in the microhardnesses of unbleached groups stored at 25°C and 37°C.

Based on the results of this study, the null hypothesis was rejected, because the use of a 10% carbamide peroxide bleaching agent at 37°C did affect the microhardness of the resin-based restorative materials. This finding is in accordance with those of previous studies, which reported that the microhardness of all dental restorative materials except ceramic was reduced after the application of bleaching agents.[17-19] Conversely, the finding is contrary to those of other studies reporting that the use of bleaching agents had no effect on the microhardness of composite resin, compomer, and glass-ionomer cement.[6,20]

The bleaching gel used in this study contained 10% carbamide peroxide. This type of gel is unstable and degrades immediately into approximately one-third hydrogen peroxide and two-thirds urea on contact with tissue and saliva. After the degradation of carbamide peroxide, the hydrogen peroxide then breaks down into free radicals, which may induce oxidative cleavage of polymer chains and then lead to chemical softening of the dental materials.[6,21] Carbamide peroxide had a greater effect on the microhardness of resin-based composite and glass-ionomer materials at a higher environmental temperature (37°C) compared with a low temperature (25°C), and this might be due to the release of more free radicals at the higher temperature.[17]

The composition of the artificial saliva used in this study acted as an accelerator in the degradation of carbamide peroxide, to mimic the oral environment, and may minimize the adverse effects of bleaching and inhibit microhardness reduction by means of salivary remineralization potential due to the presence of calcium and phosphate ions.[22] In contrast, the findings of this study showed that the use of artificial saliva as a storage medium during and after the bleaching procedure had no benefit,

and the reduction in microhardness of the different resin-based composite materials was not inhibited.

The microhardness reduction percentages of the different resin-based composite materials (Filtek Z250, Filtek Z350, Filtek P90, and Valux Plus), after exposure to the same bleaching regimen, were 0.97%, 12.59%, 18.39%, and 12.08%, respectively, which might be related to the different resin matrices, filler contents, and particle sizes. This is in agreement with the results of a study by Hannig and others.[23]

In our study, nanofilled (Z350), silorane-based low-shrink (P90), and hybrid (Valux) resin-based composite materials showed significant reductions in microhardness values after the bleaching procedure, compared with the minimal and nonsignificant reduction in microhardness demonstrated by the microhybrid (Z250) composite.

This might be related to the presence of a higher amount of TEGDMA in the Z350 and Valux Plus resin-based composite materials and the absence of TEGDMA in Z250. The incorporation of TEGDMA diluent monomers in the resin matrix may make the resin matrix less resistant to bleaching agents and may increase the softening of resin composite material.[24] In contrast, the reduction in microhardness of the Z350 was higher than that of Valux Plus, which might be related to the presence of the high molecular weight of the resin matrix and the lower filler content in Z350 compared with Valux Plus.[25]

According to the results, the silorane-based low-shrink (P90) material showed more reduction in microhardness compared with the other tested materials; however, this might be due to the presence of a new ring-opening silorane resin matrix and lower filler content. This type of resin matrix might be softer than other resin matrices (Bis-GMA, UDMA, and Bis-EMA) and easily soluble by bleaching agents.

Furthermore and based on the findings of this current study, bleaching agents should not be used indiscriminately in the patient's mouth, and the teeth that have extensive tooth-colored restorations should not be exposed to bleaching agents or at least protected. Finally, patients should be informed that the physical properties of tooth-colored restorations might be affected by the bleaching procedure, and the restorations might be softened. This could potentially predispose to increased adherence of cariogenic bacteria, surface wear rate, stain absorption, and potential marginal/edge strengths of these restorations and that they may need to be replaced.

CONCLUSIONS

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

1. A 10% carbamide peroxide bleaching agent had an adverse effect on the microhardness of nanofilled, silorane-based low-shrink, and hybrid types of resin-based composite materials compared with the microhybrid type.
2. The microhardness reduction in different resin-based composite materials after bleaching was not inhibited by the use of artificial saliva storage media during and after the bleaching procedure.

Acknowledgement

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Conflict of Interest

The author of this article certifies that there is 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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Temperature Increase at the Light Guide Tip of 15 Contemporary LED Units and Thermal Variation at the Pulpal Floor of Cavities: An Infrared Thermographic Analysis

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Clinical Relevance

The thermal variation at the pulpal floor of dental cavities during photopolymerization is associated with the measured irradiance level of LED curing units.

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SUMMARY

In this study, a comprehensive investigation on the temperature increase at the light guide tip of several commercial light-emitting diode (LED) light-curing units (LCUs) and the asso-

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ciated thermal variation (ΔT) at the pulpal floor of dental cavities was carried out. In total, 15 LEDs from all generations were investigated, testing a quartz-tungsten-halogen (QTH) unit as a reference. The irradiance level was measured with a power meter, and spectral distribution was analyzed using a spectrometer. Temperature increase at the tip was measured with a type-K thermocouple connected to a thermometer, while ΔT at the pulpal floor was measured by an infrared photodetector in class V cavities, with a 1-mm-thick dentin pulpal floor. The relationship among measured irradiance, ΔT at the tip, and ΔT at the pulpal floor was investigated using regression analyses. Large discrepancies between the expected and measured irradiances were detected for some LCUs. Most of the LCUs showed an emission spectrum narrower than the QTH unit, with emission peaks usually between 450 and 470 nm. The temperature increase at the tip followed a logarithmic growth for LCUs with irradiance ≥1000 mW/cm^2, with ΔT at the tip following the measured irradiance linearly (R^2=0.67). Linear temperature increase at the pulpal floor over the 40-second exposure time was observed for several LCUs, with linear association between ΔT at the pulpal floor and measured irradiance (R^2=0.39) or ΔT at the tip (R^2=0.28). In conclusion, contemporary LED units show varied irradiance levels that affect the temperature increase at the light guide tip and, as a consequence, the thermal variation at the pulpal floor of dental cavities.

INTRODUCTION

Dental dimethacrylate-based restorative composites polymerize via free-radical polymerization under irradiation with visible light. Development of the polymerization reaction is influenced by many factors related to the composite, such as comonomer formulation,[1-4] shade,[5] translucency,[6] and filler loading/size,[7] as well as to the curing unit, such as irradiance level,[8-10] exposure time,[11,12] emission spectrum,[13] and thermal variation.[14,15] Sufficient light energy reaching the composite is necessary to ensure adequate polymerization; the spectral irradiance of the light also has to overlap as much as possible the absorption spectrum of the photosensitizer contained in the material, that is, camphorquinone (CQ).[3,4]

Blue light-emitting diodes (LEDs) were introduced to overcome shortcomings of the traditional quartz-tungsten-halogen (QTH) light-curing units (LCUs). LEDs consume little power in operating and do not require filters to produce blue light. The main difference in the emission radiation is the narrower spectrum of wavelengths of the LEDs, usually centered on 470 nm, which is the wavelength at which CQ has its maximum energy absorption.[16] The semiconductors used for light emission in LEDs, instead of the hot metal filaments in QTH bulbs, generate less internal heating and undergo little degradation over the course of the time. First-generation LEDs emitted low irradiance, hence they generated low heat and were considered "cold-light devices." However, as high-irradiance, second- and third-generation LEDs were introduced, the increase in irradiance was associated with a thermal increase.[9]

It has been reported that external heating may enhance the conversion kinetics of resin composites and increase the network cross-linking.[14,15] Increased temperature may enhance the mobility of reactive species during polymerization, ultimately resulting in further conversion. Therefore, LCUs with low irradiance levels, despite tending to reduce the heat generation, could pose a risk to increased cytotoxicity and poorer mechanical properties of the restorative due to lower conversion. There is also a controversial issue regarding the critical temperature increase that may cause injury to the dental pulp. Zach and Cohen[17] reported that an increase of 5.5°C within the pulp chamber caused irreversible damage to monkey teeth. Another investigation, however, showed that a temperature increase of 11.2°C did not damage the pulp.[18] Laboratory studies usually analyze the temperature variation at the light guide tip of the LCU, but the thermal variation (ΔT) yielded at the pulpal floor or chamber[19,20] is probably more relevant as regards the biological issues related to high-irradiance curing units.

The aim of the present study was to provide a comprehensive investigation on the temperature increase at the light guide tip of commercially available LED LCUs and the associated ΔT at the pulpal floor of class V dental cavities during photopolymerization procedures. In total, 15 LEDs from all generations were investigated, testing a QTH unit as a reference. The hypothesis tested was that increased temperature at the light guide tip of the LCUs would be associated with increased ΔT at the pulpal floor of the cavities.

MATERIALS AND METHODS

LED Units Tested, Irradiance, and Emission Spectrum

Fifteen commercial LED units were tested, as shown in Table 1. The conventional QTH unit Optilux 501 (Kerr, Orange, CA, USA) was tested as a reference. The units (when applicable) were fully charged before testing. The irradiance level of each unit was measured five times with a calibrated power meter (Ophir Optronics, Jerusalem, Israel), and the spectral distribution was analyzed using a computer-controlled spectrometer (USB2000, Ocean Optics, Dunedin, FL, USA). The radiant exposure was calculated on the basis of a 40-second exposure time.

Temperature Increase at the Light Guide Tip

Measurements were carried out under controlled temperature (25°C) and humidity (45%) conditions using a type-K thermocouple (Salcas, São Paulo, SP, Brazil) connected to a digital thermometer accurate to 0.1°C, with 0.1-second response time. After calibration in water baths with temperatures ranging between 25°C and 40°C, the thermocouple was positioned in contact with the light guide tip. The temperature was recorded every 5 seconds during the total 40-second exposure time.

ΔT at the Pulpal Floor

The lingual faces of 150 bovine incisors were removed. Standard class V cavities (2.5 mm wide × 3 mm long) were prepared 1 mm above the cemento-enamel junction in the buccal faces using water-cooled #3099 cylindrical diamond burs (KG Sorensen, Barueri, SP, Brazil) in a high speed handpiece. Burs were replaced after every four preparations. The cavity depth was controlled in order to standardize a 1-mm dentin thickness at the pulpal floor of all cavities. The teeth were held in position using a custom-made device, and temperature at the pulpal floor was measured by infrared thermographic analysis using a handheld quantum well infrared photodetector (ThermaCAM SC 3000, FLIR Systems Inc, Boston, MA, USA) with 0.01°C accuracy and 0.01-second response time. The thermographic camera was directed to the pulpal chamber through the exposed lingual face of the teeth at a 10-cm distance between the sample and camera. The camera was calibrated considering the dentin emissivity to be 0.91 within the temperature range of 20°C to 100°C,[21] with data acquisition at 60 Hz. Measurements were carried out under controlled temperature (25°C) and humidity (45%) conditions. The LED units were placed in contact with the buccal aspects of the teeth, and light exposure was carried out for 40 seconds (n=10); data were processed using the software ThermaCAM Research 2001 (FLIR Systems Inc).

Statistical Analysis

Data for ΔT measured by the thermocouple/thermometer setup and ΔT data from the thermographic analysis were separately submitted to one-way analysis of variance followed by the Tukey's *post hoc* test. The relationship among measured irradiance, ΔT at the light guide tip, and ΔT at the pulpal floor was investigated using linear regression analyses. All analyses were conducted at a 5% significance level.

RESULTS

Irradiance, Energy Dose, and Emission Spectrum

Table 1 shows the characteristics of the LCUs tested. In order to facilitate comparisons, the LCUs were separated into four groups. Large discrepancies between the expected and measured irradiance levels were detected for most of the LCUs, especially those with measured irradiance <800 mW/cm^2. Results for the calculated energy dose are shown in Table 2; a wide range between 8 and 48 J/cm^2 was calculated. The spectral distribution of the lights is shown in Figure 1. Most of the LCUs showed an emission spectrum narrower than the QTH unit (OPL), with emission peaks usually centered on the range between 450 and 470 nm.

Temperature Increase at the Light Guide Tip

Figure 2 shows the profiles of temperature increase at the tip over the 40-second exposure time. Results for ΔT at the tip are shown in Table 2. The statistical analysis showed significant differences among the groups (p<0.001). The LCUs with measured irradiance >800 mW/cm^2 showed significantly higher ΔT at the tip than the LCUs with lower irradiance levels. The QTH unit (OPL) showed significantly higher ΔT than all LED units with irradiance ≤800 mW/cm^2. The ΔT profiles followed a logarithmic growth pattern for the LCUs with irradiance ≥1000 mW/cm^2.

ΔT at the Pulpal Floor

Figure 3 shows the profiles of temperature increase at the pulpal floor over the 40-second exposure time; a linear temperature increase was observed for

Table 1: *Characteristics of the Light-Curing Units (LCUs) Tested*

LCU	Manufacturer	Code	Emission Mode	Expected Irradiance,[a] mW/cm^2	Measured Irradiance, mW/cm^2
LCUs<450 mW/cm^2					
Ultra Blue IV Plus	DMC, São Carlos, SP, Brazil	UTB	Continuous	600	200
Biolux	Bioart, São Carlos, SP, Brazil	BIO	Soft-start	300 (5 s)/1000 (35 s)	200 (5 s)/400 (35 s)
Radii Plus	SDI, Bayswater, Victoria, Australia	RDP	Continuous	1500	410
Optilux 501 (QTH)	Demetron Kerr, Orange, CA, USA	OPL	Continuous	850	448
LCUs<800 mW/cm^2					
LEC 470 II	MM Optics, São Carlos, SP, Brazil	LEC	Continuous	400	475
Mais	Mais, Ribeirão Preto, SP, Brazil	MAI	Continuous	700	500
UltraLight III	Sanders Medical, S. R. Sapucaí, MG, Brazil	UTL	Soft-start	1200	200 (5 s)/600 (35 s)
Elipar Freelight 2	3M ESPE, St. Paul, MN, USA	EFL	Continuous	1200	795
LCUs≤1000 mW/cm^2					
LEDemetron	Demetron Kerr	LDM	Continuous	800	800
Smart Lite PS	Dentsply Caulk, Milford, DE, USA	SML	Continuous	950	995
Celalux	Voco, Cuxhaven, Germany	CEL	Continuous	1000	1000
Ultra-Lume LED 5	Ultradent, South Jordan, UT, USA	ULM	Continuous	800	1000
LCUs>1000 mW/cm^2					
Demi	Demetron Kerr	DEM	Pulsatile	1100	1015
Bluephase	Ivoclar Vivadent, Schaan, Liechtenstein	BLP	Continuous	1200	1180
Blue Star I	Microdont, São Paulo, SP, Brazil	BST	Continuous	1000	1200
FLASHlite 1401	Discus Dental, Culver City, CA, USA	FLH	Continuous	1400	1200

[a] *As provided by the manufacturer.*

Table 2: *Means (SD) for Energy Dose, Thermal Variation (ΔT) at the Tip, and ΔT at the Pulpal Floor*

LCU	Energy Dose,[a] J/cm^2	ΔT (Tip), °C	ΔT (Pulpal Floor), °C
LCUs<450 mW/cm^2			
UTB	8	3.8 (0.1) H	2.4 (0.04) H
BIO	15	4.6 (1.6) H	3.4 (0.02) GH
RDP	16.4	7.1 (0.1) GH	5 (0.08) EFG
OPL (QTH)	17.9	15.3 (0.8) E	6.3 (1.8) CDEF
LCUs<800 mW/cm^2			
LEC	19	9.8 (0.3) FG	4.7 (0.09) FG
MAI	20	9.3 (0.3) GH	3.9 (0.06) GH
UTL	22	7.8 (0.4) GH	3.4 (0.9) GH
EFL	31.8	12.1 (0.2) FG	4.9 (0.1) EFG
LCUs≤1000 mW/cm^2			
LDM	32	12 (1.0) FG	6.7 (1.17) CDE
SML	39.8	22 (0.8) D	11.3 (0.07) A
CEL	40	21.6 (0.8) D	4.9 (0.09) FG
ULM	40	38.8 (0.5) B	9 (1.7) AB
LCUs>1000 mW/cm^2			
DEM	40.6	28 (5.9) C	2.3 (0.12) H
BLP	47.2	20 (0.3) D	10.6 (0.15) BC
BST	48	26.5 (0.3) C	7.2 (0.12) CD
FLH	48	45 (2.0) A	7.7 (0.13) ABC

[a] *Calculated on the basis of a 40-second exposure time. In each column, distinct letters indicate significant differences (p<0.05).*

several LCUs. Except for OPL (QTH), none of the LCUs with measured irradiance up to 800 mW/cm^2 showed maximum temperature increase above 5.5°C, while only CEL showed maximum temperature increase below 5.5°C for the LCUs with measured irradiance >800 mW/cm^2. Results for ΔT at the pulpal floor are shown in Table 2. The statistical analysis showed significant differences among groups ($p<0.001$). Similarly to the ΔT at the light guide tip, the LCUs with measured irradiance >800 mW/cm^2, except for CEL and DEM, showed significantly higher ΔT at the tip than the LCUs with lower irradiance levels.

Linear Regression Analyses

Regression plots are shown in Figure 4. It was observed that ΔT at the light guide tip of the LCUs follows linearly the measured irradiance ($R^2=0.67$; $p<0.001$) and ΔT at the pulpal floor ($R^2=0.28$; $p=0.04$). The relationship ΔT the pulpal floor and measured irradiance is also linear ($R^2=0.39$; $p<0.01$).

DISCUSSION

The present results indicate that large discrepancies between the expected and measured irradiance levels emitted by some LED LCUs were observed. The measured irradiances were, sometimes, less than one-third the irradiance the manufacturers originally claimed. This evidence reinforces the fact that clinicians should be cautious about manufacturers' claims. Each resin composite has a minimum light exposure time indicated for proper polymerization, and clinicians tend to follow this time; however, shorter exposure times could sound reasonable when high-intensity units are used. A way to avoid problems is to always check the irradiance level of the LCUs, regardless of the manufacturers' claims, and always follow the photoactivation time indicated for the composite. The present results show that the energy doses calculated based on a 40-second photoactivation time also varied significantly, although the radiant exposure calculated for most of the LCUs was above 20 J/cm^2. Care should be taken again because a 40-second exposure time is usually twice the time indicated for most of the resin composites available in the market, and very low radiant exposures could be delivered by some LCUs in shorter photoactivation times. In addition, it has been reported that the irradiance delivered from LCUs is not uniform across the light tip,[22] which may also hinder calculation of energy doses applied to the restorative materials.

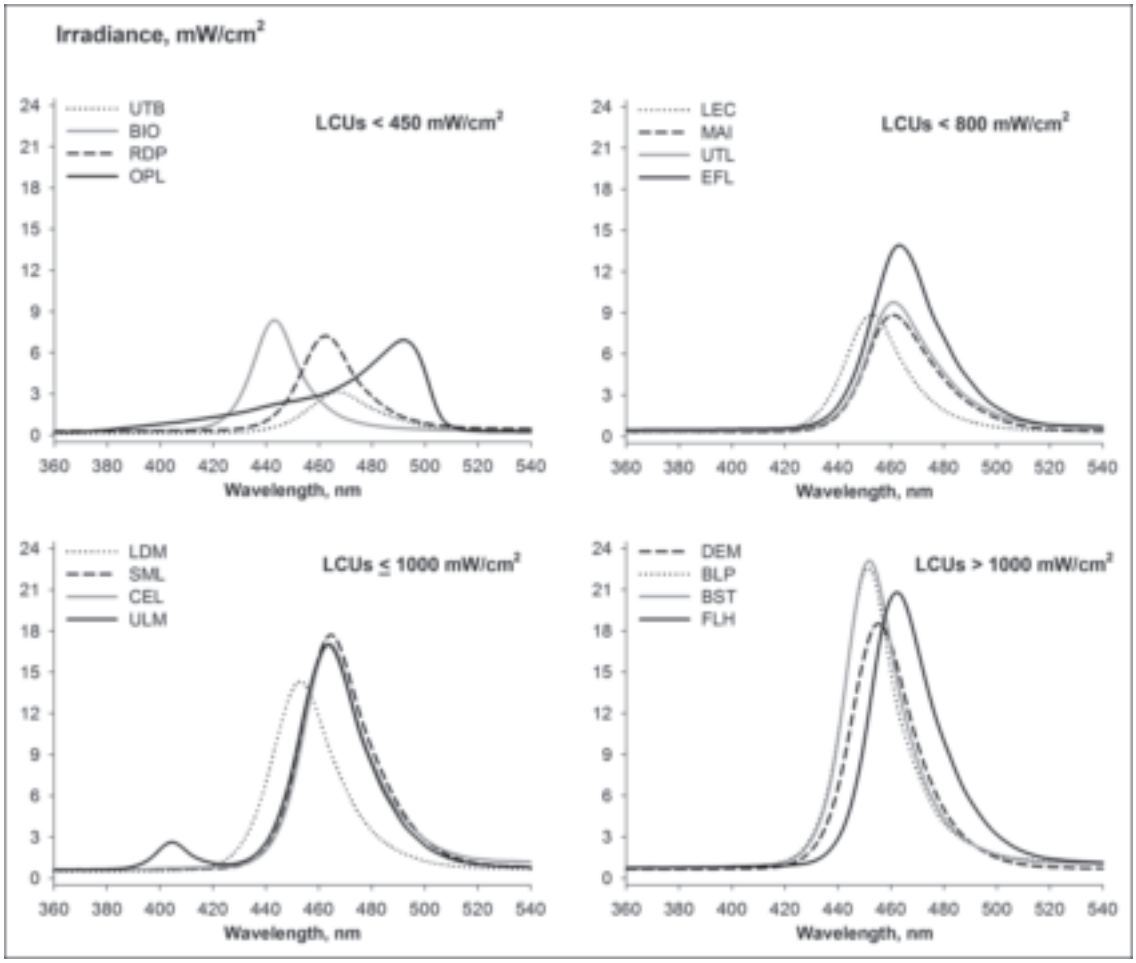

Figure 1. *Spectral emission of the light-curing units (LCUs) tested: Ultra Blue IV Plus (UTB), Biolux (BIO), Radii Plus (RDP), Optilux 501-QTH (OPL), LEC 470 II (LEC), Mais (MAI), UltraLight III (UTL), Elipar Freelight 2 (EFL), LEDemetron (LDM), Smart Lite PS (SML), Celalux (CEL), Ultra-Lume LED 5 (ULM), Demi (DEM), Bluephase (BLP), Blue Star I (BST), and FLASHlite 1401 (FLH).*

The light spectral emission of the LEDs tested was usually centered on the 450- to 470-nm range, with narrower emission distribution compared with the QTH unit. This is important information because efficient correlation between the light spectral irradiance and the absorption spectrum of the photosensitizer contained in the restorative may overcome problems related to low light irradiance levels.[19] It is known that free-radical polymerization of methacrylates, among other factors, is dependent on the number of photons available in the area around the peak of major absorption of the photosensitizer (for CQ, at 468 nm).[16] Variation in radiant exposure may significantly impact the polymerization kinetics of photoactivated restoratives as well the mechanical properties of the resulting polymer.[11]

Regarding the thermal variation results, the ΔT at the light guide tip of the LCUs was linearly associated with the measured irradiance of the LCU. This is explained by the fact that higher light power outputs are associated with increased generation of radiation energy. Compared with the QTH, 7 out of the 15 LEDs tested yielded significantly higher temperature increase at the tip device. This indicates that LEDs used for dental light-curing purposes might not be considered "cold-light devices" as a general rule. For some LEDs, the temperature increase at the tip was 153% to 194% higher than the QTH unit. The temperature increase showed a logarithmic growth for LCUs with irradiance $\geq$1000 mW/cm^2, indicating that the heat generated is not dissipated by the LCU itself.

The ΔT results at the pulpal floor provided additional evidence that dental LEDs generate high thermal energy. Only two LEDs from the eight units that presented irradiance $\geq$800 mW/cm^2 generated temperature increase below 5.5°C, which here is used as a reference based on the work by Zach and

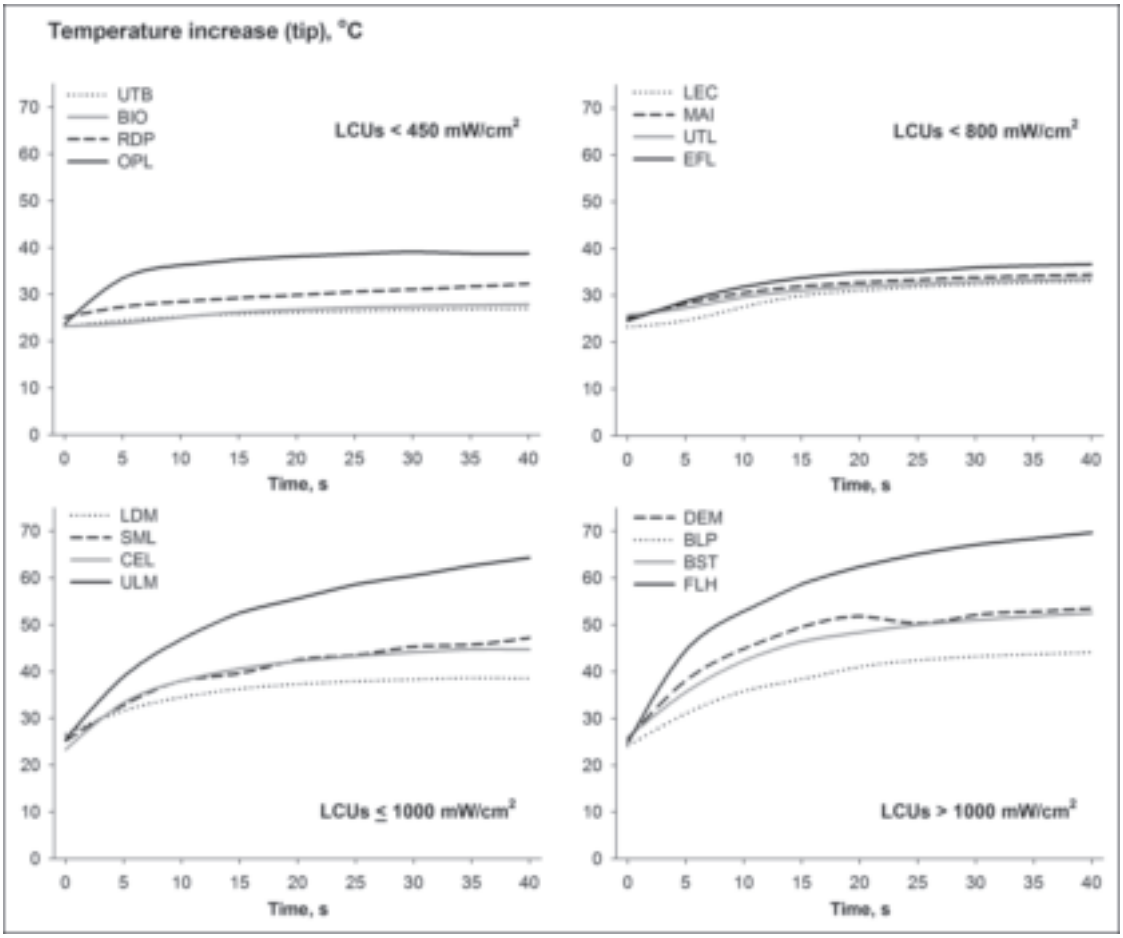

Figure 2. *Profiles of temperature increase at the light guide tip over the 40-second exposure time. The temperature increase followed a logarithmic growth pattern for the LCUs with irradiance ≥1000 mW/cm².*

Cohen.[17] An interesting result was found for DEM, which emits a pulsatile light; despite the high ΔT at the tip of this LCU, the ΔT at the pulpal floor was below most of the other LCUs. This is explained by the intermittent light exposure allowing more time for the heat to dissipate by the tooth structure. In spite of this example, the ΔT at the pulpal floor of cavities followed linearly both the measured irradiance of the LCU (R^2=0.39) and ΔT at the light guide tip (R^2=0.28). Therefore, the hypothesis tested is accepted. As the irradiance level can be measured clinically with a radiometer, despite its inaccuracy as sometimes reported,[23] and the correlation between irradiance and ΔT at the pulpal floor was linear for both the measured irradiance of the LCU (R^2=0.39) and ΔT at the light guide tip (R^2=0.28), the measured irradiance of the LCU might be taken as a feasible tool to predict the ΔT at the pulpal floor during photopolymerization procedures. The methods used here, however, have limitations, such as the absence of pulpal fluid perfusion, use of bovine teeth, and baseline temperatures below the intraoral temperature, and these should also be taken into account when extrapolating the results.

The present study was not focused on determining critical temperature levels that may be harmful to the pulpal tissues. For instance, the results reported by Zach and Cohen[17] that an intrapulpal temperature rise above 5.5°C might cause pulp cell vitality injuries difficult to recover are for a sustained temperature increase, but the temperature increases during light-curing procedures are relatively brief. The focus of the present study was rather to conduct a comprehensive investigation of several LED LCUs available on the market and investigate the relationship of irradiance level and thermal variations. When the remaining dentin is thick (ie, in shallow to medium cavities), light scattering may predominate over light absorption because the dentin absorption coefficient is low for the wavelengths emitted by dental LCUs.[24] Special concern lies for bonding procedures in deep cavities, where photoactivation

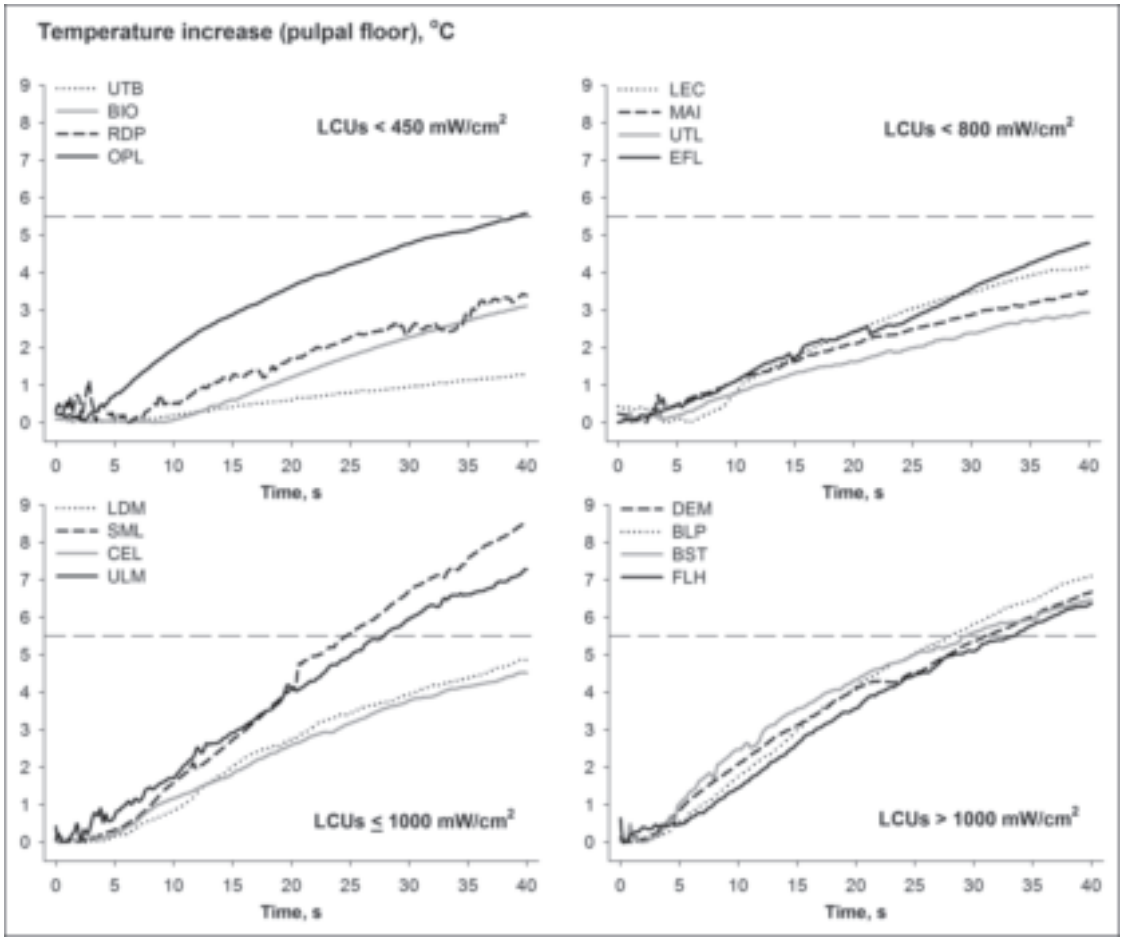

Figure 3. *Profiles of temperature increase at the pulpal floor over the 40-second exposure time. Data are shown not taking into account the baseline temperature (25°C) in order to allow observation of the maximum temperature increase compared with the 5.5°C used as a reference.[17] A linear increase was observed for several LCUs.*

of the adhesive is carried out without any layer of restorative resin that could act as a barrier for thermal conduction.[19,25]

Clinicians should be familiar with the factors that are involved in temperature increase during photoactivation procedures in dental cavities, especially the irradiance level emitted by the LCU and the thickness of the remaining dentin. As the temperature at the pulpal floor increases linearly over the exposure time for several LCUs, clinicians should be aware of the possibility of overheating the area when using prolonged photoactivation protocols,[20] especially during the initial restorative steps, when the dentin is more exposed. An alternative to reduce the thermal increase at the pulpal floor in deep cavities could be the use of a glass-ionomer base, but the presence of such a base may decrease restoration survival compared with total-etch restorations.[26,27] Therefore, clinical studies investigating the survival and postoperative sensitivity of composite restorations performed using different LCUs are still necessary.

CONCLUSION

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

- Large discrepancies between the irradiance emitted by some LED units and the irradiance level claimed by the manufacturers were observed.
- The thermal variation at the light guide tip followed a logarithmic growth pattern for units with irradiance $\geq$1000 mW/cm^2, while a linear temperature increase at the pulpal floor was observed for several units.
- The measured irradiance of LED units was linearly associated with the thermal variation at the pulpal floor of dental cavities during photopolymerization.

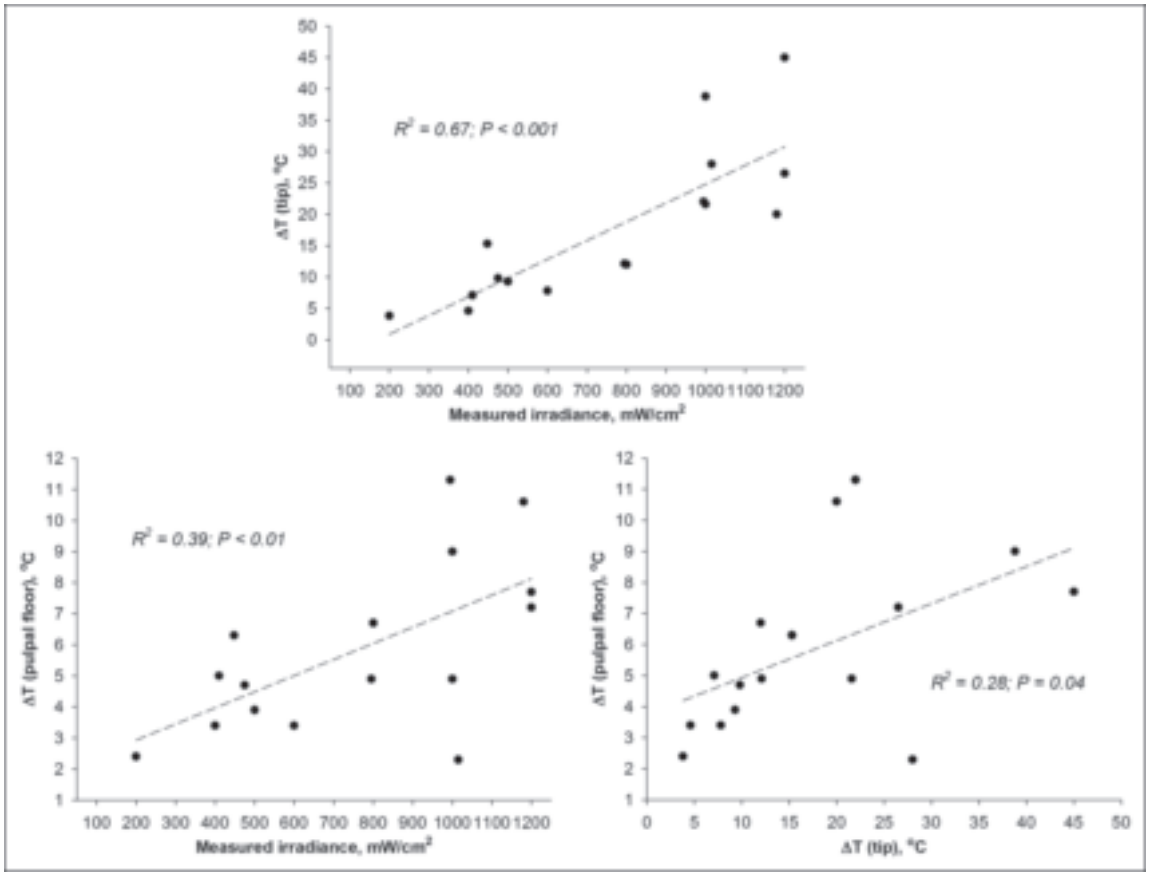

Figure 4. *Regression plots showing that the thermal variation (ΔT) at the light guide tip follows linearly the measured irradiance of the LCUs (R^2=0.67; p<0.001) and ΔT at the pulpal floor (R^2=0.28; p=0.04); the relationship ΔT the pulpal floor and measured irradiance is also linear (R^2=0.39; p<0.01).*

Conflict of Interest

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

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Labeled vs Actual Concentration of Bleaching Agents

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Clinical Relevance

Actual bleaching agent concentration needs to be what is indicated on the label. This study evaluates the differences in label vs. actual concentration of bleaching agents in dentist dispensed and over the counter products in four countries.

SUMMARY

The purpose of this study was to determine if the actual concentration of bleaching agents available in four different countries were the same as the label indicated and within the recommendations of the International Standard on Tooth Whitening. The method recommended for assaying peroxide by the United States Pharmacopeia was used to determine concentrations. All products in the United States and China were within the standard when products were tested immediately upon delivery at testing sites. One product in Saudi Arabia and three products in Brazil had greater than 30% concentration loss. Three of 24 products in the United States did not meet the International Standard when they were tested at month of expiration.

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INTRODUCTION

Tooth whitening has become a $1.2 billion industry. Patients are requesting bleaching procedures at an ever-increasing rate and desire results as quickly as possible. Dentists are aware that rapidity and degree of whitening are dependent on many variables, including contact time and concentration of the active ingredient.

Studies[1,2] have documented discrepancies between the listed and actual concentrations of the active material in bleaching products. The concentration of bleaching agents in previously assayed products has varied from 1.08 above to 3.55 below the posted

concentration. Until recently, the difference in concentration was reported as a difference from the posted label concentration. The current standard for indicating differences in listed and actual bleaching agent concentrations is to indicate the percentage loss from the label concentration. A recently established International Standard for Bleaching Products requires that the actual concentration of active ingredient "shall not exceed 10% or lower than 30% of the manufacturer's stated concentration" over the stated lifetime of the products.[3]

The purpose of this current study was to gather tooth-whitening agents from four different parts of the world and to determine their concentrations and if the bleaching agents in the United States were within the recommendations of the International Standard at the month of expiration. Differences from the labeled concentration may occur during manufacturing, shipping, or storage of the tooth-whitening agents. Refrigeration during transportation from the manufacturer to dentist's office, when recommended, may vary from the manufacturer's recommendations, causing a more rapid degradation of the agents. This present study examines the labeled vs measured levels of active bleaching agents in tooth-whitening products prescribed by dentists or available over the counter in China, Brazil, Saudi Arabia, and the United States. The date of manufacture was unknown for the bleaching products tested; however, all tests were accomplished shortly after the products were received by the institutions conducting the testing.

The International Standard requires that products retain a certain percentage of the concentration indicated on the label until the date of expiration. The initial evaluation was accomplished when products were acquired to determine why some products may be less effective than expected. The evaluation at product expiration was performed to determine if the use life of the agents was within the requirement of the International Standard.

METHODS AND MATERIALS

An undergraduate student wrote to all the manufacturers of tooth-whitening agents available in the United States, requesting they forward a sample of each of their products for a comprehensive photo of commercially available bleaching products. Three months were spent collecting products. Thirty-five products were received, and they were stored at room temperature after arrival at the university. The over-the-counter products were purchased at local retailers. All assays to determine the initial concentration were performed by the end of the three months. The products were maintained at room temperature in the United States. Another student determined the concentration of the products on the month of expiration. Twenty-four of the products were available for assaying at that time. In China, Saudi Arabia, and Brazil, all products that were available in their countries were purchased and kept at room temperature until they were assayed. The various participants were able to collect 13 products in China, 7 products in Saudi Arabia, and 15 products in Brazil.

The same method of determining the amount of peroxide in the agents was used at all testing sites, which is the one recommended by the United States Pharmacopeia[4] and the International Standard.[3] The specific steps in this chemical analysis have been used in multiple studies.[5–9] The testing sites were sent the specific steps and asked to become familiar with the procedure. All of the participants who performed the tests are published authors in tooth whitening.

Prior to determining the amount of peroxide in a bleaching agent, a researcher at each site indicated on a data sheet the current date, manufacturer, product, expiration date, peroxide type, peroxide concentration, and trial number. An empty 250-mL beaker was then weighed on a scale that was accurate to three decimal points. Approximately 2 g of the bleaching product was placed in the empty beaker, and another weight was taken. The sample weight was calculated by subtracting the empty beaker weight from the beaker with the sample.

Deionized water was added to the 100-mL mark on the beaker. A stir bar was added, and the beaker was allowed to mix on a stir plate until a homogeneous mixture was attained. Twenty milliliters of glacial acetic acid was added, and the beaker was immediately covered with a watch glass. Approximately 2 g of potassium iodide was added to the solution and allowed to dissolve, which turned the solution to a light shade of yellow. Three drops of ammonium molybdate were added, and the solution was allowed to again become homogenous. The beaker was then transferred to a darkened cupboard. The darkened cupboard was used to allow the chemicals to fully associate to ensure complete reaction with the available peroxide agent.

Once the sample had been in a darkened area for at least 10 minutes, it was placed on the stir plate. Gradually, 0.025 N sodium thiosulfate was triturated into the solution, using a 50-mL burette, until the

Table 1: *Bleaching Agents Available to Dentists Only in the United States Listed by Manufacturer, With Lot Number, Type of Peroxide, Label Concentration, Average Concentration, and Concentration Difference (cont.)*

Manufacturer	Product	Lot No.	Pr Ty	Label Conc	Average	% Diff
Agents with concentrations within 15% of label						
Spectrum Dental	Contrast AM	06192010	HP	22.00	23.33	6.00
Discus Dental	Night White ACP	6208022	CP	22.00	23.15	5.20
Premier	Perfecta Rev	2547	HP	14.00	14.49	3.50
Premier	Dental Whitening Systems	16062006	CP	16.00	16.47	2.90
Premier	Perfecta Ultra	2265	HP	6.00	6.17	2.80
Discus Dental	Night White ACP	6219081	CP	10.00	10.22	2.70
Discus Dental	Night White Excel 3 Turbo	6213074	HP	6.00	6.11	1.80
Temrex	Star White	11168-0306	CP	16.00	16.26	1.60
Premier	Dental Whitening Systems	11042706	CP	11.00	11.09	0.80
Ivoclar Vivadent	Vivastyle Touch Up	JL1017	CP	10.00	10.07	0.70
Ultradent Products	Opalescence PF10	B2HNF	CP	10.00	9.96	0.40
Patterson Dental	Tooth Whitening Gel	B26KS	CP	16.00	16.03	0.20
Ultradent Products	Opalescence	B27BN	CP	10.00	9.90	−1.00
Nu Radiance	Nu Radiance Touch-up Kit	060613–0900	CP	16.00	15.83	−1.10
SDI	Pola Day	68454	HP	7.50	7.32	−2.40
Spectrum Dental	Contrast PM	6181007	CP	15.00	14.58	−2.80
Ultradent Products	Opalescence PF15	B283D	CP	15.00	14.47	−3.50
Dentsply	Nupro White Gold	60718	CP	15.00	14.38	−4.10
Ivoclar Vivadent	Vivastyle Plus	JL1017	CP	10.00	9.57	−4.30
Ultradent Products	Opalescence PF20	B25V3	CP	20.00	19.02	−4.90
Discus Dental	Day White	6226026	HP	9.50	9.00	−5.30
Spectrum Dental	Contrast PM	6180014	CP	10.00	9.41	−5.90
Premier	Perfecta	B27GZ	CP	16.00	14.97	−6.40

Table 1: Continued.

Manufacturer	Product	Lot No.	Pr Ty	Label Conc	Average	% Diff
Premier	Perfecta Bravo	2493	HP	9.00	8.36	−7.10
Premier	Perfecta	B27GZ	CP	11.00	10.12	−8.00
Premier	Perfecta	B27GZ	CP	13.00	11.86	−8.80
Patterson Dental	Tooth Whitening Gel	B26KT	CP	22.00	20.07	−8.80
Spectrum Dental	Contrast PM	6165030	CP	15.00	13.58	−9.50
Premier	Perfecta	B251Q	CP	21.00	18.92	−9.90
SDI	Pola Night	60405	CP	16.00	14.41	−10.00
SDI	Pola Night	68400	CP	22.00	19.59	−11.00
Patterson Dental	Tooth Whitening Gel	B09N8	CP	11.00	9.38	−14.70
Agents with concentrations between 15% and 30% lower than label indicates						
Discus Dental	Night White ACP	6205027	CP	16.00	13.32	−16.80
SDI	Pola Paint	51220	CP	8.00	6.65	−16.90
Discus Dental	Day White	6215024	HP	7.50	5.50	−27.00

Abbreviations: CP, carbamide peroxide; HP, hydrogen peroxide.

sample turned a pale shade of yellow. Three milliliters of a 1.0% starch indicator was added to the solution, turning the solution a dark purple. More sodium thiosulfate was titrated into the solution, using a 10-mL burette, until the solution turned colorless, which was the end point of the assay. All chemical analyses of concentrations were performed in triplicate.

The concentration of the bleaching agent was determined by the following formula:

$$\text{Hydrogen Peroxide (HP)\%} = 1.704 \times \text{TSmL} \times (0.025/\text{PWg})$$

$$\text{Carbamide Peroxide (CP)\%} = 4.704 \times \text{TSmo} \times (0.025/\text{PWg})$$

where TS is sodium thiosulfate and PW is product weight.

RESULTS

United States

Thirty-two products dispensed to dentists were within 15% of the active agent concentration listed on the label (Table 1). Three products had a 15% lower but not more than a 30% lower concentration of active agent than that listed on the label. All of the tooth-whitening agents dispensed to dentists in the United States that were assayed in this study were within the requirements established by the International Standard upon delivery to the testing site.

In the United States, the concentration of the active agent in the bleaching products available over the counter was also assayed. Manufacturers are not required by the Food and Drug Administration to list the active agent concentration of cosmetic products, only to list the ingredients found in the product. The new International Standard requires manufacturers of all tooth-whitening products to list

Table 2: *Over-the-Counter Bleaching Agents in the United States Listed by Manufacturer, With Lot Number, Type of Peroxide, and Average Concentration*

Manufacturer	Product	Lot No.	Pr Ty	Label Conc	Average
Lumalite	GentleBright Plus	6C091/6C101	HP	None	0.94
Lumalite	StayBright Plus	F609060	HP	None	7.30
Nu Radiance	Duet 30	060524–0800	CP	None	12.62
Nu Radiance	Forte with Calcium	060424–1300	CP	None	22.70
Procter & Gamble	Crest Whitening Rinse	95659415	HP	None	1.54
Proctor & Gamble	Crest Night Effects Gel	61525614TO	HP	None	3.33
Proctor & Gamble	Crest Strips Premium Plus 10 day	625BT4	HP	None	9.29
Proctor & Gamble	Crest Whitestrips Classic 14 day	6221BT2	HP	None	6.18
Proctor & Gamble	Crest Whitestrips Premium	6254BT4	HP	None	9.77
Proctor & Gamble	Crest Whitestrips Renewal 10 day	6017BT2	HP	None	7.93
Proctor & Gamble	Crest Whitestrips Daily Multicare	7180BT3	HP	None	6.07
TeleBrands	White Light	WLPGR5D	CP	None	21.22
Plus White	5 Min Speed Whitening	7610	HP	None	6.06
Oral B	Rembrandt 2hr White	266057	HP	None	6.12
GlaxoSmithKline	Aquafresh White Trays	6L11C1	HP	None	10.32
Dentco	Equate Dental Whitening Strips	7E03A	CP	None	10.02
Johnson & Johnson	Rembrandt 2hr Whiten Kit	0887AR290874	HP	None	5.70

Abbreviations: CP, carbamide peroxide; HP, hydrogen peroxide.

the concentrations of the active bleaching agent on the packaging. Since the products were not required to identify the concentration on the label, it was not possible to identify the concentration differences at the time of testing. The over-the-counter product concentrations of carbamide peroxide (CP) ranged from 10% to 23% CP, and the hydrogen peroxide (HP) concentrations ranged from .09% to 10% HP (Table 2).

Twenty-four products were tested at the month of expiration. Three products were found to have concentrations less than that accepted by the International Standard (Table 3).

China

The concentration testing for the tooth-whitening agents that were available on the Chinese market was accomplished at Wuhan University in Wuhan, China. Thirteen products were secured and assayed. Nine of the products had CP as the active agent, with agent concentrations ranging from 8% to 19% CP. Four of the products had HP as the active agent,

Table 3: *Bleaching Agents in the United States Assayed During the Month of Expiration, Listed by Manufacturer, With Lot Number, Type of Peroxide, Label Concentration, Mean Concentration of Three Trials, and Concentration Difference*

Manufacturer	Brand Name	Lot No.	Type	Label Conc	Mean Conc	% Difference
Premier	Perfecta Bravo	2493	HP	9.00	9.14	2
Spectrum Dental	Contrast AM	06192010	HP	22.00	22.07	0
Ultradent Products	Opalescence PF10	B2HNF	CP	10.00	9.96	0
Discus Dental	Night White Excel 3 Turbo	6213074	HP	6.00	5.79	−4
Premier	Dental Whitening Systems	11042706	CP	11.00	10.08	−8
Ultradent Products	Opalescence PF20	B25V3	CP	20.00	18.22	−9
Discus Dental	Day White	6215024	HP	7.50	6.77	−10
Discus Dental	Night White ACP	6205027	CP	16.00	14.22	−11
Discus Dental	Night White ACP	6219081	CP	10.00	8.82	−12
SDI	Pola Day	68454	HP	7.50	6.50	−13
Discus Dental	Night White ACP	6208022	CP	22.00	18.93	−14
Patterson Dental	Tooth Whitening Gel	B26KS	CP	16.00	13.72	−14
Premier	Dental Whitening Systems	16062006	CP	16.00	13.81	−14
Premier	Perfecta Ultra	2265	HP	6.00	5.89	−14
Agents with concentrations between 15% and 30% lower than label indicates						
Ultradent Products	Opalescence	B27BN	CP	10.00	8.42	−16
Dentsply	Nupro White Gold	60718	CP	15.00	12.32	−18
Premier	Perfecta	B27GZ	CP	11.00	8.82	−20
Patterson Dental	Tooth Whitening Gel	B26KT	CP	20.00	17.50	−22
SDI	Pola Paint	51220	CP	8.00	0.26	−26
SDI	Pola Night	68400	CP	22.00	15.96	−27
Spectrum Dental	Contrast PM	6165030	CP	15.00	10.76	−28
Agents with concentrations more than 30% lower than label indicates						
Spectrum Dental	Contrast PM	6180014	CP	10.00	7.05	−30
Discus Dental	Day White	6226026	HP	9.50	6.36	−33
Spectrum Dental	Contrast PM	6181007	CP	15.00	9.60	−36

Abbreviations: CP, carbamide peroxide; HP, hydrogen peroxide.

Table 4: *Bleaching Agents in China Listed by Manufacturer, With Product Name, Type of Peroxide, Label Concentration, Average Concentration, and Concentration Difference*

Manufacturer	Product	Pr Ty	Label Conc	Average	% Diff
Agents with concentrations within 15% of label					
*Kernel Bio Tech	Whitening Strip (16 CP)	CP	16	17.34	8.4
*CCA	Plus White (6 HP)	HP	6	6.27	4.5
Ultradent	Opalescence (10 CP)	CP	10	10.24	2.4
*Crest	Whitestrips Premium (10 HP)	HP	10	10.19	1.9
Ultradent	Opalescence (20 CP)	CP	20	19.19	−4
Ultradent	Confi-white Tooth whitening Gel (15 CP)	CP	15	14.38	−4.1
Ultradent	Opalescence (15 CP)	CP	15	14.01	−6.6
Ultradent	Confi-white Tooth whitening Gel (10 CP)	CP	10	9.28	−7.2
Discus Dental	Nite White (9.5 HP)	HP	9.5	8.79	−7.5
*Onuge	Professional Whitening strip (10 CP)	CP	10	9.13	−9.7
Agents with concentrations between 15% and 30% lower than label indicates					
*AWG	Teeth Whitening Gel (8 HP)	HP	8	6.69	−16.4
Discus Dental	Nite White (10 CP)	CP	10	8.27	−17.3
*Onuge	Dental Whitening Strip (8 CP)	CP	8	6.56	−18

Abbreviations: CP, carbamide peroxide; HP, hydrogen peroxide.
** Products available over-the-counter.*

with agent concentrations ranging from 6-10% HP. Six of the products were available over the counter, and the other seven were available from dental offices. Ten of the products were within 15% of the concentration on the label. Three products had concentrations that were lower than 15% of the indicated concentration but were not more than 30% lower than the listed active agent concentration on the label (Table 4).

Brazil

The concentration testing for the bleaching products that were available on the Brazilian market was accomplished at the University of Santa Catarina in Florianopolis, Brazil. Fifteen products were secured and assayed. Twelve of the products contained CP and had concentrations ranging from 9% to 37% CP. Three of the products contained HP and had concentrations ranging from 6% to 7.5% HP. No tooth-whitening products were available over the counter. Six of the products were within 15% of the concentration on the label. Six products had concentrations that were lower than 15% of the indicated concentration but were not more than 30% lower than the listed concentration of the active agent. Three of the products had a loss of more than 30% of the concentration indicated on the label (Table 5).

Table 5: *Bleaching Agents in Brazil Listed by Manufacturer, With Product Name, Type of Peroxide, Label Concentration, Average Concentration, and Concentration Difference*

Manufacturer	Product	Pr Ty	Label Conc	Average	% Diff
Agents with concentrations within 15% of label					
Ultradent Products	Opalescence PF (15 CP)	CP	15	13.86	−7.6
Ultradent	Opalescence PF (20 CP)	CP	20	18.26	−8.7
Voco	Perfect Bleach (10 CP)	CP	10	9.02	−9.8
SS White	Review 16F (16 CP)	CP	16	14.22	−11.1
FGM	White Class (7.5 HP)	HP	7.5	6.61	−11.9
Villevie	Mix Day (6 HP)	HP	6	5.27	−12.2
Agents with concentrations between 15% and 30% lower than label indicates					
Ultradent	Opalescence PF (10 CP)	CP	10	8.42	−15.8
FMG	Whiteness Standard (10 CP)	CP	10	7.26	−17.4
FGM	White Class (6 HP)	HP	6	4.93	−17.8
FGM	Whiteness Standard (16 CP)	CP	16	12.51	−21.8
Vigodent	Whiteness Perfect (16 CP)	CP	16	11.6	−27.50
FGM	Magic Bleaching (16 CP)	CP	16	11.38	−28.9
Agents with concentrations more than 30% lower than label indicates					
Vigodent	Magic Bleaching (10 CP)	CP	10	6.66	−33.4
SSWhite	Review 10F (10 CP)	CP	10	5.59	−44.1
Vigodent	Magic Bleaching (37 CP)	CP	37	18.33	−50.5

Abbreviations: CP, carbamide peroxide; HP, hydrogen peroxide.

Saudi Arabia

The concentration testing for the bleaching agents that were available on the Saudi Arabian market was accomplished at King Saud University in Riyadh, Kingdom of Saudi Arabia. Seven products were secured and assayed. The labels indicated that six of the products contained CP and had concentrations ranging from 10–22% CP; one of the products contained HP, and the label indicated a 7.5% HP concentration of the active agent. No tooth-whitening products were available over the counter. One of the products was within 15% of the concentration on the label. Five products had concentrations that were lower than 15% of the indicated concentration but were not more than 30% lower than the listed concentration of active agent. One of the products had a loss of more than 30% of the concentration indicated on the label (Table 6).

Table 6: *Bleaching Agents in Saudi Arabia Listed by Manufacturer, With Product Name, Type of Peroxide, Label Concentration, Average Concentration, and Concentration Difference*

Manufacturer	Product	Pr Ty	Label Conc	Average	% Diff
Agents with concentrations within 15% of label					
Ultradent Products	Opalescence (10 CP)	CP	10	9.17	−8.3
Agents with concentrations between 15% and 30% lower than label indicates					
Ultradent	Opalescence PF (20 CP)	CP	20	16.26	−18.7
Discus Dental	Nite White (16 CP)	CP	16	12.71	−20.6
Discus Dental	Nite White (22 CP)	CP	22	17.03	−22.6
Ultradent Products	Opalescence PF (15 CP)	CP	15	11.36	−24.3
Discus Dental	Nite White (10 CP)	CP	10	7.41	−25.9
Agents with concentrations more than 30% lower than label indicates					
Discus Dental	Day White (7.5 HP)	HP	7.5	4.62	−38.4

Abbreviations: CP, carbamide peroxide; HP, hydrogen peroxide.

DISCUSSION

It is well accepted that bleaching effectiveness depends on the time the agent is in contact with the teeth and on the concentration of the agent. The *Clinical Research Associates Newsletter* has stated that "storage for extended time or in warm temperature, faulty packaging, and other problems can cause bleaches to lose potency."[9] The Clinical Research Associates performed their study using a different assay methodology. Of the 12 products they assayed, eight were within 1 of the concentration indicated on the label. Four products were more than 1 higher than the label indicated, and one product was more than 1 lower than the label indicated.

Previous studies in which concentrations were assayed evaluated products as a combined total of all similar products by specific manufacturers and not as individual products. In a report in 2000, the mean decrease in the concentrations of 10%, 15%, and 20% in Contrast PM products was 3.55 less than the label indicated. Rembrandt products of 10%, 15%, and 22% were 1.08 higher than the label indicated.[1] In a report published in 2003, Stark White products of 10%, 16%, and 22% were found to be 2.64 less than the label indicated.[2]

In Brazil, tooth-whitening agents are required to put the date the product was manufactured, instead of the lot number, on the label. The manufacturing date was not evident on the products in the other countries. It is possible to use the lot number to determine the time of production by contacting the manufacturer, if there is a need to know that at some point in time.

In the past, the consumer had no way of knowing the concentration of active agents in the products sold over the counter in the United States. The International Organization of Standardization now requires manufacturers to list the concentrations of active agents on all tooth-whitening agents. Patients are now able to make an informed decision as to the concentration of over-the-counter products they are purchasing.

Manufacturers have a responsibility to deliver products to the dental practitioner with the bleaching agent concentrations that are listed on the label. It is known that HP is not as stable as CP. The urea in CP stabilizes the HP. HP degrades less rapidly in cold and away from sunlight. Dental practitioners

need to keep the products that recommend refrigeration in a cool area to lower the rate of degradation before use.

Manufacturers need to review the expiration dates they place on tooth-whitening agents to ensure the product they market remains within the labeled concentration required by the International Organization of Standardization. Universities around the world need to assay tooth-whitening agents and publish the results in their national dental journals to indicate which ones are at lower concentration than the label indicates at the time of delivery and those that are within the standard at the month of expiration. This will encourage manufacturers to reevaluate the priority they place on maintaining concentrations of products at the appropriate level.

Manufacturers need to adjust the expiration dates, place another agent in the active agent to reduce the degradation of their products, or encourage dental practitioners to keep their products in a cool place so the products will be at the full strength indicated on the label when patients use their products. This will give practitioners the confidence to expect predicted results.

CONCLUSION

The tooth-whitening products available in the United States and China were all within the newly established International Standard. One product in Saudi Arabia and three products in Brazil had a loss of at least 30% or more of the concentration indicated on the label by the manufacturer. These products were assayed after securing the products in the respective country. When testing was accomplished at the month of expiration in the United States, three products had a loss of more than 30% of the concentration indicated on the label. Products with a loss of at least 30% of the listed concentration at any time before the expiration date do not meet the International Standards for tooth-whitening agents.

Conflict of Interest

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

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Departments

ANNOUNCEMENTS

Attention Members of the Academy of Operative Dentistry! The Planning Committee has made arrangements to move the annual meeting of the Academy to the Wednesday through Friday format, starting next year, 2014! The details are as follows:

HOTEL: The Drake, Chicago

MEETING DATES: Wednesday–Friday, February 19–21, 2014

NOTE: The AARD meets Sat–Sun, February 22–23, 2014

COMMITTEE MEETINGS: Wednesday

ESSAY SESSION: Thursday all day and Friday AM

TABLE CLINICS: Friday PM

CODE Meeting: Thursday 4:30 PM

CAMBRA Meeting: Wednesday PM

ABOD meetings and Luncheon: Wednesday

The room rate is $159 for a standard room and we have reserved many nice rooms overlooking the lake. The hotel has been recently remodeled and the rooms and facilities look spectacular. We will be sharing the venue with another meeting in 2014, but in 2015 the entire facility will be ours. We hope you will bear with us as we re-adjust again, in order to better serve our Members.

Sincerely, Richard G. Stevenson III, AOD Secretary

Academy of Operative Dentistry

2013 Hollenback Recipient

George Hollenback

Dr. Max Anderson

Dr. Anderson has been a leader in the area of research related to dental caries and has generated significant grants and discovery related to caries prevention and caries management. He is also a master clinician, a superb teacher, and a great seeker of ways to promote oral health through research and transfer of new information to dental practitioners. He had a tremendous impact on dentistry in the U.S. Navy and other federal services prior to (and after) his retirement as a Naval Dental Officer. Additionally, he has had great impact on dental education. He has lectured for the annual meeting of the Academy several times, most recently in 2011 when he delivered the Buonocore Memorial Lecture, "Advancing Science: Its Current and Future Impacts on Dentistry." He has an extensive list of publications and invited lectures. But the primary reason for his deserving the Hollenback Award has been his leadership in translation of research findings transforming clinical practice with an open eye for economic realities. This has led to advances in prevention and treatment of dental caries in the Navy, in dental education, and in the insurance industry.

He earned his DDS degree at the University of Nebraska in 1976, completed a U.S. Navy General Practice Residency in 1977, and received his MS degree in Operative Dentistry from the University of Michigan in 1983. He also received a M.Ed. degree from George Washington University in 1988. Dr. Anderson later served as Professor and Chairman, Operative Dentistry Department, Naval Dental School, Bethesda; Specialty Advisor to the Chief of the Navy Bureau of Medicine (the Navy's surgeon general); Assistant Professor, Department of Restorative Dentistry, University of Washington; Professor and Chairman, Department of Restorative Dentistry, Indiana University; Vice President, Dental Director, and Chief Science Officer at Washington Dental Service (part of the Delta organization); and CEO, C3 Scientific Corporation (studying and promoting new technology in the prevention of dental disease). For health reasons, he stepped down as CEO of C3 Jian, Inc. but he continues to do extensive consulting work for several companies and dental schools.

He has held the position of Editor of our journal, *Operative Dentistry,* and has served on the Editorial Board of that journal since 1994. He has also served the Academy as Councilor and has chaired the Research Committee. He is currently Associate Editor of *The Journal of Evidence Based Dental Practice*, one of the two major journals in the area of evidence-based dentistry. He has been a clinical consultant to *The Dental Advisor* and editorial advisor to the *Journal of the American College of Dentists*. He served the American Board of Operative Dentistry as a member of the Executive Council, an Examiner, and a member of the Examination Committee. He is a fellow in the American College of Dentists and the International College of Dentists and member of the American Dental Association, the American Academy of Restorative Dentistry, the American and International Associations for Dental Research, the Federation Dentaire Internationale, and other professional organizations.

Max's energy is infectious and has stimulated many in the profession and in the Academy to action. His contributions in the areas of caries prevention and treatment and in operative dentistry have been nothing short of superb. He is uniquely qualified to be awarded the Hollenback Prize.

Online Only

Effects of Preheating and Precooling on the Hardness and Shrinkage of a Composite Resin Cured With QTH and LED

FH Osternack • DBM Caldas • JB Almeida • EM Souza • RF Mazur

Clinical Relevance:

The composite hardness was not affected by different pretreatment temperatures, whereas the shrinkage was not affected by the temperatures only when the composite was cured with an LED-curing unit.

http://dx.doi.org/10.2341/11-411-L

Effect of Er,Cr:YSGG Laser, Air Abrasion, and Silane Application on Repaired Shear Bond Strength of Composites

SD Cho • P Rajitrangson • BA Matis • JA Platt

Clinical Relevance:

Use of Er,Cr:YSGG (erbium, chromium: yttrium-scandium-gallium-garnet) laser does not improve the shear bond strength of the repaired resin; however, air abrasion with 50-μm aluminum oxide particles results in higher shear repair bond strength, and therefore is recommended prior to repair. Application of a silane coupling agent does not improve the shear bond strength of the repaired resin.

http://dx.doi.org/10.2341/11-054-L

Conservative Treatment of Complicated Oblique Crown-root Fractures of Molars: A Report of Five Representative Cases

P Wang • W He • L Ni • Q Lu • H Sun

Clinical Relevance:

Crown-root fractures of molars with extensive loss of tooth structure extending well below the alveolar crest can be successfully treated with a conservative method.

http://dx.doi.org/10.2341/12-371-S

Evaluation of Resin Composite Translucency by Two Different Methods

D-H Kim • S-H Park

Clinical Relevance:

Composite translucency varies by manufacturer. This information should be considered when selecting materials and clinical techniques to improve clinical performance.

http://dx.doi.org/10.2341/12-085-L

Crown Discoloration Induced by Endodontic Sealers: Spectrophotometric Measurement of Commission International de l'Eclairage's L*, a*, b* Chromatic Parameters

K Ioannidis • P Beltes • T Lambrianidis • D Kapagiannidis • V Karagiannis

Clinical Relevance:

This study emphasizes the need for attention after root canal obturation. The prevention of tooth staining post-operatively is still a clinical issue that affects patients and dentists. Clinicians must be able to recognize the etiology of local intrinsic staining and even in cases of sealer-induced discoloration must be able diagnose the type of sealer that was used. The prevention of crown discoloration in endodontically-treated teeth is currently taught in all accredited graduate and post-graduate programs.

http://dx.doi.org/10.2341/11-266-L

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