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

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Guest Editorial

Hand Instrumentation

BA Matis • M Lund

At the initial meeting of the Academy of Operative Dentistry in February 1972, David Granger posed a question during his keynote address titled "What are you Operative Dentistry?" That is a question that continually requires a response of those who provide clinical operative treatment.

Part of the response is anchored in the constant need to demonstrate excellence with each of the options a clinician selects as treatment. Materials needed for treatment are to be used as recommended by the manufacturer and according to good clinical practices taught during dental school training. Long term results are dependent on the affinity of the restoring materials as related to the preparation walls of the teeth.

A clinician must give careful attention to the design and execution of all tooth preparations. These preparations must reflect the details and fineness which will lead to long term stability of the restorations. G V Black presented our initial designs for tooth preparations employing restorative materials of his time. Through the years the design of these preparations has been modified to take advantage of the improvement in dental materials as well as the change and upgrading of our dental knowledge. Newer materials, such as resin based composites and improved ceramics have been introduced since GV Black's time, but the need for caries removal will continue to be the reason for cavity preparation. As a result of this research the materials should reflect improved convenience and also an enhanced longevity.

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The improvement of dental materials is highly appreciated but, according to retrospective studies and insurance data, the clinical restorations are not maintaining the desired level of longevity. The replacement of restorations occurs at a frequency greater than one should expect with improved materials and knowledge.

The bottom line is that clinical success and longevity are intimately related to the skill and attention to detail of the operator. When high speed handpieces were fully established as the optimum method of cutting dental tissues, the attention to the state of the enamel at the interproximal cavo-surface took on less importance. Unfortunately, the burs have definite limitations in providing acceptable cavo-surface margins. This is a continuing concern of restorative clinicians.

Clinical researchers have observed that burs at high speed left enamel in unsatisfactory condition, particularly at the interproximal margins. (1–5) A variety of options have been proposed to minimize this problem, including variations in bur design and the use of discs and hand instrumentation.

The creation of smooth enamel is very compelling, and it is helpful if clinicians do their best to think and visualize at a microscopic level.

If a practitioner attempts to complete conservative interproximal preparations exclusively with use of high speed burs, it can easily cause injury to the adjoining tooth. In a conservative interproximal preparation there is no space for a bur to function without the risk of damage to the adjacent tooth. Some have estimated as high as 60% of adjacent teeth are marred during interproximal tooth prepa-

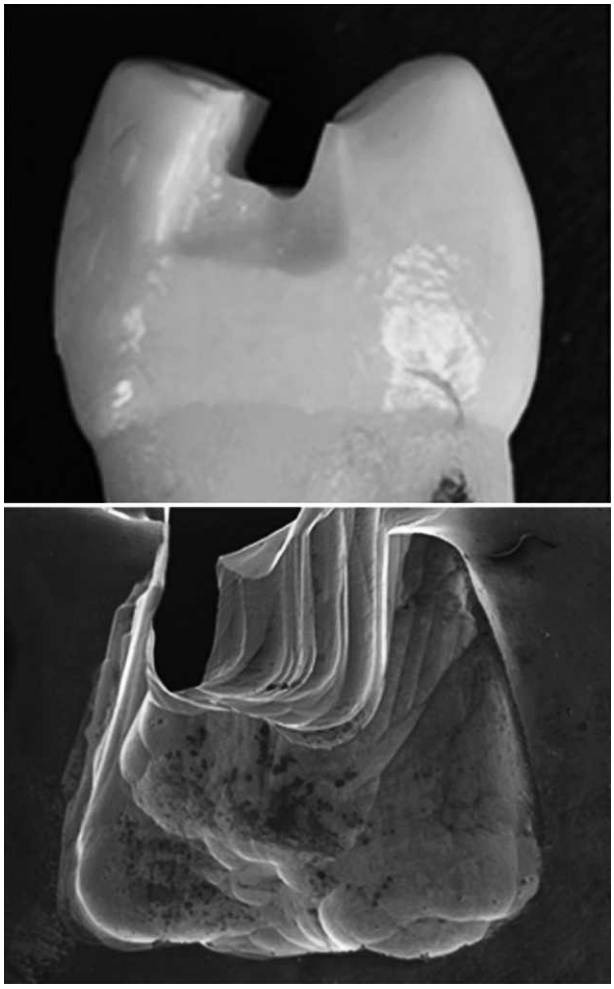

Figure 1. *Mesial of bicuspid prepared with rotary instruments only: a) natural tooth b) preparation magnified 35X.*

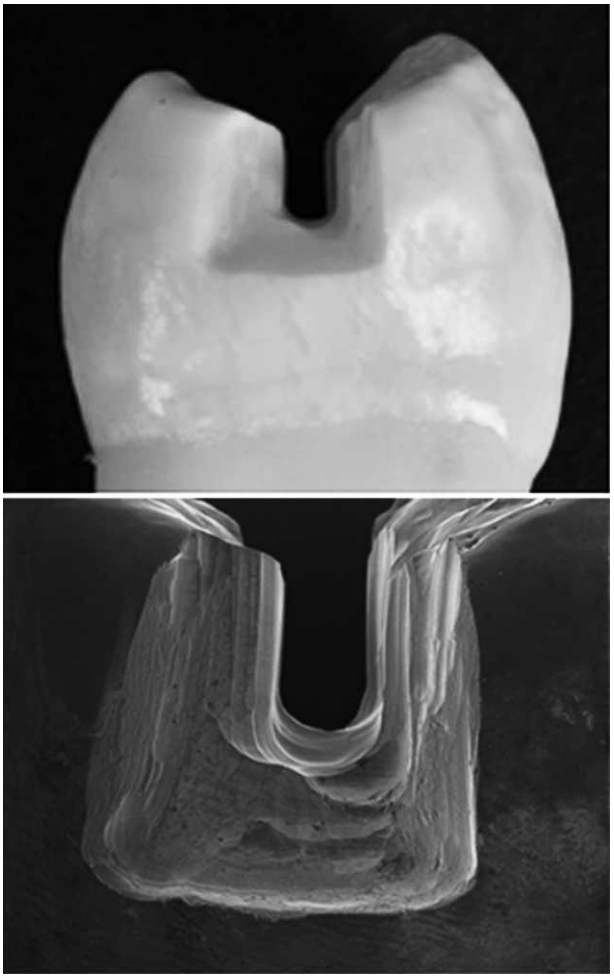

Figure 2. *Mesial of bicuspid prepared with rotary and hand instruments: a) natural tooth, b) preparation magnified 35X.*

ration in the posterior sections. The normal action of a bur at high speed, as it cuts the cavo-surface of interproximal enamel also leaves the enamel microscopically irregular. This must be corrected by the use of hand instrumentation to provide smooth cavo-surface margins. The facial lingual and gingival margins, especially with amalgam and cast restoration, require a smooth finish to receive the materials used for the restorations. The only way this can be done adequately is by the careful use of hand instruments used in a planeing or scraping action.

To be effective clinically, these instruments must be maintained at the highest level of sharpness. A carbon steel instrument will stay sharp longer than a stainless steel instrument but they are very subject to corrosion when subjected to autoclave sterilization.(6) This can be minimized by using dry heat sterilization (the turnaround time is increased) or chemical sterilization that requires ventilation to the external environment

Hand instruments are expected to require frequent resharpening. This involves using a high quality sharpening stone that may be stationary or revolving. As far as possible, the sharpening process is directed against the cutting edge of the instrument.

Several researchers have explored the action of high speed dental burs on enamel and have provided photographic evidence indicating less than perfect margins. To gain additional information we placed natural teeth in a controlled manikin setting in an ideal proximal contact relationship. Using the best clinical skill possible, interproximal preparations were made on the lower posterior teeth. These preparations, by design, were made using a 330 bur only (Figure 1) or a 330 bur plus hand instrument finishing (Figure 2). The SEM prepara-

tions were photographed at 35X. The preparations formed exclusively by burs provided microscopic irregularities that would leave a restoration with less than ideal margins. The irregularities were markedly reduced when a sharp hand instrument was applied to margins. This reinforces the fact that hand instrumentation contributes in a positive manner to the improvement of our restorative preparations.

When concerned with the details of operative preparations, visual acuity is mandatory. Thus it is helpful to use rubber dam isolation, which is a traditional recommendation for the operative discipline. This has a positive impact on both our vision and access to our margins. It also is very helpful in maintaining the sensitive physical and chemical requirements of our restorative materials.

Our opening paragraph contained a question asking us to reflect on our identity. G. V. Black let it be known that we are to function in the role of being continual students of our discipline. Many years ago Peter K. Thomas would make the statement "If you have it on the shelf, you can deliver". Thus, the question is best answered by those who are willing to develop clinical skills and enhance their knowledge level, so as to solve our clinical challenges.

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Gold Inlay Procedures for Extensive Cervical Lesions

RV Tucker • RD Tucker

This clinical technique article presents a design and procedure for Class V inlays that simplify the operation. Operating time is minimal. Both preparation and impression combined require less than an hour to complete, and the time required for seating and finishing is approximately the same. The inlays shown below were prepared from 9:00 AM to 10:00 AM (including time for intraoral photography), and seating/finishing was done from 4:00 PM to 5:00 PM on the same day (laboratory work was done in-house).

Figure 1. *Prototype of the inlay preparation on an Ivorine tooth.*

The preparation has an outline similar to a Class V gold foil (Ferrier design) but can produce extremely durable restorations for extensive carious lesions that would be too large for direct gold application. Gingival margins are rounded to follow the gingival tissue contour, and retention is derived from the incisal wall paralleling the opposing gingival wall. The proximal walls offer resistance form, but the flair of these walls does not contribute to the retentive form.

These inlays are subjected to minimal displacement forces and are not in a functional area of the tooth, which may help provide the long-term success of this restoration. Type I gold is recommended to facilitate finishing.

Conflict of Interest

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

(Accepted 16 December 2011)

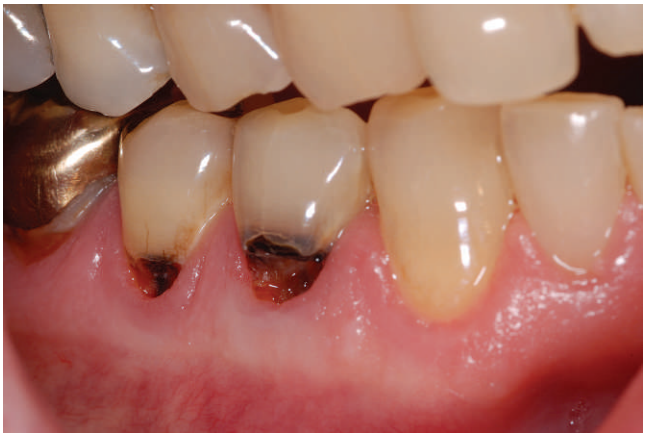

Figure 2. *Extensive erosion and caries at the gingival area of the mandibular right first and second premolars.*

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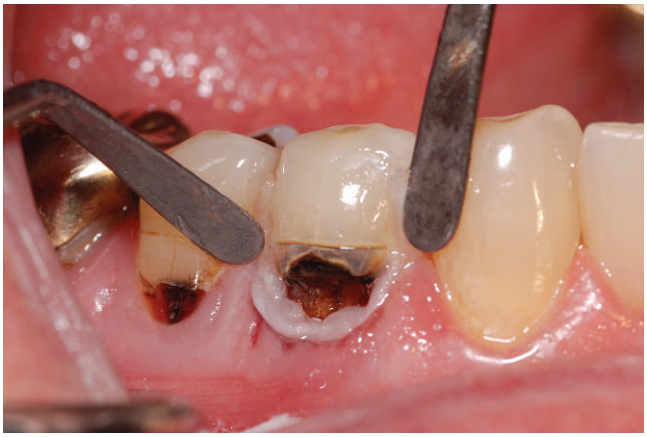

Figure 3. *The gingival tissues are retracted with a double cord (Gingipak, Belport Co, Camarillo, CA, USA) saturated with aluminum chloride. (Hemodent, PremierDental, Plymouth Meeting, PA, USA)*

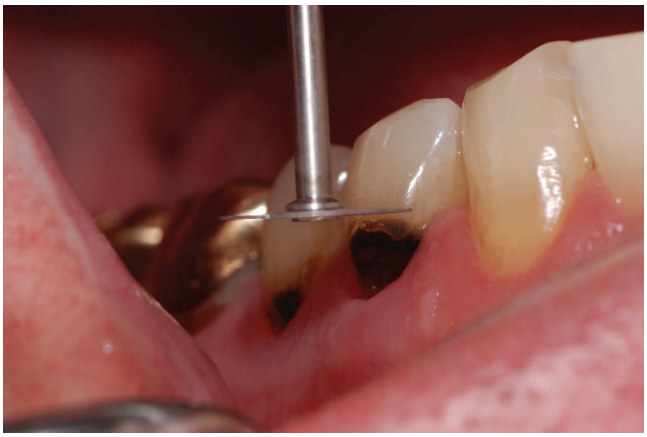

Figure 4. *The occlusal wall is prepared with a small diamond disk and is cut parallel to the occlusal plane of the rest of the arch.*

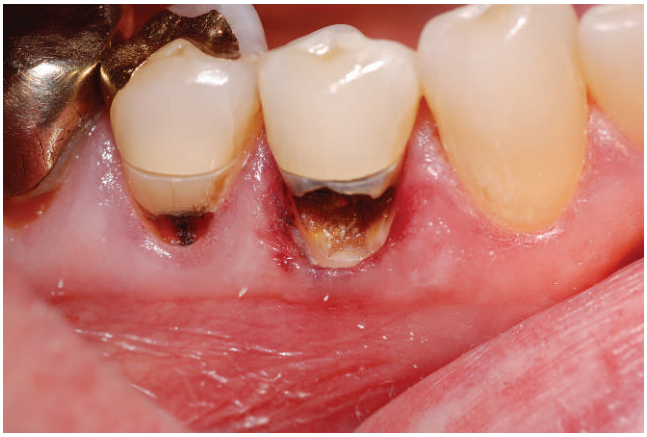

Figure 5. *Margins are exposed as cords are removed. (Note: No rubber dam is placed.)*

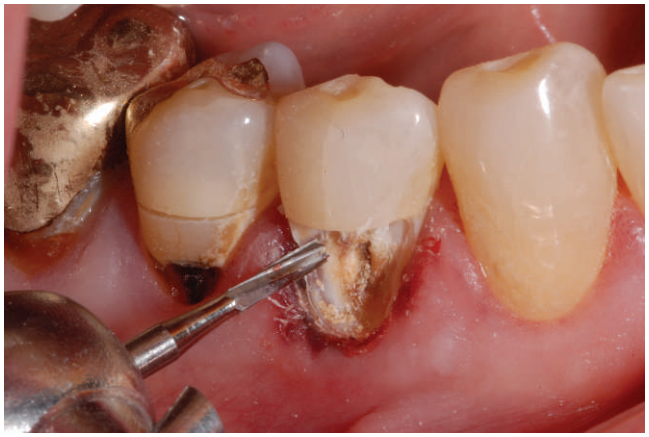

Figure 6. *A #56 bur is used to round the proximal and gingival walls and to flatten the axial wall.*

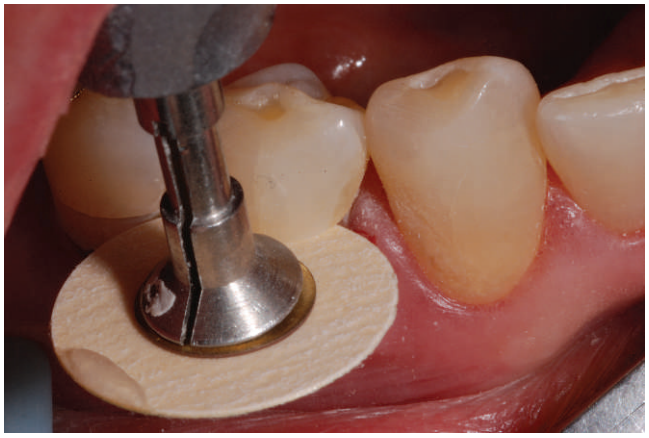

Figure 7. *A fine cuttle disk is used to smooth the incisal walls that were left slightly roughened by the diamond disk.*

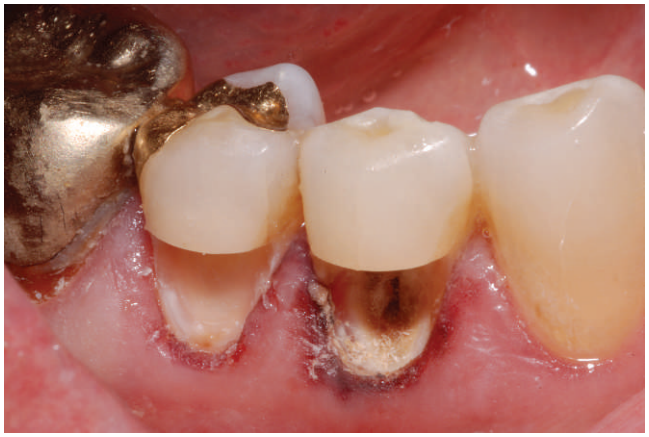

Figure 8. *Cavity preparations are finished (note that the incisal point angles were slightly rounded by the #56 bur). The gingival tissues are retracted with a single strand of packing cord (or two strands if the depth of the sulcus allows it).*

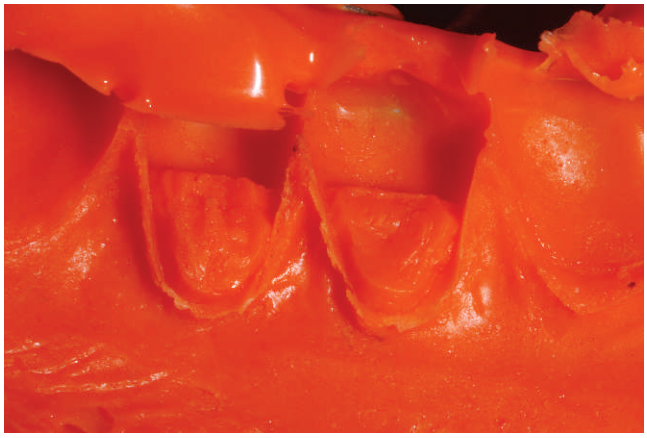

Figure 9. *The impression material is injected into the open sulcus and cavity using an impression tray that can be easily removed. Cavities are temporized with a soft plastic provisional material. (Fermit, Ivoclar Vivadent, Amherst, NY, USA)*

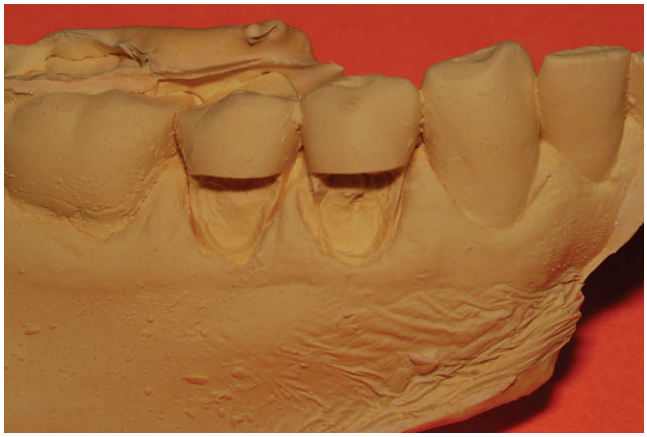

Figure 10. *Impression is poured with die stone.*

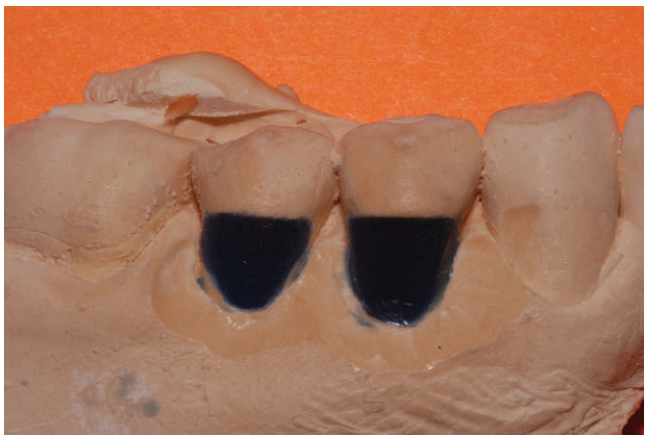

Figure 11. *Wax patterns are formed on the dies.*

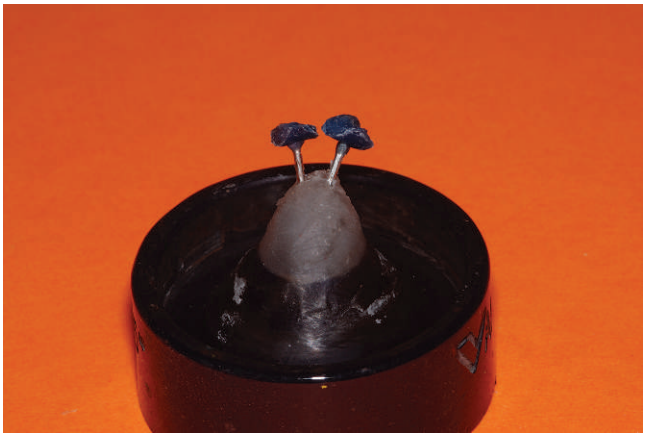

Figure 12. *Patterns are sprued and placed on the sprue former.*

Figure 13. *Patterns are invested in the casting ring using 15 cc of water and 50 g of investment (Novacast, Whip-Mix Co, Exeter, KY, USA) for maximum expansion. Castings are cut from the sprue but not finished. The sprue remnant is reduced with a small diamond point after cementation when the cement is set.*

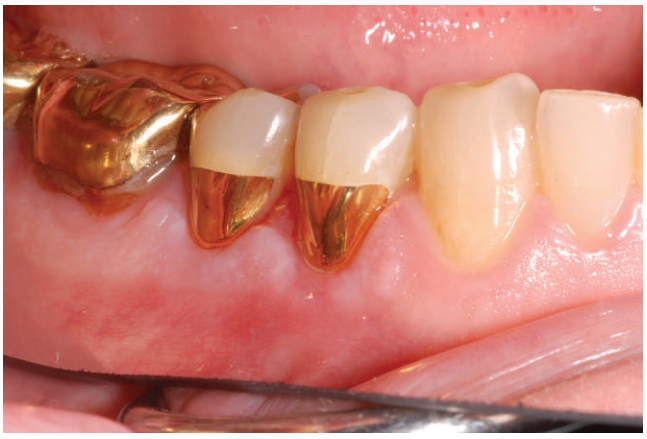

Figure 14. *One half–inch disks of medium garnet, fine sand, and fine cuttle are used to finish the inlays (including the margins).*

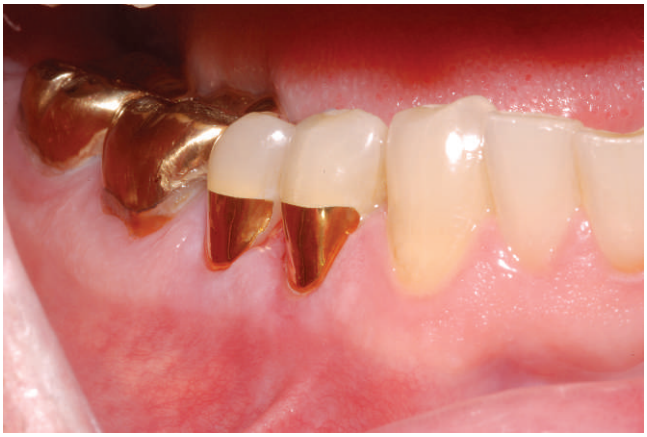

Figure 15. *Finishing is completed with a rubber cup (ribbed, not webbed) and fine flour of pumice, followed by 15-μm and 1-μm aluminum oxide. (Micro Abrasives Co, Westfield, MA, USA) (Note that the inlays are not pre-finished prior to cementation but are only cut from the sprue.)*

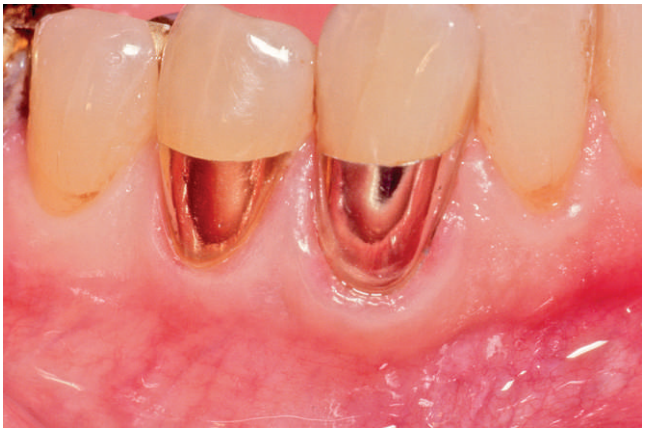

Figure 16. *A similar inlay and gold foil have served more than 30 years.*

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The 7/8 Crown: A Lost Art

ST McGill • JR Holmes

Clinical Relevance

In the right situation, A 7/8 gold crown can be the most esthetic, as well as, the most durable alternative to a ceramic restoration.

SUMMARY

Historically, the longevity of teeth restored with gold inlays, onlays, crowns, and partial veneer restorations is excellent. However, in-office computer-aided design and computer-aided manufacturing restorations, laboratory-constructed all-ceramic bonded restorations, and conventional ceramo-metal restorations are more common. The high price of gold, the difficulty of the preparation, and the fact that most dental schools are de-emphasizing the teaching of partial veneer restorations has created a situation whereby the 7/8 crown is rarely viewed as the treatment of choice. Time and experience will determine if the new ceramic materials, along with the all-important bonding agents, can achieve the success of a well-done, all-gold restoration.

Patients today have increasing esthetic demands, and many new materials and techniques are advocated to deal with this issue. All-ceramic crowns, both fabricated and milled, as well as ceramo-metal restorations are commonly used with varying degrees of success. Some meet esthetic demands while failing from a strength standpoint, and others fall short from an esthetic perspective despite being serviceable over many years. Some obviously satisfy both requirements.

One option that is seldom used is the 7/8 crown, which, to a great degree, has become "a lost art." In the right situation it can be the most esthetic as well as the most durable alternative.

INDICATIONS AND CONTRAINDICATIONS

The primary indication for a 7/8 crown is to restore a maxillary first molar where the mesiobuccal enamel surface is intact. This eliminates the need for porcelain (or some other ceramic material),

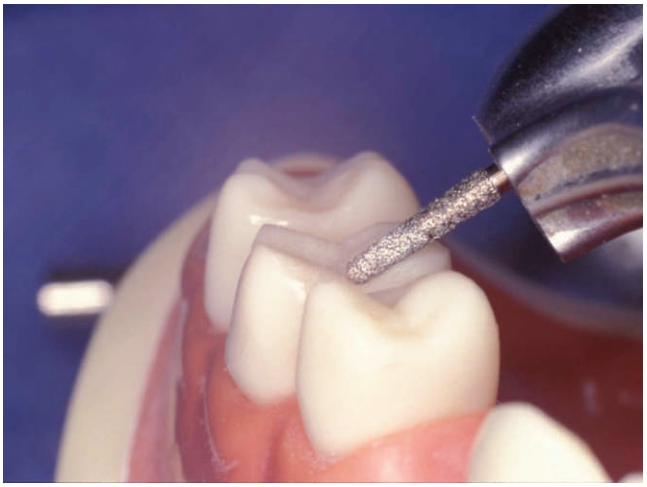

Figure 1. *Occlusal reduction.*

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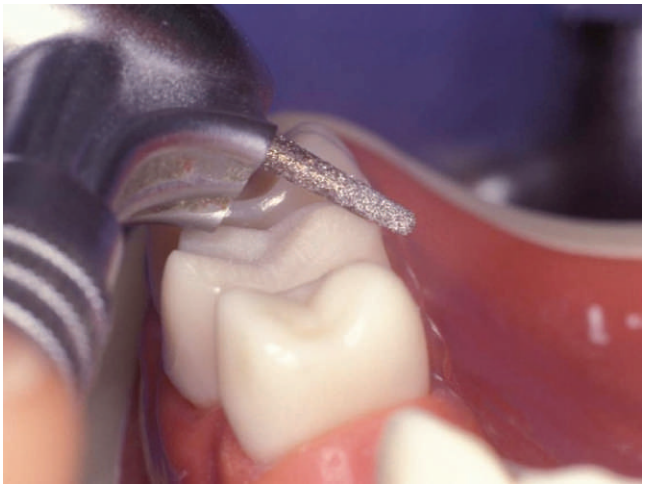

Figure 2. *Functional cusp bevel.*

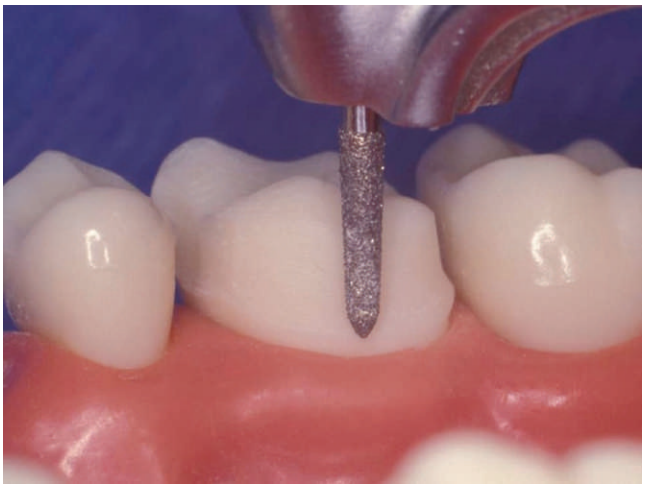

Figure 3. *Axial reduction and proximal extension.*

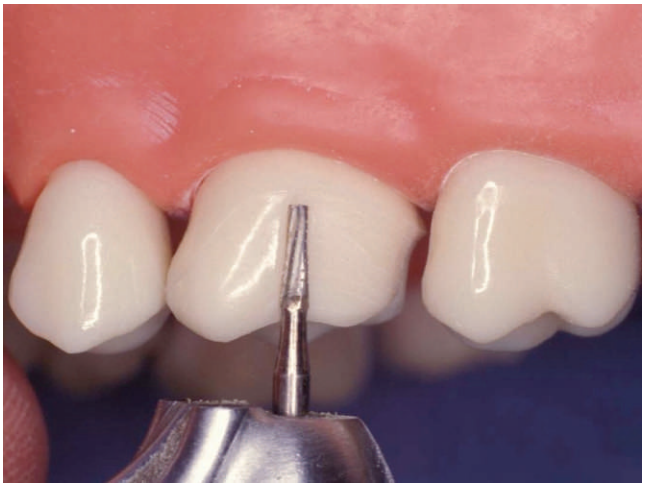

Figure 4. *Proximal and buccal grooves.*

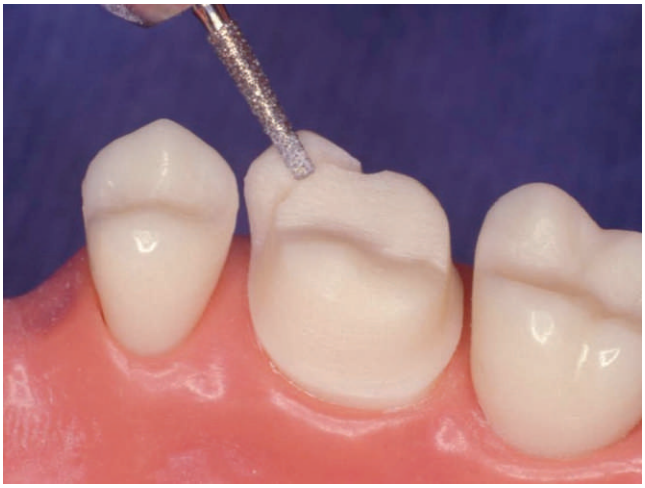

Figure 5. *Occlusal offset.*

which is not as durable as gold, and it also has the esthetic advantage of maintaining natural tooth structure.[1]

The primary contraindication for a 7/8 crown is when there is some defect or esthetically compromised quality in the buccal enamel of the mesiobuccal cusp.

STEPS IN TOOTH PREPARATION

Tooth preparation for a 7/8 crown is more difficult than that of a full veneer crown and needs to be accomplished precisely. This relative difficulty is

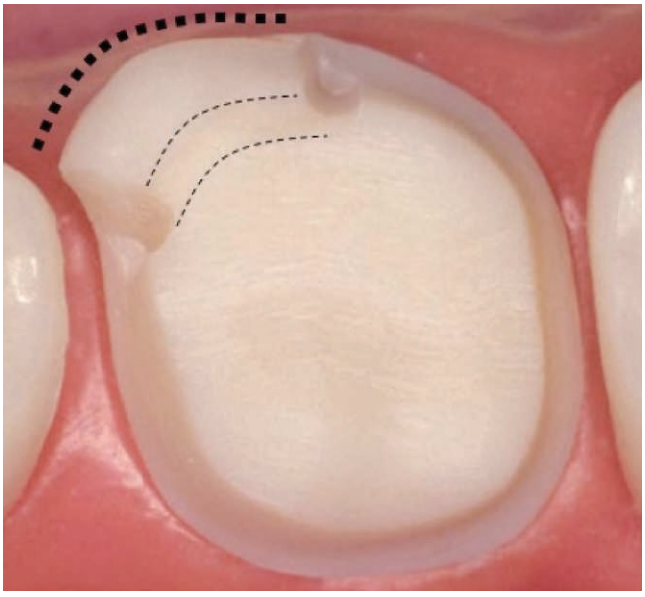

Figure 6. *Occlusal offset follows contour of mesiobuccal surface.*

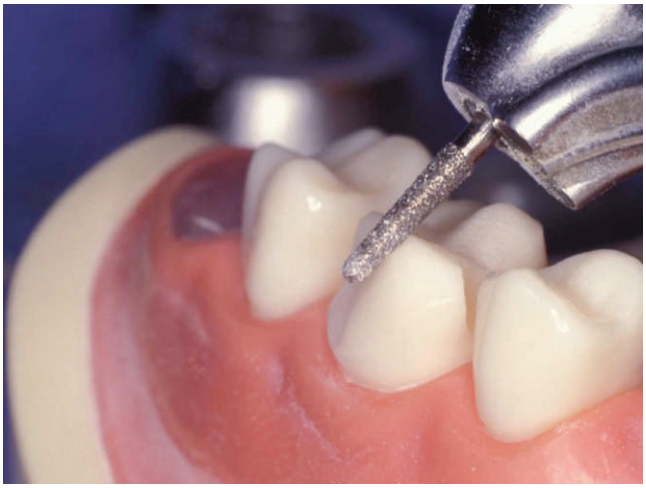

Figure 7. *Buccal reverse bevel.*

probably the primary reason this type of restoration is rarely used. The steps are as follows:

Step 1: Occlusal Reduction[2]

Occlusal reduction is accomplished in the same manner as for a full-veneer gold crown on the lingual cusps and distobuccal cusp (Figure 1). The reduction on the mesiobuccal cusp tapers out from the normal amount in the central groove area to about 0.3 mm at the cusp tip in order to avoid a resultant excessive display of metal when the mesiobuccal cusp reverse bevel is placed (see step 6 below).

Step 2: Functional Cusp Bevel

A functional cusp bevel is placed on both lingual cusps to provide additional metal thickness in this area (Figure 2). The bevel is placed at an angle consistent with the opposing tooth cusp/fossa angle (usually 30°-45°).

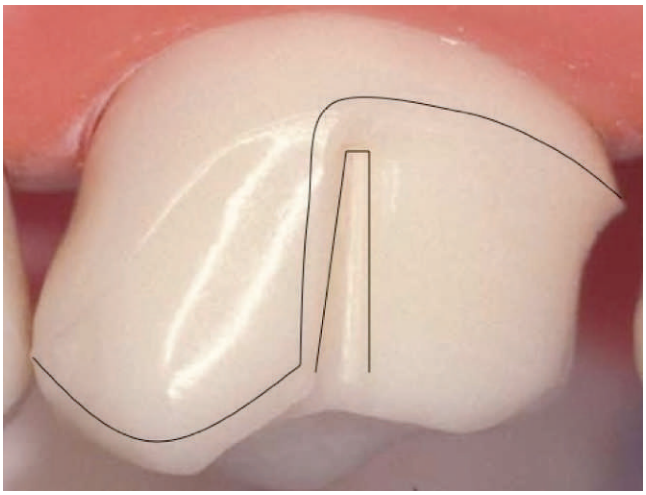

Figure 8. *Buccal reverse bevel (note position relative to groove).*

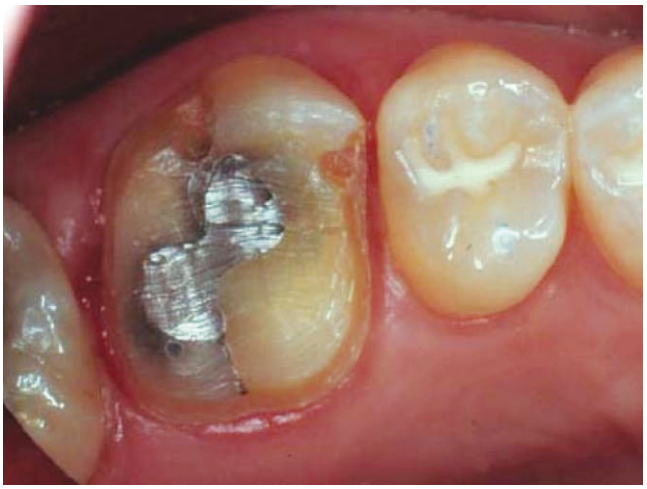

Figure 9. *The 7/8 crown preparation (note remnants of the crack still visible on the lingual).*

Step 3: Axial Reduction and Proximal Extension

The axial reduction should extend from a point that includes the buccal groove around the distal and lingual surfaces and includes the mesial surface to just short of the mesiobuccal line angle (ie, 7/8 of the axial surface). Proximal extension should result in contact being broken slightly at the mesiobuccal (Figure 3). A chamfer finish line is recommended.

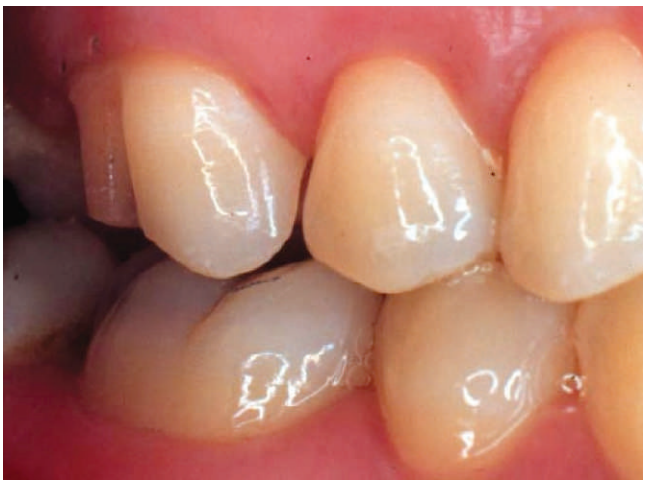

Figure 10. *The 7/8 crown preparation (buccal view) showing mesial extent of preparation (hidden behind height of contour of tooth no. 4), 0.75-mm reverse bevel of mesiobuccal cusp, and buccal groove (hidden behind height of contour of mesiobuccal cusp of the tooth itself).*

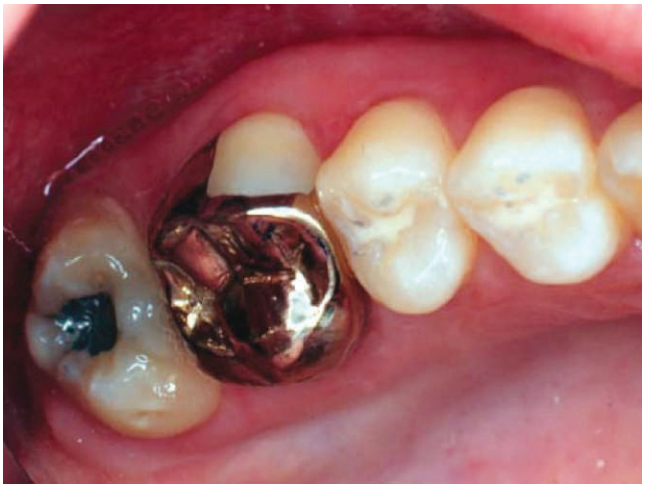

Figure 11. *Completed restoration (occlusal view).*

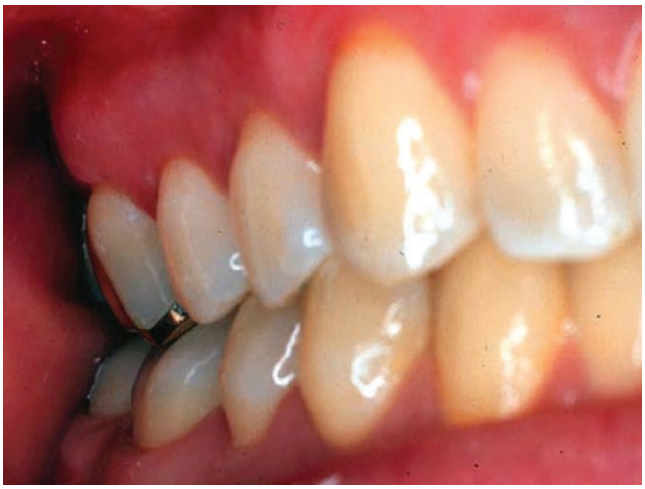

Figure 12. *Completed restoration (retracted facial view).*

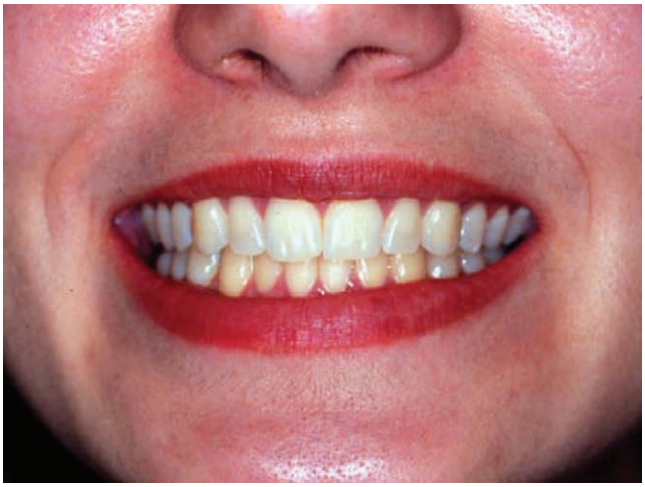

Figure 13. *Completed restoration (close-up view). Gold is not visible.*

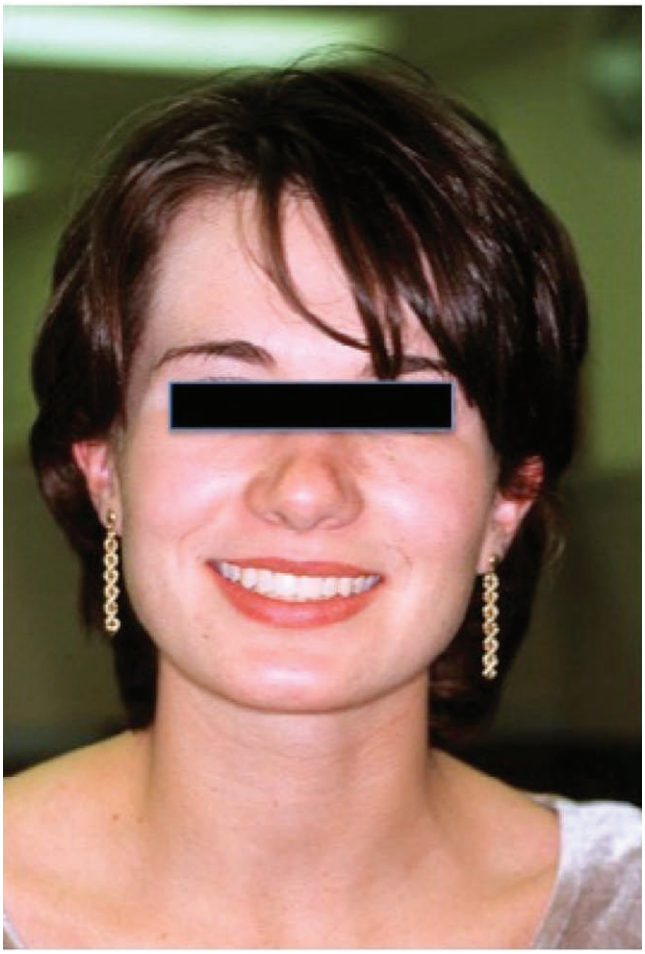

Figure 14. *Completed restoration (conversational distance) exhibiting esthetic success with maximum strength.*

Step 4: Proximal and Buccal Grooves

The proximal and buccal grooves should be placed parallel to each other and in line with the overall path of draw (Figure 4). A flat-end diamond or carbide is recommended. The entire groove should be within the axial reduction of the preparation and not extend fully to the cavosurface angle The proximal groove should be placed near the cavosurface mesiobuccal proximal extension without undermining the enamel surface (see also Figure 6).

Step 5: Occlusal Offset

The occlusal offset is a shallow trough that connects the two grooves and provides rigidity to the casting (Figures 5 and 6). It provides a bulk of metal to reinforce the thin reverse bevel of the mesiobuccal cusp and acts as an auxiliary sprue during the casting process. It is prepared with a flat-end diamond or carbide bur held at a 45° angle. The

occlusal offset should follow the contour of the external surface of the tooth.

Step 6: Buccal Reverse Bevel

The buccal reverse bevel is a thin band of metal covering and protecting the mesiobuccal cusp and prepared with a diamond at about a 45° angle (Figures 7 and 8). It is approximately 0.75 mm in dimension (occluso-gingivally) and follows the normal contour of the unprepared mesiobuccal cusp. If properly prepared it will strengthen the cusp and not be visible against the dark background of the mouth.

CLINICAL CASE

The patient is a 25-year-old esthetically conscious woman with a cracked maxillary first molar. The tooth was symptomatic to heat, cold, and biting pressure (Figures 9–14). Upon completion of the restoration, the tooth was restored to normal function with complete esthetic success. With proper maintenance the clinician can comfortably assure the patient many years of service without concern of breakage had ceramic materials been used. In addition, the tooth has been more conservatively prepared.

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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Multidisciplinary Approach in the Rehabilitation of Missing Lateral Incisors: A New Trend in Daily Practice

LQ Closs • EG Reston • F Tessarollo
MPM Freitas • G Broliato

Clinical Relevance

The clinical case described is a multidisciplinary clinical technique article. It showcases the need for a multidisciplinary approach to certain restorative cases and offers a number of possible options in addition to the one utilized for this patient.

SUMMARY

This article reports the case of a patient with bilateral hypodontia of the maxillary lateral incisors who was dissatisfied with the outcome of initial orthodontic treatment, highlighting the importance of a multidisciplinary interaction among Restorative Dentistry, Orthodontics, and Implantology to achieve satisfactory esthetics and functional results.

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INTRODUCTION

Several alterations may influence the establishment of occlusion during tooth development, including hypodontia, also known as tooth agenesis, partial anodontia, or oligodontia, which is characterized by one or more congenitally missing teeth.[1,2] The prevalence of permanent tooth hypodontia ranges from 3.5% to 6.5% in the general population, is more frequent among females (by a 3:2 ratio),[1,3,4] and displays no ethnic prevalence.[2]

The literature unanimously reports that this condition is caused by disturbances during the initial stages of tooth formation (initiation and proliferation),[1–3,5] is more frequent in the region of the permanent maxillary lateral incisors and mandibu-

lar second premolars,[1–3,6–11] and occurs bilaterally in most cases.[4]

The greatest challenge when treating patients with hypodontia of the maxillary lateral incisors is achieving satisfactory esthetics and function.[12–14] The first step is to establish the desired objectives and goals, considering the limitations of each individual case.

In the case of agenesis, the treatment options may include space maintenance for later rehabilitation with prostheses, dental implants, or orthodontic space closure, followed by restorative treatment when needed.[1,10,13,15–17] Space maintenance is often selected in the presence of adequate occlusal relationships, less flattened facial profile, and establishment of Class I canine relationship.[14] This treatment has often been indicated as a result of the increasing use of endosseous implants and the difficulty in achieving satisfactory esthetic outcomes by closing the edentulous space, especially in the case of unilateral hypodontia.[12]

DESCRIPTION OF THE TECHNIQUE (CASE REPORT, OPTIONS, AND GOALS)

Patient E.D., a 34-year-old male, attended the graduate clinic of the School of Dentistry for orthodontic retreatment with the chief complaint of unsatisfactory esthetics. During analysis of the patient history, the patient reported congenitally missing maxillary lateral incisors and previous orthodontic treatment with space closure by moving the canines into the edentulous space, followed by esthetic reconstruction of the maxillary anterior teeth.

Extraoral analysis indicated a relatively symmetrical face, marked nasolabial sulcus, straight profile with slight anterior maxillary deficiency, increased lower facial third, good lip sealing, and a low smile line (Figure 1.a,). Intraoral evaluation (Figure 1.b-d revealed a Class III molar relationship subdivision right; normal overjet and overbite; the absence of permanent maxillary lateral incisors and maxillary and mandibular third molars; the presence of maxillary and mandibular diastemas (positive discrepancy of 3 mm in the maxillary arch and 4.5 mm in the mandibular arch); maxillary canines positioned in the region of the maxillary lateral incisors; and a 3-mm deviation of the mandibular dental midline to the left side.

The initial panoramic radiograph revealed slightly short and parallel roots. Analysis of the lateral cephalogram demonstrated a skeletal Class III pattern with anterior maxillary deficiency, protruded maxillary incisors, and slightly retroclined mandibular incisors.

The study of the case presented different treatment options with regard to the following: 1) Space opening at the region of maxillary right and left canines for later placement of implants, prosthetic and esthetic rehabilitation of the maxillary anterior teeth; 2) Repositioning the maxillary canines to their original position and space maintenance for placement of implants at the region of maxillary lateral incisors; 3) Orthodontic closure of the maxillary and mandibular spaces by repositioning of the incisors

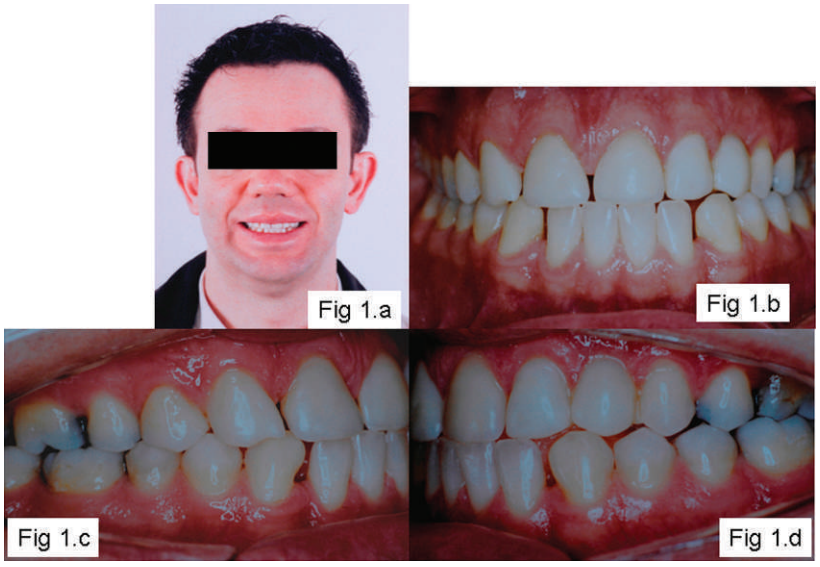

Figure 1. *(a) Initial smiling photograph. (b) Frontal smiling photograph. (c) Right intraoral view. (d) Left intraoral view.*

and canines, yet without midline correction; and 4) Closure of the maxillary and mandibular anterior spacing by esthetic restoration of the teeth, also without midline correction.

The treatment goals established were to achieve more favorable esthetics and functional occlusion by opening space at the region of the maxillary canines, followed by rehabilitation with implant/prosthesis for anatomic restoration of the maxillary anterior teeth. This would enhance the maxillary anterior dental proportions, improving the smile esthetics and harmony.

DESCRIPTION OF TECHNIQUE (TREATMENT PROGRESS)

First Stage: Orthodontic Treatment

The first stage of orthodontic treatment consisted of placing bands on the molars and bonding a fixed appliance to align and level with round archwires. This was followed by mesial movement of the permanent maxillary canines using elastics and open nickel-titanium (NiTi) coil springs, recreating space between the canines and the first premolars (Figure 2.a).

In the mandibular arch, alignment and leveling of teeth was achieved, and the diastemas were closed to achieve a more satisfactory position of the mandibular midline in relation to the maxillary midline, which was used as a reference. After using rectangular archwires, the fixed appliance was removed, (Figure 2.b) and the patient received maxillary (removable circumferential Hawley retainer) and mandibular (bonded wire between the mandibular right and left first premolars) retainers. The total time of orthodontic treatment was 36 months.

Second Stage: Treatment With Implant/ Prosthesis (Restorative Procedures)

After completing the orthodontic treatment, the patient was referred for restorative treatment, comprising placement of implants for teeth 13 and 23.

Tooth bleaching was performed in the lower arch to establish a gold standard for shade, as a result of the soundness of the teeth. For that purpose, an alginate impression was taken and a plaster dental cast was obtained. A vacuum-formed, custom-made tray was then fabricated and carefully trimmed to be nonirritating to the soft tissues. Tooth bleaching was performed with 10% carbamide peroxide, at home and overnight for 14 days. The patient was asked to return once a week to evaluate the bleaching effects. The patient received written instructions concerning the use of the gel, cleaning of the tray, risks of sensitivity, and dietary control to avoid highly staining foods, such as coffee and teas. Satisfactory bleaching was achieved at completion of this period; nevertheless, the treatment was continued for seven days to stabilize the outcome, avoiding early relapse of the dental shade.

During bleaching, the upper anterior teeth were prepared for placement of ceramic restorations. The upper incisors were prepared for ceramic veneers (Figure 2.c) and the upper canines for full crowns over implants. The patient presented with indirect

Figure 2. *(a) Orthodontic treatment for space reopening. (b) Final result after orthodontic treatment. (c) Anterior teeth preparation for complete crowns. (d) Temporary crowns cemented.*

restorations made with a glass polymer; therefore, there was no need for a huge change in the existing preparations, except when greater resistance and esthetics were necessary for the new ceramic restorations. Provisional acrylic crowns (Figure 2.d) were then placed and used during the period of tooth bleaching and laboratory procedures. After preparation, impressions were taken with polyvinyl siloxane in two stages: the putty material was initially used and then relieved to accommodate the light paste, allowing for an improved impression of the marginal areas (Figure 3.c).

Ceramic veneers were planned for the upper incisors. Porcelain-fused-to-metal (metal-ceramic) crowns for the upper canines were fabricated on endosseous implants.(Figure 3.a-d) After esthetic and functional adjustments, the crowns were glazed and cemented(Figure 4.a-d). The veneers were cemented using a self-etching cement, and the porcelain-fused-to-metal crowns were cemented on

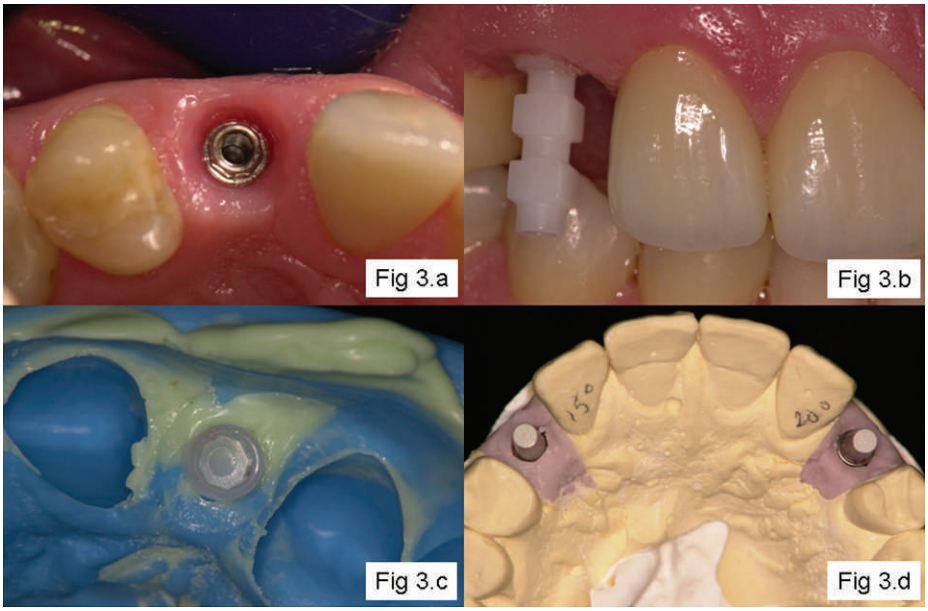

Figure 3. *(a) Occlusal view of the canine implant. (b) Impression component. (c) Impression with addition silicon. (d) Laboratory phase.*

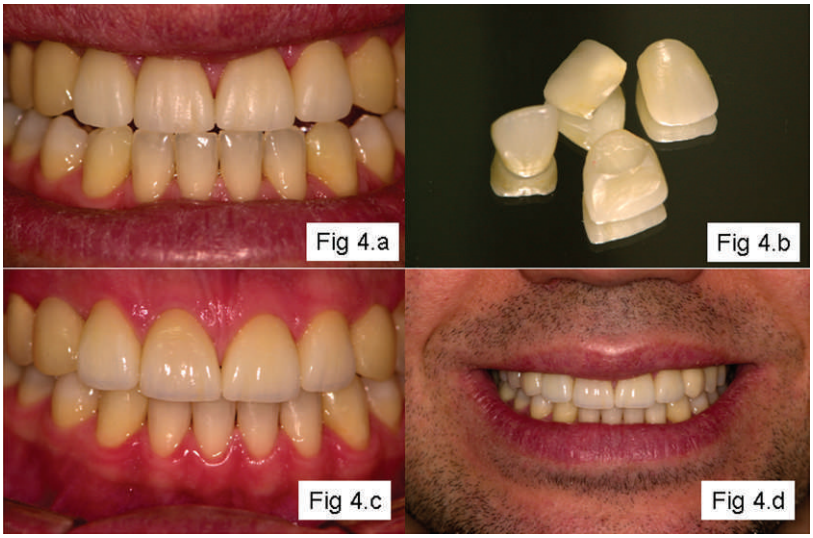

Figure 4. *(a) Pre-cementation adjustments. (b) Crowns before cementation. (c) Close-up of cemented crowns. (d) Patient smile showing pleasant harmony.*

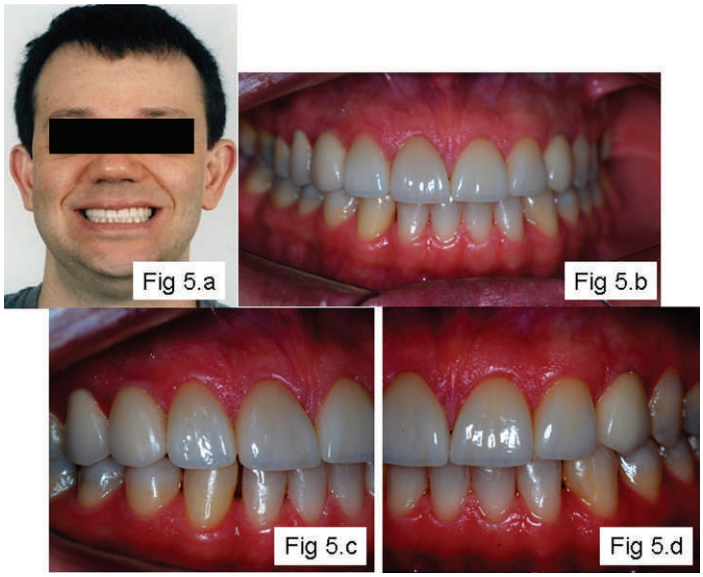

Figure 5. *(a) Twelve-month control time. (b) Gingival contour shows excellent response to ceramic contact. (c) Lateral view: Right side, at 12 months control time. (d) Lateral view: Left side, at 12 months control time.*

the metallic implant abutments using glass ionomer cement. The excess cement was carefully removed, and the patient received oral hygiene instructions. The final results present a natural smile and perfect harmony with healthy gingival tissues (Figure 5.a-d).

List of Materials

The materials utilized in treating the patient included the following:

- Brackets Roth prescription .022 × .025-inch (Ormco, Orange, CA, USA)
- Transbond XT, UNITEK (3M ESPE Dental Products, St Paul, MN, USA)
- 12-mm implants Narrow Neck (Straumann, Basel, Switzerland)
- Nite White 10% (Discus Dental, Culver City, CA, USA)
- Express (3M ESPE Dental Products)
- Empress IPV (Ivoclar Vivadent, Zurich, Switzerland)
- Rely X Unicem (3M ESPE Dental Products)
- Meron Cement (VOCO, Cuxehaven, Germany).

Potential Problems

The decision involved in re-treating a case is a difficult one, particularly when it involves changes in tooth position and structural changes of tooth morphology.

Instead of closing all of the existing spaces in the arch, the option of reopening spaces and proclining anterior teeth allowed for an improvement in facial esthetics with better lip support. On the other hand, the treatment period was extended, and there were more risks of root resorption. If space closure is indicated, the dental professionals should consider the possible adverse effects, including unfavorable achievement of canine guidance, flattened facial profile, consequent increase of the nasolabial angle, and deep bite.[3] Notwithstanding, this treatment option presents various advantages, such as immediate intervention without the need for skeletal maturation for implant placement in growing patients; improved gingival contour of the region; stability; and reduced treatment costs, because no prostheses or implants are required.[5,12]

Restorative treatment in the anterior teeth, particularly that involving implants, is a challenge. It is not only a technical issue but also a psychological matter, divided between patient's expectations and limitations in operator skill.

Retention for tooth movement should offer long-term control to avoid relapse.

Summary of Advantages and Disadvantages

The treatment outcome for this patient was very favorable, since it achieved a wider maxillary arch by opening space with placement of implant/prosthesis in the region of the maxillary canines and anatomic restoration of the maxillary right and left

central and lateral incisors. This procedure provided better proportion of the tooth crowns in the maxillary anterior region as well as enhanced gingival contour, favoring an esthetic smile.

Concerning the dental relationships between the maxillary and mandibular arches, there was a great improvement in terms of anterior tooth position and correction of the mandibular midline, highlighting the treatment benefits when considering the limitations of retreatment in an adult patient.

The overall treatment was long and dependent upon patient participation and comprehension. On the other hand, as a result of the use of this multidisciplinary approach, it was possible to create a good occlusal relationship that met with the patient's satisfaction.

CONCLUSION

The treatment, comprising an interaction between Restorative Dentistry, Orthodontics, and Implantology, allowed a favorable functional and esthetic outcome in an unfavorable skeletal Class III case with bilateral hypodontia. The treatment approach was based on the patient's expectations related to the correct morphology, esthetics, and function.

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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Clinical Research

Clinical Comparative Study of the Effectiveness of and Tooth Sensitivity to 10% and 20% Carbamide Peroxide Home-use and 35% and 38% Hydrogen Peroxide In-office Bleaching Materials Containing Desensitizing Agents

RT Basting • FLB Amaral • FMG França
FM Flório

Clinical Relevance

For the in-office technique, lower prevalence of tooth sensitivity may be expected when using in-office 38% hydrogen peroxide (HP) agent when compared with the 35% HP agent, which may be related to the presence, type, and concentration of desensitizing agents in the bleaching agents. The use of 10% carbamide peroxide (CP) or 20% CP home-use and 35% HP or 38% HP in-office treatments may have the same effectiveness in bleaching teeth.

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SUMMARY

The aim of this study was to compare the effectiveness of and tooth sensitivity to 10% and 20% carbamide peroxide (CP) home-use bleaching agents and 35% and 38% hydrogen peroxide (HP) in-office bleaching agents, all of

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which contain desensitizing agents, in a clinical trial. Four agents were evaluated: 10% CP and 20% CP (Opalescence PF 10% and Opalescence PF 20%, Ultradent, both with 0.5% potassium nitrate and 0.11% fluoride ions), 38% HP (Opalescence Boost PF, Ultradent, with 3% potassium nitrate and 1.1% fluoride ions), and 35% HP (Pola Office, SDI, with potassium nitrate). The initial screening procedure included 100 volunteers, aged 18 to 42, with no previous sensitivity or bleaching treatment and with any tooth shade. Volunteers were randomly assigned among the technique/bleaching agent groups. A run-in period was performed 1 week before the beginning of the bleaching treatment. For the home-use bleaching technique, each volunteer was instructed to dispense gel (10% CP or 20% CP) into the trays and then insert them into his or her mouth for at least two hours per night for three weeks. For the in-office bleaching technique, the bleaching agents (38% HP or 35% HP) were prepared and used following the manufacturer's instructions, with three applications performed in each session. Three sessions were carried out with an interval of seven days between each session. The participants were evaluated before, at one week, two weeks, and three weeks after the beginning of the bleaching treatment, and again one and two weeks after the bleaching treatment ended. A shade guide (Vita Classical, Vita) was used by a blinded examiner to perform shade evaluations before bleaching and two weeks after the end of bleaching. At the time of the shade evaluations, tooth sensitivity was also recorded by asking the volunteers to classify the sensitivity during bleaching treatment as absent, mild, moderate, or severe. The present study found that 13.8% of the volunteers withdrew from the study due to tooth sensitivity, and 43.2% of the participants experienced some type of sensitivity during bleaching treatment. The χ^2 test showed that there was a significant prevalence of tooth sensitivity during bleaching treatment using the home-use 20% CP agent, with 71.4% of volunteers reporting any level of tooth sensitivity ($p=0.0032$). A low prevalence of tooth sensitivity was observed for volunteers who used the in-office 38% HP agent (15.0%). The Wilcoxon test ($p<0.05$) showed that all of the bleaching treatments were effective in bleaching teeth and that there were no differences between the final color shade results among the treatments (Kruskal-Wallis, $p<0.05$). This study showed that 43.2% of all the volunteers experienced mild or moderate tooth sensitivity during the treatment with bleaching agents. A higher prevalence of tooth sensitivity was observed for 71.4% of the volunteers who used the 20% CP home-use bleaching agent, which may be ascribed to the peroxide concentration and/or the time/length the agent was in contact with the dental structures.

INTRODUCTION

Tooth bleaching is an increasingly requested dental treatment because it is considered to be a more conservative approach to improve the color of teeth without invasive procedures such as crowns or laminated veneers. Carbamide peroxide (CP) is a well-accepted agent for home-use bleaching supervised by a dentist; the gel is applied to the external surfaces of the teeth using a customized tray.[1] In the past, a 10% CP was considered as the standard product for the home-use bleaching technique.[2] In an attempt to increase the efficacy of the bleaching agents, higher concentrations of CP were used,[3–7] as well as different concentrations of hydrogen peroxide (HP), ranging from 3% to 10%.[6,8] The main advantages of the home-use technique are the ease of use, reduced chair time, and a low incidence of tooth sensitivity and gingival irritation.[9–12] Also, some home-use agents include fluoride and/or other desensitizing products, such as potassium nitrate, in formulations that may reduce tooth sensitivity.[11] However, the in-office technique has emerged as more popular than home use because highly concentrated products may promote faster tooth whitening (the higher the bleaching solution concentration, the more quickly a shade change will occur).

The in-office systems typically use a high concentration of HP (15% to 38%) and make possible the use of light-activation devices (eg, plasma arc, light-emitting diodes, diode laser, and xenon halogen lamps) with the purpose of accelerating the whitening process. However, the use of light sources for in-office tooth whitening is still controversial.[13–15] The dentist is in complete control of the process throughout the treatment and has the option to end the treatment at any time. Usually the color change results can be observed after a single visit. Despite the advantage of the in-office method to quickly achieve tooth whitening, tooth sensitivity is usually reported.[13–19] As in the home-use agents, some manufacturers have incorporated fluoride or desen-

sitizing products into the in-office gel formulas to decrease tooth sensitivity. However, there is no information about the addition of these products regarding the decrease in tooth sensitivity and effectiveness in bleaching.

A number of clinical trials have compared the performance of high- and low-concentration agents used for home-use or in-office tooth bleaching, and some have shown a similar whitening effect regardless of the concentrations and techniques used.[9,12,20,21] Nevertheless, the incidence of tooth sensitivity or irritation gingival is more common when the agent concentration[19,21,22] or bleaching time[5,23,24] is increased.

However, due to the different techniques available for bleaching teeth (home use or in office), various concentrations of bleaching agents available in the market, and the addition of fluoride or desensitizing products in bleaching agents, it may be difficult for the dentist to choose the technique and agent that will prove to be the most effective for and least sensitive to the patients. Therefore, the purpose of this study was to compare the effectiveness of and the dental sensitivity to 10% and 20% CP home-use bleaching agents and 35% and 38% HP in-office bleaching materials containing desensitizing agents in a clinical trial. The null hypothesis tested was that there are no differences in efficacy and dental sensitivity with the use of these bleaching gels, regardless of their concentration, the technique used (home use or in office), or the presence of desensitizing agents.

MATERIALS AND METHODS

Ethics, Sample Size, Eligibility Criteria, Randomization, and Blinding

The protocol was reviewed and approved by the Research Ethical Committee of São Leopoldo Mandic School of Dentistry, Campinas, São Paulo, Brazil, prior to the start of the study. A total of 100 participants took part in this study. All participants signed an approved human informed consent form.

It was determined that a sample size of 80 volunteers would be necessary, with 20 volunteers per group. The sample size was increased to 25 volunteers per group to account for potential loss of participants or their refusal to participate.

The initial screening procedure included an anamnesis, an intraoral assessment, and a medical history form to determine the eligibility of each volunteer to enter the study. The study excluded pregnant and breast-feeding women, as well as people with active caries, periodontal disease, previous hypersensitivity, tetracycline-stained teeth, and who had received a prior bleaching treatment. The study required each participant to have six upper and six lower anterior teeth with no more than one-sixth of each buccal surface covered with a restorative material. The study included volunteers of either gender, aged 18 to 42 years, and with any tooth shade. The bleaching technique and concentration of the agent to be used (Table 1) were randomly attributed to the volunteers in an attempt to obtain an equal number (25) of volunteers per bleaching agent group by the use of a randomization table to allocate the participants to each study group. However, 94 volunteers (76 women and 18 men) were accepted to participate in this study after signing the informed consent form and meeting the inclusion/exclusion criteria of the study.

A shade guide (Vitapan Classical, Vita, Bad Säkingen, Germany) was used to perform initial baseline shade selection of the middle third of the central incisor. The researcher who evaluated the tooth shade did not know the technique or bleaching agent each volunteer used. No attempt was made to exclude participants with a lighter tooth shade of the central incisors (shade A1, for example) except the lightest one (B1), because other teeth would be darker than the shade presented by tooth 11 and the participant may require a bleaching treatment to improve the color of all teeth. At this moment, a statistical analysis was applied to detect whether there were differences in color shade of the volunteers among groups. The Fisher exact test showed that there was a homogeneous distribution of initial tooth shade color of volunteers among the technique and bleaching agents groups ($p=0.113$).

Bleaching Procedure

One week before starting treatment, a run-in period was performed for all participants to standardize the toothbrush (Oral B Classic, Procter & Gamble, São Paulo, SP, Brazil) and 1500 ppm fluoride dentifrice (Colgate Máxima Proteção Anticáries, Colgate-Palmolive, São Berbardo do Campo, SP, Brazil) used.

For the home-use bleaching techniques, alginate impressions (Jeltrate, Dentsply International, Milford, DE, USA) of both arches of each participant were obtained to prepare stone molds (Gesso pedra, Vigodent S/A Ind. Com., Rio de Janeiro, RJ, Brazil). No preparations with reservoirs were made because no differences in effectiveness[25] and no higher rates and intensity of gingival inflammation[26] have been

Table 1: *Bleaching Techniques, Bleaching Agents, Composition, Manufacturer, pH Measure, and Lot Number of the Agents Used in the Study*

Bleaching Techniques	Bleaching Agents	Composition[a]	Manufacturer	pH Measured	Lot Number
Home-use bleaching technique	Opalescence PF 10%	10% carbamide peroxide, 0.5% potassium nitrate, and 0.11% fluoride ions (1000 ppm); pH ~6.5	Ultradent Products, South Jordan, UT, USA	7.1	B51JR
	Opalescence PF 20%	20% carbamide peroxide, 0.5% potassium nitrate, and 0.11% fluoride ions (1000 ppm); pH ~6.5	Ultradent Products, South Jordan, UT, USA	7.2	B3NVC
In-office bleaching technique	Opalescence Boost PF 38%	38% hydrogen peroxide, 3% potassium nitrate, and 1.1% fluoride ions (10000 ppm); pH ~7.0	Ultradent Products, South Jordan, UT, USA	6.6	B3VFR; B563J
	Pola Office 35%	Liquid: 35% hydrogen peroxide, distilled water, and stabilizers. Powder: thickener, catalyst, pigments, and potassium nitrate (unknown concentration); pH ~7.0	SDI Limited, Bayswater, Victoria, Australia	2.6	083011; 082776; 082547

[a] *The exact percentage of these additives is proprietary.*

found. All teeth of both arches were to be bleached and thus were included in the trays. The trays and three bleaching gel tubes were given to each volunteer with instructions to dispense the gel into both trays and then insert them into the mouth for at least two hours per night for three weeks.[12]

For the in-office bleaching technique, the bleaching agent was prepared and used following the manufacturer's instructions. The gingivae of all teeth to be bleached were isolated with either OpalDam (Ultradent, South Jordan, UT, USA) light cured resin (for Opalescence Xtra Boost/ Ultradent, South Jordan, UT, USA) or Gingival Barrier (SDI Limited, Bayswater, Victoria, Australia) (for Pola Office/ SDI Limited, Bayswater, Victoria, Australia). To prevent saliva from flowing through embrasures of anterior teeth, a saliva ejector and cotton rolls were used in the sublingual region. An expanded lip retractor was used to protect lips.

For Opalescence Boost PF, the activator was mixed into the bleaching agent using the proper syringe. For Pola Office, the powder was mixed into the liquid using a brush applicator to obtain a homogeneous gel. For both products, the mixture was then applied 1–2 mm thick on the buccal surfaces of the teeth (second premolar to second premolar) of both arches and remained on for eight minutes. No heat or special lamps were used to complete the process. The agent was removed using suction and gauze only for a new application. After the last application, teeth were rinsed with water and the gingival isolation and lip retractor were removed. A total of three applications were completed in each session. There were three sessions with an interval time of seven days between each session.

All participants were advised to avoid darkened foods and beverages during bleaching as much as possible and to not use any kind of mouth rinses. For the home-use bleaching group of volunteers, written instructions concerning the proper use of the bleaching agent were given. Instructions were also given to call the main researcher or to cease using the treatment solutions if tooth sensitivity or gingival irritation was perceived as too great to tolerate.

At one, two, and three weeks after the beginning of the bleaching treatment, the participants of the home-use bleaching were assessed; at the same time, participants of the in-office technique were receiving their bleaching treatments. All participants were also evaluated one and two weeks after the end of the bleaching treatment. At the final evaluation appointment, the blinded researcher determined tooth shade by following the same protocol used at

Table 2: *Prevalence of Tooth Sensitivity Reported by Volunteers (Absolute and Percentage) During Bleaching Treatment According to Technique/ Bleaching Agent*[a]

	Absence		Presence		Total
	n	%	n	%	n
Home-use 10% CP	12	63.2	7	36.8	19
Home-use 20% CP	6	28.6	15	71.4	21
In-office 35% HP	11	52.4	10	47.6	21
In-office 38% HP	17	85.0	3	15.0	20
Total	46	56.8	35	43.2	81

[a] *χ^2 test, $p = 0.0032$.*

baseline. Tooth sensitivity also was recorded at this time by the same blinded researcher asking the volunteers to classify the sensitivity during bleaching treatment as absent, mild, moderate, or severe. If the sensitivity was severe enough that the volunteer stopped using the bleaching agent, the volunteer was withdrawn from the study.

Although the manufacturers stated the pH of the agents, an evaluation was made by using a fresh portion of each agent either extruded by the syringe (home-use agents) or recently mixed (in-office agents). A measurement in triplicate was performed using a pHmeter (MS Tecnopon Equipamentos Especiais Ltda, Piracicaba, SP, Brazil) (Table 1).

Statistical Analysis

The data were tabulated in an Excel program for each volunteer according to bleaching technique/ concentration, gender, tooth sensitivity, and tooth shade of the right central upper incisor and submitted to exploratory analysis. The selected tab in the shade guide was converted to previously established numeric values[9,13,21] ranging from 1 (B1) to 16 (C4) in decreasing order of value: B1, A1, B2, D2, A2, C1, C2, D4, A3, D3, B3, A3.5, B4, C3, A4, and C4. The smaller the numeric value, the lighter the tooth. The comparison between shade color before and after each treatment was analyzed by the Wilcoxon nonparametric test. The comparisons of shade color between volunteers among the technique and bleaching agents groups before and after the bleaching treatments were analyzed by the Kruskal-Wallis test. The associations among variables were analyzed by the χ^2 test (Bioestat 5.0 statistical program, Mamirauá Maintainable Development Institute, Belém, Brazil) or the Fisher exact test (Release 9.2, SAS Institute Inc, Cary, NC, USA) when at least one of the variables was less than 5. The significance level was 5%.

RESULTS

There was a homogeneous distribution of volunteers among the technique and agent bleaching groups, with 25 volunteers for 20% CP, 24 volunteers for 10% CP, 24 volunteers for 35% HP, and 21 volunteers for 38% HP. Some volunteers withdrew from the experiment due to extreme sensitivity during the bleaching treatment. A total of 13.8% of the volunteers withdrew from the study: five from 10% CP, four from 20% CP, three from 35% HP, and one from 38% HP.

There was a significant prevalence of tooth sensitivity during the bleaching treatment with the home-use 20% CP agent, with 71.4% of the volunteers reporting any level of tooth sensitivity (p=0.0032). A low prevalence of tooth sensitivity was observed for volunteers who used the in-office 38% HP agent (15.0%). The present study found that 43.2% of the participants experienced some type of sensitivity during the bleaching treatment (Table 2).

When tooth sensitivity was reported (Table 3), there was mild sensitivity when volunteers used the 10% CP home-use agent (85.7%). Severe sensitivity that did not compromise the continuity of the bleaching treatment was reported by volunteers who used 20% CP home-use agent (6.7%) and 35% HP in-office agent (10.0%).

There were no significant differences among groups in tooth color shade of volunteers after the end of the treatments among technique/bleaching agents groups (Table 4). All volunteers obtained a lighter shade color after the bleaching treatment, showing its effectiveness (Table 5). All the technique/bleaching agents had the same effectiveness (Table 6).

DISCUSSION

Tooth sensitivity is the most common adverse side effect of bleaching. It is related to the increase in enamel and dentin permeability and the consequent easy passage of the peroxide through the enamel and dentin to the pulp.[23,27,28] Although the great majority of people are able to tolerate tooth whitening, sensitivity related to tooth whitening is a critical

Table 3: *Intensity of Perceived Tooth Sensitivity Reported by Volunteers (Absolute and Percentage) During Bleaching Treatment According to Technique/ Bleaching Agent*

	Mild		Moderate		Severe	
	n	%	N	%	n	%
Home-use 10% CP	6	85.7	1	14.3	0	0.0
Home-use 20% CP	10	66.7	4	26.6	1	6.7
In-office 35% HP	6	60.0	3	30.0	1	10.0
In-office 38% HP	2	66.7	1	33.3	0	0.0
Total	24	68.7	9	25.6	2	5.7

problem. Studies have shown that the prevalence of sensitivity during home-use or in-office bleaching treatments varies from 0% to 100% of participants.[9,10,13,20,21,23,29,30] Bernardon and others[21] reported a higher rate of tooth sensitivity for the in-office bleaching treatment compared with the home-use technique, although other studies showed similar levels of tooth sensitivity when comparing both techniques.[9,19,20,31] This suggests that tooth sensitivity is not only related to the high peroxide concentration used in the in-office techniques but is also a symptom that may vary greatly from person to person.[10] In this study, 43.2% of volunteers experienced some sensitivity during the treatment with bleaching agents. With home-use bleaching agents, 71.4% who used 20% CP experienced tooth sensitivity vs 15% of volunteers who used the in-office 38%

Table 5: *Prevalence of Volunteers Who Showed Color Change According to Vita Shade Guide Scale*[a]

Initial Shade Color Tooth	Final Shade Color Tooth				
	B1	A1	A2	C2	B3
A1	9	(–)	(–)	(–)	(–)
B2	3	2	(–)	(–)	(–)
A2	16	12	(–)	(–)	(–)
A3	5	12	3	(–)	(–)
B3	(–)	1	(–)	(–)	(–)
A3.5	1	3	5	(–)	(–)
B4	1	(–)	(–)	(–)	(–)
C3	(–)	(–)	(–)	2	(–)
A4	(–)	(–)	2	(–)	1

[a] *Fisher exact test,* p = *0.7291.*

Table 4: *Prevalence of Color Shade in Volunteers (Absolute and Percentage) at the End of Bleaching Treatment According to Technique/Bleaching Agent*[a]

Shade	Home-use 10% CP		Home-use 20% CP		In-office 35% HP		In-office 38% HP		Total	
	n	%	n	%	n	%	n	%	n	%
B1	10	28.6	14	40.0	4	11.4	7	20.0	35	44.8
A1	5	16.7	5	16.7	13	43.3	7	23.3	30	38.5
A2	3	30.0	1	10.0	3	30.0	3	30.0	10	12.8
C2	1	50.0	0	0.0	0	0.0	1	50.0	2	2.6
B3	0	0.0	0	0.0	0	0.0	1	100.0	1	1.3
Total	24	30.8	25	32.1	24	30.8	21	26.9	78	100

[a] *Fisher exact test, p = 0.0501.*

Table 6: *Median, Minimum, and Maximum Values of Shade Color Tooth Before and After Bleaching Treatments and the Comparison by Wilcoxon Test*

Technique/Bleaching Agent	Before			After		
	Median[a]	Minimum	Maximum	Median	Minimum	Maximum
Home-use 10% CP	5 Aab	2	15	1 Ba	1	7
Home-use 20% CP	5 Ab	2	12	1 Ba	1	2
In-office 35% HP	9 Aa	5	15	2 Ba	1	5
In-office 38% HP	5 Aab	3	15	2 Ba	1	11

[a] *Medians followed by different letters (capital letters in rows and lowercase in columns) are different by Wilcoxon ($p<0.05$) and Kruskal-Wallis ($p<0.05$) tests, respectively.*

HP. This also shows that sensitivity may not only be related to the peroxide concentration but most likely is related to the time/length the application is in contact with the dental structure (higher for home-use agents), as well as to the presence, type, and concentration of desensitizing agents in the composition. Thus, the null hypothesis regarding dental sensitivity response was rejected. Moreover, the tooth sensitivity was considered mild or moderate, and only 13.8% of the participants in both techniques experienced enough extreme sensitivity to force them to withdraw from the study. Schulte and others[29] found that sensitivity was severe enough to cause 14% of the participants to discontinue the home-use bleaching 10% CP agent, although other studies showed no volunteers who withdrew from the study when using the home-use agents.[10,20] In this study 13.8% of the volunteers declined continuing the treatment due to sensitivity: 9.5% from the home-use treatment and 4.3% from the in-office bleaching treatment.

Although tooth sensitivity is generally reported immediately after the application of the in-office agents[13] or during the first few days of using the home-use bleaching treatment,[10] these events are generally mild and resolved during or on completion of the treatment.[16,32] In this study, tooth sensitivity records were reported at the end of the treatment as a way to evaluate the volunteer's perception of the bleaching technique used.

In an attempt to decrease or limit the side effects of dental sensitivity during bleaching, manufacturers have introduced different desensitizing agents into the composition of the bleaching agent, such as potassium nitrate, sodium fluoride, or amorphous calcium phosphate.[33] Dentists have done their part by using different techniques prior to or in association with the bleaching treatment, such as using of fluorides as desensitizing agents on a tray, or prescribing these products as mouth rinses or dentifrices, or topically applying them on the external surfaces of the teeth.[30,32,34,35]

The home-use agents evaluated in this study contain potassium nitrate and sodium fluoride, which have been shown to efficiently and significantly reduce postoperative sensitivity.[11] It is believed that potassium nitrate reduces dental sensitivity by decreasing the ability of nerve fibers in the dental pulp to repolarize after an initial depolarization due to pain sensation. Fluoride may be added to the bleaching agent's composition because it also may decrease sensitivity by blocking the dentin tubules, thus reducing fluid flow to the pulp chamber.[36] Some studies showed that the use of 10% CP with potassium nitrate and fluoride[37] or the use of 16% CP with amorphous calcium phosphate[38] significantly reduced the amount of sensitivity. Also, Matis and others[33] found no differences in sensitivity when comparing 15% CP containing potassium nitrate and fluoride with 16% CP containing amorphous calcium phosphate. Although the same concentration of desensitizing agents (0.5% potassium nitrate and 0.11% sodium fluoride) were formulated for different concentrations of the home-use bleaching agents (10% and 20% CP), a significantly higher sensitivity was experienced by the volunteers who used the 20% CP (71.4%) than by those who used the 10% CP (36.8%) (Table 2), using the same protocol

for both. For the group of volunteers who used the 20% CP agent, there was a higher prevalence of moderate or severe sensitivity than for those who used the 10% CP (Table 3). Thus, in comparing the home-use products, it can be suggested that a high concentration of CP may be related to a higher prevalence of tooth sensitivity.[19,21,22]

For the in-office bleaching treatments, a higher prevalence of tooth sensitivity was experienced by those volunteers who used the 35% HP agent than by those who used the 38% HP agent (47.6% and 15%, respectively, reporting some level of tooth sensitivity). Although the manufacturer of 35% HP does not mention the concentration of potassium nitrate contained in the formula, the results of a lower prevalence of tooth sensitivity for 38% HP may be related to the type and concentration of the desensitizing agents (3% potassium nitrate and 1.1% fluoride ions). This corroborates Al Shethri and others[17] who found no differences in tooth sensitivity when comparing 35% HP with 38% HP in-office agents. Thus, for the in-office bleaching agents, tooth sensitivity may not be related to the concentration of the bleaching agent used, as opposed to what was found for the at-home agents, but to the type and concentration of desensitizing agents used.

This study also confirmed that low-concentration bleaching agents can provide effects similar to those obtained with high concentrations, as shown by Kihn and others,[4] Matis and others,[5] Braun and others,[7] and Leonard and others.[23] Therefore, the null hypothesis, when considering the efficacy of bleaching, was accepted. A meta-analysis of seven clinical studies indicated a significant mean change from baseline of 6.4 shade-guide units, according to the Vitapan guide scale (Vita), by the use of tray-based bleaching systems using 10% CP gels.[39] In this study, a median change from baseline of 4 to 7 shade-guide units was observed for all techniques, confirming that all bleaching treatments were effective, without any differences of final color shade obtained with all treatments. Also, regardless of the initial color shade of the upper central incisors, 83% of the volunteers obtained the lighter shade colors (B1 or A1) of the Vita guide scale after treatment. In this study, the shade color was evaluated with a subjective method: visual examination with the aid of the shade guide. Although an objective method (such as the use of a spectrophotometer) would be more precise and without the influence of the examiner and illumination conditions, similar results regarding color change were observed in studies that used both evaluation methods,[9,13,21] showing that the subjective method is a reliable, practical, and useful method to evaluate color changes.

The effectiveness of the bleaching treatment is one of the major factors to be considered when choosing a bleaching technique or agent, but longevity, safety, and the patient's convenience should also play an important role in selecting the bleaching treatment. This study found no clinically significant differences in bleaching, which corroborates Giachetti and others,[31] who performed a clinical trial comparing at-home bleaching treatments with in-office bleaching treatments. Meireles and others[22] and Giachetti and others[31] showed that a higher CP concentration does not increase the longevity of the whitening effect of home-use tooth-bleaching agents. Da Costa and others[19] also verified that subjects preferred, and would recommend, the home-use bleaching technique over the in-office technique.

The results of this study indicate that 10% CP or 20% CP home-use treatments and 35% HP or 38% HP in-office treatments are effective bleaching procedures to whiten teeth. However, the 20% CP home-use treatment was found to produce more sensitivity than other techniques/agents, even though desensitizing agents were incorporated into the product. The technique preference of the dentist and patient, composition and concentration of the bleaching agents, side effects involved (such as tooth sensitivity), and effectiveness must be taken into consideration when choosing the safest bleaching treatment for the patient.

CONCLUSION

This study showed that 43.2% of the volunteers experienced mild or moderate tooth sensitivity during the treatment with bleaching agents. A higher prevalence of tooth sensitivity was observed for 71.4% of the volunteers who used the 20% CP home-use bleaching agent. This may be ascribed to the peroxide concentration and the time/length application of the agents in contact with the dental structure. For the in-office technique, a low prevalence of tooth sensitivity was observed for the volunteers who used the 38% HP agent when compared with those who used the 35% HP agent. This may be related to the presence, type, and concentration of the desensitizing agents in the composition. The use of the 10% CP or 20% CP home-use and the 35% HP or 38% HP in-office treatments have the same effectiveness in bleaching teeth.

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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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Laboratory Research

Adhesion of Indirect MOD Resin Composite Inlays Luted With Self-adhesive and Self-etching Resin Cements

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Clinical Relevance

The dentin bond strengths of indirect composite inlay restorations cemented with self-adhesive and self-etching resin cements were reduced after loading, while microleakage increased. There were no significant differences in microtensile bond strengths and microleakage between the three resin cements.

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SUMMARY

This study investigated the effect of loading on the bond strength to dentin and microleakage of MOD indirect composite restorations bonded with self-adhesive and self-etching resin cements with or without acid etching of the proximal enamel margins. Class II MOD cavities were prepared in 48 molar teeth into dentin and divided into three groups of 16 teeth. Impressions were taken and indirect composite inlays fabricated (Estenia C & B). The enamel margins of the proximal boxes of half the specimens were phosphoric acid etched, and the inlays were cemented with one of three cements (Panavia F 2.0, SA Cement, or Rely X Unicem). After luting, eight teeth in each cement group were mechanically loaded at 2.5 cycles/s for 250,000 cycles. Unloaded teeth acted as controls. Teeth were stored in Rhodamine B solution for 24 hours, sectioned buccolingually at the proximal boxes to examine microleakage using confocal microscopy, and further sectioned for μTBS testing of the resin-dentin interface. Analysis of variance was performed to assess the effect of

loading and acid etching on microleakage and bond strength. Acid etching had no effect on microleakage. No significant difference in the dentin bond strengths between the three cements existed after loading. Panavia F 2.0 exhibited a significant reduction in bond strength. With regard to microleakage at the proximal boxes, loading had no effect on dye penetration at the cavity floor. However, at the axial walls, loading had a significant deleterious effect on Panavia F 2.0. No difference in microleakage existed between the three cements at both sites before and after loading. In conclusion, the two tested self-adhesive cements exhibited similar bond strengths before and after loading to the self-etching resin cement. Loading reduced dentin bond strengths and increased microleakage at the resin-dentin interface. However, acid etching of the enamel margins had no significant effect on microleakage in the approximal regions of the bonded inlays.

INTRODUCTION

Clinicians are often faced with the challenge of restoring a tooth that has lost a substantial amount of tooth structure through caries or the combined effects of erosion, abrasion, and attrition. The patient often desires an esthetic, tooth-colored restoration, and therefore manufacturers offer a choice of resin composite or all-ceramic materials for construction of an inlay or onlay. In the case of resin composite made by the indirect technique, because fabrication and polymerization have taken place outside the mouth, this allows the composite to be placed in the cavity without any further shrinkage.[1] Moreover, inlays fabricated from hybrid resin composite are purported to exhibit similar physical properties to dentin, whereas those fabricated from ceramic cannot compensate for tooth deformation under occlusal loading.[1]

Several types of resin cement are available to cement an indirect restoration. These differ in their pretreatment of the tooth surface prior to application of the resin cement and have been classified as etch-and-rinse adhesives, self-etching adhesives, and self-adhesive cements.[2] Self-adhesive cements have attracted the interest of both manufacturers and clinicians alike because they do not require any prior treatment of the dentin surface and are straightforward to use. Self-adhesive cements are reported to be able to adhere to tooth structure because they contain acidic monomers that can simultaneously demineralize and infiltrate the tooth structure, enabling micromechanical retention of the resin.[2]

It is important that *in vitro* testing of adhesive restorations tries to simulate the oral environment, and tests that involve accelerated aging through water storage, thermocycling, and cyclic loading have been developed.[3-6] While there have been several studies on the mechanical loading of direct composite restorations,[4-7] to date there has been only one published study on the effect of loading on the bond strength of indirect composite restorations.[8] However, this research was carried out on Class II cavities in premolar teeth. To date, there has been no published research on the adhesion of indirect composite restorations in larger MOD cavities in molar teeth with respect to microtensile bond strength and microleakage measurement within the same tooth.

Several *in vitro* studies on indirect restorations bonded with resin cements have looked at microleakage.[9,10] However, the validity of microleakage evaluation as a predictor of the clinical performance of materials has been called into question, and it has been suggested that research instead focus on laboratory tests that are validated with regard to their ability to predict the clinical performance of restorative materials despite the fact that no laboratory test can simultaneously reproduce all the conditions encountered in the oral environment.[11] Since clinical trials often include marginal integrity among the evaluation criteria,[12] combining microleakage measurements with another *in vitro* test, such as bond strength testing within the same tooth, may provide useful information on the durability of the bonded interfaces of indirect restorations.[13]

Therefore, the aim of this experiment was to investigate the effect of mechanical loading on the microtensile bond strength and microleakage of MOD indirect composite restorations bonded with one self-etching resin cement and two self-adhesive resin cements. In addition, the effect of additional phosphoric acid etching of the proximal enamel margins on microleakage was investigated. The null hypothesis was that loading would have no effect on the bond strength and microleakage of the tested cements.

MATERIALS AND METHODS

Forty-eight noncarious human lower third molars, extracted in accordance with the rules of the local ethics committee (King's College London Dental

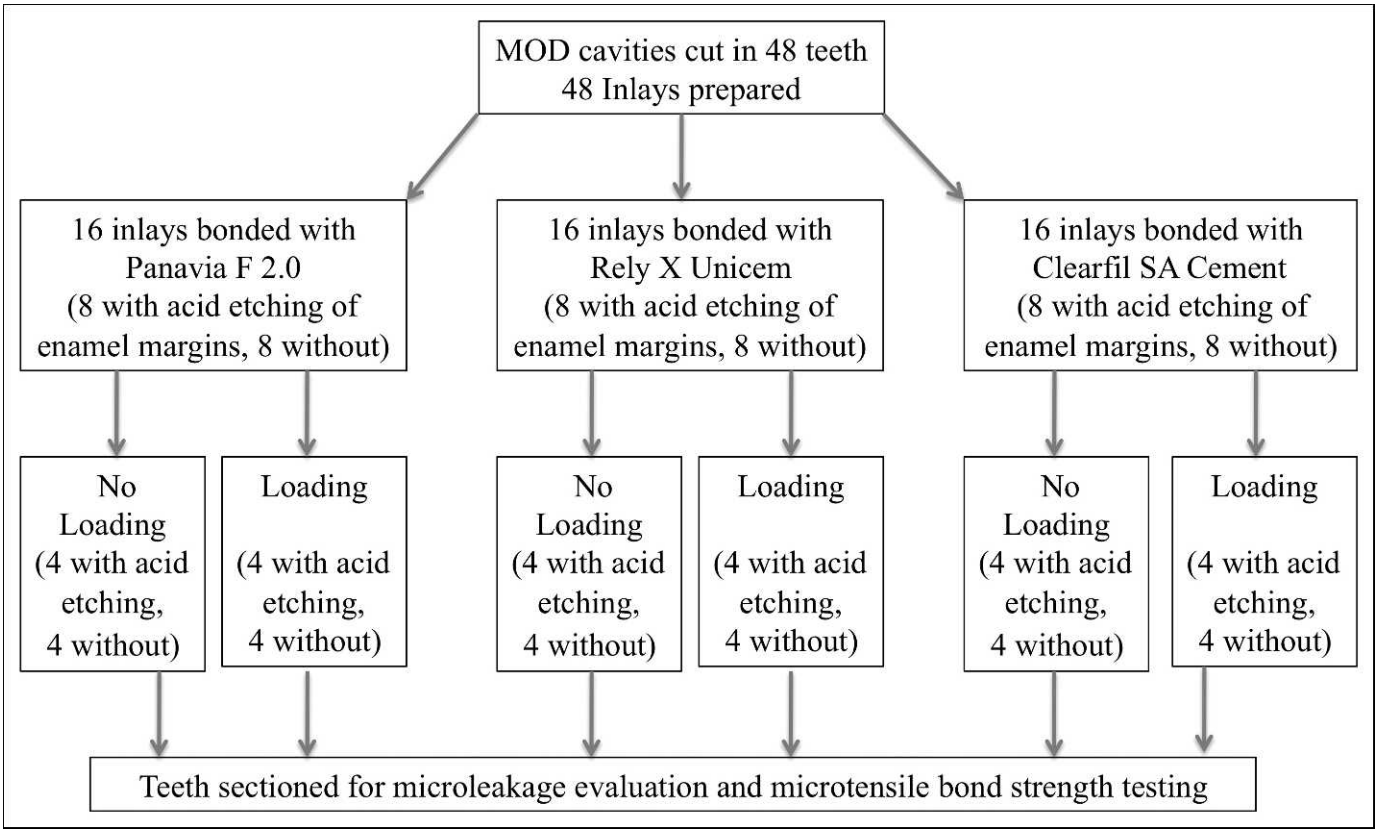

Figure 1. *Flowchart depicting the experimental design*

Institute Research Ethics Committee approval 04/Q0704/57) and after obtaining informed consent of the patients, were used in the study. Only caries-free lower third molars with no visible cracks and of similar dimensions were used. The teeth were stored in tap water at 4°C, undisinfected, in order to avoid chemical media–induced artifacts and were used within one month of extraction.

Each tooth was positioned in the center of the test chamber of the mechanical loading device to ensure that loading would be in the center of each restoration through the vertical axis of the tooth. To ensure that this position was maintained, an aligning method was developed and a dental milling machine used (Galloni, Milan, Italy). On a copolyester translucent disc (Erkodent, Pfalzgrafenweiler, Germany), a circular outline was scribed with the same diameter as the test chamber of the loading device. This disc was used to determine the center of the polysiloxane matrix. Then a rosehead bur (1 mm) was attached to the drill chuck of the milling machine and set in the center of the polysiloxane matrix. Each tooth was fixed with red beading wax (Kemdent, Dental Products Ltd, Purton, UK) to the rosehead bur. Autopolymerizing acrylic resin (cold cure modeling acrylic, Mr Dental, Old Woking, UK) was poured in the polysiloxane matrix from a height of 10 cm to ensure uniform filling of the matrix. Then the root base of each tooth was embedded in the acrylic resin to complete stabilization of the tooth.

Preparation Design

Figure 1 illustrates how the specimens were prepared. Each tooth was prepared with a diamond bur to receive an MOD inlay (FG 845C, Sybron Kerr, Orange, CA, USA) mounted on a high-speed hand piece under water coolant. The dimensions of the preparations were 4 mm buccolingually, 3 mm deep at the isthmus, and 4 mm deep at the mesial and distal boxes, and the boxes were also 1.5 mm at the base toward the pulp. The cavities were prepared 1-1.5 mm above the cemento-enamel junction, and the boxes were prepared with butt margins gingivally. All the internal line angles were smoothed to reduce the possibility of stress concentrations. The burs were replaced after every four preparations in order to ensure high cutting efficiency. All cavity dimensions were strictly standardized during preparation by securing specimens to a microscope stage converted into a specimen holder/cutting guide. Each

Table 1: *Resin Cements Used and Their Manufacturers and Composition*

Adhesive	Manufacturer	Composition
Panavia F 2.0 (batch no. 41247)	Kuraray Medical (Okayama, Japan)	ED Primer II: Primer A—HEMA, MDP, chemical initiator, water, 5-NMSA
		Primer B—5-NMSA, chemical initiator, water Panavia F 2.0
		A Paste—quartz, glass, MDP, methacrylate, photoinitiator
		B Paste—barium glass, NaF, methacrylates, chemical initiator
Rely X Unicem (batch no. 346518)	3M ESPE (St Paul, MN USA)	Powder—silica, glass fillers, calcium hydroxide, chemical-curing initiators, light-curing initiators
		Liquid—methacrylated phosphoric esters, dimethacrylates, chemical-curing initiators
Clearfil SA Cement (batch no. 06AAA)	Kuraray Medical (Okayama, Japan)	Paste A—Bis-GMA, TEGDMA, MDP, silanated filler, hydrophobic aromatic dimethacrylate, benzoyl peroxide, initiator
		Paste B—Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated fillers, surface-treated NaF, accelerators, pigments

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; MDP, 10-Methacryloyloxydecyl dihydrogen phosphate; 5-NMSA, N-methacryloyl 5-aminosalicylic acid; NaF, Sodium fluoride; Bis-GMA; Bis phenol-A diglycidylmethacrylate; TEGDMA; tri-ethylene glycol dimethacrylate.

cavity was cut by keeping the position of the hand piece constant and slowly moving the stage in the x, y, and z directions.

The prepared teeth were randomly divided into three groups of 16 teeth each, which were assigned to be bonded with one of three cements: Panavia F 2.0 (Kuraray Medical Inc, Okayama, Japan), Clearfil SA Cement (Kuraray Medical), and Rely X Unicem (3M ESPE, St Paul, MN, USA; Table 1).

Fabrication of Inlays

In order to make an impression of the prepared cavity, equal amounts of the base and catalyst of the low-viscosity poly vinyl siloxane (PVS) impression paste (Panasil initial contact light, Kettenbach GmbH & Co KG, Eschenburg, Germany) were syringed into the prepared cavity and on the surrounding tooth. At the same time, a glass specimen vial, 2 cm deep, was filled with high-viscosity PVS (Panasil putty fast set, Kettenbach) and seated onto the light-bodied material until the rim of the glass vial touched the dental stone embedding the tooth. This ensured that the impression of the cavity was embedded in a thickness of impression material of at least 10 mm. The cavity was temporarily restored using acrylic resin (Duraseal, Reliance, IL, USA). The impression was cast in a hard natural stone (Moonstone, Bracon Dental Laboratory Products, Etchingham, UK), which was mixed in the recommended water-to-powder ratio of 23 cc to 100 g.

The indirect composite resin inlay was fabricated using Estenia C & B (Kuraray Medical) in accordance with the manufacturer's instructions. In order to closely reproduce the clinical situation, cusps and fissures were created in accordance with the existing morphology of the tooth being restored. The base of the cusps was gently rounded to create a shallow central fissure that enabled accurate positioning and seating of the tip of the loading device at the center of the inlay both in a buccolingual and a mesiodistal direction.

Cementation of Inlays

Prior to cementing the inlays, half of the teeth in each group (eight) had the enamel margins of the prepared cavities etched with 37% phosphoric acid (K-etchant gel, Kuraray Medical) using a sponge microapplicator. After silanating the fitting surface of the inlay (Clearfil Ceramic Primer, Kuraray Medical), it was cemented in the prepared cavity using one of the three cements (Table 1). All the bonded specimens were stored in 37°C distilled water for 24 hours.

Mechanical Loading

Eight teeth in each group were fatigued in a water bath maintained at 37°C (JB1, Grant Instruments Ltd, Shepreth, UK). The fatiguing regime consisted of 250,000 cycles of 80-N loads at a rate of 2.5 loads per second. Static loading was applied vertically via a 2-mm-wide, round-ended, stainless-steel shaft attached to a LAL90 linear actuator (SMAC Europe Ltd, Horsham, UK), which generates force and motion using speaker coil technology to the midpoint of the composite restoration both mesiodistally and buccolingually. The actuator was operated via computer coding stored in an LAC1 controller (SMAC Europe) linked to a computer by an RS232 interface, allowing the input of loading parameters using the HyperTerminal program (Hilgraeve Inc, Monroe, MI, USA). The remaining eight teeth in each group were stored in water at 37°C for an equivalent time span.

Microleakage Evaluation

The method of specimen preparation for microleakage evaluation is illustrated in Figure 1.

There were four groups (nonetched enamel margins unloaded, nonetched enamel margins loaded, etched enamel margins unloaded, and etched enamel margins loaded) of four teeth for each of the three resin cements with respect to the evaluation of microleakage at the approximal boxes of the restored cavities.

The teeth were sealed with two layers of nail varnish up to 1.0 mm from the restoration margins after the root apices were sealed with wax. They were then immersed in a 0.25% solution of Rhodamine B in distilled water, for 24 hours. After storage, the teeth were thoroughly cleaned in an ultrasonic water bath (Biosonic, Coltène/Whaledent Inc, Cuyahoga Falls, OH, USA). Each tooth was sectioned twice buccolingually with a diamond wafering blade (Benetec Ltd, London, UK), yielding two end tooth sections close to the axial wall–gingival floor line angles of the proximal boxes. In preparation for examination using confocal microscopy, the sections were manually wet polished using 1,000-grit carborundum paper (Struers, Solihull, UK) for 20 seconds each. After polishing, the sections were ultrasonicated (Biosonic) in distilled water for three minutes each. Microscopy followed, in which each specimen was examined with a tandem scanning confocal microscope (TSM, Noran Instruments, Middleton, WI, USA) using a 20/0.80× NA oil immersion objective lens. Following calibration of an acetate measuring sheet to the objective lens' output to an iXon 885 EM-CCD camera (Andor Technology, Northern Ireland, UK), each margin could be scored for dye penetration. Detection of Rhodamine B infiltration was possible via suitable emission and excitation filters: 546 nm (green) and 600 nm (red), respectively. Using the CCD in fixed-gain mode in order to isolate the fluorescent signal, images were relayed to an LCD monitor via iQ capture software IQ (Andor Technology). Dye penetration was measured using the calibrated scale on the acetate sheet at each wall separately and expressed as a percentage. Dye penetration into enamel and dentin was not considered independently. For the axial walls, each tooth had four walls measured: buccal and lingual walls of both the mesial and the distal specimens. For the cavity floor, measurements for the mesial and distal slices were considered together.

Microtensile Bond Test

The method of specimen preparation for the microtensile bond strength test is illustrated in Figure 1. Sectioning of the two approximal slabs for microleakage evaluation left the central region of the bonded inlay to be used for microtensile bond strength measurement. This enabled evaluation of the bond between the inlay-resin cement and the cavity floor, which was dentin at a depth of 3 mm, measured from the enamel occlusal surface.

Since adhesion to the dentin of the cavity floor was to be evaluated and not the enamel cavity margins, the nonetched and etched enamel groups of bonded specimens were pooled together. This resulted in two groups of eight teeth (unloaded and loaded) for each of the three resin cements.

The remaining bonded specimens were sectioned buccolingually and then in a mesiodistal direction to obtain beams with an approximate surface area of 1 mm^2. The dimensions of each beam were checked using a digital caliper before the microtensile bond test was performed. A maximum of 32 beams per

group of eight teeth could be harvested for testing. In order to prevent the beams from becoming dehydrated prior to testing, they were stored at 100% humidity on moistened gauze in 7-ml glass vials. Each specimen was attached to a customized microtensile jig with cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA), which was mounted on a linear actuator (LAL 300, SMAC Europe)and stressed until failure at a speed of 1 mm/min.

Statistical Analysis

Data were analysed using Stata 10 (Stata Corp, College Station, TX, USA) software. Mean values of proportional microleakage for the four axial walls and the two cavity floors in each tooth were calculated. These microleakage data were not normally distributed and were described using a median and interquartile range. Three-way analysis of variance (ANOVA) was used to test the effect of type of cement, loading status, and etching on microleakage. For this analysis, the microleakage data were transformed to the square root of the proportion. Two-way ANOVA was used to test the effect of type of cement and loading status on microtensile bond strength. Subsequent post-ANOVA tests were performed using a Bonferroni correction for multiple comparisons. A *p*-value less than 0.05 was regarded as indicating statistical significance ($p<0.05$).

RESULTS

The results of the microtensile bond strength test and microleakage evaluation are presented in Tables 2, 3, and 4. A maximum of 32 beams per group could be harvested for testing. There were no pretest failures.

With regard to the microtensile bond strength test, when the specimens were not loaded, the bond strength of SA Cement was significantly less than both Panavia F 2.0 and Rely X Unicem. All three cements showed a significant reduction in bond strength after loading. However, there was no significant difference in bond strength between the three cements after loading (Table 2).

With regard to dye penetration into the adhesive interfaces, three-way ANOVA indicated that etching had no statistically significant effect on dye penetration and was excluded as a factor in subsequent analysis.

Panavia F 2.0 showed a lower level of microleakage at the axial walls than the other cements prior to loading, the difference being statistically significant in comparison with SA Cement ($p=0.036$). All three

Table 2: *Mean (SD) Bond Strengths (MPa) by Treatment and Loading*

Cement	Loading		*p*-Value
	No	Yes	
Panavia F 2.0	20.0 (4.1)	15.8 (3.9)	<0.001
SA Cement	14.1 (3.6)*	12.1 (2.6)	0.011
Rely X Unicem	18.0 (4.2)	15.5 (4.3)	0.021

** Significantly different from Panavia F 2.0 and Rely X Unicem (p=0.001).*

Table 3: *Median (Interquartile Range) of Percentage Microleakage at Axial Walls by Cement and Loading*

Cement	Loading		*p*-Value
	No	Yes	
Panavia F 2.0	12.7 (11.2–23.7)	38.4 (31.5-45.1)	<0.001
SA Cement	29.2 (24.2–34.5)*	45.5 (36.4–52.5)	0.040
Rely X Unicem	21.8 (19.8-33.4)	36.3 (31.4–44.9)	0.114
p- value	0.028	0.596	

** Significantly different from Panavia F 2.0 (p=0.036).*

Table 4: *Median (Interquartile range) of Percentage Microleakage at Cavity Floor by Cement and Loading*

Cement	Loading		*p*-Value
	No	Yes	
Panavia F 2.0	13.5 (10.5-38.8)	56.2 (26.1–76.9)	0.059
SA Cement	52.0 (39.5–67.7)*	69.6 (52.2–78.5)	0.604
Rely X Unicem	35.8 (21.3–54.3)	47.8 (35.1–63.0)	0.477
p-value	0.050	0.612	

** Significantly different from Panavia F 2.0 (p=0.047).*

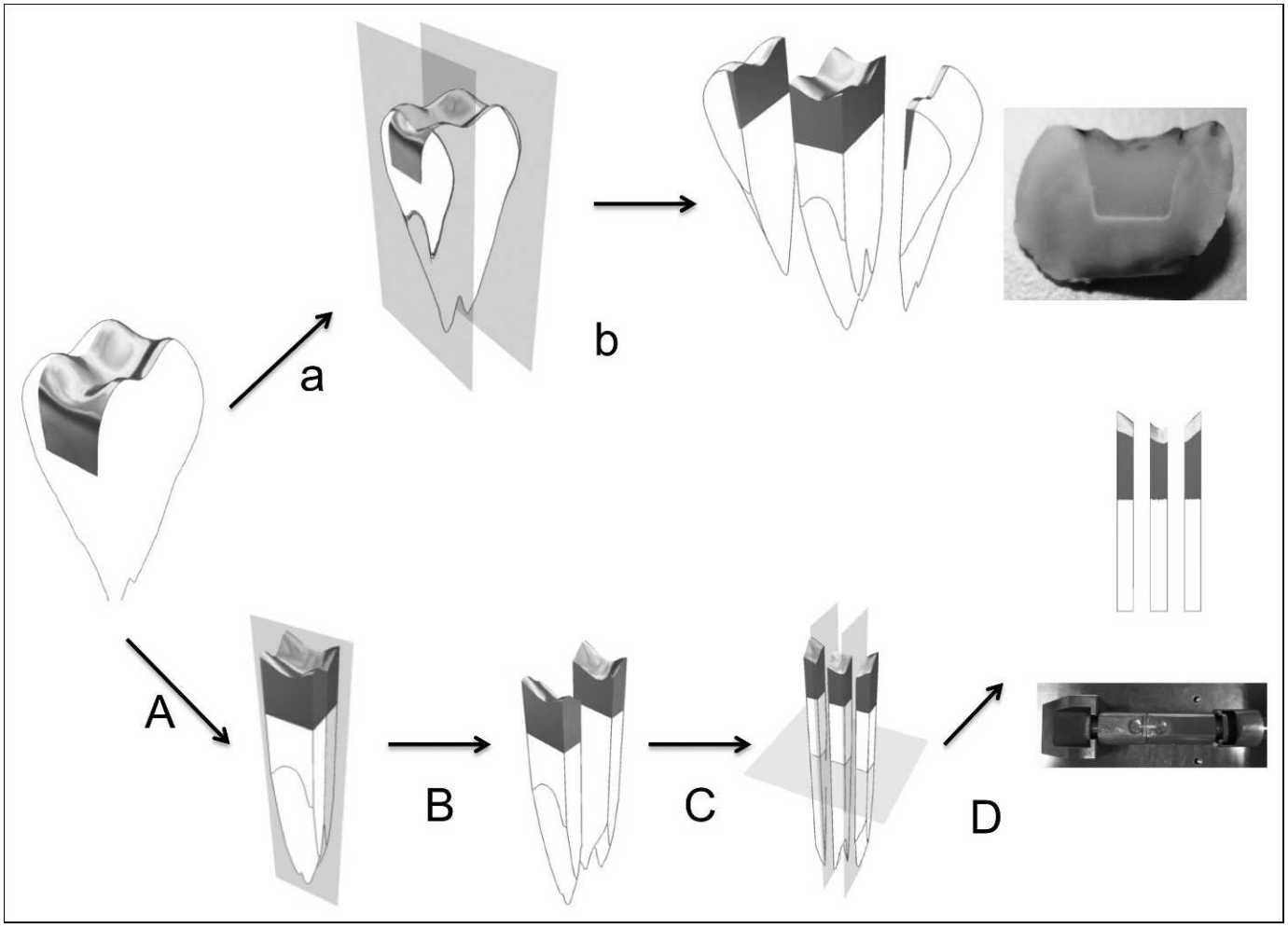

Figure 2. *Schematic representation of the method of specimen preparation for dye penetration and microtensile bond strength test of loaded specimens. (a and b): Two slices obtained for microleakage by cross-sectioning the tooth buccolingually close to the pulpal floor–axial wall line angle. (A): Occlusal portion of restoration selected for bond strength evaluation. (B): Buccolingual, then mesiodistal, slicing (C) to obtain beams for the microtensile bond strength test (D).*

cements exhibited an increase in dye penetration after loading, although the difference was statistically significant only in the case of Panavia F 2.0 and SA Cement (Table 3).

Panavia F 2.0 showed a lower level of microleakage at the cavity floor than the other cements prior to loading, the difference being statistically significant in comparison with SA Cement (p=0.047). All three cements exhibited an increase in dye penetration after loading, although none of the differences were statistically significant (Table 4). Representative observations of microleakage in the TSM images are shown in Figures 2 and 3.

The modes of failure of the beams are shown in Table 5. Almost all the beams of both the unloaded and the loaded groups of the three cements failed at the cement-dentin interface.

DISCUSSION

Large cavities in posterior teeth are challenging to restore from the point of view of creating a restoration with the correct anatomical form and proximal contacts. However, to date, there has been no information published on the *in vitro* durability of indirect composite inlays bonded in large MOD cavities of human molar teeth with either a self-etching or a self-adhesive cement. *In vitro* mechanical load cycling of restorations underwater is an important method for evaluating their clinical potential.[5,7] A force of 80 N was chosen as an average of the masticatory forces observed by Anderson,[14] and the loading condition of 250,000 cycles has been verified as one year of clinical wear.[15] The load was applied to the midpoint of the occlusal portion of the restoration. It has been

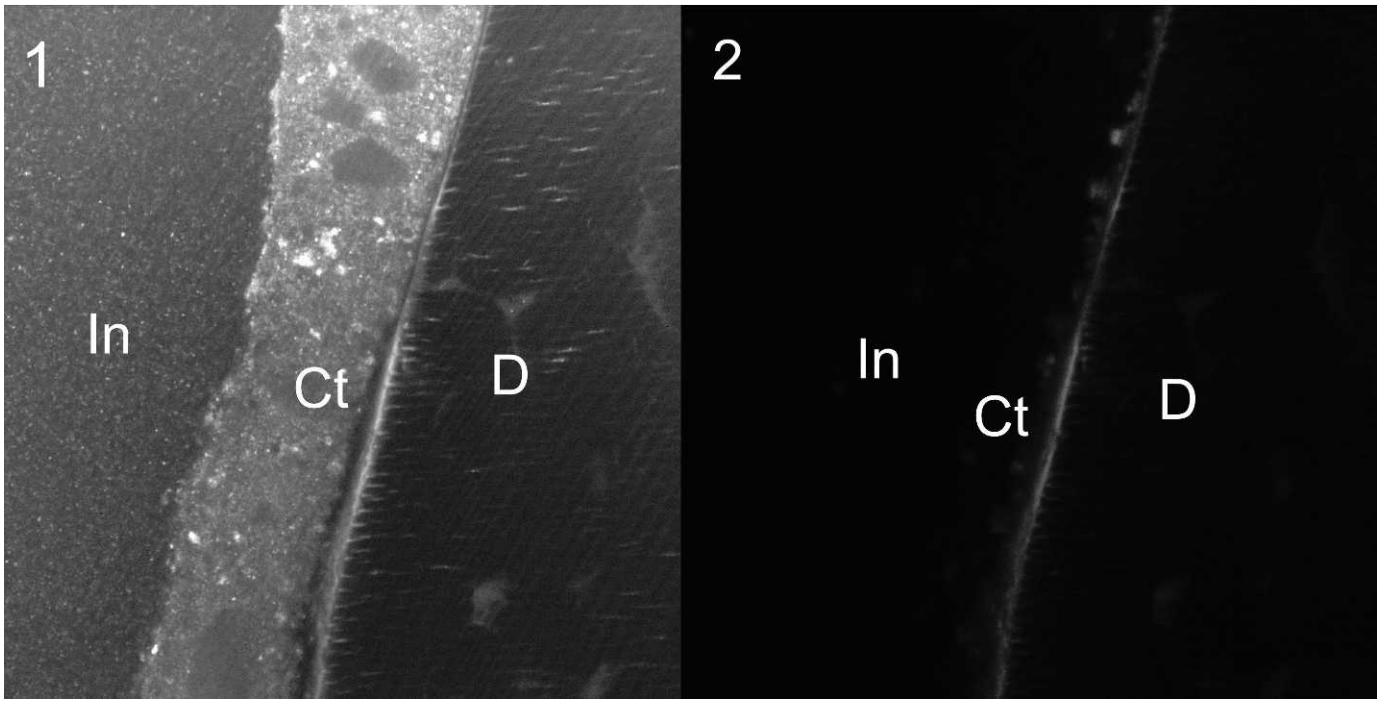

Figure 3. *Representative confocal scanning micrographs of fluorescence evaluation of an unloaded, etched Panavia F 2.0 axial wall specimen. (1): Reflection (546-nm illumination). (2): Fluorescence (546/600-nm excitation/emission) images of an area of axial wall. In, inlay; Ct, resin cement; D, dentin 20/0.80× oil immersion objective.*

reported that the application of a compressive load in the middle of the restoration would create tensile stresses along the bonded interface at the mesial and distal aspects of the restoration.[16] It was suggested that this would mimic the situation when occluso-proximal restorations are loaded directly by the opposing teeth during mastication.[16] The present study evaluated two self-adhesive cements and one

Table 5: *Failure Mode by Treatment and Loading*

	Cohesive in Inlay	Failure at Inlay-Cement Interface	Failure at Cement-Dentin Interface	Cohesive in Dentin
Panavia F 2.0				
No loading	0	0	31	1
Loaded	0	0	30	2
SA Cement				
No loading	0	0	31	1
Loaded	0	0	30	2
Rely X Unicem				
No loading	0	0	31	1
Loaded	0	0	31	1

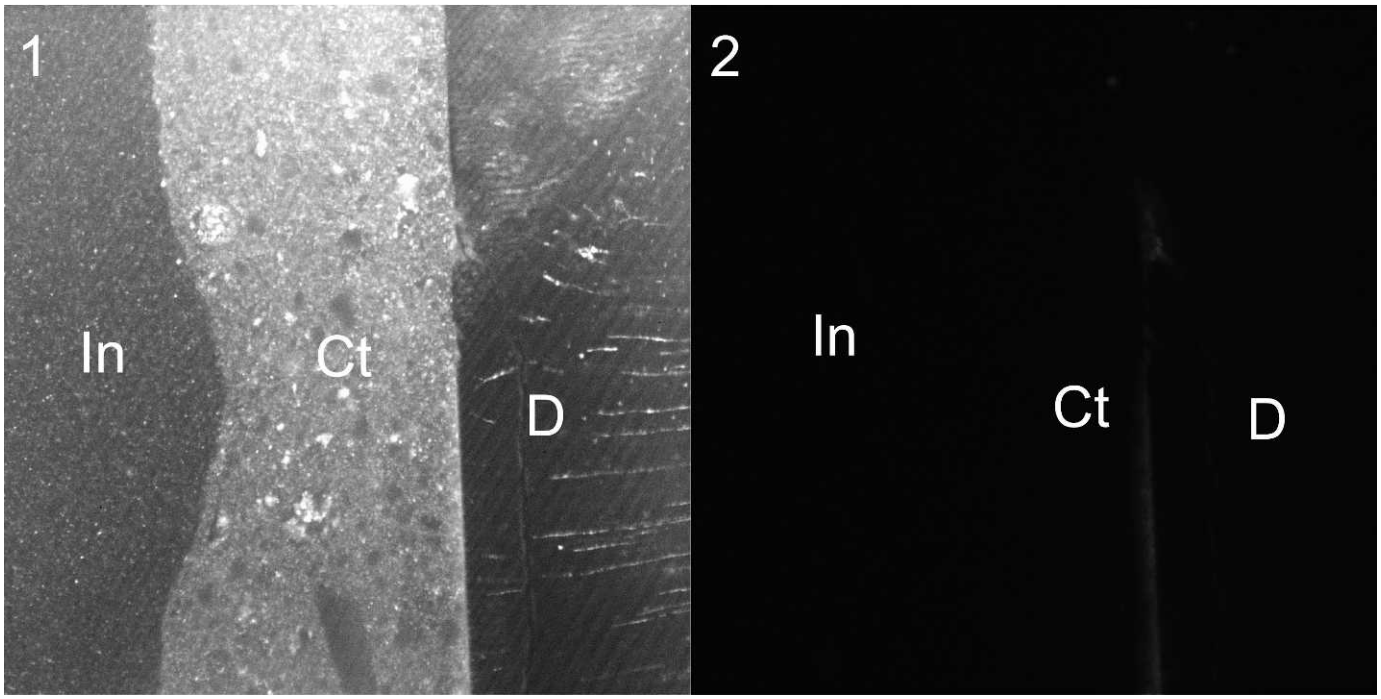

Figure 4. *Representative confocal scanning micrographs of fluorescence evaluation of a loaded, etched Panavia F 2.0 axial wall specimen. (1): Reflection (546-nm illumination). (2): Fluorescence (546/600-nm excitation/emission) images of an area of axial wall. In, inlay; Ct, resin cement; D, dentin 20/0.80× oil immersion objective.*

self-etching resin cement that acted as the control material. After mechanical loading of the restorations, both the self-etching and the self-adhesive resin cements exhibited a significant reduction in bond strength; however, the three tested cements had equivocal bond strengths and exhibited a similar mode of failure. This result indicates that using a self-adhesive cement to cement an indirect composite inlay would offer comparable bond strength and durability to a self-etching resin cement.

To ensure that the cavities were prepared into dentin of a standardized depth and therefore similar morphology, only caries-free upper third molars that were of a similar size were selected for the study. For the microleakage evaluation, dye penetration was measured at both axial walls of the proximal boxes of the cavities and on the gingival floor. Moreover, the enamel margins of the proximal boxes of half the prepared cavities in each group were etched with phosphoric acid prior to application of the cement. To date, there has been no previously published data on the microleakage of MOD indirect resin composite inlays cemented in molar teeth with self-etching or self-adhesive cements. In the present experiment, it was found that acid etching of the enamel margins of the proximal boxes had no statistical effect on dye penetration in the cements. All the resin cements exhibited an increase in dye penetration after loading, with Panavia F 2.0 and SA Cement exhibiting a significant increase at the axial walls. On the cavity floors, all the resin cements exhibited increased dye penetration after loading, but the increases were not significant. However, there was no difference in dye penetration between the three cements after loading both at the axial walls and on the cavity floors. Moreover, dye penetration, when present, was observed in all the specimens at the resin cement–dentin interface and not at the inlay–resin cement interface. Previous research has investigated the nanoleakage patterns of Panavia F 2.0 and Rely X Unicem in which indirect resin composite blocks of Estenia C & B were bonded to flat occlusal dentin surfaces.[2] It was found that Panavia F 2.0 exhibited a significantly greater silver particle penetration than Rely X Unicem.[2] The authors attributed this difference to the cements' different mechanisms of adhesion to dentin. They reported that in the case of Panavia F 2.0, incomplete penetration of resin monomers into the demineralized dentin surface may result in the formation of nanospaces. On the other hand, the adhesion of Rely X Unicem to dentin occurs through the interaction of ionised phosphoric acid-methacrylate with dentin.[2] An increased chemical action with the calcium from hydroxyapatite has been reported.[17] The other self-

adhesive cement investigated in the present study, SA Cement, is a dual-cured resin cement that contains the adhesive phosphate monomer MDP but does not require the prior application of a self-etching primer. These findings indicate that in a bonded indirect composite restoration, the interface at which failure is most likely to occur is the resin cement–dentin interface and that strengthening of this interface is necessary to improve its resistance to mechanical loading. Observation of the failure modes in the present experiment support this supposition in that almost all the specimens failed at the resin-dentin interface.

The present experiment employed the microtensile bond strength test to evaluate the resin cement–dentin bond strengths. The advantages and disadvantages of the microtensile bond strength test have been comprehensively discussed in several recent review articles.[18-20] It has been reported that the microtensile bond strength test enables multiple specimens to be obtained from one tooth, reducing the number of teeth required to around five, and facilitates improved stress distribution at the resin-dentin interface as well as good control of potential regional differences in tooth structure.[18,19] On the other hand, it has been pointed out that it is difficult to measure low bond strengths, and this can result in pretest failures and specimens dehydrating rapidly.[18-20] In the present experiment, resin cement–dentin bond strengths were measured at the base of the cavities in the occlusal region following removal of approximal slices for evaluation of microleakage. The sectioned beams were kept moist prior to being bonded to a specially designed jig that ensured correct alignment of the beams and standardization within the test setup.

In our previous experiment, resin coating of the dentin surface with a dentin bonding agent and flowable composite improved the microleakage of CAD/CAM Cerec inlays cemented with resin cement.[11] It has also been reported that resin coating of the dentin improved the bond strength of MO inlays fabricated from indirect composite resin.[21] Therefore, further research is needed on whether resin coating the dentin substrate might improve the bond strength and reduce the microleakage of indirect resin composite restorations bonded with self-adhesive resin cements. Moreover, further *in vitro* research is needed on indirect resin restorations that are bonded and tested in cavities under conditions that more closely reproduce the clinical environment.

CONCLUSIONS

It can be concluded that the two tested self-adhesive cements exhibited similar bond strengths before and after loading to the self-etching resin cement. After loading, resin cement–dentin bond strengths were significantly reduced. Dye penetration was observed at the resin cement–dentin interfaces of the cemented inlays but not at the resin-cement-inlay interface. Increases in dye penetration were observed in all three cements after loading, and the increases were significant for Panavia F 2.0 and SA Cement at the axial walls. However, after loading, there were no significant differences between the three cements. Within the limitations of the present experiment, prior acid etching of enamel margins of the prepared cavities had no significant effect on microleakage in the approximal regions of the bonded inlays.

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Shear Bond Strength of Composite to Deep Dentin After Treatment With Two Different Collagen Cross-linking Agents at Varying Time Intervals

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S Mahalaxmi

Clinical Relevance

Deep dentin does not serve as a suitable bonding substrate for contemporary bonding agents. The use of natural collagen cross-linkers like sodium ascorbate and proanthocyanidin as dentin pretreatment agents greatly improves bond strength to deep dentin.

SUMMARY

Objective: This in vitro study evaluated the shear bond strength of composite resin to deep dentin using a total etch adhesive after treatment with two collagen cross-linking agents at varying time intervals.

Materials and Methods: Thirty freshly extracted human maxillary central incisors were sectioned longitudinally into equal mesial and distal halves (n=60). The proximal deep dentin was exposed, maintaining a remaining dentin thickness (RDT) of approximately 1 mm. The specimens were randomly divided into three groups based on the surface treatment of dentin prior to bonding as follows: group I (n=12, control): no prior dentin surface treatment; group II (n=24): dentin surface pretreated with 10% sodium ascorbate; and group III (n=24): dentin surface pretreated with 6.5% proanthocyanidin. Groups II and III were further subdivided into two subgroups of 12 specimens each, based on the pretreatment time of five minutes (subgroup A) and 10 minutes (subgroup B). Shear bond strength of the specimens was tested with a

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universal testing machine, and the data were statistically analyzed.

Results: Significantly higher shear bond strength to deep dentin was observed in teeth treated with 10% sodium ascorbate (group II) and 6.5% proanthocyanidin (group III) compared to the control group (group I). Among the collagen cross-linkers used, specimens treated with proanthocyanidin showed significantly higher shear bond strength values than those treated with sodium ascorbate. No significant difference was observed between the five-minute and 10-minute pretreatment times in groups II and III.

Conclusion: It can be concluded that dentin surface pretreatment with both 10% sodium ascorbate and 6.5% proanthocyanidin resulted in significant improvement in bond strength of resin composite to deep dentin.

INTRODUCTION

Continuous developments in dentin bonding systems have led to their widespread use in daily dental practice.[1] Total etch adhesive systems, which remove the smear layer with phosphoric acid and combine the function of primer and adhesive in one bottle, have been widely used because of their documented long-term clinical success.[2,3] Although contemporary dentin bonding systems show improvement in better handling and bonding characteristics, the achievement of a strong and stable bond to deep dentin remains a challenge in restorative dentistry.

Dentin is a complex hydrated biological composite material with structural components and properties that vary with location.[4] Various studies have shown that a reduction in bond strength occurs when resin composite is bonded to deep dentin. This significant influence of dentin depth on the bond strength of dentin bonding systems can be attributed to the complexities in the structure of deep dentin, such as an increase in the number of tubules and their diameters with much less intertubular dentin matrix, as compared to superficial dentin.[5-7] In addition, the higher water content in deep dentin (22 volume % as compared to 1 volume % in superficial dentin) may dilute the organic solvents of some bonding systems, resulting in separation of monomers from the soluble phase and leading to the formation of resin globules in water.[8]

Successful bonding to dentin depends largely on proper resin infiltration of the demineralized microporous collagen fibril scaffold (hybrid layer) and the formation of resin tags.[9,10] Since the durability of the bond between dentin and adhesive system depends largely on the structural integrity and mechanical properties of acid-demineralized collagen fibers, any attempt to stabilize collagen will help enhance the bonding.[11] Mechanical properties of collagen can be improved by an increase in the formation of intra- and intermolecular and intermicrofibrillar cross-links. This can be achieved by the use of various collagen cross-linkers, both synthetic and natural, on the dentin substrate prior to the bonding procedure.[12,13] Naturally occurring collagen cross-linkers such as sodium ascorbate and proanthocyanidin have been reported to increase the collagen cross-linking in sound and caries-affected dentin,[12-14] but their effects on bonding to deep dentin have not been reported in the literature. Hence, the aim of this *in vitro* study was to determine the shear bond strength of composite resin to deep dentin using a total etch adhesive after treatment with different collagen cross-linking agents at varying time intervals.

MATERIALS AND METHODS

The materials used in this study and their composition are given in Table 1.

Preparation of Solutions

Two solutions were prepared for this study: 1) 10 g of sodium ascorbate powder (sd fiNE cHEM Ltd, Mumbai, India) were dissolved in 100 mL of distilled water to make 10% sodium ascorbate solution, and 2) 6.5 g of grape seed extract in the form of powder (Puritans Pride Inc, Oakdale, NY, USA) were collected from the capsules and dissolved in 100 mL of distilled water to make 6.5% proanthocyanidin solution.

Specimen Preparation

Thirty freshly extracted human maxillary central incisors were collected for the study, adhering to the protocol of the Institutional Review Board of SRM University (SRMU/M&HS/SRMDC/2010/M.D.S-PG Student/114). The teeth were cleaned of debris and stored in 0.2% thymol until use. Each tooth was sectioned longitudinally, parallel to the long axis of the tooth, into an equal mesial and a distal half by means of a low-speed diamond disc (HI-DI Diamond Precision Tools Ltd, London, UK) under copious water supply (Figure 1a,b). The contents of the pulp chamber were then removed ultrasonically to know its extent.

Table 1: *Materials Used in This Study and Their Composition*

Study	Materials	Composition
1	Sodium ascorbate (sd fiNE cHEM Ltd, Mumbai, India)	100-g powder containing 99.1% sodium ascorbate
2	Proanthocyanidin Grape Seed Extract (Puritans Pride Inc, Oakdale, NY, USA)	100-mg capsule containing 97.3% proanthocyanidin
3	Total Etch Adhesive System (Dentsply DeTrey GmbH, Konstanz, Germany)	
	Etchant: DeTrey Conditioner 36	36% ortho-phosphoric acid
	Adhesive: Prime & Bond NT	Dipentaerythritol penta acrylate monophosphate (PENTA), di- and tri- methacrylate resins, amorphous silicon dioxide nanofillers, photoinitiators, stabilizers, cetylamine hydrofluoride, acetone
4	Composite resin: Ceram X Nano Ceramic Restorative (Dentsply DeTrey GmbH)	Methacrylate modified polysiloxane, dimethacrylate resin, pigments, stabilizers, camphoroquinone, ethyl-4 (dimethylamino) benzoate, barium-aluminum-borosilicate glass, methacrylate functionalized silicon dioxide nanofiller

Using a diamond disc, the dentin in the proximal wall of each half of the crown incisal to the cemento-enamel junction was removed until the remaining dentin thickness (RDT) was approximately 1 mm (Figure 1c) as measured with a metal caliper (Iwanson Spring metal caliper, I.D-Tech, Sialkot, Pakistan) from the outer surface of the prepared proximal portion of crown to the pulp chamber. All the specimens were immersed in an ultrasonic bath to remove the smear layer. The roots of the specimens were then mounted in self-cure acrylic resin. The number of specimens thus obtained was 60. The prepared proximal surfaces of the specimens were acid-etched with 36% ortho-phosphoric acid (DeTrey Conditioner 36, Dentsply DeTrey GmbH, Konstanz, Germany) for 15 seconds, rinsed with water for 15 seconds, and blot dried. These specimens were randomly divided into three groups based on the surface treatment of dentin as follows:

Group I (n=12, control): No dentin pretreatment was done. Adhesive bonding and composite buildup was done according to the bonding protocol as described here. Two successive coats of the adhesive Prime & Bond NT (Dentsply DeTrey GmbH) were applied on the prepared proximal dentin surface of the specimens according to the manufacturer's instructions, and light curing was done (Astralis 3 light curing unit (530 mW/cm^2), Ivoclar Vivadent, Schaan, Liechtenstein) for 40 seconds. Composite buildup was done (Figure 1d) by placement of two increments of 2-mm-thick composite resin (Ceram X Nano Ceramic Restorative, Dentsply DeTrey GmbH) using a 3-mm-diameter plastic tube as a matrix, with each increment being light cured for 40 seconds.

Group II (n=24): 10% sodium ascorbate pretreatment. This group was further subdivided into two subgroups based on the pretreatment time as follows:

Subgroup IIA (n=12): The etched dentin surface was treated with 10% sodium ascorbate solution for five minutes and rinsed with water, followed by the bonding/buildup procedure as described above.

Subgroup IIB (n=12): The etched dentin surface was treated with 10% sodium ascorbate solution for 10 minutes and rinsed with water. Adhesive bonding and composite buildup followed as described above.

Group III (n=24): 6.5% proanthocyanidin pretreatment. This group was further subdivided into two subgroups based on the pretreatment time as follows:

Subgroup IIIA (n=12): The etched dentin surface was treated with 6.5% proanthocyanidin solu-

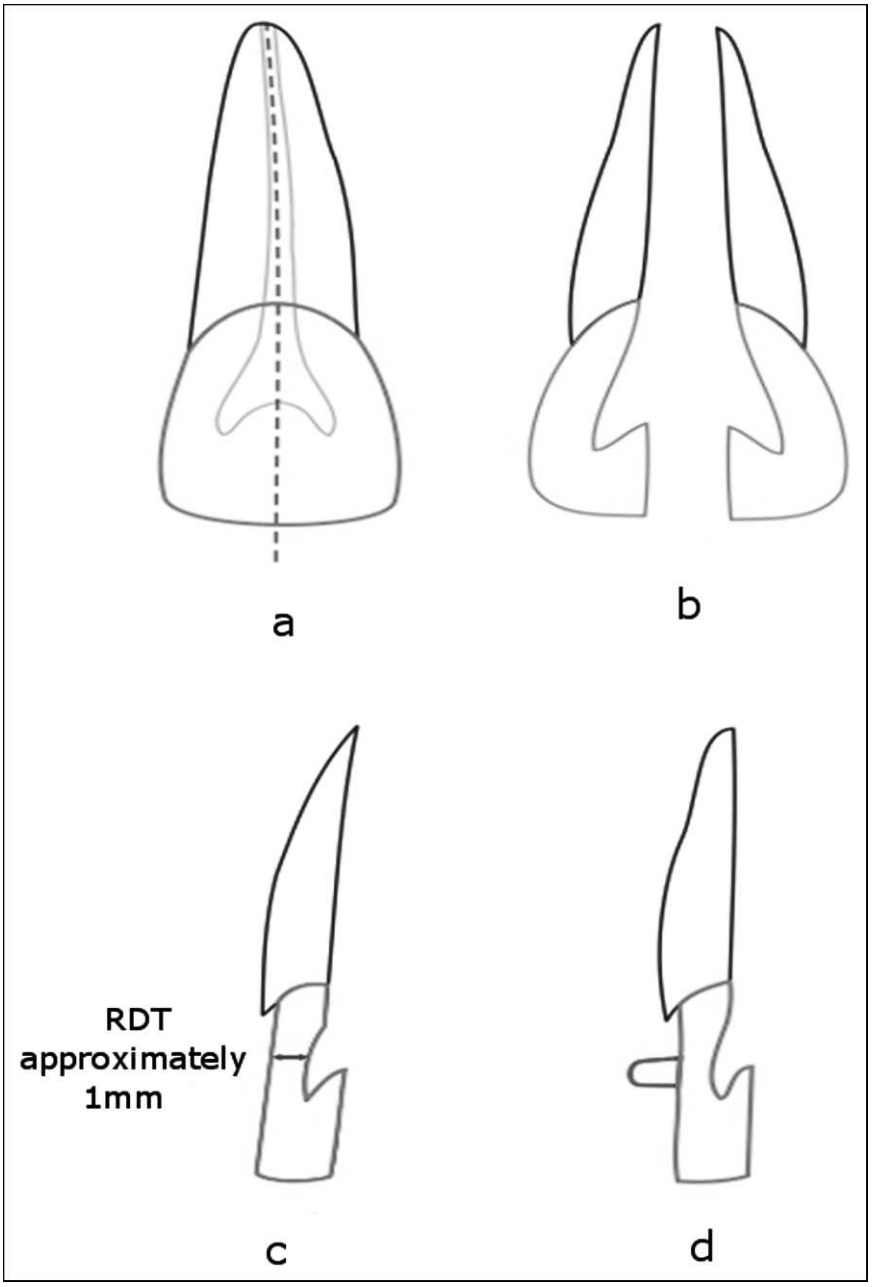

Figure 1. *Specimen preparation for shear bond strength testing. (a and b): Sectioning the tooth labio-palatally to obtain an equal mesial and a distal half. (c): The dentin in the proximal wall of each half of the crown incisal to the cemento-enamel junction was removed until the remaining dentin thickness was approximately 1 mm. (d): Composite resin buildup.*

tion for five minutes and rinsed with water, followed by the bonding/buildup procedure as described above.

Subgroup IIIB (n=12): The etched dentin surface was treated with 6.5% proanthocyanidin solution for 10 minutes and rinsed with water. Adhesive bonding and composite buildup followed as described above.

Shear Bond Strength Testing

All the specimens were then stored in distilled water at 37°C for 24 hours. Shear bond strength was determined using a universal testing machine (LR 100K, Lloyd Instruments, Largo, Florida, USA) at a crosshead speed of 1 mm/min. The results were tabulated and statistically analyzed. Paired *t*-test was used to calculate the *p*-value ($p<0.001$).

Table 2: *Comparison of Mean Shear Bond Strength of Different Study Groups*

Group	Mean ± SD (Mpa)	Significant groups ($p<0.001$)
I	17.84 ± 0.56[a]	IIIA, IIIB, IIA, IIB vs. I
IIA	22.12 ± 0.90[b]	IIIA vs. IIA
IIB	23.05 ± 0.60[b]	
IIIA	27.57 ± 0.92[c]	IIIB vs. IIB
IIIB	27.85 ± 1.01[c]	

[a] *For each column, same superscript letters indicate no statistically significant difference (p>0.05) between the groups and different superscript letters indicate a statistically significant difference (p<0.001) between the groups.*

RESULTS

The results of this study are shown in Table 2. The results showed that the mean shear bond strength values of subgroup IIA (22.12 ± 0.90), IIB (23.05 ± 0.60), IIIA (27.57 ± 0.92), and IIIB (27.85 ± 1.01) were significantly higher than the mean shear bond strength value in the control group (17.84 ± 0.56; $p<0.001$). Both five-minute and 10-minute pretreatment times of 6.5% proanthocyanidin (IIIA [27.57 ± 0.92] and IIIB [27.85 ± 1.01]) yielded significantly higher shear bond strength values than the corresponding pretreatment times of 10% sodium ascorbate (IIA [22.12 ± 0.90] and IIB [23.05 ± 0.60]; $p<0.001$). No significant difference in shear bond strength values was found between the two pretreatment times in both the 10% sodium ascorbate and the 6.5% proanthocyanidin groups ($p>0.05$).

DISCUSSION

The structure and properties of dentin are the principal determinants of many procedures in restorative dentistry.[15] Adhesive bonding to dentin will be most effective only if the hybrid layer that is formed between resin monomers and collagen fibrils is structurally stable.[14] Dentin contains micrometer-diameter tubules surrounded by highly mineralized peritubular dentin embedded within a partially mineralized intertubular dentin.[4,16] The collagen-rich intertubular matrix area decreases from about 96% at the dentino-enamel junction to nearly 12% at the predentin. The amount of collagen fibrils per unit volume of dentin decreases from superficial to deep dentin. Studies have shown that large dentinal tubules with much less collagen-rich intertubular dentin matrix make bonding to deep dentin unpredictable.[4,7,17]

Fibrillar type I collagen accounts for 90% of the organic matrix of dentin, while the remaining 10% consists of noncollagenous proteins, such as phosphoproteins and proteoglycans.[12,18] Type I collagen serves as a scaffold for the deposition of apatite mineral phase and provides viscoelasticity to the tissue by forming a rigid, strong, space-filling biomaterial.[15] Structural integrity and mechanical properties of the collagen fibrils of acid demineralized superficial and deep dentin play an important role in the determination of bond strength and its durability.[11] Covalent inter- and intramolecular cross-links are the basis for stability, tensile strength, and viscoelasticity of the collagen fibrils. Various chemicals, both synthetic and natural, have the ability to increase these collagen cross-links.[12,15,19]

Sodium ascorbate is an important component in the synthesis of hydroxyproline and hydroxylysine in collagen. Hydroxyproline serves to stabilize the collagen triple helix, and hydroxylysine is necessary for the formation of intermolecular cross-links in collagen.[20]

Proanthocyanidins (PA) are naturally occurring bioflavonoids found in high concentrations in grape seed, pine bark, cranberries, lemon tree bark, and hazel nut tree leaves.[21] Although PA from grape seed extract has been shown to effectively cross-link collagen in *in vitro* studies,[14,19,22,23] their effect on the bond strength of resin composites to deep dentin remains an unexplored area in restorative dentistry.

A novel method of sample preparation was adopted in the current study. Maxillary central incisors were chosen because of greater cervico-incisal length of the crown (10.5 mm).[24] In addition, the pulp chamber is located in the center of the crown, equidistant from the dentinal walls.[25] Since the pulp chamber is broad mesiodistally, sectioning was done labiopalatally to achieve two equal halves. When observing the cross section of maxillary central incisors, mesial and distal outlines of the pulp chamber were nearly straight. Thus, more uniform RDT is available for standardization of deep dentin. Radiographic confirmation of RDT was not required in this study, as it was directly measured from the pulp chamber to the proximal surface of tooth.

In the present study, group I (control) recorded a mean shear bond strength value of 17.84 ± 0.56

MPa to deep dentin, which is less than the optimal bond strength of resin composite to superficial dentin (20–23 MPa).[26,27] This is in accordance with previous studies done by Suzuki and others,[28] Olsson and others,[29] and Yazici and others,[30] who showed that bond strength of resin composite to deep dentin can be as low as 10.3-16.7 MPa.

Results of this *in vitro* study showed an increase in shear bond strength after pretreatment with 10% sodium ascorbate and 6.5% proanthocyanidin as compared to control group. This can be attributed to improved dentin collagen stability, obtained from an increase in the number of collagen cross-links, achieved by the use of these collagen cross-linkers.

Group III (6.5% proanthocyanidin) showed a significantly higher bond strength to deep dentin compared to group II (10% sodium ascorbate) in both the subgroups ($p<0.001$). This is in accordance with the findings of Macedo and others,[14] who showed that the application of 6.5% grape seed extract and 5% gluteraldehyde to caries-affected and sound dentin significantly improved the microtensile bond strength of composite to dentin. Similarly, Walter and others[31] showed that 0.5% proanthocyanidin efficiently stabilized collagen and increased its resistance to caries compared to 0.625% genipin and 5% gluteraldehyde. Bedran-Russo and others[12] found that naturally occurring cross-linking agents such as 0.5% proanthocyanidin and 0.625% genipin are capable of stabilizing demineralized dentin collagen more effectively compared to 5% gluteraldehyde.

This could be attributed to the specificity of proanthocyanidin to facilitate the enzyme proline hydroxylase, which catalyzes the hydroxylation of proline, an essential step in collagen biosynthesis. Proanthocyanidins and proteins have been shown to interact in four different ways: 1) covalent interactions, 2) ionic interactions, 3) hydrogen bonding interactions, or 4) hydrophobic interactions.[14,22,23] Castellan and others[19] showed that when demineralized dentin is treated with PA, it results in improved mechanical properties and reduced water absorption as a result of the dense collagen network formed by the use of exogenous cross-linkers.

Macedo and others[14] showed that treatment of dentin surface with 6.5% proanthocyanidin for 1 hour resulted in an increase in the microtensile bond strength of the specimens. Hence, in this study, in order to establish more clinically relevant and feasible application times, lesser application times of five and 10 minutes were chosen. The present study showed no statistical significant difference in the shear bond strength values between the two pretreatment times in both 10% sodium ascorbate and 6.5% PA groups.

In the present study, when deep dentin was treated with sodium ascorbate, the bond strength reached values (22.12–23.05 MPa), comparable with the optimal bond strength values of superficial dentin. But after dentin surface treatment with proanthocyanidin, the deep dentin bond strength significantly increased to levels (27.57-27.85 MPa) that are higher than the optimal bond strength values of superficial dentin. Hence, it can be safely recommended that the use of collagen cross-linkers be employed as an effective chairside procedure to overcome the disadvantage of reduced bond strength of composite resin to deep dentin. Although this research allowed for an effective evaluation of the potential effect of these cross-linkers on deep dentin, the time used is still excessive from a clinical point of view. Hence, other application times in the range of 30–60 seconds should be taken into consideration in future experiments to develop a clinically relevant strategy for improving deep dentin bond strength.

CONCLUSIONS

Under the limitations of this *in vitro* study, the following conclusions can be drawn:

1. Dentin surface pretreatment with both 10% sodium ascorbate and 6.5% proanthocyanidin resulted in significant improvement in the bond strength of resin composite to deep dentin compared to the control group.
2. The use of 6.5% proanthocyanidin as a collagen cross-linker on deep dentin significantly improved the shear bond strength values compared to the use of 10% sodium ascorbate.
3. There was no significant difference in the shear bond strength values between the five-minute and 10-minute pretreatment times in both the 10% sodium ascorbate and the 6.5% proanthocyanidin groups.

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Effect of Composite Resin Contamination with Powdered and Unpowdered Latex Gloves on Its Shear Bond Strength to Bovine Dentin

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AA Ajami • PA Oskoee • NM Abbasi

Clinical Relevance

In order to avoid the negative effect of contamination of composite resins with powdered latex gloves on the bond strength of two-step self-etching adhesive systems, the use of unpowdered latex gloves is recommended with these adhesive systems.

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SUMMARY

The aim of the present study was to evaluate the effect of composite resin contamination with powdered and unpowdered latex gloves on the shear bond strength of etch-and-rinse and two-step self-etch adhesive systems. Standard flat dentin surfaces were prepared on the facial aspect of 120 bovine incisors and randomly assigned into two (n=60) groups: group 1: Single Bond (SB), group 2: Clearfil SE Bond (CSE). Furthermore, each group was randomly subdivided into three (n=20) based on the type of composite contamination (without contamination, contamination with powdered latex gloves, and contamination with unpowdered latex gloves). The adhesives were applied and resin composite bonded to the dentin. After thermocycling, the specimens were subjected to a shear bond strength test. Two-way analysis of variance (ANOVA) and a post hoc Bonferroni test were used for statistical analysis. One-way ANOVA was used to compare shear bond strength values in each group. Statistical

significance was set at $p < 0.02$. Two-way ANOVA showed that the shear bond strength was significantly influenced by the type of composite surface contamination ($p=0.001$). In the SB group there were no significant differences between different surface treatments ($p=0.08$). In the CSE group a significant difference was observed between the subgroup without contamination and the subgroup with powdered latex glove contamination ($p=0.01$); however, no significant differences were observed between the other subgroups.

INTRODUCTION

During the past decade adhesive dentistry has witnessed major advances. Restorative techniques, such as the restoration of various surfaces with composite resins, bonded fixed prostheses, and porcelain laminate veneers have now gained widespread acceptance. Various intraoral and extraoral factors can decrease adhesion properties. Although technique sensitivity has decreased in some instances, adhesive dentistry still requires a clean substrate for proper bonding.[1-4] The adhesion site might be contaminated by saliva, blood, or dental gloves during the procedure, resulting in defective bonding. Improper bonding leads to margin discoloration, recurrent caries, postoperative hypersensitivity, and pulpal irritation.[4-10]

At present, infection control during dental procedures is a major concern. Facial masks, gowns, gloves, and appropriate glasses have been suggested by infection control centers and authorities for infection control.[11] The majority of commercial gloves available are made of latex and are powdered with cornstarch to facilitate wearing and removal of the gloves.[12-14] Unfortunately, some cases of starch side effects during medical and dental procedures have been reported, which include contamination of wounds and development of granulomatous lesions subsequent to surgery.[15-19]

Starch powder particles, which contain latex proteins, can spread by air during wearing or removal of gloves and contaminate the surface of dental equipment, instruments, materials, and the surgical field.[20] The detrimental effects of powdered gloves in clinical dentistry have only been briefly discussed in dental literature.[21,22] Several researchers have reported delay in the setting reaction of polyvinyl siloxane impression materials as a result of contact with these gloves. Delay in the setting reaction might result from the starch itself or the sulfur-containing accelerator particles present in the latex that have penetrated into the starch.[23-27] It has also been reported that contamination of radiographic films with these gloves during processing of the films has a deleterious effect on image quality.[28]

Since the introduction of latex gloves some questions have been raised concerning the effect of this material on resin bonds for restorative procedures, with no consensus in this regard.[29] In some studies the contamination of the bonding surface with gloves has significantly decreased bond strength[29]; however, another study has not been able to demonstrate any significant effect on bond strength.[20] All previous studies on this topic have been carried out with the use of etch-and-rinse adhesive systems.[20,29] Now self-etch adhesive systems have been introduced, which have incorporated etching and priming steps into a single step and have decreased chair time through elimination of the rinsing step with a concomitant decrease in technique sensitivity.[30] In addition, the effect of contamination of bonding surface with latex gloves has been evaluated,[20,29] but the effect of composite resin surface contamination has not been studied.

Therefore, the aim of the present study was to evaluate the effect of contamination of a composite resin surface with powdered and unpowdered latex gloves on shear bond strength of composite resin to bovine tooth dentin with the use of etch-and-rinse and self-etch adhesive systems.

METHODS AND MATERIALS

A total of 120 bovine incisors were used in the present study. The teeth were collected from Tabriz Industrial Slaughterhouse after approval was granted by the Ethics Committee of the Research Deputy of Tabriz University of Medical Sciences. After the removal of any remaining tissues tags from tooth surfaces, the teeth were cleaned with pumice and brush in a low-speed handpiece under constant water spray. Then the teeth were stored in distilled water at 4°C until used for the purpose of the study.

In all the specimens the roots were cut away with a diamond saw in a straight handpiece. Then the facial surfaces of the teeth were abraded with a diamond bur under air/water spray until the superficial layer of dentin was exposed; the dentin surfaces were smoothed with 400-grit and 600-grit abrasive paper to achieve a standard smooth surface. The flat dentin surface was buried inside a plastic syringe filled with self-curing acrylic resin in a manner in which the prepared surface was perpen-

dicular to the horizontal line; the syringe had an inner diameter of 8 mm and had been filled with acrylic resin to a height of 2.5 cm. A total of 120 plastic molds, with a diameter of 3 mm and a height of 2 mm, were prepared for bonding the restorative composite resin to the surface of the samples.

Then the samples were prepared in two groups, as follows, based on the type of the adhesive used.

Group 1: Etch-and-Rinse Adhesive System

In this group, Single Bond (SB) adhesive system (3M ESPE, St Paul, MN, USA) was used. The surface of the samples was etched with 35 wt% of phosphoric acid gel (Scotch Bond Etchant, 3M ESPE) for 15 seconds and rinsed for 15 seconds with water spray; then the surfaces were dried, but not desiccated, for one to two seconds. The SB adhesive was applied to the surfaces in two consecutive layers according to the manufacturer's instructions.

Next, the adhesive was dried with a gentle current of air for two to five seconds and light-cured for 10 seconds at a light intensity of 400 mW/cm^2 at a distance of 1 mm from the surface using the Astralis 7 light-curing unit (Ivoclar Vivadent GmbH, Bremschlstr, Austria). The light intensity of the light-curing unit was checked before the procedure using a Coltolux light meter (Coltene/Whaledent Inc, Cuyahoga Falls, Ohio, USA).

Group 2: Two-Step Self-Etching Adhesive System

In this group, the Clearfil SE Bond (CSE) adhesive system (Kuraray, Okayama, Japan) was used. The surface of the samples was primed for 20 seconds with CSE primer. After application of the bonding, the Astralis 7 light-curing unit was used for the curing process at a light intensity of 400 mW/ cm^2 at a distance of 1 mm.

In the next stage, the samples in each group were randomly subdivided into three groups of 20 based on the surface contamination used:

- Subgroup 1 (control; without contamination): The plastic mold, with a diameter of 3 mm and a height of 2 mm, was placed on the glass slab, filled with composite resin (Filtek Z 250, 3M ESPE), and then placed on the bonding surface. A dental explorer was used to remove excess material. The mold was cured for 40 seconds at a light intensity of 400 mW/ cm^2. The plastic mold was cut away with a no. 11 scalpel blade.
- Subgroup 2 (contamination with powdered latex gloves): In this group, composite resin was packed into the plastic mold on the glass slab. Then its surface was contaminated for two minutes with the outer surface of powdered latex gloves (Supermax Glove Manufacturing, Selangor, Malaysia) in a dark room to prevent composite resin polymerization. The contaminated surface of composite resin was bonded to the tooth structure. One new glove piece was used for each specimen. The remaining steps were similar to those described for subgroup 1.
- Subgroup 3 (contamination with unpowdered latex gloves): All the steps were similar to those described for subgroup 2 except for the fact that the surface of composite resin was contaminated with unpowdered latex gloves (Supermax Glove Manufacturing).

All the specimens were stored in distilled water at 37°C in an incubator until the shear bond strength test. Table 1 summarizes the composition of the materials used in the present study.

Once all the samples were ready, they underwent a thermocycling procedure consisting of 500 cycles between 55°C ± 2°C and 5°C ± 2°C with a dwell time of 30 seconds and a transfer time of 15 seconds to closely simulate the oral cavity conditions before the shear bond strength test.

Finally, all the samples underwent a shearing force with a knife-edge crosshead at 0.5-cm^2 surface area in a universal testing machine (H5K-S Model, Hounsfield Test Equipment, Surrey, England); the strain rate was 0.5 mm/min. The force was applied at the composite cylinder-tooth interface. The maximum force at failure was recorded in newtons, then converted to megapascals by subdividing it to bonding surface area.

The specimens were evaluated under a stereomicroscope (SMZ800, Nikon, Tokyo, Japan) at 20× for failure mode. Failure modes were classified as cohesive (failure inside the composite resin), adhesive (at the interface of tooth-composite resin), and mixed failure (when more than 25% of the failure was adhesive[31]) (Figure 1).

To observe the surface contaminations on the surface of various latex gloves and also the clean and contaminated composite resin surfaces, two samples measuring 1×1 cm from each type of latex gloves and two samples from each type of clean and contaminated composite resins with powdered and unpowdered latex gloves were prepared; these samples were similar in size to the cylinders used

Table 1: *The Materials Used in the Present Study*

Batch Numbers	Manufacturer	Description & Composition	Material
N202942	3M ESPE, St Paul, MN, USA	35 wt% phosphoric acid gel	Scotch Bond Etchant
N202333	3M ESPE, St Paul, MN, USA	Etch-and-rinse adhesive system contains 2-HEMA, Bis-GMA, dimethacrylates, amines, methacrylate functional copolymer of polyacrylic and polyitaconic acid, ethanol, water	Single Bond
Primer: 01027A, Bond: 01531A	Kuraray, Okayama, Japan	Self-etching adhesive system contains 10-MDP, HEMA, camphorquinone, N,N-diethanol-P-toluidine, Bis-GMA, silanated colloidal silica	Clearfil SE Bond
N142256	3M ESPE, St Paul, MN, USA	Visible-light activated, radiopaque, restorative composite; filler is zirconia/silica; filler loading is 60% by volume (without silane treatment), with a particle size range of 0.01 to 3.5 μm; contains Bis-GMA, UDMA, and Bis-EMA resins	Filtek Z250
30235616	Supermax Glove Manufacturing, Selangor, Malaysia	Natural rubber latex + cornstarch	Supermax powdered glove
S3322679	Supermax Glove Manufacturing, Selangor, Malaysia	Natural rubber latex	Supermax unpowdered glove

Abbreviations: Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate; Bis-GMA, bisphenol A diglycidyl ether dimethacryalate; HEMA, hydrosyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; UDMA, urethane dimethacrylate.

for bonding. The prepared samples were gold-sputtered and evaluated under a scanning electron microscope (SEM; Vega-II, Tescan sro, Libusinia Trida, Czech Republic) at 1000×.

Subsequent to evaluating the normal distribution of data with the Kolmogorov-Smirnov test and the equality of variances between the groups with the Levene test, two-way analysis of variance (ANOVA) was used for statistical analysis. A post hoc Bonferroni test was used for the two-by-two comparison of the groups. In addition, the statistical significance of differences in bond strength values between the subgroups of each adhesive system was evaluated by one-way ANOVA and a post hoc Bonferroni test. Statistical significance was set at $p < 0.02$.

RESULTS

Table 2 separately shows the descriptive statistics (bond strength values in megapascals) and failure modes for each group and its subgroups. Two-way ANOVA showed that differences in the mean bond strength values based on the type of the adhesive system were not statistically significant ($p=0.42$). However, the shear bond strength values were significantly under the influence of surface contamination ($p=0.001$). The interaction between the variables of adhesive system type and the type of surface contamination was not statistically significant ($p=0.75$) either. Two-by-two comparison of surface contaminations with a post hoc Bonferroni test demonstrated statistically significant differences between noncontaminated groups and the groups contaminated with powdered latex gloves ($p=0.02$). However, the differences between different surface contaminations were not significant ($p>0.02$).

In comparison of the subgroups based on different surface contaminations in each adhesive group, one-way ANOVA showed that in the SB group there were no significant differences between the subgroups ($p=0.08$). In the CSE group, there were significant differences between the noncontaminated subgroup and the subgroup contaminated with powdered latex gloves ($p=0.01$); however, the differences between other subgroups were not significant ($p=0.13$).

All the failure modes listed in Table 2 were observed in the composite resin structure, and no cohesive failures were observed in tooth structures. Furthermore, in the CSE adhesive system subgroup

contaminated with powdered gloves, cohesive failures were more numerous than the mixed failures.

SEM images of surface contaminations on each type of gloves and composite resin surface are shown in Figure 2.

DISCUSSION

Coincident with the previously noted increase in use of gloves by dental health care workers has been the relatively rapid development of adhesive dentistry. Inadvertent contamination of the surface of composite resin with latex gloves during restorative procedures is a common problem in operative dentistry, which might have a detrimental effect on the shear bond strength of adhesive bonds to dentin.[10,29]

In the present study, the effect of contamination of composite resin with powdered and unpowdered latex gloves on the shear bond strength of SB and CSE adhesives was investigated.

The results of the present study showed that in the SB group no significant differences were observed between the subgroups; however, in the CSE group significant differences were observed between the noncontaminated subgroup and the subgroup contaminated with powdered latex gloves.

In a study carried out by Sanders and others[29] with the etch-and-rinse system ProBond no significant differences were observed in composite bond strength to enamel between groups contaminated with powdered latex gloves and groups contaminated with unpowdered latex gloves, which is consistent with the results of the present study in the SB group. In that study[29] the adhesive system itself had been directly contaminated and enamel surface bonding had been evaluated, but in the present study the surface of composite resin was contaminated after curing the adhesive system. In addition, the results of a study by Roberts and Bartoloni[20] showed that the bond strength of dentin surfaces contaminated with powdered and unpowdered latex gloves, whether before etching or after the application of etch-and-rinse system Excite and before placement of composite resin, had no significant differences with those of the control group (not contaminated with latex gloves). During evaluation with unaided eyes it was observed that contamination of the dentin surface with latex gloves results in the removal of a considerable portion of the air-inhibited resin layer.[20] This removed resin layer was visible on finger tips and on the latex gloves; the surface of dentin had lost a large part of its glossy appearance,

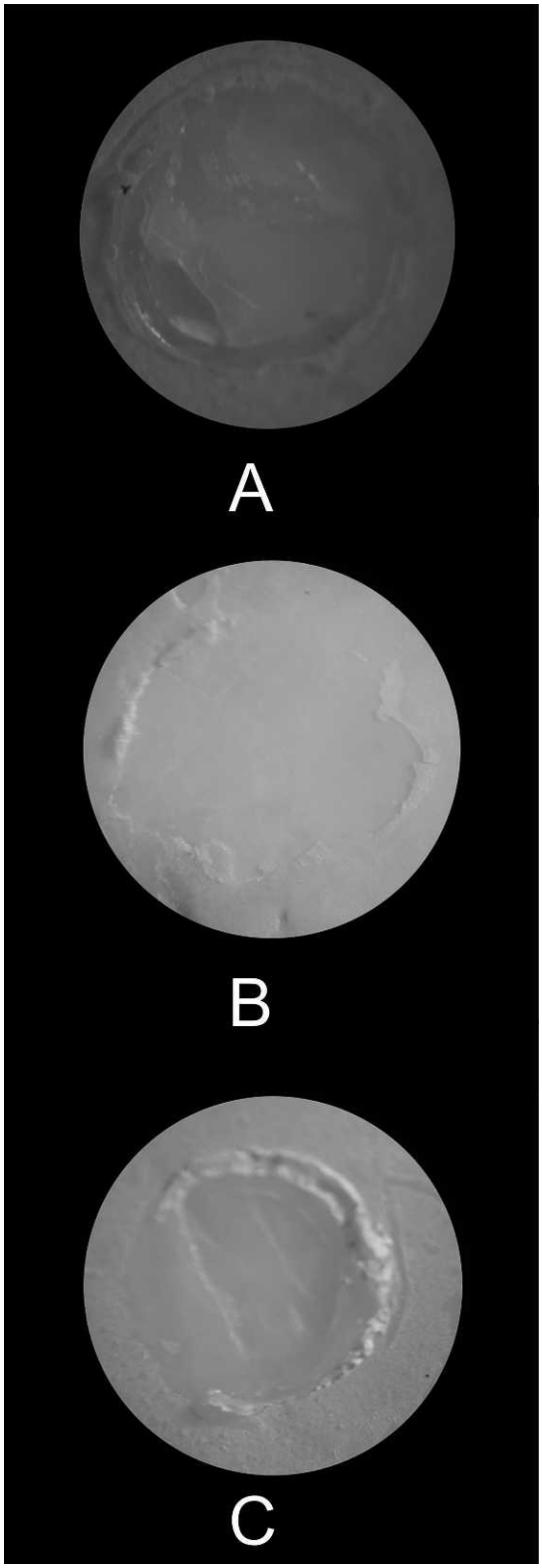

Figure 1. *Different types of failure modes. (A): Cohesive. (B): Adhesive. (C): Mixed.*

Table 2: *Means, Standard Deviations and Standard Error of Shear Bond Strength Values (MPa) and Failure Modes (%)*

Adhesive System	Contaminations	Bond Strength (MPa)			Failure Type, n (%)		
		Mean	SD	Standard Error	Adhesive	Cohesive	Mixed
Single Bond	No contamination	13.55 Aa	4.21	0.94	10 (50)	5 (25)	5 (25)
	Contaminated with powdered glove	9.89 Aa	6.55	1.46	14 (70)	3 (15)	3 (15)
	Contaminated with unpowdered glove	10.33 Aa	5.68	.27	10 (50)	4 (20)	6 (30)
Clearfil SE Bond	No contamination	13.17 Ab	8.16	1.82	13 (65)	4 (20)	3 (15)
	Contaminated with powdered glove	7.98 Ac	3.68	0.82	10 (50)	1 (5)	9 (45)
	Contaminated with unpowdered glove	9.89 Abc	3.99	0.89	11 (55)	3 (15)	6 (35)

Same small capital means no statistically significant differences between adhesive systems.
Same lowercase indicates no statistically significant differences between the subgroups in each adhesive system.

but it was surprising that the bond strength had not decreased significantly.[20]

The lack of contamination influence on the shear bond strength of these fifth-generation adhesives might be attributed to the fact that even in the presence of bonded surface contamination, it is probable that composite resin polymerization reactions are not easily compromised, and even in case of destruction the severity of the problem is not so great as to have a noticeable detrimental effect on composite resin bond strength.

It appears differences in the chemical composition and manipulation characteristics of different adhesive systems play an important role in the effect of contamination on bond strength. CSE belongs to the mild self-etching adhesive systems (pH=1.9) and transmission electron microscope images show that when this adhesive is applied, a shallow 1-μm thick hybrid layer is formed at the resin-dentin interface. The dentin surface is somewhat demineralized and hydroxyapatite crystals are visible inside the hybrid layer; in contrast, when SB is used the hybrid layer is thicker because etching is carried out in one stage and priming/bonding is achieved in the other.[30] Furthermore, according to manufacturer's instructions, SB is applied in two layers, which might be a factor in an increase in the bonding hybrid layer. It appears penetration of starch particles in the oxygen-inhibited layer does not interfere with the SB bonding given the greater thickness of the bonding layer in SB compared with CSE; however, penetration of starch particles in CSE into the depths of the bonding layer contacting the adhesive and dentin can produce defects that are centers for stress concentration, propagating cracks and decreasing bond strength. In addition, it is likely that the presence of starch granules can trap oxygen molecules and prevent polymerization of the adjacent resin layer, increasing the defects. This phenomenon will lead to greater problems if the bonded layer is thin.

In addition, cornstarch in latex gloves cross-links with epichlorohydrin containing not more than 2% magnesium oxide as a dispersive agent. Epichlorohydrin, which renders the cornstarch absorbable, also is used as a solvent for natural and synthetic resins. The presence of any residual epichlorohydrin possibly could account for the decrease in bond strength we observed.[10]

Given the use of two different generations of adhesive systems in the present study and the differences in the results despite identical procedures and contaminations, the chemical interference of epichlorohydrin cross-linked cornstarch powder with polymerization reactions of the adhesive systems used might have had a role in the significant decrease in the bond strength of the CSE adhesive system. Because we did not ascertain the causes of the starch-related adherence problems, we recommend further studies with a wider range of self-etch adhesive systems so that the results can be extended to clinical situations.

Furthermore, evaluation of the effect of contamination with starch particles on the conversion rate of

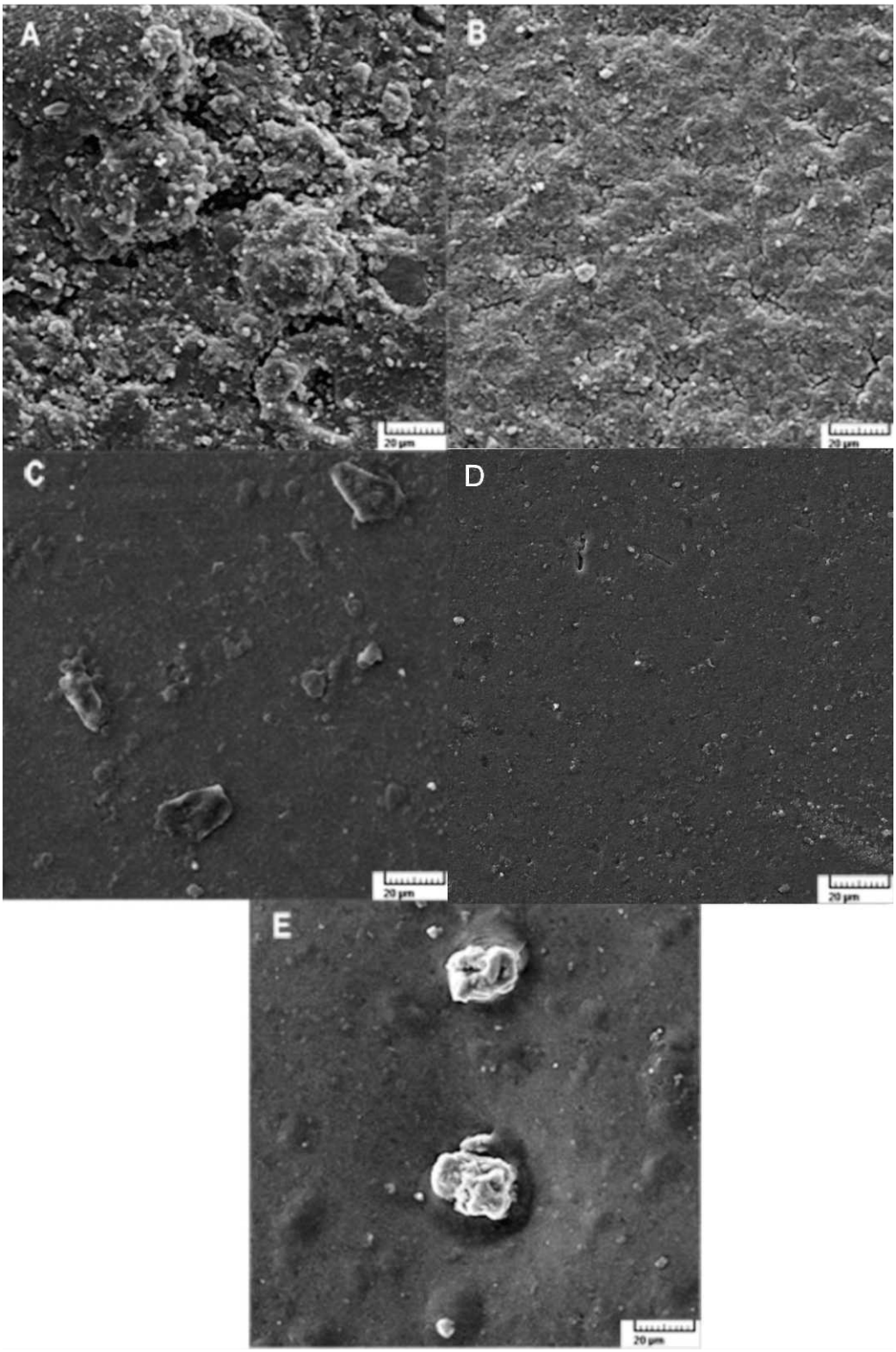

Figure 2. *SEM images of surface contaminations on each type of gloves and composite resin surface. (A): Unpowdered latex glove. (B): Powdered latex glove. (C): Clean composite surface. (D): Composite surface contaminated with unpowdered latex glove. (E): Composite surface contaminated with powdered latex glove.*

composite resins and adhesive systems can help to better understand and analyze the results. In materials that have a low conversion rate, the number of double bonds (C=C) or free radicals is higher; therefore, a higher bond strength is achieved in the presence of starch compared with materials that have a high conversion rate.

The bond failure location provides data about the quality of the bond between the tooth structure and the adhesive. Adhesive failure might be an indication of the wetting ability or the chemical reaction with the dental substrate of the adhesive. In the present study no differences were observed between the percentages of adhesive failures in the CSE and SB groups. In addition, the lower number of adhesive failures, compared with the mixed ones, in the subgroup contaminated with powdered gloves of the CSE adhesive in comparison with other subgroups of the same system might indicate the negative effect of contamination with powdered gloves on bond strength, which is consistent with the results of bond strength analysis.

CONCLUSION

The results of the present study showed that

1. Contamination of composite with powdered and unpowdered latex gloves did not influence the shear bond strength in SB dentin bonding system.
2. Contamination of composite resin with powdered latex gloves decreased the shear bond strength in CSE dentin bonding system.

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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Hydration and Dehydration Periods of Crown Fragments Prior to Reattachment

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Clinical Relevance

In the case of bonding the fragment of a fractured tooth, giving the fragment time to become rehydrated and considering treatment during a second appointment (scheduled 24 hours or more after the first visit) can lead to better bonding.

SUMMARY

Introduction: **Tooth fragment bonding is an excellent treatment option in dealing with traumatic injuries of the anterior teeth. Rewetting the tooth fragment has been shown to increase restoration durability. The present study examined the effect various dry and wet storage periods had on the reattached fragment's bond to the tooth.**

Materials and Methods: **One hundred and eight human mandibular incisors were fractured and assigned to undergo a dehydration period of 30 minutes, six hours, 24 hours, or three days before the rewetting procedure. After fracturing the teeth and drying the fragments, each of the specimens was assigned to one of the three main groups (A, B, or C) intended to evaluate the effect of different rehydration periods. Groups A and B underwent a 30-minute and a 24-hour rewetting period, respectively. Group C served as a control (without a rewetting stage). Tooth fragments were then reattached and prepared for the strength test. Force was applied on the lingual side of the tooth at a 1 mm/min rate until failure.**

Results: **The mean loads (N) required to fracture the restored teeth were as follows: 204.43 ± 33.48 N, 322.59 ± 34.62 N, and 253.25 ± 29.05 N for groups A, B, and C, respectively. Two-way analysis of variance ($p<0.05$) showed that**

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rehydration and dehydration periods as well as their interaction caused significant differences in the strength of the final restoration. Multiple comparison tests showed that, in general, significant differences were not seen among different dehydration times prior to the rewetting stage ($p>0.05$), except in the case of the 30-minute dehydrated specimens ($p<0.05$).

Conclusion: Compared to a 30-minute period, a 24-hour rehydration of the tooth fragment before treatment seems to salvage enough moisture to result in an increase in reattachment strength.

INTRODUCTION

The fragment bonding technique was first reported as a temporary treatment for crown fractures.[1] Since then, tremendous development has occurred in terms of both the materials used and in the treatment procedure itself. Crown fractures are very common among traumatic dental injuries, particularly in the permanent dentition. Among these injuries, uncomplicated crown fractures (crown fractures without pulp involvement) stand out as the most frequent type of fracture[2–5] and also represent the condition for which this technique (fragment bonding) is most frequently used. The numerous benefits of this technique with regard to both the patient and the dentist have made it a treatment of choice, especially in children.[6–10] The fragment bonding technique has been included in the recent guidelines for trauma management[11] and is considered a realistic, conservative, and cost-effective approach that provides long-lasting esthetics and restores function.[12,13]

Some laboratory studies[8,12] of this technique have shown results similar to those associated with intact teeth. In addition, the major shortcomings of composite restorations, the high failure rates in Class IV restorations, and their questionable long-term prognosis[4,7,14] have made the fragment bonding technique a more favorable approach. Preparation of a full crown or laminate veneer sacrifices tooth structure and may also be contradicted in younger patients.[7] Nevertheless, planning the final treatment with regard to the availability, adaptation, size, and number of fragments is of utmost importance.[7]

Contributions from several investigations have focused on the factors having an effect on the longevity of the restoration and the potential of this technique to yield better strength. Supplementary preparation of the bonding surfaces, which include tooth substance removal, has been shown to increase fracture strength.[6,15] Even though these additional preparations increase fracture strength, they prevent a precise fit between the two parts and are also in conflict with the conservative nature of this method. However, regardless of the bonding material and the technique used to reattach the tooth fragment, wetting and drying of the fragment appears to have a considerable effect on the fracture strength and appearance of the restoration.[15]

Various hydration and dehydration periods have been investigated.[15,16] Previous investigations show a downward trend in fracture strength as the dehydration time increases from five seconds to 24 hours. Rewetting the fragment for at least 24 hours after a 24-hour dehydration period had been suggested.[16] Others[15] demonstrated a 30-minute rehydration of the fragment to be sufficient after 48 hours of drying.

However, an optimum time that is both practical and beneficial in clinical conditions has yet to be determined. More research has to be done to determine whether the treatment should be carried out immediately or during a second appointment. Furthermore, the effect that different periods of dry storage may have on the fragment is also a matter of uncertainty.

The purpose of this study was to determine the effects of various drying and wetting storage periods on the fractured fragment and to reach a final conclusion as to which rehydration period is both better and more practical under clinical conditions. It was hypothesized that rehydration of dehydrated tooth fragments and the duration of this procedure affect the bond of the reattached fragment to the tooth.

MATERIALS AND METHODS

One hundred and eight intact human mandibular incisors (free of any cracks, caries, or structural defects and extracted because of periodontal problems) were selected. The dental plaque on the teeth was removed using an ultrasonic scaling device. Teeth were stored for less than four weeks in a physiological saline solution in 5°C refrigeration until the experiment was carried out.

The teeth were divided into a total of nine groups (A1-A4, B1-B4, and C1) on a simple random selection basis. Every subgroup contained 12 incisors. The numbers in each group name indicated the drying period of the specimens, and the letters (A, B, and C)

on each label specify the rehydration period considered for the group. The specimens were studied in three main experimental groups: group A, group B, and group C. Group C, with only one subgroup (C1) of 12 incisors, served as the control group (Figure 1).

The tooth crowns were prepared for fracturing. A line was traced on each tooth 3 mm from the incisal edge and parallel to it. Then, using a diamond disc (reference no. 918F, D+Z, Diamant, Lemgo, Germany), an enamel-deep fracture line was made on the lingual side of each tooth along the traced line. On the labial side of the line, a small notch was made in the middle of the surface to prevent the blade to be used later from slipping (Figure 2). The crowns were then fractured by a perpendicular force applied labio-lingually. Force was applied on the enamel cut by a blade on the lingual surface of the tooth and by another blade positioned in the opposite direction on the labial surface (Figure 3). The surgical blades (No. 21, Aesculap AG, Tuttlingen, Germany) were replaced after every third sample. Any teeth displaying a fracture pattern different from the predetermined line or an unclear fracture line were discarded at this stage. The apical parts were kept in distilled water and at an ambient temperature afterward.

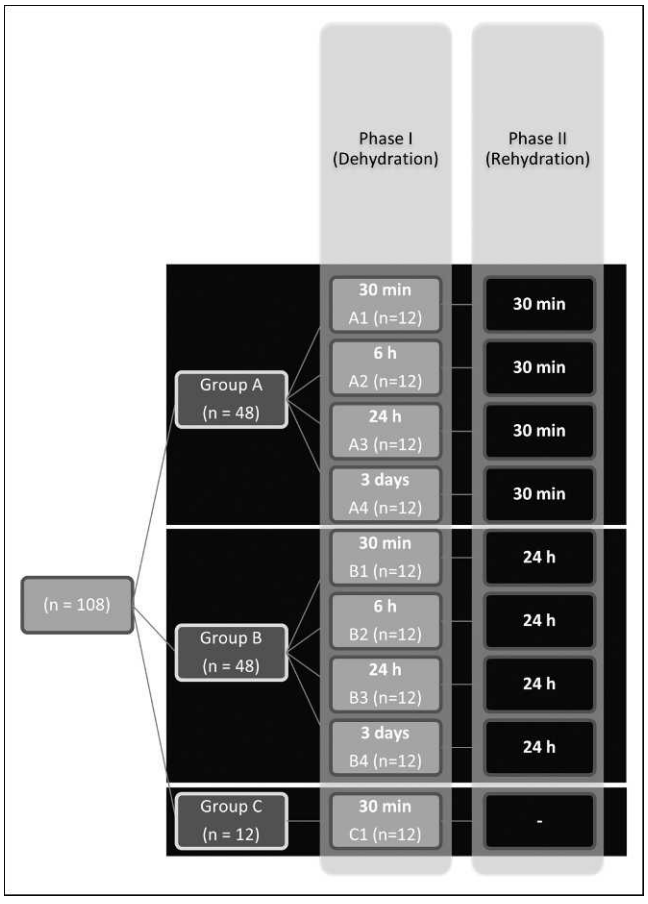

Figure 1. *Diagram illustrating the grouping of the teeth based on the dehydration and rehydration periods applied.*

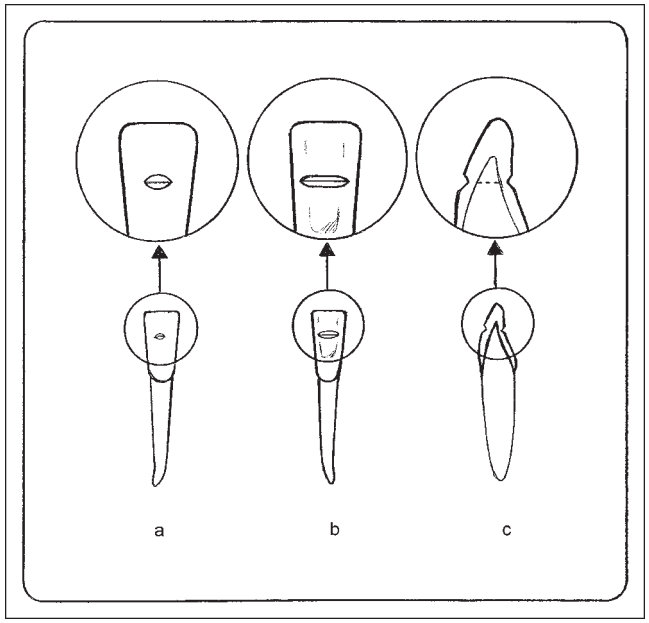

Figure 2. *Preparations on the tooth surface prior to fracturing from a labial (a), lingual (b), and proximal (c) view.*

Four dehydration periods in an ambient temperature (22°C, relative humidity of approximately 10%) were tested: group 1 (A1, B1, and C1) fragments were kept dry for 30 minutes; group 2 (A2 and B2) fragments for six hours; group 3 (A3 and B3) fragments for 24 hours; and group 4 (A4 and B4) fragments for three days.

Two wetting periods were administered after drying and before reattachment of the fragments. Group A (A1-A4) fragments were all immersed in distilled water for 30 minutes, while the group B (B1-B4) fragments followed this procedure for 24 hours. Group C (C1) fragments were reattached after the 30-minute drying time without a rewetting period.

The crown fragments were all reattached using the same technique. The fracture surfaces were etched using a 35% phosphoric acid gel (Ultra-Etch, Ultradent Products Inc, South Jordan, UT, USA) for 15 seconds and were then rinsed and gently air-dried to keep the surface moist. The bonding agent (Single Bond [batch no. 6KR], 3M ESPE, St Paul, MN, USA) was then used according to the manufacturer's instructions. Two coats of Single Bond were applied on the surfaces: the first layer, intended for priming, was applied for 10 seconds, and then the second

Figure 3. *Fracturing device used to fracture the teeth with two surgical blades mounted in a vise, 3 mm from the incisal edge of the incisor.*

layer was applied until a glossy look appeared on the surface. At each stage the bonding agent was air-thinned to remove any excess. Light curing was preformed for 20 seconds on each surface following the manufacturer's instructions using a light curing unit (Coltolux 75, Coltene/Whaledent Inc, Mahwah, NJ, USA) with a wavelength of 480 nm. A layer of low-viscous composite resin (Filtek Flow [batch no. 5GP], 3M ESPE) was placed on both bonding surfaces, and then the remnant and the fragment were pressed together into the proper position. After checking the exact realignment of the two reattached pieces, light curing was performed on the labial and lingual surfaces for 40 seconds. Excess composite resin was removed from all sides using a scalpel. The teeth were embedded by hand in an acrylic block, situated such that the long axis of the tooth would be parallel to the central axis of the block and at the same time the enamel cut would be parallel to the block surface. The crowns were exposed from above the tooth cingulum. Samples were put away in 37°C water in an incubator for 24 hours before the debonding test took place.

The specimens were mounted in a universal testing machine (Zwick Roell, ZO 20, Ulm, Germany). Stress was applied to specimens at a 1 mm/min rate until failure, using a 1-mm–wide chisel-shaped device. The attached device was placed perpendicular to the palatal surface of the tooth, 3 mm away from the incisal edge and adjacent to the reattachment line. The forces required to fracture specimens were calculated in Newtons (N). Recorded data were entered into a database, and statistical analysis was performed using SPSS version 11.5 (SPSS Inc, Chicago, IL, USA). Differences between all experimental groups were compared using two-way analysis of variance (ANOVA) followed by multiple comparison tests (Tukey *post hoc* tests). The significance level for all tests was set at 0.05. The remaining tooth particles were kept for further investigation concerning the fracture and detachment pattern.

RESULTS

Table 1 presents the mean forces required to fracture teeth in every group and subgroup separately. The two-way ANOVA revealed that the fracture load means were affected by both main factors (dehydration and rehydration periods) ($p<0.001$) and their interaction ($p<0.001$). The 30-minute dehydrated subgroups (A1, B1, and C1) had higher fracture load means ($p<0.001$). Fracture loads were also significantly lower in group A (with 30-minute rehydration) and higher in group B (with 24-hour

Table 1: *Ultimate Fracture Load Means (N) and Standard Deviations (SDs) of Various Dehydration (According to Their Subgroup Number [1–4]) and Rehydration (According to Their Group [A, B, and C]) Periods*[a]

Group	Subgroup	No. of Specimens	Dehydration Period	Rehydration Period	Mean, N	SD, N	Significance[b] ($p<0.05$)
A	A1	12	30 min	30 min	295.58	25.02	A
	A2	12	6 h	30 min	173.33	32.98	B
	A3	12	24 h	30 min	179.17	38.53	B
	A4	12	3 d	30 min	169.67	35.19	B
	Total (A)	48		30 min	204.43	33.48	
B	B1	12	30 min	24 h	331.00	29.87	C
	B2	12	6 h	24 h	324.75	31.87	C
	B3	12	24 h	24 h	313.38	37.62	AC
	B4	12	3 d	24 h	321.25	38.65	AC
	Total (B)	48		24 h	322.59	34.62	
C	C1	12	30 min	—	253.25	29.05	D
Total		108			262.43	73.83	

[a] *Subgroups with the same online capital letter did not differ significantly.*
[b] *Two-way analysis of variance (ANOVA) followed by Tukey tests.*

rehydration) ($p<0.001$). Multiple comparison tests showed statistically significant differences between the control group (30-minute dehydrated without rehydration) and each of the other groups and subgroups ($p<0.02$). The control group showed a lower mean compared to group B ($p<0.001$) and its subgroups and a higher mean compared to group A ($p<0.001$) and its subgroups. The mean fracture forces in group B (24-hour rehydrated) were the highest among all, whereas no significant differences were seen among the B1-B4 subgroups ($p>0.05$). The lowest means of fracture forces were indicated in the A2, A3, and A4 subgroups (with no significant difference among these subgroups [$p>0.05$]). Subgroup A1 (30 minute dehydration, 30 minute rehydration) means showed significant differences with most other subgroups, higher than those of subgroups A2, A3, A4, and C1 ($p<0.001$), but lower compared to subgroups B1 and B2 ($p<0.05$). The outcomes for the three-day dehydrated (A4 and B4) subgroups did not differ significantly from subgroups that were dehydrated for 24 hours (A3 and B3) and six hours (A2 and B2).

All specimens fractured and detached at the fragment-tooth interface, showing a similar pattern under 4× magnification.

DISCUSSION

The amount of strength recovery by fragment bonding still remains a controversial issue.[17] Some[6,13–15,17,18] believe reattachment with the hydration (or without dehydrating) of the surfaces and without any additional preparation restores approximately 50% of the fracture strength of the original tooth. Other studies[8,17,19,20] have reported this restoration to exhibit strength close to that associated with intact teeth. Although the improvements in dental materials can favor the use of this technique, it still may not be considered a standalone and definite treatment by some. However, the impact of hydration exists in spite of additional preparations on the surface,[15] usually intended to

increase the restoration durability. In addition, the greater part of the dentin surface stays unchanged in some of the preferred preparations (eg, the "overcontour technique" which requires a superficial 0.3-mm–deep preparation into the buccal surface and on the fracture line).[17] In addition, appropriate hydration reestablishes esthetics and natural color.[2,9] Hence, hydration of the fragment should be considered a fundamental step in treatment of fractured teeth.

The method used to fracture teeth in this study was intended to simulate the fracture pattern and surface anatomy in traumatic situations and the micromechanical adaptation of the surfaces under clinical conditions. To obtain standardized fragments and dictate a similar fracture pattern in all specimens, the initial enamel cut line was made. Although the cross-head speed used in the debonding test does not simulate clinical conditions, and although the results may differ in higher velocities (relatively lower strengths), this technique has been accepted in similar investigations.[21–23] Similar to previous studies,[24,25] the combination of an adhesive and a low-viscous resin was intended to improve the mechanical properties of the treatment.[13,21,26,27] The etch-and-rinse technique employed has proven to result in restorations with greater longevity compared to enamel etching alone.[28]

The drying and wetting durations of the fragments were intended to resemble different possibilities in actual practice. The 30-minute rehydration period is similar to the time that treatment is carried out at the same appointment (with a 30-minute rehydration carried out by the dentist before treatment). The 24-hour rehydration is similar to the time between an initial visit and a second appointment the dental practitioner schedules for treatment. On the other hand, various drying periods were considered to simulate various times from the incident until the patient may consult a dentist.

The mean loads that caused fracture in each group represent a pattern of dehydration that begins at a very high rate, from the moment of fracture. The dehydration process does not reach its final state in 30 minutes, but it appears to have approached a plateau-like trend within six hours. Any further drying (up to three days) seems to be almost ineffective in the final strength of the tooth since no significant difference or decreasing trend is seen. These results correspond with those of Farik and others,[16] who also showed that the manner in which fracture strengths weakened with time was steeper at the beginning and became less noticeable later on. Even so, there is a point of difference: in this study drying the fragments for as few as 30 minutes diminished fracture strengths, compared to the over one-hour limit in the previous study.

In contrast to a recent study by Capp and others[15] that considered 30 minutes of rehydration sufficient, this research found that the results for 30-minute rehydration were unsatisfactory compared to those associated with a 24-hour period. There was one exception to this: when the fragment was dried for only 30 minutes and rehydrated for 30 minutes afterward (subgroup A1). This subgroup was the main reason the effect of dehydration periods as a factor and the interaction between the two experimental factors were statistically significant (since both became statistically insignificant with the exclusion of this dehydration period). Although the results for the A1 subgroup were significantly lower than those for the 24-hour rehydrated group, they were remarkably improved compared to those of the other subgroups that were rehydrated for 30 minutes (A2-A4). Therefore, it seems reasonable that if the patient is treated immediately after the accident (not far off the 30-minute limit), an acceptable treatment could be achieved with a 30-minute rehydration of the fragment (especially when it is not seen as a long-term treatment). However, regardless of the drying time, it appears to be advantageous to proceed with a 24-hour rehydration of the fragment. On the other hand, dental injuries should be classified as emergencies at all times, and as a result, it is believed that patients should be treated as soon as possible to improve the overall prognosis of both the pulp and the restoration.[4] Therefore, if the treatment is to be postponed to a second appointment, proper protection of the pulp and the remaining tooth structure should be taken into consideration, particularly to avoid any adverse biological or physical side effects. For example, the use of a hydrophilic restorative material to seal the exposed dentinal tubules would be appropriate.[5] The dentist may also prefer temporary restoratives to be able to regain the fit between the fragment and the remaining tooth structure.

There are some times during which the rehydration of the fragment may seem unnecessary: for example, when complete removal of the fragment's dentin is scheduled to take place during the treatment. This is because the dehydration process mainly affects dentin.[15] Another matter to take into account is that based on the bonding system used and its characteristics, the impact of dentin dehydration on bonding may differ.[29,30] For this reason,

dehydration may have a different influence on bonding strengths when bonding agents other than Single Bond are utilized.

In the present study, it was impractical to calculate the bonded surface area in each specimen. Thus, to reduce any side effects related to this issue, and so the outcome would be able to represent fracture strengths, the number of specimens considered for this experiment was increased to 12 for each group. The teeth with differing crown widths, lengths, and thicknesses were excluded in the initial selection, and the teeth showing dissimilar fracture patterns were excluded after the fracture test.

An interesting observation that was not part of the research was that the specimens dried for a longer period appeared to lose their adaptability with the apical part more than the specimens with a shorter drying period. This may partially be because of an irreversible contraction in the dentin structure during dehydration. Undoubtedly, further research has to be done to prove such a point.

CONCLUSIONS

In this in vitro study,

- Twenty-four hour rehydrated specimens of dehydrated tooth fragments exhibited stronger bonds in comparison with the 30-minute rehydrated specimens.
- When the fragment had been dried for 30 minutes or less, rehydration for 30 minutes afterwards seemed to significantly improve the bonding strength of the reattachment.

Conflict of Interest Declaration

The author of this manuscript 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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Effect of Chemical and Mechanical Degradation on Surface Roughness of Three Glass Ionomers and a Nanofilled Resin Composite

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Clinical Relevance

The nanofilled glass ionomer provides intermediate resistance to chemical and mechanical degradation among the glass ionomer cements (conventional and resin-modified) and nanofilled composite.

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SUMMARY

Nanofillers have been incorporated into glass ionomer (GI) restorative materials to improve their mechanical and surface properties. The aim of this present laboratory study was to compare the superficial roughness (*Ra*) of nanofilled GI (Ketac N100) with that of conventional GI (Fuji IX GP), resin-modified GI (Vitremer), and a nanofilled resin composite (Filtek Supreme) after pH cycling and toothbrush abrasion. Ten specimens of each material were made using Teflon molds, which were polished using aluminum-oxide abrasive disks. Three measurements of *Ra* were made of each specimen to serve as baseline values. The specimens were submitted to pH cycling for 10 days in a demineralization solution for six

hours (pH 4.3) and were then stored in remineralization solution for 18 hours (pH 7.0). *Ra* measurements were recorded after the pH cycling. Specimens were then submitted to toothbrush abrasion in a brushing machine with a 200*g* load for 30,000 cycles at 250 cycles/min. The *Ra* values were then recorded. The surface morphology of specimens from each group was analyzed using a scanning electron microscope. Data were analyzed by analysis of variance, Tukey, and *t*-tests. After toothbrushing, only Fuji IX GP (1.10 ± 0.80) showed *Ra* values that were statistically different from those of the other materials evaluated. Ketac N100 (0.68 ± 0.16) showed intermediate *Ra* values, but it did not differ statistically from the results associated with Vitremer (1.04 ± 0.46) and Filtek (0.30 ± 0.15). Ketac N100 showed intermediate values of superficial roughness among the conventional glass ionomer cement, resin-modified glass ionomer cements, and the nanofilled resin after chemical and mechanical degradation.

INTRODUCTION

Glass ionomer (GI) restorative materials have been widely used in dentistry, mainly in Class V and primary tooth restorations.[1,2] Chemical adhesion to tooth structure, long-term fluoride release, ability to uptake fluoride, and low coefficients of thermal expansion are characteristics that make GI clinically attractive.[3–5]

The properties of GI have improved in recent years as the result of an increased percentage of filler particle loading, the incorporation of resin monomers into the cement, or both.[6] Resin-modified GI cements (RMGIs), one of the major categories of commercial GIs, were developed to partly overcome the problems associated with conventional GIs, such as poor handling characteristics, sensitivity to moisture during initial setting, and poor physicomechanical strength.[3,7,8] Recently, a new category of GI restorative materials was introduced for the restoration of primary teeth and small cavities in permanent teeth: the nanofilled RMGIs (Ketac N100 or Ketac Nano Light-Curing; 3M ESPE, St Paul, MN, USA), a paste/paste material.[9] Its primary curing mechanism involves light activation, and no redox reactions or self-curing occur during setting. The major innovation of this material involves the incorporation of nanotechnology, which allows a highly packed filler composition (~ 69%), of which approximately two-thirds are nanofillers.[10] The nanofillers improve the mechanical strength and optical properties of the restorative material.[11]

The resistance of dental restorations to superficial degradation has an appreciable influence on their clinical performance.[12] In the oral cavity, the degradation of restorative materials can be caused by low pH (due to cariogenic biofilm and acidic drinks), water sorption, erosion, and wear.[12–14] These factors involve degradation of the resin matrix, filler, or matrix-filler interface on the surface of the materials, leading to increased surface roughness and wear rate.[15,16]

Laboratory studies assess the surface integrity of restorative materials using mechanical degradation tests, such as wear simulation and brushing abrasion, which evaluate the surface texture and wear/abrasion resistance of materials.[17–20] Corrosive wear or mechanical degradation results from the joint action of chemical and mechanical forces and is also associated with the mechanical removal of corroded layers that form on the surface of a material through a reaction with its environment.[21] However, it is also important to determine how the surface of restorative materials performs as a consequence of the chemical degradation that also occurs in the oral cavity. The dynamic variations in the oral pH, due to cariogenic challenges, can develop superficial degradation of restorative materials from the low pH.[16,22] Furthermore, some studies[15,23,24] have demonstrated that materials can be more susceptible to brushing abrasion at lower pH values. The acidic storage medium erodes the surface of conventional GI, for example, and causes hydrolysis and dissolution mainly in the polyalkenoate matrix.[25–27] This degradation has a detrimental and irreversible effect on the cement surface. As a clinical consequence, the low environmental pH, in association with toothbrushing abrasion, can result in a loss of contour and increasing surface roughness, which increase the rate of wear and can be responsible for bacterial biofilm and stain accumulation.[28]

Based on the fact that the incorporation of nanofillers in restorative materials can improve the polish and abrasion resistance of restorations,[11] it seems relevant to evaluate whether a nanofilled, resin-modified GI (Ketac N100, 3M ESPE) has lower superficial degradation after pH cycling and abrasion testing when compared with a conventional or resin-modified GI. Therefore, the null hypothesis tested in this current study was that the nanofilled RMGI subjected to a pH-cycling model (chemical degradation) prior to a three-body abrasion test (mechanical degradation) does not differ in the roughness (*Ra*) and

surface morphology to that of a conventional GI, RMGI, or nanofilled resin composite.

MATERIALS AND METHODS

Materials

Four direct restorative materials, including one conventional GI (Fuji IX GP; GC America Inc, Alsip, IL, USA), one RMGI (Vitremer; 3M ESPE), one nanofilled RMGI (Ketac N100; 3M ESPE), and one nanocomposite resin (Filtek Supreme; 3M ESPE) were evaluated in this current study. Brand names, manufacturers, basic compositions, mean filler size, and batch numbers are indicated in Table 1.

Specimen Preparation

Ten specimens of each restorative material (n=10) were fabricated using Teflon molds (4.0-mm diameter and 2.0 mm thick), according to the manufacturers' instructions. The GI restorative materials were mixed and placed in one increment into the mold. The nanocomposite was also placed into the mold in one increment. The surface of the restorative materials was covered with a polyester strip and a glass slab. A load of 500*g* was applied for 30 seconds to expel excess material from the matrix. Vitremer, Ketac N100, and Filtek Supreme specimens were polymerized with a light-curing unit (Elipar Trilight; 3M ESPE). The output of the QTH curing light used to polymerized specimens was found to be 690 mW/cm^2 determined with a curing light meter (Hilux Dental Curing Light Meter; Benliglu Dental Inc, Turkey). Fuji IX GP specimens were allowed to set for five minutes, according to the manufacturers' instructions. All specimens were stored in 100% relative humidity at 37°C for 24 hours.

Finishing and polishing were carried out using a sequence of medium, fine, and superfine aluminum-oxide abrasive disks (Sof-Lex Pop On; 3M Dental Products, St Paul, MN, USA). Each disc was applied in a single direction for 15 seconds using air-water spray by a single calibrated operator based on a previous pilot study,[16] which tested the direction and time of oxide abrasive disk application on the surface roughness of each material. A new disc was used for each specimen. After the polishing procedures were completed, the specimens were ultrasonically cleaned (Model T1440D; Odontobrás Ltda, Ribeirão Preto, SP, Brazil) in distilled deionized water for 10 minutes to remove polishing debris and stored in 100% relative humidity.

Surface Roughness Measurements

Before the pH-cycling regime and abrasion testing, the surface roughness of all specimens was analyzed

Table 1: *Restorative Materials Evaluated in this Study*

Restorative Material (n=10)	Basic Composition	Mean Filler Size[a]	Batch No.
Fuji IX GP (GC America Inc, Alsip, IL, USA)	Powder: polyacrylic acid, aluminum fluorosilicate glass	4.4 μm	0706061/0706051
	Liquid: polyacrylic acid, tartaric acid and water		
Vitremer (3M ESPE Dental Products, St Paul, MN, USA)	Powder: fluoroaluminosilicate glass; redox system	3.0 μm	6LN/6FC
	Liquid: aqueous solution of a modified polyalkenoic acid, HEMA		
Ketac N100 (3M ESPE Dental Products)	Paste A: silane-treated glass; silane-treated ZrO2 silica; silane-treated silica; PEGDMA; HEMA; Bis-GMA; TEGDMA	1 μm (cluster) 5-25 nm (nanofiller) <3.0 μm (glass)	M3M3
	Paste B: silane-treated ceramic; silane-treated silica; water; HEMA; acrylic/itaconic acid copolymer		
Filtek Supreme (3M ESPE Dental Products)	Bis-GMA, Bis-EMA, UDMA, TEGDMA zirconia/silica cluster filler and a nonagglomerated silica filler	20–75 nm	7CJ

Abbreviations: Bis-EMA, ethoxylated bisphenol-A dimethacrylate; Bis-GMA, bisphenol glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *According to the manufacturers' information.*

using a Surfcorder SE1700 surface roughness–measuring instrument (Kosaka Corp, Tokyo, Japan), equipped with a 2-μm-radius diamond needle, which traversed the surface at a constant speed of 0.05 mm/s with a force of 0.7 mN. The cutoff value was set at 0.08 mm (Gauss filter). The surface roughness was characterized by *Ra* (μm). *Ra* is an arithmetic average of the peaks and valleys of the specimen surface, it is recorded as absolute values within the evaluation length, and it is generally expressed in units of height. The roughness parameters were obtained via Surface Analysis (Kosaka Corp), and the mean *Ra* values were calculated using the following formula, where the roughness curve is expressed in $y = f(x)$ and L is the reference length:

$$Ra = \frac{1}{L}\int_0^L |f(x)|dx$$

Three readings were taken from each specimen, with tracings at three different locations—parallel, perpendicular, and transverse to the finishing and polishing scratch directions. The average of these readings, the mean *Ra*, was used as the baseline *Ra* value for each sample.

pH-Cycling Model

The specimens were subjected to a pH-cycling regime, as proposed by Featherstone and others[29] and Silva and others.[22] The demineralization solution contained 2.0 mM calcium and 2.0 mM phosphate in a buffer solution of 74.0 mM acetate at pH 4.3. The remineralization solution consisted of 1.5 mM calcium and 0.9 mM phosphate in a buffer solution of 0.1 mM Tris (hydroxymethyl-aminomethane) at pH 7.0. The specimens were first immersed in 5 mL of demineralization solution for six hours at 37°C, rinsed with distilled deionized water, and then stored in 5 mL of remineralization solution for 18 hours at 37°C.[22] These experimental conditions were repeated for 10 uninterrupted cycles of demineralization and remineralization. The solutions were changed every day.

After the pH-cycling regime, all specimens were submitted to surface roughness testing, as described previously.

Three-body Abrasion Testing

After pH cycling, the toothbrushing test was conducted in an automatic toothbrushing machine using a 200*g* load for 30,000 cycles at 250 cycles/min. Colgate Total dentifrice (Colgate Palmolive Ind. e Com. Ltda, SB Campo, São Paulo, Brazil) that had been diluted in distilled water (1:2) was used as an abrasive third body. This dentifrice was considered to be a moderate abrasive toothpaste, according to the Relative Dentin Abrasion (~70) adopted by DIN EN ISO 11609.[30] The specimens were washed in an ultrasonic bath for 10 minutes. The final *Ra* readings were taken from each specimen, as described previously, in a direction opposite that of the toothbrushing movement.

Surface Morphology Assessment

Three specimens of each material at each experimental period (baseline, after pH cycling, and after toothbrushing) were analyzed to determine the superficial morphology using a scanning electron microscope (SEM). The nine specimens of each material were mounted on a holder using double-sided adhesive carbon tape in order to illustrate the effect of pH cycling or/and toothbrushing on the material surfaces. The specimens were sputter-coated with gold in a vacuum appliance (Balzers-SCD 050 Sputter Coater; Liechtenstein, Germany) and examined with a Model JEOL JSM 5600 LV SEM (Tokyo, Japan) operating at 3000× magnification.

Statistical Analysis

The roughness data were evaluated to check the equality of variances and to ensure a normal distribution. After this, the roughness data were submitted to a paired *t*-test to analyze the differences in surface roughness before any experimental procedures (baseline), after pH cycling, and after the abrasion test. Two-way analysis of variance (ANOVA) and Tukey test were used to compare the roughness among materials. A level of $\alpha = 0.05$ was used to demonstrate statistical significance.

RESULTS

Table 2 presents the comparison of superficial roughness measurements (*Ra*) of each material among the experimental periods (treatment)—baseline, after pH cycling, and after toothbrushing—and among restorative materials in each treatment. When the comparisons of *Ra* were made for each material among the treatments, only Fuji IX GP showed a statistically significant increase in surface roughness (0.15 ± 0.10 μm at baseline, 0.27 ± 0.20 μm after pH cycling, and 1.10 ± 0.80 μm after toothbrushing; $p=0.005$). For Vitremer, Ketac N100, and Filtek Supreme restorative materials there was only a statistically significant increase in surface roughness ($p<0.001$) between the baseline (0.20 ±

Table 2: *Superficial Roughness Measurements (μm) of Restorative Materials Tested After pH-Cycling Regime and Toothbrush Abrasion*[ab]

Material/ Treatment	Baseline	pH-Cycling Regime	Tooth-brushing
Fuji IX GP	0.15 ± 0.10[a,A]	0.27 ± 0.20[b,A]	1.10 ± 0.80[c,A]
Vitremer	0.20 ± 0.16[a,A]	0.23 ± 0.08[a,A]	1.04 ± 0.46[b,A]
Ketac N100	0.14 ± 0.07[a,A]	0.18 ± 0.12[a,A]	0.68 ± 0.16[b,AB]
Filtek Supreme	0.09 ± 0.03[a,A]	0.11 ± 0.05[a,A]	0.30 ± 0.15[b,B]

[a] *Values (mean ± standard deviation) expressed in μm.*
[b] *Lowercase letters refer to comparisons of each material among the experimental periods (baseline, after pH-cycling regime, and after toothbrushing). Values followed by the same lowercase letters in each row do not differ statistically (paired t-test, p>0.05). Uppercase letters refer to comparisons among restorative materials in each experimental period. Values followed by the same uppercase letters in the columns do not differ statistically (two-way analysis of variance [ANOVA] and Tukey test, p>0.05).*

0.16 μm, 0.14 ± 0.07 μm, and 0.09 ± 0.03 μm, respectively) and after toothbrushing treatment (1.04 ± 0.46 μm, 0.68 ± 0.16 μm, and 0.30 ± 0.15 μm, respectively). Therefore, the greatest surface roughness values were found after toothbrushing for all materials.

The ANOVA showed a significant interaction between restorative materials and treatment (p=0.003). Because the interaction was significant, a comparison of the behavior of the different materials in each treatment was made. There was no statistically significant difference among the roughness values for all of the materials after pH cycling (p=0.089). Fuji IX GP (1.10 ± 0.80 μm) and Vitremer (1.04 ± 0.46 μm) had the highest roughness values after toothbrushing; however, no statistically significant difference in surface roughness existed between Fuji IX GP and Vitremer. Ketac N100 (0.68 ± 0.16 μm) showed intermediate roughness values after toothbrushing. This material did not have a statistically significant difference in surface roughness between Fuji IX GP and Vitremer, but it also did not have a statistically significant difference in surface roughness with Filtek Supreme (0.30 ± 0.15 μm). Filtek Supreme showed the lowest superficial roughness values.

The scanning electron micrographs in Figure 1 show details of the surface morphology of the studied materials. The conventional GI and RMGI cements presented a large number of cracks in the microstructures, probably caused by dehydration during preparation for SEM analysis (Figure 1B,E). Fuji IX GP, Vitremer, and Ketac N100 showed moderate irregularities in surface morphology after pH cycling (Figure 1B,E,H), and the surface of Filtek Supreme seemed minimally affected (Figure 1K). However, all materials showed a discernible loss of material after toothbrushing, leading to irregular surfaces and protruding filler particles, showing the abrasion aspect of the matrix (Figure 1C,F,I,L). There was a clearly visible difference between the fillers of Ketac N100 (Figure 1I) and the other GIs with regard to shape and size. Ketac N100 has a more regular shape and the lowest size particles when compared with the other GIs. Furthermore, Ketac N100 presents nanoclusters with similar size and shape, which Filtek Supreme does not (Figure 1L).

DISCUSSION

The present study investigated the superficial roughness of a nanofilled GI after a chemical challenge and toothbrushing abrasion and compared those data with conventional GI, RMGI, and nanofilled composite. The pH-cycling regime used in the current study was an *in vitro* chemical degradation method that simulated alterations in the pH of the oral cavity and mimics clinical conditions whereby there is always a dynamic between demineralization and remineralization.[22] The method of acid challenge by the dynamic pH-cycling model used in the current study was proposed by Featherstone and others.[29] This method involves six hours of acid challenge and 18 hours of remineralization per day, which is a reasonable estimate for subjects who frequently consume sugar.[29] Moreover, Silva and others[22] showed that the surface properties of restorative materials were influenced by pH variation in the demineralizing solution of pH cycling. The acidic pH led to a greater alteration in hardness and surface morphology of GI cements.[22]

In this present study, the dynamic pH cycling did not affect the superficial roughness of RMGI, nanofilled GI, and nanofilled resin; only the conventional GI showed an increase in superficial roughness after pH cycling (Table 2). Since the conventional GI did not have resin monomers in its composition, an acid-base reaction occurs after the formation of a calcium and aluminum polyalkenoate matrix, in which the filler particles are embedded and surrounded by a silica hydrogel.[31] While resin-based materials undergo the cleavage of polymer chains to form oligomers and monomers, the ionomeric cements present a complex process of absorption, disintegra-

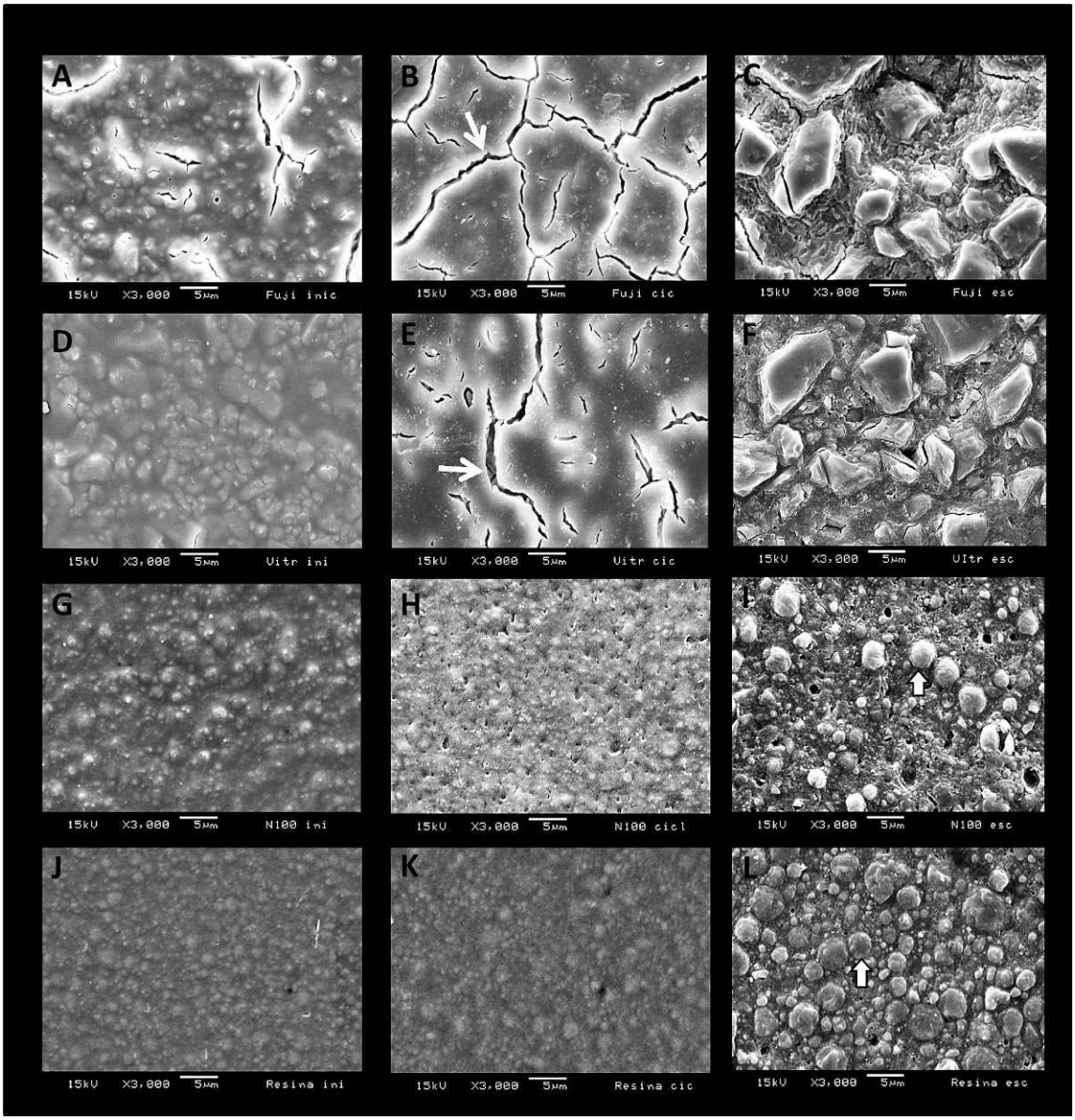

Figure 1. *Scanning electron micrographs of the surface morphology of Fuji IX GP (A-C), Vitremer (-F), Ketac N100 (G-I), and Filtek Supreme (J-L). The first column shows the surface morphology of restorative materials without treatment (control). The second column represents the surface morphology of restorative materials after pH cycling with a large number of cracks on the Fuji IX and Vitremer surfaces (→) and undetectable fillers (B and E). Filtek Supreme shows minimal alteration in surface morphology (K), and Ketac N100 shows minimal degradation of the matrix. The third column represents the surface morphology of restorative materials after toothbrushing; an irregular surface and protruding filler particles were visualized for all materials (C, F, I, and L). Open arrows indicate nanoclusters and nanofillers of Ketac N100 and Filtek Supreme (I and L).*

tion, and outward transportation of ions.[12] The acidic degradation begins with water absorption that diffuses internally through the matrix, filler interfaces, pores, and other defects, accelerated by a low pH and causing filler-matrix debonding or even hydrolytic degradation of the filler-matrix interface.[32,33]

No statistically significant difference was found among the roughness values of all materials tested after the pH cycling (Table 2). It is likely that there was no statistically significant difference among the roughness values as a result of the large standard deviations obtained, mainly for Fuji IX GP and Ketac N100. Moreover, Filtek Supreme showed minimal alterations in surface morphology after pH cycling, as observed by SEM (Figure 1K), while the GIs showed few irregularities with minimal degradation of the matrix (Figure 1H) and a surface layer with

undetectable fillers (Figure 1B,E). While other studies[15,24] have shown that a pH-cycling regime similar to the one used in the current study (six hours of demineralization/18 hours of remineralization for 10 uninterrupted days) affected the surface roughness of restorative materials, it is possible that the period of pH cycling (10 days) was not long enough to cause significant hydrolytic and corrosive degradation among the GI restorative materials and nanocomposite, regardless of their different compositions. It is possible that if the period of the pH-cycling regime was increased, differences in the roughness and morphology could be found among the GIs and composite.

The design of the toothbrush abrasion used in this current study, 30,000 strokes, is equivalent to three years of brushing in the clinical situation.[34] However, the abrasion regimen used in this study certainly does not correspond to 10 episodes of variation in pH in the clinical situation. This extensive number of brushing strokes was performed in order to develop grooves to analyze the surface roughness. After toothbrushing abrasion, GIs and composite resins behaved differently with regard to their surface roughness and morphology (Table 2; Figure 1). Possible explanations for these differences are the inherent resistance of the major constituents (polymer matrix and glass filler particles), size and shape of the filler particles, adhesion between the particles and matrix, and the setting reaction of all materials.[35]

While the pH cycling did not present a roughness difference between RMGI and conventional GI (Table 2), it was not possible to find any studies regarding superficial roughness analyses between conventional GI and RMGI after pH-cycling and abrasion tests. In this present study, the conventional GI and RMGI did not show significant differences in roughness values after toothbrushing abrasion. As both GIs present larger and irregular glass filler particles with similar mean size (around 3–4 μm) (Table 1), the toothbrushing abrasion provided a discernible loss of material, causing these filler particles to be lost or to protrude from the surface (Figure 1C,F), while differences in roughness values were not found between them. Moreover, the difference between the abrasion of the matrix and filler particles might have been large enough to provide an anisotropic degradation, which gradually slowed down as the filler became exposed to the surface.[15] Thus, the differences of composition between the matrix of conventional GIs and RMGIs—conventional GI has a polyalkenoate matrix and RMGI has a polyalkenoate and poly–2-hydroxyethyl methacrylate (HEMA) matrix—were minimized, and the superficial roughness after abrasion was similar.

Although the roughness value of the nanofilled GI Ketac N100 did not statistically differ from those of Vitremer and Fuji IX GP, it behaved like an intermediate material between a GI and a nanocomposite resin when abrasion resistance was considered (Table 2). A reasonable explanation for this result is the composition of Ketac N100, which includes other resin monomers in addition to HEMA, is similar in composition to RMGI, with bisphenol glycidyl methacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA). Additionally, this cement represents a blend of fluoraluminosilicate technology (40%) and nanotechnology (60%), including silica cluster fillers, nanoagglomerated silica fillers, and acid -reactive glass fillers that are smaller than those of resin-modified glass ionomers (Ketac™ N100-Technical Product Profile).[9] Filtek Supreme nanocomposite presents nanofillers and nanoclusters and the resin matrix had other resin monomers, such as the ethoxylated version of Bis-GMA (Bis-EMA) and urethane dimethacrylate (UDMA).[36] Thus, Ketac N100 had an intermediate composition between RMGIs and nanocomposites, presenting intermediate superficial characteristics and abrasion resistance. Ketac N100 presented an appearance more like a polyacid-modified resin composite based on resin components and structure; however, further studies must be conducted to investigate this fact.

The incorporation of nanofillers in restorative materials improves the abrasive resistance because it promotes a higher filler loading with smaller particle size and provides a reduction in the interparticle spacing, which effectively protects the softer matrix, reduces the incidence of filler exfoliation, and enhances the overall resistance of the material to abrasion.[37] Ketac N100 has a highly packed filler composition (~69%), of which approximately two-thirds are nanofillers.[10] Figure 1I and L show the nanoclusters of nanofilled GIC and nanocomposite. According to the manufacturer information, the aggregated "nanoclusters" in Ketac N100 are in the 1-μm size range but are composed of 5- to 20-nm spherical particles that have been lightly sintered together to form a porous structure interpenetrated with resin monomers (Ketac™ N100-Technical Product Profile).[9] As the surface of the "nanocluster"/resin combination is subjected to stress and toothbrushing abrasion, the smaller

nanosized particles, which make up the clusters, tend to break apart rather than the entire particle being plucked from the resin matrix, leaving the surfaces with defects smaller than the wavelength of light and providing satisfactory polish and optical properties.[11] In this current study, these characteristics contributed to the abrasion resistance of Ketac N100, which was not statistically significantly different from that of the nanocomposite (Table 2). Similar results were found by de Paula and others,[17] in whose study the surface roughness of Ketac N100 did not differ from that of a nanocomposite after biomechanical degradation. However, the larger and irregular filler particles present in Vitremer and Fuji IX GP (Figure 1C,F) made it easier to "pluck out" a whole filler particle from the resin matrix, which could act as an additional abrasive agent once it has detached from the surface and is held against the specimen.[38] In addition to the filler size, the hydrophilic property of the Vitremer polymer matrix, as a result of the hydroxyl groups of HEMA,[39] and the insufficient coherent entanglement between the ionic cross-linked polyalkenoate network and the polymeric chains[3,35] could have provided a greater chemical and mechanical superficial degradation than was associated with Ketac N100 and Filtek Supreme.

The nanocomposite Filtek Supreme showed lower roughness values than did the conventional GI and RMGI. The presence of nanofillers and nanoclusters, as explained previously, contributes to this result. In addition, the presence of Bis-EMA can promote hydrolytic and biochemical stability as a result of the hydrophobicity of this monomer when compared to Bis-GMA.[13] The greater part of TEGDMA of Filtek Supreme was replaced with a blend of UDMA and Bis-EMA. The absence of a phenol ring in the monomer chain of UDMA leads to greater flexibility and toughness when compared with Bis-GMA, making the UDMA resins more reactive, with higher conversion and cross-link density than are associated with the Bis-GMA polymers, contributing to the matrix being more resistant to abrasion.[36]

Based on the results of this current study, the hypothesis that a nanofilled RMGI subjected to a pH-cycling model prior to a three-body abrasion test would not differ in surface roughness and micromorphology from a conventional GI, RMGI, and nanofilled resin composite was rejected. Ketac N100 showed statistically significant intermediate roughness values between GI and a nanocomposite resin after pH cycling and toothbrushing abrasion.

CONCLUSION

Concerning the surface roughness and surface morphology after chemical and mechanical degradation, the nanofilled GI restorative material, Ketac N100, behaved as an intermediate material among GI restorative materials (conventional and resin-modified) and a nanocomposite.

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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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Effect of Hydrogen Peroxide at 35% on the Morphology of Enamel and Interference in the De-remineralization Process: An *In Situ* Study

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SB Berger • ALF Briso

Clinical Relevance

The 35% hydrogen peroxide bleaching gel potentiated the structural and histomorphological changes induced by the accumulation of biofilm on the enamel surface.

SUMMARY

This study evaluated the microhardness and histomorphology of bovine enamel when 35% hydrogen peroxide is used. A total of 44 specimens were adapted to removable devices used by 11 individuals subjected to dental caries challenge. A decrease in microhardness was observed for all groups after the cariogenic challenge. Microscopic analysis revealed that fragments subjected to cariogenic challenge associated with bleaching had more intense superficial histologic changes, but the depth of the lesions remained unchanged. It was concluded that 35% hydrogen peroxide enhanced the reduction in hardness and histomorphologic changes in the enamel surface exposed to cariogenic challenge.

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INTRODUCTION

Achieving a beautiful and healthy smile is a constant concern of dental professionals and patients. Therefore, cosmetic dental treatments can be decisive in

raising self-esteem and are increasingly requested by patients and stimulated by the media, which has given emphasis to health being associated with beauty.[1] In this context, the presence of dental discoloration, whether intrinsic or extrinsic, has stimulated the development and improvement of bleaching techniques.[2,3]

Among the most common products for tooth bleaching are those based on hydrogen peroxide at 35%, which should be applied in dental offices under the supervision of dental professionals.[4]

Regardless of the technique or product used, the mechanism of action of bleaching agents is based on a complex oxidation process with release of reactive forms of oxygen, which penetrate through the pores of enamel rods and reach the dentin, causing the breakdown of organic molecules and resulting in lighter, smaller, and clearer compounds.[5]

Despite the good clinical results of bleaching techniques, their usage has been associated with decreased wear resistance of enamel and dentin, an increase in surface roughness, a decrease in microhardness, and major histomorphologic changes.[2,4] However, the wide variation in dosages used in bleaching treatments, along with the fact that most studies are carried out *in vitro,* makes it difficult to compare results and to extrapolate clinical conditions in which the teeth are continuously subjected to demineralization and remineralization cycles.

In situ studies represent an intermediate stage between laboratory experiments and clinical trials.[6] These are attempts to reproduce the process to be studied under the influence of biological factors, such as the protective effect of saliva.[6] Therefore, intraoral models allow some clinical reality, while preserving the sensitivity and accuracy of laboratory models, because the analysis can be performed outside the oral cavity.

Little is known about the effects of bleaching materials on early caries lesions because lesion diagnosis is difficult or uninterpretable by most dentists. Moreover, in an era when esthetics is of great value for people, many professional bleaching centers in malls may practice bleaching techniques without ensuring the adequacy of the oral environment. So, there is a need to know the effects of hydrogen peroxide in high concentrations on the microhardness and histomorphology of demineralized enamel.

Thus, because of the limited number of studies on the effects of bleaching agents on incipient lesions, it is important to evaluate the structural and morphologic aspects of enamel after the application of 35% hydrogen peroxide to demineralized enamel by introducing an intense cariogenic challenge. The null hypothesis tested was that 35% hydrogen peroxide would not affect the structural features and histomorphology of the enamel surface when subjected to cariogenic challenge.

MATERIALS AND METHODS

Preparation and Selection of Samples

This study was approved by the Ethics in Research Board (P. 2008-01237). Eleven volunteers between 19 and 26 years of age, who were aware of the experiment through the "letter informing the volunteer" and who signed a "term of consent," were selected After receiving all relevant information concerning the research, volunteers answered the health history questionnaire and were clinically examined for criteria of exclusion (Table 1).

Once selected, volunteers received a kit containing a toothbrush (Indicator Plus, Oral B, São Paulo, Brazil), a fluoridated toothpaste (Pro-Health, Oral B), dental floss (Essential Floss, Oral B), a vial with 20% sucrose, a case to store the unit during meals, and a list of guidelines.

Experimental units were obtained from the teeth of slaughtered cattle aged approximately 30 months.

Table 1: *Exclusion Criteria Applied to Select Volunteers for This Research*

EXCLUSION CRITERIA
Pregnant or nursing volunteers
Smoking volunteers
Volunteers with fixed or removable prostheses
Volunteers with braces
Volunteers with caries activities
Volunteers with periodontal disease
Take drugs that affect salivary flow (Antidepressants, narcotics and diuretics)
Presence of digestive disorders
Unable to attend pre-booked appointments for the laboratory procedures

The teeth were cleaned and cut into sections, resulting in 100 pieces measuring 4 × 4 × 2 mm. The fragments were ground flat and polished on a polishing unit (Aropol and Arotec SA Industria e Comercio Ltda, Cotia, Sao Paulo, Brazil) with the use of aluminum oxide sandpaper in grits of 600, 800, and 1200, at low speed under water cooling. Final polishing was performed with felt disks soaked in 1 μm diamond paste (Arotec SA Industria e Comercio Ltda) for five minutes. Between changes of sandpaper, the fragments were cleaned in an ultrasonic tank with distilled water (Branson 2200® **ultrasonic** cleaner, Shelton, CT, USA) to remove debris left by the sandpaper on the enamel surface.

The 100 polished fragments, none of which were found to have cracks, were subjected to initial readings of microhardness and received three indentations in the central region of each fragment with a Knoop-type indenter (HMV-2000, Shimadzu, Columbia, MD, USA) with a static load of 25 g for five seconds. Microhardness values were used to select 44 enamel blocks. For this, the general average was calculated for the 100 blocks (246), and extreme values (above and below this average) were excluded.

Making of Intraoral Palatal Devices

After preparation and selection of enamel blocks, the manufacture of intraoral palatal devices (IPDs) was started by molding the upper arch of each volunteer with alginate (Avagel, Dentsply Industria e Comercio Ltda, Petropolis, Rio de Janeiro, Brazil) and casting in dental stone (Durone IV, Dentsply Indústria e Comércio Ltda). Later, the palatal devices were made of acrylic resin containing four 4 × 4 × 3 mm niches, two on each side, which served to fix the blocks of bovine enamel.

Specimens were arranged in the device to avoid interference from site-specific factors. Thus, two blocks were placed in the anterior region of the device: one exposed to hydrogen peroxide at 35%, and one, a control, the same in the posterior region. Specimens were fixed on the intraoral device with sticky wax (Kota Industrial e Comercio Ltda, Sao Paulo, Brazil), positioned 1 mm below the surface of the resin. To enhance the accumulation of biofilm on the enamel samples, a polyethylene screen was fixed on the acrylic covering the specimens.

Treatment of Samples

Volunteers used the IPD for seven days for initial formation of the biofilm; subsequently they were instructed to apply a drop of 20% sucrose solution on the enamel, eight times a day, every two hours. After the dripping, the device remained at rest for five minutes before returning to the oral cavity, allowing the sucrose to spread on the biofilm. The first daily exposure to sucrose was at 8 AM and the last at 10 PM. A new sucrose solution was prepared every two days.

After seven days, samples were carefully removed from the IPD and were submitted to 35% hydrogen peroxide bleaching gel (Whiteness HP Maxx, FGM Produtos Odontológicos Ltda, Santa Catarina, Brazil). The product was handled according to the manufacturer's specifications (three drops of peroxide for each drop of thickener), and 0.06 ml of bleach was applied to each fragment, remaining in contact with the enamel for 15 minutes. This procedure was repeated three times in each session. Then, the samples were washed with distilled-deionized water to waste from the gel, carefully dried with absorbent paper, and placed back into their niches. Three bleaching sessions were held with seven days between sessions. Afterward, the samples were removed and were kept in deionized water with daily changes for seven days.

Knoop Microhardness

After the experimental procedures had been performed, the samples were subjected to a final enamel surface microhardness test. Three new indentations were made in the center of the sample, 100 μm away from the site of the initial microhardness reading. The average of the three indentations was used for statistical analysis.

The Knoop microhardness values were transformed into percentage of loss of hardness (% LH) obtained by the following formula:

$$\%LH = 1 - KHN(I) - KHN(F)/KHN(I) \times 100$$

where KHN(I) is the average of initial microhardness of the group, and KHN(F) is the average of final microhardness. The results obtained were submitted to analysis of variance at 5% (repeated measures ANOVA).

Scanning Electron Microscopy (SEM)

Twenty-two samples (11 in the control group and 11 in the bleached group) were fixed with double-sided carbon tape (Electron Microscopy Sciences 19034, Washington, DC, USA) on stubs and were sputter-coated with gold, using the vacuum camera (Balzers SCD 050 Sputter Coater, Balzers Aktiengesellschaft Union, Fürstentum, Liechtenstein, Germany), at a current of 45 mA for 160 seconds. Evaluation was

performed using a scanning electron microscope (JSM 5600, Joel Inc, Peabody, MA, USA) with magnifications of 1000× and 3000×, taking into account the enamel surface morphology. Representative areas were photographed for benchmarking.

Polarized Light Microscopy (PLM)

The remaining 22 samples were analyzed by PLM. They were longitudinally sectioned with double-faced diamond disks (7020 KG Sorensen Ind e Com Ltda, Barueri, SP, Brazil) to obtain two samples of each block of enamel. The samples were polished with aluminum oxide sandpaper in grits of 600 and 1200 and at low speed and were water cooled to obtain slices that were approximately 100 μm thick.

The specimens were placed on glass slides, immersed in distilled water, and covered with a coverslip. Analysis was performed using a polarized light microscope (Axiophot Zeiss DSM-940 A, Oberkochen, Germany) at magnifications of 25× and 100×. The images, taken by a digital camera attached to the microscope, were analyzed as to the shape and depth of lesions using AxioVision 4 software (Zeiss, Oberkochen, Germany).

RESULTS

Mean and standard deviation values of microhardness before and after bleaching are shown in Table 2. Results show that specimens of the control group and those of the bleaching treatment group had similar microhardness values in the initial period (p=0.7871). However, at the end of the experiment, a general decrease was seen in microhardness values, which was especially more significant in the bleached group (p=0.0069).

Statistical analyses of the data showed that the control group had a reduction of 19.2% for microhardness (submitted to cariogenic challenge), and the bleached group (submitted to cariogenic challenge and bleaching) had a reduction of 24.9% for microhardness.

Table 2: *Mean (Standard Deviation) of Knoop Hardness (KHN) Before and After Bleaching Treatment According to Experimental Groups**

Group	Mean (SD)	
	Initial	Final
Control	237.3 (17.9)^{Aa}	191.7 (15.8)^{Ba}
Bleached	238.9 (21.3)^{Aa}	179.4 (12.5)^{Bb}

* *Capital letters compare columns; lowercase letters compare lines.*

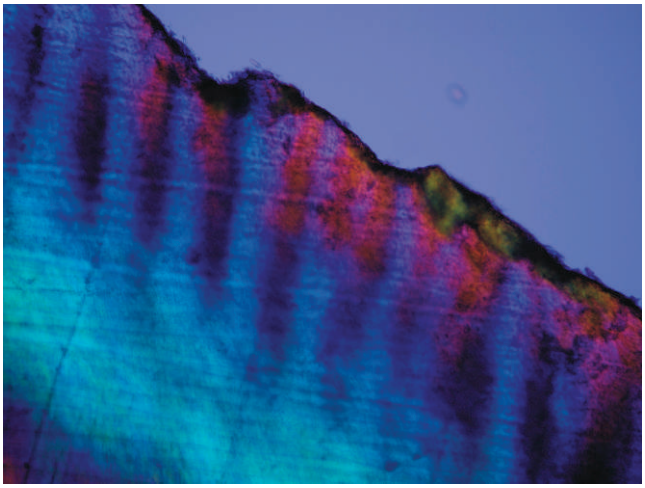

Figure 1. *Image from polarized light microscopy of the bleached group in a cut where the cavitated region represented by a more brownish region in the image can be clinically seen (100×).*

PLM analysis showed that both the bleached group (Figures 1 and 2) and the control group

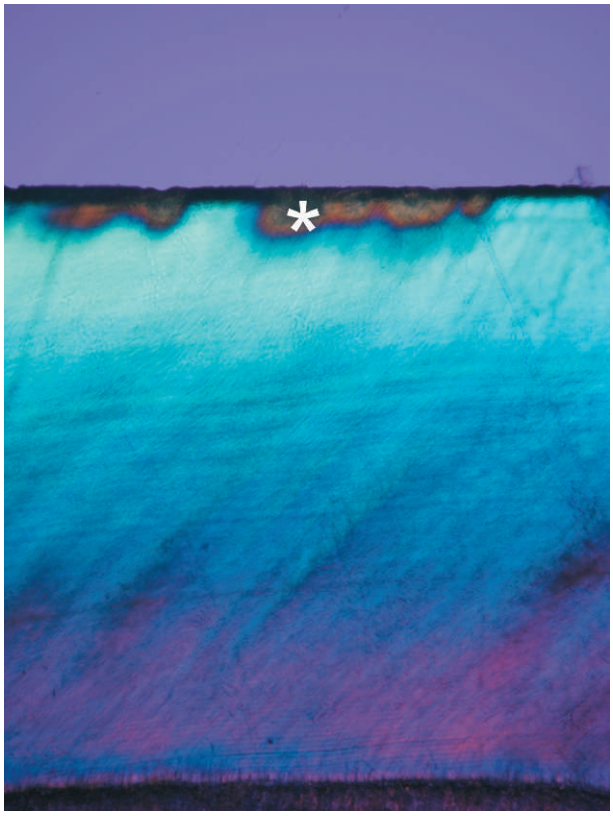

Figure 2. *Image from polarized light microscopy of the bleached group, showing an intense birefringence difference in enamel. The asterisk (*) shows areas of enamel demineralization (100×).*

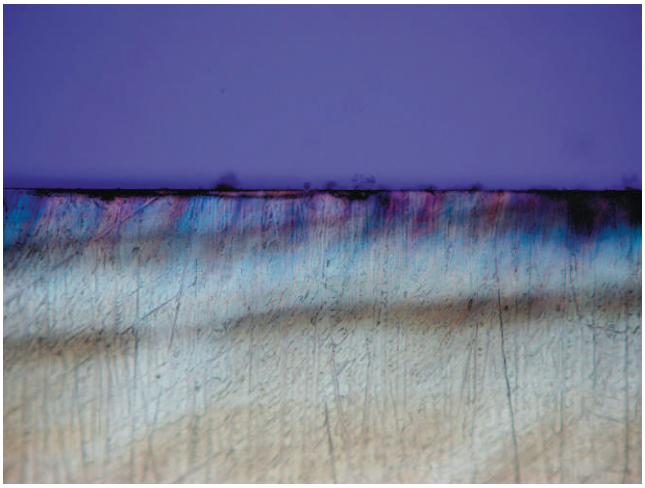

Figure 3. *Image from polarized light microscopy of a cut in the control group, with areas of subsurface demineralization (darkened region) (100×).*

(Figures 3 and 4) had areas of demineralization in all specimens, represented by regions of negative birefringence. This proves the effectiveness of the cariogenic challenge. However, it is worthwhile to

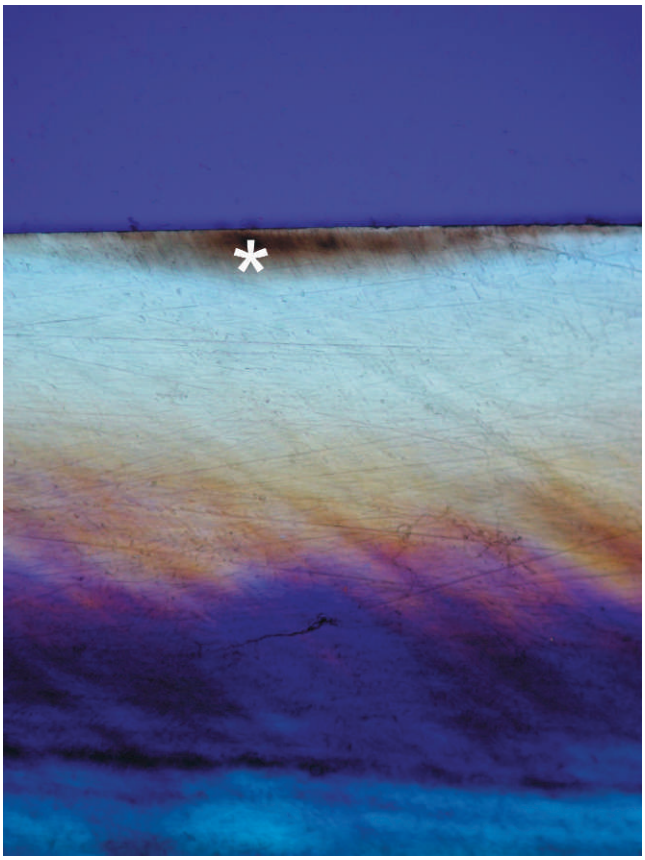

Figure 4. *Image from polarized light microscopy of a specimen of the control group, showing isolated areas of demineralization (darkened region). The asterisk (*) shows area of enamel demineralization (100×).*

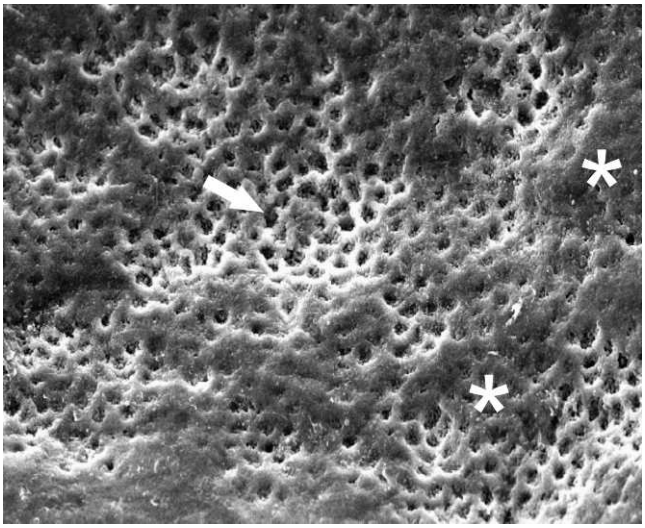

Figure 5. *Photomicrograph of a specimen of the control group showing areas of aprismatic layer removal and exposure of the prisms (→) and better conserved regions (*) (1000×).*

emphasize that histologic changes in the bleached enamel were sharper and reached most of the surface, causing cavitation in some specimens. The depth of demineralization varied from 58.85 μm to 103.52 μm in the control group (average, 74.89 μm) and from 63.71 μm to 132.06 μm in the bleached group (average, 88.13 μm). Surface analysis in scanning electron microscopy showed that the control group (Figures 5 and 6) and the bleached group (Figures 7 and 8) exhibited surface morphologic changes similar to dissolution of the central region of the prisms and increased surface porosity. Greater removal of the aprismatic layer was noted in the group exposed to hydrogen peroxide.

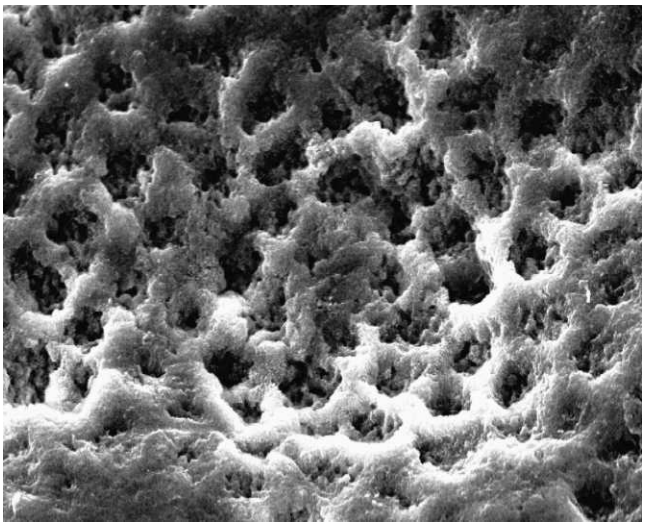

Figure 6. *Detail of morphologic changes. Removal of the aprismatic layer and removal of the center of the prisms can be seen (3000×).*

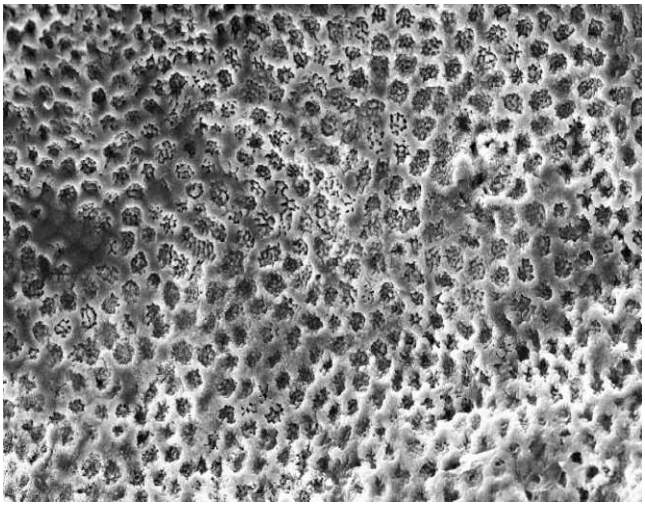

Figure 7. *Photomicrograph of a specimen of the bleached group, showing morphologic changes that are more homogeneous than in the control group and present on the whole surface (1000×).*

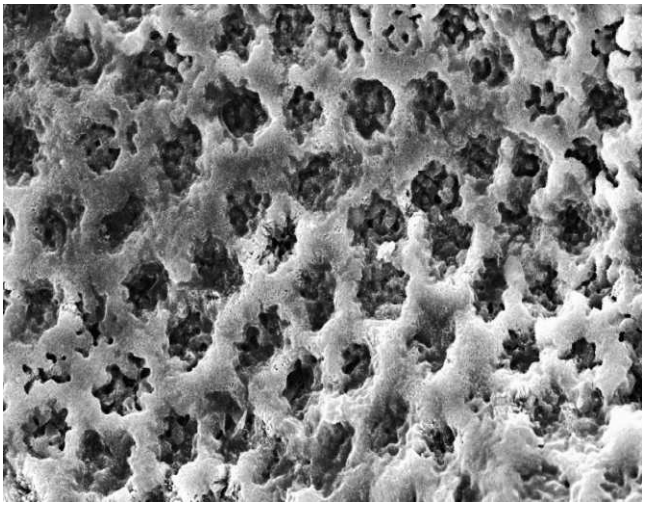

Figure 8. *Detail of morphologic changes observed in the bleached group. Despite the greater presence of demineralized areas, the pattern of changes was similar to that in the control group (3000×).*

DISCUSSION

In situ models provide conditions of the oral cavity and allow all factors related to the development of carious lesions to be present in the experiment. Thus, the dental substrate, the formation of plaque with cariogenic potential, the presence of carbohydrates, and the time are properly valued in the results.[6,7]

The accumulation of biofilm was achieved by fixing the specimens 1 mm below the level of the site, and by covering the sites with a plastic screen that allowed the formation of a uniform and thick bacterial plaque possessing great demineralization ability, consistent with regions of high accumulation of biofilm. This was associated with frequent dripping of 20% sucrose solution, which has been effective for the formation of carious lesions.[6,8–10]

Current results show that bleached and control groups experienced a significant decrease in microhardness, which was most pronounced in the bleached group. It is believed that in the control group, frequent exposure to sucrose caused a change in the microbial composition of the plaque, selecting for acidogenic and aciduria microorganisms; this brought the pH below the critical level, causing demineralization and consequently a reduction in microhardness.[11] Aires and others[8] showed that *Streptococcus mutans,* in the presence of sucrose, produce large quantities of extracellular polysaccharides, which are responsible for the increase in cariogenicity of the plaque.

SEM analysis showed that specimens from the control group had surface features similar to those observed in the bleached group, which were the surface changes described by Silverstone,[12] who reported the type 1 conditioning pattern with dissolution of the center of enamel prisms. This suggests that biofilm formed in the presence of 20% sucrose produces micromorphologic damage as great as, or more significant than, that caused by hydrogen peroxide. However, it is noteworthy that changes in the control group reached a smaller surface area, and that this most likely occurred as a result of the noninfluence of products from degradation of hydrogen peroxide.[10,11,13–15]

PLM analysis targeted primarily the qualitative evaluation of mineral loss or gain, allowing the ability to analyze the specimen in depth and to identify incipient enamel lesions.[9,16] Specimens from the control group had isolated areas of superficial demineralization that were less intense, but had a depth of lesion similar to that of bleached specimens. This emphasizes the effectiveness of the cariogenic challenge and the surface demineralizing action of peroxide. It is believed that the presence of highly reactive oxygen free radicals and prolonged contact with the bleaching agent of high concentration and low pH[17] are responsible for the increased sensitivity of the substrate to cariogenic challenge.

Because of close contact of the bleaching agent with the tooth structure, microscopic changes in the enamel surface have been reported. The acidic pH of bleaching agents, the time of application, and the composition of products are some of the factors discussed in the literature as being responsible for these superficial changes.[4,18,19]

In this context, Marson and others[20] found that Whiteness HP Maxx ranges in pH from 6 to 5 over time. This fact makes the enamel prone to decalcification, decreasing its microhardness.[21] It is known that peroxides can affect not only the surface but also the interprismatic and intraprismatic portions of the enamel, promoting mild deproteinization. Thus, any mineral element associated with enamel proteins is also removed, which would explain the loss of calcium and phosphorus in these areas, thus contributing to the occurrence of microstructural damage and possibly even to changes in microhardness.[22,23]

The most frequent alterations observed through SEM in the bleached group were removal of the aprismatic layer of enamel, exposure of prisms, and partial dissolution of this structure. These changes are potentially related to the cariogenic challenge and the great reactivity of oxygen.[13] In analysis through polarized light microscopy, surface demineralization, subsurface carious lesions, and cavitated regions were observed in the bleached group, confirming the potentiated action that hydrogen peroxide exerts on surfaces with an accumulation of cariogenic biofilm.

The noncavitated carious lesions observed through PLM showed a characteristic that is already well documented in the literature[16] in which two distinct zones are quite evident: the surface area represented by a relatively intact layer and the body of the lesion with intense demineralization observed as darkened regions.[12]

Throughout the present study, the specimens remained in the oral cavity in contact with saliva, which was not able to inhibit demineralization. Many studies suggest that the action of saliva may reverse mineral loss,[24,25] because stimulation of the flow of saliva increases the constituents, such as carbonic acid, hydrogen carbonate, hydrogen phosphate, calcium, and fluoride, which are associated with increased buffer capacity and maintenance of the balance between demineralization and remineralization phenomena.[26] However, frequent and substantial accumulation of bacterial plaque prevented direct contact between these constituents and the enamel surface.[27,28]

In this current study, bleaching gel was applied to the enamel before it was submitted to cariogenic challenge. After bleaching treatment on these carious lesions, a statistically significant loss of enamel microhardness occurred. The carious enamel is more susceptible to the demineralization effect of the bleaching gel because it becomes more porous (Figures 5 and 7). The current study simulated a dynamic process of demineralization/remineralization, hypothesizing that saliva would be able to mitigate the effects caused by 35% hydrogen peroxide; however, this effect was not observed. The interval of seven days may not have been sufficient to reverse the effects of the bleaching treatment.

After the bleaching cycle, the samples were removed from the palatal devices and were stored in deionized water for seven days. The hypothesis was that if the samples had remained in the oral environment for this same period of time, demineralization of the bleached samples could have been minimized.

PLM and SEM analyses showed that histomorphologic changes caused by frequent exposure to sucrose were possibly caused by changes in the microbial composition of the plaque and allowed an acid environment that led to demineralization, increased by the unspecific action of oxygen radicals released by the bleaching agent, and also by the low pH that the product presents. Given these findings, the influence of hydrogen peroxide and its action on ionic imbalance with mineral loss of the dental substrate are evident.[17,29]

As a consequence of the current results, the null hypothesis should be rejected because 35% hydrogen peroxide bleaching gel potentiated the structural and histomorphologic changes induced by the accumulation of biofilm on the enamel surface. However, additional studies should be conducted, including the use of other materials and bleaching techniques, to minimize doubts and conclusions on the effects of bleaching healthy or demineralized dental tissue. Only then will application of bleaching agents be proven safe and to have satisfactory cosmetic and biological results.

It is concluded that application of 35% hydrogen peroxide bleaching gel intensified changes in the bovine enamel surface that had been subjected to an *in situ* cariogenic challenge.

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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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Influence of Hydrogen Peroxide Bleaching Gels on Color, Opacity, and Fluorescence of Composite Resins

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AB Borges

Clinical Relevance

Bleaching therapies with 20% or 35% hydrogen peroxide gels influenced composite resin color and fluorescence. No influence on opacity was observed.

SUMMARY

The aim of the present study was to evaluate the effect of 20% and 35% hydrogen peroxide bleaching gels on the color, opacity, and fluorescence of composite resins. Seven composite resin brands were tested and 30 specimens, 3-mm in diameter and 2-mm thick, of each material were fabricated, for a total of 210 specimens. The specimens of each tested material were divided into three subgroups (n=10) according to the bleaching therapy tested: 20% hydrogen peroxide gel, 35% hydroxide peroxide gel, and the control group. The baseline color, opacity, and fluorescence were assessed by spectrophotometry. Four 30-minute bleaching gel applications, two hours in total, were performed. The control group did not receive bleaching treatment and was stored in deionized water. Final assessments were performed, and data were analyzed by two-way analysis of variance and Tukey tests ($p<0.05$). Color changes were significant for different tested bleaching therapies ($p<0.0001$), with the greatest color change observed for 35% hydrogen peroxide gel. No difference in opacity was detected for all analyzed parameters. Fluorescence changes were influenced by composite resin brand ($p<0.0001$) and bleaching therapy ($p=0.0016$) used. No significant differences in fluorescence between different bleaching gel concentrations were detected by Tukey test. The

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greatest fluorescence alteration was detected on the brand Z350. It was concluded that 35% hydrogen peroxide bleaching gel generated the greatest color change among all evaluated materials. No statistical opacity changes were detected for all tested variables, and significant fluorescence changes were dependent on the material and bleaching therapy, regardless of the gel concentration.

INTRODUCTION

Recent beauty standards place a high value on oral esthetics. Therefore, the materials and techniques of esthetic dental procedures, such as composite resin restorations and bleaching therapies, are under constant development.[1]

Tooth discoloration compromises esthetics and represents one of the major complaints of patients and a major reason for dental appointments. Bleaching procedures are nowadays largely employed by clinicians to reduce discolorations and improve or return tooth color. Dental bleaching is a conservative esthetic treatment that produces safe and acceptable results in a short period of time, assuming the technique and its indications are well known by clinicians.[2]

Because of the increased use of bleaching techniques, several studies have been performed to determine possible effects on dental structures. Although these studies on the effect of bleaching gels over soft and hard tissues are present in the literature, there is contradictive information regarding the interactions between esthetic restorative materials and bleaching gels, especially for those composed of high peroxide concentrations.[3–5] This contradiction is of concern because of the large number of people undergoing bleaching procedures who also present with esthetic restorations. Bleaching gels might compromise the composite resin's mechanical characteristics, surface hardness, and roughness,[6–8] as well as its optical characteristics such as translucency and color.[1,9,10]

The development of composite resins that esthetically mimic human enamel and dentin according to color, opacity, and fluorescence has been a significant contribution to restorative dentistry. Human teeth are characterized by a translucency variety, which is dependent on enamel thickness, enamel crystal structures, and different mineralization patterns. Translucency affects the restoration's esthetics and represents how intense the light penetrates into both the tooth structure and the restoration prior to being reflected to the external environment. Fluorescence is another important property to be reproduced by composite resins. When illuminated with a light source containing ultraviolet rays, such as sunlight, human teeth emit a bluish-white light perceptible to the naked eye.

It is known that dental materials' optical properties, translucency, opacity, and fluorescence are dependent on the composite resin's components.[10,11] Therefore, to better reproduce these properties, the composition of inorganic fillers has been studied by adding fluorescent particles and different sizes and shapes of fillers. The literature, however, does not present accurate information regarding different types of composite resins and their optical properties when submitted to bleaching therapies. Considering that bleaching therapies are often performed in patients presenting composite resin restorations, the aim of the present study was to evaluate the effect of bleaching treatments with 20% or 35% hydrogen peroxide gels on different types of composite resins regarding their color, opacity, and fluorescence.

METHODS AND MATERIALS

Seven composite resin brands, shown in Table 1, were tested. Two hundred ten specimens, 3-mm in diameter and 2-mm thick, were fabricated using a silicon matrix, with 30 specimens of each composite resin brand. All specimens were fabricated in shade A3. Specifications of tested composite resins are presented in Table 1.

Specimens were light activated for 40 seconds with 550 mW/cm^2 light intensity using an LED emitter (Schuster Comercio de Equipamentos Odontologicos Ltda, Santa Maria, RS, Brazil) and stored in Eppendorf vials with 2 mL of deionized water for 24 hours. Specimens were further polished with #4000 sandpaper using a circular polishing machine (Poliplan 2, Panambra, São Paulo, SP, Brazil).

The specimens of each composite resin group were then subdivided into three subgroups (n=10) according to bleaching therapy. Subgroup 1 received bleaching therapy with 20% hydrogen peroxide gel (Whiteness HP 20–FGM, Produtos Odontologicos Ltda, Joinville-SC, Brazil), subgroup 2 received bleaching therapy with 35% hydrogen peroxide gel (Whiteness HP 35–FGM, Produtos Odontologicos Ltda), and subgroup 3 (control) received no bleaching treatment but was immersed in deionized water during the entire research period.

Table 1: *Types of Composite Resin Tested in the Present Study*

Market Brand	Classification Based on Particles	Manufacturer	Composition
Admira	Microhybrid Ormocer	VOCO GMBH, Cuxhaven, Germany	Complex inorganic-organic copolymers tridimensionally polymerized (ormocers), aliphatic dimethacrylates; 56% of 0.7-μm fillers in volume
Amaris	Microhybrid	VOCO GMBH, Cuxhaven, Germany	Bis-GMA and UDMA 20-nm silica and 5- to 20-nm zirconia particles
Estelite Sigma	Microhybrid with spherical submicron fillers	Tokuyama Dental, Shibuya, Tokyo, Japan	Methacrylates and 82% by weight of silica-zirconia filler sized between 0.1 and 0.3 μm
Esthet X	Microhybrid	Dentsply, Petrópolis, RJ, Brazil	Bis-GMA, Bis-EMA, triethylene glycol dimethacrylate (TEGDMA), camphorquinone, stabilizers, tints, barium aluminofluoroborosilicate (BAFG) with silicone dioxide particles (1 μm) silica nanoparticles (0.04 μm)
Venus	Microhybrid	Heraeus Kulzer, Grüner, Germany	Bis-GMA and TEGDMA and submicron barium glass particles with mean size of 0.7 μm and filler volume greater than 78% by weight
Filtek Z 350	Nanofilled	3M ESPE, St Paul, MN, USA	Bis-GMA, Bis-EMA, UDMA, and TEGDMA; 20-nm nanosilica, and 5- to 20-nm zirconia nanoagglomerates
GrandioSO	Nanohybrid	VOCO GMBH, Cuxhaven, Germany	Bis-GMA, Bis-EMA, UDMA, and TEGDMA with 87% of filler by volume

Specimens were stabilized on a glass slide using utility wax. The bleaching gel was applied to completely cover the specimens' surface. Four 30-minute applications comprised the two-hour bleaching protocol. Bleaching gel was removed with an endodontic cannula between bleaching procedures, while the last application was rinsed with deionized water.

Two readings were performed: the baseline and the final. The baseline color, opacity, and fluorescence readings were performed 24 hours after the specimens' fabrication. The final readings were performed 24 hours after the bleaching therapy. Color changes (ΔE) for each treatment were assessed by variation of L* (ΔL*), a* (Δa*), and b* (Δb*) values, by subtracting the final data from baseline data. Readings were performed using a spectrophotometer CM-2600D (Konica Minolta, Osaka, Japan) calibrated for small samples reading (SAV). Color determination was performed following the CIE (Commission Internationale de l'Eclairage) L*a*b* model, by means of Spectra Magic NX software (Konica Minolta, Osaka, Japan), using standard illuminant D65 on reflectance mode.

The equipment was adjusted to perform three consecutive readings and to calculate the mean L*, a*, and b* values. For standardization, readings were performed with specimens over white and black epoxy resin backgrounds. ISO standard #2469 was followed during the measurements. The spectrophotometer was connected to an Acer Aspire 3624WXMi computer for data storage.

The statistical analysis was performed by two-way analysis of variance (ANOVA) and Tukey tests, with the level of significance at 5%.

RESULTS

For color analysis, two-way ANOVA detected significant differences for different bleaching therapies ($p<0.0001$). The type of composite resin had no statistical significance ($p=0.3006$). The bleaching therapy influence on color variations performed by Tukey test is shown in Table 2.

Opacity variations were not statistically influenced by composite resin type and bleaching therapy ($p=0.1410$ and $p=0.3872$, respectively). However,

Table 2: *Tukey Test Analysis, Mean Values, and Standard Deviation (±SD) of Color Variation (ΔE) According to Different Bleaching Therapies*

Bleaching Therapy	Mean	±SD	Homogeneous Groups[a]	
Control	1.90	1.35	A	
20%	2.50	2.43	A	
35%	3.44	1.77		B

[a] *Groups with same letter are statistically similar.*

Table 4: *Tukey Test Analysis, Mean Values, and Standard Deviation (±SD) of Fluorescence Variation According to Different Bleaching Therapies*

Bleaching Therapy	Mean	±SD	Homogeneous Groups[a]	
Control	0.70	1.52	A	
20%	1.38	1.61		B
35%	1.47	1.37		B

[a] *Groups with same letter are statistically similar.*

the interaction of both factors was statistically significant ($p=0.0316$).

Fluorescence changes were statistically influenced by composite resin type ($p<0.0001$) and bleaching therapy ($p=0.0016$). The interaction of both variables also influenced the fluorescence changes ($p=0.0137$). The Tukey test analyses for both variables are presented in Tables 3 and 4.

DISCUSSION

The effect of bleaching procedures on dental materials is important as this therapy is largely employed by clinicians, and patients usually present composite resin restorations. Recent composite resins mimic tooth color, opalescence, and fluorescence properties. Thus, it seems important to evaluate all of these properties on composite resins after bleaching therapies.

Table 3: *Tukey Test Analysis, Mean Values, and Standard Deviation (±SD) of Fluorescence Variation According to Different Composite Resin Types*

Composite Resin Types	Mean	±SD	Homogeneous Groups[a]		
Venus	0.42	1.66	A		
Grandio SO	0.80	2.15	A		
Amaris	0.96	0.70	A	B	
Estelite	1.05	1.17	A	B	C
Esthet X	1.11	1.18	A	B	C
Admira	1.90	1.39		B	C
Z350	2.05	1.51			C

[a] *Groups with same letter are statistically similar.*

The influence of bleaching procedures on total color alteration (ΔE values) of composite resins was observed in previous studies.[5,9] In this study, the multiple comparisons test revealed that 35% hydrogen peroxide bleaching therapy resulted in greater color variations compared with both 20% hydrogen peroxide and control groups, while no differences were detected between 20% hydrogen peroxide and the control group. This leads to the assumption that not only does the hydrogen peroxide influence color change, but the concentration of hydrogen peroxide gels will also influence the degree of color change. Although this study detected color changes with hydrogen peroxide, there are studies that observed similar alterations with other types of bleaching agents, such as carbamide peroxide.[12]

This study's observations of color changes contradict previous studies, which reported either color stability[13] or differences in color changes among different materials.[9,10,14] The type of composite resin used has been reported as a factor influencing color changes after bleaching therapies due to its organic matrix[9] or mainly to its inorganic composition.[9–11,15,16] However, in the present study, although the tested composite resins presented different fillers, this difference did not influence the color change. As the organic matrix of the tested materials was very similar, this matrix might also play a role on how color changes or stabilizes after bleaching therapies. Moreover, the degree of conversion of the composite resin matrix to polymer may influence color stability,[5] because non-reacted monomers could be attacked and degraded by bleaching solutions.

The following main factors might be responsible for color changes: first, the oxidation of readily accessible surface pigments; second, the oxidation

of amine compounds, which are responsible for composite resin color stability[4,5]; and third, the composite surface microcrack areas.[9] The microcracking areas are related to different filler components of composite resins,[17] and this fact might be related to instant or future color changes and surface degradation of composite restorations.

Because of their filler characteristics, nanofilled composite resins are reported to present greater color stability in comparison with microfilled composite resins when submitted to bleaching treatment.[10] No microfilled composite resin was tested in the present study, and no differences were detected among nanofilled, microhybrid, and nanohybrid composite resins regarding color and opacity variations.

The optical properties of composite resins, such as color, opacity, and fluorescence, are reported to be directly influenced by their different compositions. The filler type, size, concentration, and morphological distribution into resin matrix influence light propagation and perception.[10,11,15,16]

No significant difference was detected among the tested materials with regard to their opacity variations, contradicting reported greater translucency characteristics after bleaching procedures.[5] This greater translucency was, however, not of clinical significance.[5] The opacity is related to the filler quantity and distribution, and bleaching therapies may not alter these particles. Surface gloss is reported to deteriorate after bleaching.[12,18] As gloss is related to light reflection off of the material's surface, this might influence the optical perception of these materials.[18] However, this association was not detected in a previous study assessing gloss and color of composite resins,[12] and as gloss was not a studied variable in this study, there was not a way clearly to determine this interaction between gloss and opacity or color.

Important opacity information to be addressed is the detection of an interaction between bleaching therapy and composite resin type with the present data. This means specific materials under specific bleaching regimens might behave differently, although no individual statistical differences were detected. This information should be further investigated and might be related to differences of inorganic components within the tested composites.[10,11,15,16]

For the fluorescence analysis, significant differences were detected for the assessed variables, the bleaching therapy, and the type of composite resin, as well as for the interaction of both. The composite resin brand Z350 presented the greatest fluorescence variation. This fact might be due to its composition of nanosilica and nanoagglomerated zirconia fillers. The fluorescence of restorative materials has been reported to be nonstable with aging[19]; thus, bleaching therapies might speed the aging process of restorative materials, and this may explain the observed results. Fluorescence has been shown to be dependent on the type of tested materials and, consequently, on their composition.[15,16]

The correlation between opacity and fluorescence was not the purpose of this study, and there may not be an association between the two. The opalescence of composite resin is reported to be considerably increased by the addition of TiO_2 nanofillers, while the fluorescence does not change.[11]

From a clinical standpoint, it is difficult to determine the importance of the detected statistical differences. As teeth undergo a lightening process by the bleaching therapy, the changes in composite resin color might follow the color changes of tooth structures. Thus, clinical color discrepancies after bleaching procedures are dependent on both composite resin and tooth color variations. Some reports disagree on the ΔE values that would represent perceptible clinical color changes, varying from 1 to 2[5] and greater than 3[20] or 3.7.[21] Considering this information, only subgroup 2 (35% hydrogen peroxide) would result in clinically perceptible color changes.

Although color changes detected immediately after bleaching therapies might not be clinically observed, bleaching gels lead to composite resin surface degradation and microcracks.[9] This may influence the clinical acceptability of composite resin restorations in a long-term evaluation. In a microhardness assessment study,[22] it was suggested that the oxidation promoted by hydrogen peroxide affects more than the subsurface of the restoration, thus compromising not only the restoration surface but also the body of the restoration.

The controversies of several studies might be related to differences in employed methodologies, differences of restorative materials, variations of concentration, bleaching gels presenting different pH, different periods of application of bleaching gels, and specimen storage methods.[1,13,23–25] As bleaching therapies and composite resins are under constant development, it is important to test them frequently.

Based on recent reports, it is important that clinicians have knowledge about the optical changes

of composite resins submitted to bleaching therapies when using or recommending office bleaching with hydrogen peroxide. This will provide an efficient and safe treatment. Patients should also be informed that bleaching procedures in the presence of an esthetic filling might speed its aging process or require filling substitution because ofits color variations.

CONCLUSIONS

- Thirty-five percent hydrogen peroxide gel produced the greatest color changes on the tested materials.
- No changes of opacity were detected with the tested bleaching protocols and materials.
- Fluorescence changes were statistically influenced by tested materials and bleaching protocols, regardless of the bleaching gel concentrations.

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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The Effects of Surface Roughness of Composite Resin on Biofilm Formation of *Streptococcus mutans* in the Presence of Saliva

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Clinical Relevance

Surface topography (size and depth of depressions) plays a more important role than surface roughness in biofilm formation of *Streptococcus mutans* in the presence of saliva.

SUMMARY

The purpose of this study was to investigate the effects of surface roughness of resin composite on biofilm formation of *Streptococcus mutans* in the presence of saliva. To provide uniform surface roughness on composites, disks were prepared by curing composite against 400-grit silicon carbide paper (SR400), 800-grit silicon carbide paper (SR800), or a glass slide (SRGlass). The surface roughness was examined using confocal laser microscopy. For biofilm formation, *S. mutans* was grown for 24 hours with each disk in a biofilm medium with either glucose or sucrose in the presence of fluid-phase or surface-adsorbed saliva. The adherent bacteria were quantified via enumeration of the total viable counts of bacteria. Biofilms were examined using scanning electron microscopy. This study showed that SR400 had deeper and larger, but fewer

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depressions than SR800. Compared to SRGlass and SR800, biofilm formation was significantly increased on SR400. In addition, the differences in the effect of surface roughness on the amount of biofilm formation were not significantly influenced by either the presence of saliva or the carbohydrate source. Considering that similar differences in surface roughness were observed between SR400 and SR800 and between SR800 and SRGlass, this study suggests that surface topography (size and depth of depressions) may play a more important role than surface roughness in biofilm formation of *S. mutans*.

INTRODUCTION

Although the use of resin composites in restorative dentistry is common, there are still several drawbacks to their use, such as susceptibility to plaque accumulation.[1] The resultant increase in plaque retention places patients at higher risk for secondary caries adjacent to the composites.

The major causative microorganism in the pathogenesis of dental caries in humans is *Streptococcus mutans*.[2] Adhesion and/or biofilm formation by *S. mutans* is mediated by sucrose-dependent and sucrose-independent mechanisms.[3] Sucrose-independent binding is mediated by several surface adhesins that can bind to salivary components formed on the teeth in the absence of sucrose.[4] In addition, saliva can mediate the aggregation of *S. mutans* by interaction with the cell surface adhesin (antigen I/II family) of *S. mutans* in the fluid phase.[3] On the contrary, sucrose-dependent adherence is mediated by glucan binding proteins and water-insoluble glucans produced from sucrose by glucosyltransferase enzymes (GTFs).[5] In particular, the formation of glucan matrices can enhance the virulence of the biofilm through increased total biomass and acidogenicity.[6,7]

Surface roughness has been reported to play a prominent role in biofilm formation of oral bacteria.[8,9] The effects of surface roughness on biofilm formation can be explained by the fact that a rough surface can function as a buffer against shear force and can increase the area available for biofilm formation.[9] On the contrary, other studies have reported insignificant relationships between the surface roughnesses of dental materials and the amount of biofilm formation.[10,11] These contradictions mainly may be due to the fact that the surface roughness of the dental materials was not carefully controlled. In most studies, the surfaces were roughened using various polishing techniques with rotating burs or sandpapers,[12–14] resulting in irregular surface roughness, because the time and manual pressure were difficult to control. In addition, the relationship between surface roughness and biofilm formation on composite surfaces can vary because the original differences in surface properties between the dental materials could be masked by the presence of saliva[10,15] or extracellular glucans produced by *S. mutans*.[11]

If the differences in surface roughness, saliva, and extracellular glucans are carefully controlled, the relationships between the surface roughness and biofilm formation of *S. mutans* may be more clearly defined. The purpose of this study was to investigate the effects of surface roughness of composites on biofilm formation of *S. mutans* with different carbohydrate sources in the presence of saliva. The hypothesis of this experiment was that the surface roughness of composite would not affect the biofilm formation of *S. mutans*.

MATERIALS AND METHODS

Materials

The nanofilled composite, Filtek Z350 (A2 shade, 3M ESPE, St Paul, MN, USA) was used in this study. To obtain uniform surface roughness, Teflon molds (9.0 mm in diameter and 1.0 mm in thickness) were placed on silicon carbide (SiC) papers or a glass slide. For the roughest surface group (SR400), disks were prepared on 400-grit SiC paper (Deerfos, Inchon, Korea). Disks were prepared on a glass slide for the smoothest surface group (SRGlass) and on 800-grit SiC paper (Deerfos) for the intermediately rough surface group (SR800). Experimental materials were placed into the hole in the mold until they were flush with the top of the template. A glass slide was placed on top, pushed down to assure flat dorsal surfaces, and then gently removed. The materials were handled according to the manufacturer's instruction and light cured for 40 seconds at 1000 mW/cm^2 using a LED light curing unit (Freelight2, 3M ESPE).

Measurements of Surface Roughness

The surface roughness of each sample was measured using confocal laser scanning microscopy (Axiovert 200M, Carl Zeiss, Thornwood, NY, USA). The multi-argon laser emits light with a wavelength of 633 nm, and it allows the calculation of the arithmetic mean surface roughness from a mean plane within the sampling area (245 $\times$ 245 $\times$ 60 μm). Surface roughness readings were performed on five different areas of each disk.

Saliva Collection

Unstimulated whole saliva (UWS) was collected from healthy volunteers who had no acute dental caries or periodontal lesions. Saliva collection was routinely performed between 9:00 AM and 11:00 AM to minimize the effects of diurnal variability on salivary composition. The saliva sample was centrifuged at 3500*g* for 10 minutes to remove any cellular debris, and the resulting supernatant was used after filter-sterilization through a Stericup & Steritop (Millipore, Billerica, MA, USA).

Biofilm Formation

Overnight cultures of *S. mutans* (serotype c UA 159) were transferred to a prewarmed brain heart infusion (BHI) broth and grown at 37°C in a 5% CO_2 aerobic atmosphere to the late exponential phase ($OD_{600} = 0.5$, approximately 6.5×10^7 colony forming units/mL). The cultures were then diluted 1:100 in a prewarmed biofilm medium (BM) with either 20 mM glucose (BM-glucose) or 20 mM sucrose (BM-sucrose) as a carbohydrate source as previously described.[16] Biofilm assays were performed using three different UWS treatments: fluid-phase UWS (F-UWS), surface-adsorbed UWS (S-UWS), or no UWS treatment (control).

For experiments with F-UWS, 2.0 mL of the diluted cell cultures were inoculated into the well containing a disk, concurrent with 200 μL of UWS. For the experiments with S-UWS, each disk was conditioned with 1.0 mL of UWS in the well at 37°C for 2 hours with gentle shaking, followed by three washes with phosphate buffered saline (PBS). After air drying for 30 minutes, 2.0 mL of the diluted cell cultures (without UWS) were inoculated into the well containing the disk. Two milliliters of cell suspension were inoculated to wells containing a disk without any UWS treatment for control. Biofilms were allowed to form during incubation at 37°C in a 5% CO_2 for 24 hours. The culture medium was then decanted, and the disks were washed twice with 1.0 mL sterile PBS to remove planktonic and loosely bound cells. Each disk was transferred to a conical tube containing 3 mL PBS. The adherent bacteria were detached via sonication using four 30-second pulses at 25 W with three 30-second intermittent cooling stages in a chilled ice box. The cell suspensions were serially diluted, plated on BHI agar, and incubated at 37°C in a 5% CO_2 atmosphere for two days before the colonies were counted. Colony counts were expressed as colony forming units per unit area (cm^2). All assays were performed in duplicate and independently repeated six times (n=6).

Microscopic Image

Biofilms that were examined using scanning electron microscopy (SEM), were grown as described above and analyzed at 1000× using a S-4700 microscope (Hitachi, Tokyo, Japan).

Statistical Analysis

Factorial analysis of variance (ANOVA) was used to analyze the amount of biofilm cells with respect to the surface type and UWS treatment. Multiple comparisons were performed with *t* tests using the Bonferroni correction to compare differences between the groups. Surface roughness was analyzed using one-way ANOVA, followed by Tukey honestly significant difference test. All values were considered significant when $\alpha<0.05$.

RESULTS

Comparison of Surface Roughness

There were significant differences in surface roughness between the groups. As expected, the decreasing order of surface roughness was SR400 (1.27 ± 0.06 μm), SR800 (0.80 ± 0.08 μm), and SRGlass (0.29 ± 0.05 μm) (SR400 > SR800 > SRGlass, $p<0.05$). However, the mean variation in surface roughness in each group was very small (less than 0.08 μm), indicating that uniform surface roughness was obtained. The SEM images showed very smooth appearances without any defects on SRGlass (Figure 1G,H,I). In contrast, the other groups showed rough surfaces with various sizes of depressions. SR400 showed deeper and larger depressions (Figure 1A,B,C) than did SR800 (Figure 1D,E,F) which showed more abundant depressions than did SR400.

Biofilm Formation in the Presence of Glucose

When glucose was used as the sole carbohydrate source, both F-UWS and S-UWS significantly suppressed biofilm development by *S. mutans*, compared to that in the control group (Table 1). In addition, S-UWS resulted in greater inhibition of biofilm formation than did F-UWS (no UWS treatment > F-UWS > S-UWS). There were also significant differences in biofilm development by *S. mutans* according to the surface type. Compared to SRGlass and SR800, biofilm formation was significantly enhanced on SR400 in BM-glucose (SRGlass, SR800 < SR400).

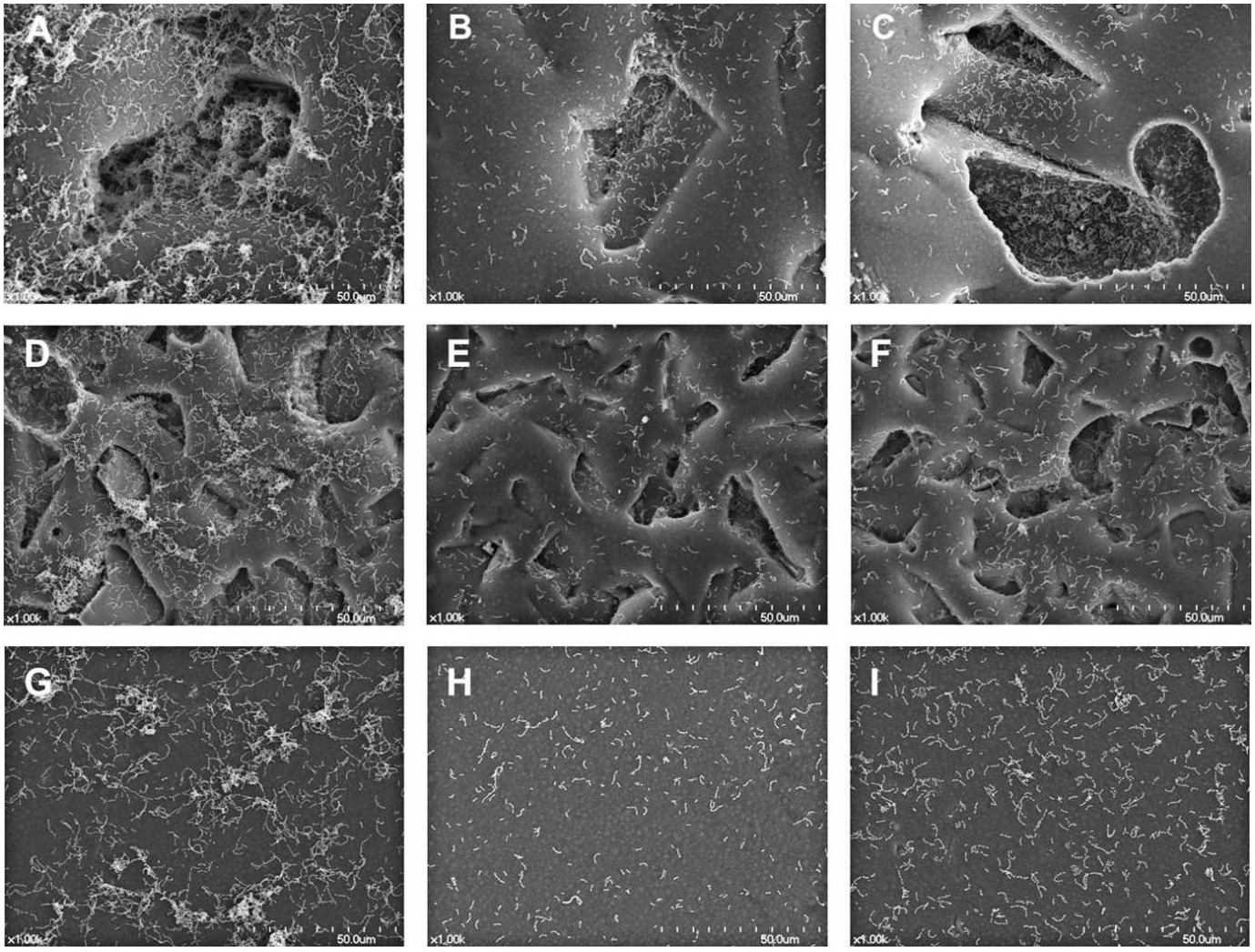

Figure 1. *Scanning electron microscopic images of biofilms of* Streptococcus mutans *formed on various composite surfaces in the presence of glucose. (A): Composite surface prepared against 400-grit silicon carbide (SiC) paper with no saliva treatment. (B): Composite surface prepared against 400-grit SiC paper with surface-adsorbed saliva. (C): Composite surface prepared against 400-grit SiC paper with fluid-phase saliva. (D): Composite surface prepared against 800-grit SiC paper with no saliva treatment. (E): Composite surface prepared against 800-grit SiC paper with surface-adsorbed saliva. (F): Composite surface prepared against 800-grit SiC paper with fluid-phase saliva. (G): Composite surface prepared against a glass slide with no saliva treatment. (H): Composite surface prepared against a glass slide with surface-adsorbed saliva. (I): Composite surface prepared against a glass slide with fluid-phase saliva. SR400 showed deeper and larger depressions (Figure 1A,B,C) than did SR800 (Figure 1D,E,F) which showed more abundant depressions than did SR400. (Data presented here are representative of three independent experiments)*

Biofilm Formation in the Presence of Sucrose

Biofilm patterns in BM-sucrose were different from those in BM-glucose (Table 2). Neither UWS treatments inhibited biofilm development in BM-sucrose. In addition, F-UWS significantly promoted biofilm development on each composite surface. There were also significant differences in biofilm development by *S. mutans* according to the surface type. Similar to biofilm patterns in BM-glucose, *S. mutans* formed more biofilms on SR400 than it did on SR800 or SRGlass in the presence of sucrose.

Comparison of Microscopic Images

Biofilms observed using SEM were consistent with the quantitative biofilm assays. Biofilm development was significantly inhibited by both UWS treatments in BM-glucose. Adherent cell clusters were shown in the absence of both UWS phases (Figure 1A,D,G) relative to the short scattered chains and microcolonies on the same surfaces in the presence of S-UWS (Figure 1B,E,H) or F-UWS (Figure 1C,F,I). There were also significant differences according to the surface type. In particular, there was an evident

Table 1: *Biofilm Formation by* Streptococcus mutans *on Various Surfaces in the Presence of Glucose for 24 Hours (The Amounts of Bacteria Were Expressed as Colony Forming Units Per Unit Area ($\times 10^7$ CFU/cm^2)*

Saliva Treatment	Surface Type			Significance*
	SR400 (Mean ± SD)[a]	SR800 (Mean ± SD)[b]	SRGlass (Mean ± SD)[c]	
No treatment control	10.65 ± 3.4	8.09 ± 3.01	7.56 ± 1.82	SR400 > SR800, SRGlass control > fluid-phase > surface-adsorbed
Surface-adsorbed	3.37 ± 1.57	2.03 ± 0.63	1.42 ± 0.75	
Fluid-phase	5.54 ± 1.27	4.35 ± 1.50	2.72 ± 0.65	

[a] *The composite surface prepared against 400-grit SiC paper.*
[b] *The composite surface prepared against 800-grit SiC paper.*
[c] *The composite surface prepared against a glass slide.*
* *Multiple comparisons were performed by t-tests using the Bonferroni correction at a significant level of $\alpha=0.05$.*

difference in biofilm formation between SR400 and SRGlass. Abundant cell aggregations were observed in deep and large depressions of rough surfaces (Figure 1A,B,C), while sporadic single cell chains or small cell aggregations were found on smooth surfaces (Figure 1G,H,I). In BM-sucrose, *S. mutans* formed superior biofilms compared to those formed in BM-glucose (Figure 2). Thick cell aggregates were found in abundant polysaccharide matrices, making it difficult to observe differences in microscopic characteristics among the specimens.

DISCUSSION

A number of studies have investigated the amount of biofilm formation on various materials with different surface roughness. In most of those studies, the surfaces of dental materials were subjected to various polishing techniques using rotating burs or sandpapers in order to create differences in surface roughness.[12-14] However, despite using the same polishing tools, heterogeneous surfaces on the specimens themselves and even among specimens in the same group occurred if the time and manual pressure required to polish the surface were not adequately controlled. In contrast to previous studies, we controlled surface roughness by curing the composite material against a glass slide or SiC papers that had been manufactured with homogeneous abrasives. As a result, three different surfaces with uniform surface roughnesses (less than 0.08 μm of the mean variation) were obtained and confirmed using confocal laser scanning microscopy. The three different surfaces were chosen to produce groups that had surface roughness that varied by more than 0.4 μm because more minor variations in surface roughness (less than 0.2 μm) have been shown to have no significant effect on bacterial adhesion.[9,17]

Table 2: *Biofilm Formation by* Streptococcus mutans *on Various Surfaces in the Presence of Sucrose for 24 Hours (The Amounts of Bacteria Were Expressed as Colony Forming Units Per Unit Area ($\times 10^8$ CFU/cm^2)*

Saliva Treatment	Surface Type			Significance*
	SR400 (Mean ± SD)[a]	SR800 (Mean ± SD)[b]	SRGlass (Mean ± SD)[c]	
No treatment control	3.30 ± 0.70	2.59 ± 0.73	2.02 ± 0.76	SR400 > SR800, SRGlass fluid-phase > control, surface-adsorbed
Surface-adsorbed	2.90 ± 1.42	2.24 ± 0.86	2.06 ± 0.78	
Fluid-phase	5.82 ± 1.83	5.01 ± 2.13	3.89 ± 1.09	

[a] *The composite surface prepared against 400-grit SiC paper.*
[b] *The composite surface prepared against 800-grit SiC paper.*
[c] *The composite surface prepared against a glass slide.*
* *Multiple comparisons were performed by t-tests using the Bonferroni correction at a significant level of $\alpha=0.05$.*

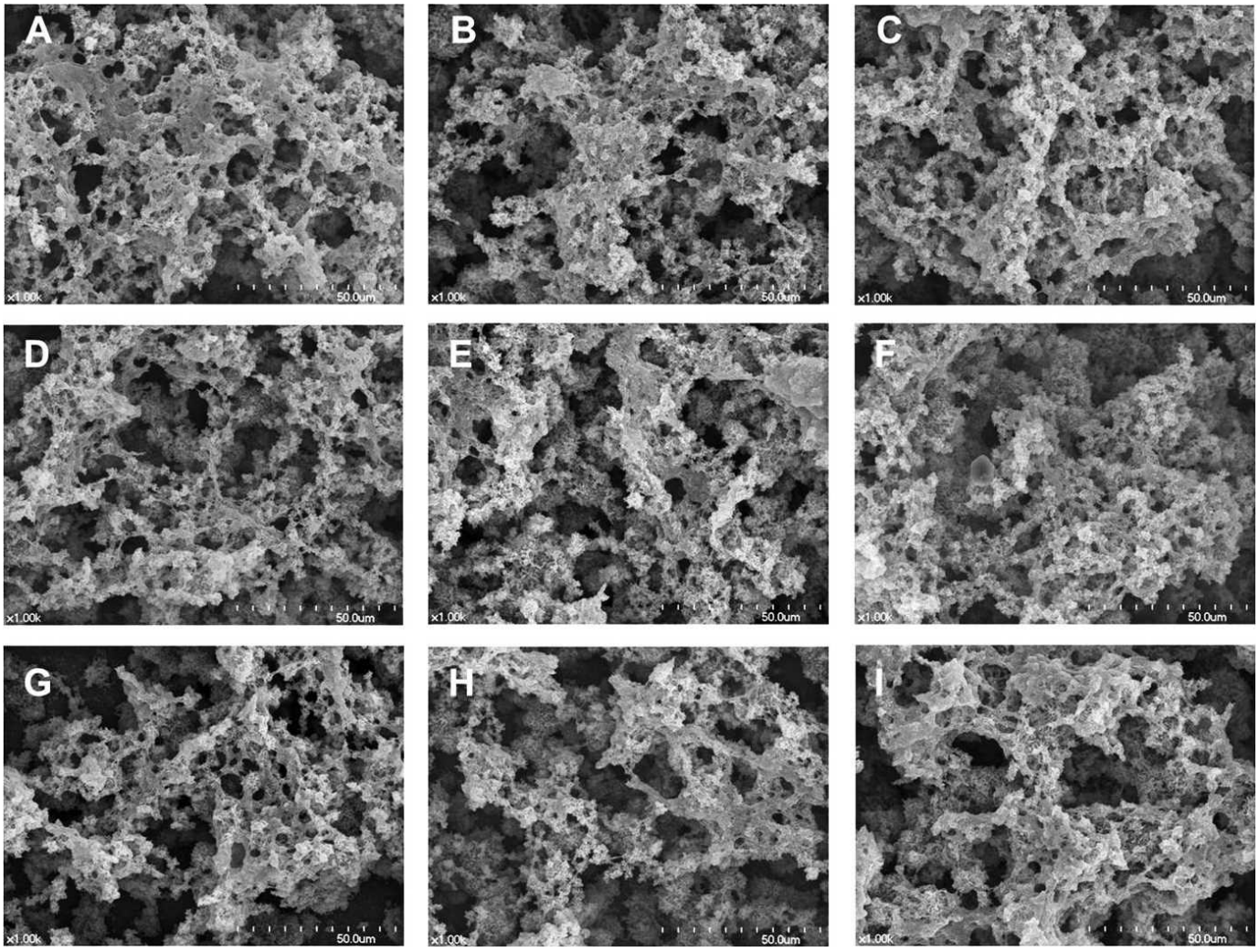

Figure 2. *Scanning electron microscopic images of biofilms of* Streptococcus mutans *formed on various composite surfaces in the presence of sucrose. (A): Composite surface prepared against 400-grit silicon carbide (SiC) paper with no saliva treatment.(B): Composite surface prepared against 400-grit SiC paper with surface-adsorbed saliva. (C): Composite surface prepared against 400-grit SiC paper with fluid-phase saliva. (D): Composite surface prepared against 800-grit SiC paper with no saliva treatment. (E): Composite surface prepared against 800-grit SiC paper with surface-adsorbed saliva. (F): Composite surface prepared against 800-grit SiC paper with fluid-phase saliva. (G): Composite surface prepared against a glass slide with no saliva treatment. (H): Composite surface prepared against a glass slide with surface-adsorbed saliva. (I): Composite surface prepared against a glass slide with fluid-phase saliva. (Data presented here are representative of three independent experiments)*

Biofilm development by *S. mutans* was significantly inhibited by both F-UWS and S-UWS in BM-glucose, with greater inhibition produced by S-UWS (Figure 1; Table 1). This indicates the different roles of UWS in adhesion of *S. mutans* according to its phase. S-UWS may inhibit adhesion of *S. mutans* by decreasing the surface free energy of the underlying material.[9] Surface modification due to saliva-coating may reduce the strength of bacterial adhesion to the substratum, resulting in a decrease in the amount of adherent bacteria. F-UWS may inhibit adhesion of *S. mutans* in a different way. Saliva has been shown to mediate the aggregation of *S. mutans* through interaction with the cell surface adhesin (antigen I/II family) of *S. mutans* in the fluid phase.[3] Bacterial aggregation induced by the interaction of F-UWS with *S. mutans* may facilitate bacterial clearance from surfaces during washing procedures, which may reduce the adhesion of *S. mutans* to the underlying surfaces. The different roles of S-UWS and F-UWS can be partially explained by the fact that the cells have an opportunity to adhere directly to the surfaces without interference of the saliva-coating in the presence of F-UWS.

In BM-glucose, there were significant differences in biofilm development by *S. mutans* according to the surface type (Table 1), but not by the presence of F-UWS or S-UWS. This demonstrates that the pres-

ence of F-UWS or S-UWS may not significantly modify the effects of surface roughness on the biofilm formation of *S. mutans*.

In BM-glucose, biofilm formation of *S. mutans* was significantly promoted on SR400, compared to that on SR800 or SRGlass. Although not significantly different, biofilm formation tended to increase on SR800 compared to that on SRGlass. The differences in biofilm formation of *S. mutans* between groups can be difficult to explain by the differences in surface roughness, because similar differences in surface roughness were observed between SR400 and SR800 and between SR800 and SRGlass. Instead, the differences in biofilm formation can be explained by the differences in surface topography between the groups. SR400 had deeper and larger depressions on its surface than did SR800 (Figure 1), which may provide more favorable places for bacterial colonization and biofilm formation due to the prevention of the dislodgement of bacterial colonies. Although SR800 showed more abundant depressions than did SR400, the shallower and smaller depressions may provide less favorable places for biofilm formation. This may explain the insignificant differences in the amounts of biofilm formation between SR800 and SRGlass. These results suggest a possibility that surface topography may play a more important role in biofilm formation of *S. mutans* than surface roughness.

In contrast to the biofilm patterns in BM-glucose, neither UWS treatment inhibited the biofilm formation ability of *S. mutans* in BM-sucrose. This may be due to the production of water-insoluble glucans by *S. mutans* in a sucrose-containing medium. One of the most important virulence factors of *S. mutans* is its production of extracellular polysaccharides (glucans) from sucrose via GTFs.[18] The glucans produce extracellular layers that promote adhesion and biofilm formation, independent of the presence of saliva. This was confirmed by SEM examinations. Biofilms in BM-sucrose showed thick cell aggregates around an enriched polysaccharide matrix, regardless of the presence of saliva (Figure 2). In addition, F-UWS significantly promoted the biofilm formation of *S. mutans* in BM-sucrose (no UWS treatment, S-UWS < F-UWS). This difference may be explained by the interactions between F-UWS and GTFs. In the presence of sucrose, saliva has been shown to promote the uptake of GTFs and to enhance glucan synthetic activities due to the aggregation of GTFs.[19] F-UWS in the media can contact GTFs present around *S. mutans* cells, which may promote biofilm formation. In the case of S-UWS, however, a portion of the salivary components can contact GTFs only on the surface. This may be the reason for reduced biofilm formation in the presence of S-UWS compared to that in the presence of F-UWS. These results may be partially attributed to the effects of enzymatically active GTFs normally present in UWS, which can assist the glucan synthesis of cell-associated GTFs derived from *S. mutans*.[20]

Similar to biofilm patterns in BM-glucose, there were also significant differences in biofilm development by *S. mutans* according to the surface type (Table 2). *S. mutans* formed more biofilms on SR400 than they did on SR800 or SRGlass. In the presence of sucrose, *S. mutans* cells mainly adhere to the surfaces using glucans synthesized by GTFs derived from cell surfaces. Therefore, surface irregularities, specifically larger and deeper depressions, may provide a greater area for glucan accumulation. This drives further development of bacterial colonization and protects bacteria against shear forces during their initial reversible binding, resulting in irreversible and stronger attachment. These results also indicate that surface topography may be an important factor even in the sucrose-dependent binding of *S. mutans* to the underlying surfaces.

It should be noted that in the human oral cavity, complex interactions of a variety of species of oral bacteria with different adhesive and physiologic capacities influence the formation of biofilms on various surfaces. Therefore, *in vitro* experiments do not reflect the complex microbial community found in the oral cavity, although this study allows for determination of the overall effect of surface roughness on microbial physiology and incorporates specific elements associated with the formation of cariogenic biofilms, such as saliva, sucrose, and culture of a proven virulent organism. Further *in vivo* investigation shall elucidate the biologic effects of surface topography on complex bacterial ecology, which will advance our understanding of the biomechanism of natural biofilm formation in the human oral cavity.

CONCLUSIONS

Understanding the relationships among surface roughness, saliva, and biofilm formation of *S. mutans* is important in preventing secondary caries around composite restorations. In this study, influences of surface roughness on biofilm development by *S. mutans* were analyzed in the presence of saliva. Our results showed that surface topography may play a more important role in biofilm formation of *S. mutans* than surface roughness.

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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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Evaluation of Outgassing, Tear Strength, and Detail Reproduction in Alginate Substitute Materials

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P Beck • LC Ramp • JO Burgess

Clinical Relevance

Alginate substitute materials are inexpensive polyvinyl siloxane (PVS) impression materials that exhibit better detail reproduction and tear strength than alginate. Alginate substitute materials do show slightly more outgassing and resulting cast porosity than traditional alginates, particularly when they are poured soon after mixing. To reduce cast surface porosity, a minimum pouring delay of 60 minutes is suggested.

SUMMARY

Objective: To compare three alginate substitute materials to an alginate impression material for cast surface porosity (outgassing), tear strength, and detail reproduction.

Materials and Methods: Detail reproduction tests were performed following American National Standards Institute/American Dental Association (ANSI/ADA) Specification No. 19. To measure tear strength, 12 samples of each material were made using a split mold, placed in a water bath until testing, and loaded in tension until failure at a rate of 500 mm/min using a universal testing machine. For cast surface porosity testing, five impressions of a Teflon mold with each material were placed in a water bath (37.8°C) for the in-mouth setting time and poured with vacuum-mixed Silky Rock die stone at 5, 10, 30, and 60 minutes from the start of mixing. The gypsum samples were analyzed with a digital microscope for surface porosity indicative of hydrogen gas release by comparing the surface obtained at each interval with four casts representing no,

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little, some, and significant porosity. Data analysis was performed using parametric and Kruskal-Wallis analysis of variance (ANOVA), Tukey/Kramer *post-hoc* tests (α=0.05), and individual Mann-Whitney U tests (α=0.0167).

Results: All alginate substitute materials passed the detail reproduction test. Tear strength of the alginate substitute materials was significantly better than alginate and formed three statistically different groups: AlgiNot had the lowest tear strength, Algin-X Ultra had the highest tear strength, and Position Penta Quick had intermediate tear strength. Significant variation in outgassing existed between materials and pouring times ($p<0.05$). All alginate substitute materials exhibited the least outgassing and cast porosity 60 minutes after mixing.

Conclusions: Detail reproduction and tear strength of alginate substitute materials were superior to traditional alginate. The outgassing effect was minimal for most materials tested. Alginate substitute materials are superior replacements for irreversible hydrocolloid.

INTRODUCTION

Traditionally, irreversible hydrocolloid (alginate) has been the material of choice for diagnostic impressions because it is inexpensive, hydrophilic, reasonably accurate, and easy to manipulate.[1,2] Advances in material refining processes, however, have produced low cost polyvinyl siloxane (PVS) impression materials, or "alginate substitutes," as alternatives to traditional alginate. For comparison, the alginate material cost for a full arch impression is approximately 90 cents, a full arch PVS impression is about $20, and a full arch impression with alginate substitute materials is $5-$7.[1] These materials may be used for the same procedures as traditional alginate materials, such as making study and orthodontic models, fabricating provisional crown and bridge restorations, and making final impressions for removable prosthodontics.[3,4] The proposed advantages of alginate substitute materials over traditional alginate are increased accuracy, prolonged dimensional stability, and the ability to repour an impression to obtain a second cast.

Earlier generations of alginate substitute materials were composed of alginate with silicone additives.[5,6] These materials demonstrated little, if any, improvements in dimensional stability and were not able to accurately transfer fine details to gypsum casts.[5] Newer generation alginate substitutes, however, are composed of refined polyvinyl siloxane. The materials are termed alginate substitutes because advances in their refining process and proprietary changes in their chemical composition have significantly reduced the price of these PVS materials. A probable cost-saving modification in alginate substitute materials is the removal of palladium. Palladium is an expensive component of PVS added to impression materials to scavenge excess hydrogen gas. Studies have suggested that hydrogen gas release from PVS materials produced bubbles on gypsum casts. Adding palladium to PVS, therefore, decreases cast porosity.[7,8] It is clinically relevant to measure the properties of alginate substitutes to determine the effects of compositional modifications of PVS on its physical and mechanical properties and compare these properties to those of traditional alginate impression material.

The American National Standards Institute and American Dental Association (ANSI/ADA) have developed standard practices for measuring properties of dental impression materials. Specification No. 18 was developed for alginates, and Specification No. 19 was developed for elastomeric impression materials.[9,10] Because alginate substitutes are composed of PVS, an elastomeric material, it is reasonable to measure their properties in accordance with Specification No. 19. Major advantages of most elastomeric impression materials over traditional alginates include increased detail reproduction[11] and strength.[12] Detail reproduction is described in Specification No. 19 as the ability of a material to reproduce a line of 50 microns scribed into a steel die.[10] An ANSI/ADA specification exists for tear strength using a notched specimen (4.3.10 of Specification No. 20); however, a more clinically relevant thin film tear strength method has been described by Lawson and others[13] and reviewed by the ADA.[14] In addition to detail reproduction and tear strength, it is also relevant to measure the cast porosity produced from alginate substitutes due to the assumed reduction of palladium scavengers in these materials.

Some studies have already examined the properties of alginate substitutes. Because these materials are relatively new, laboratory testing is scarce and clinical studies are nonexistent. A recent article by Torassian and others[15] has determined that these materials have superior dimensional stability to alginate, remaining dimensionally accurate up to a week following mixing.[15] Patel and others[16] examined the detail reproduction, gypsum compatibility, and linear dimensional accuracy of several alginate

substitute materials. All materials demonstrated superior properties to a control alginate group.

The aim of this study was to compare the detail reproduction, tear strength, and cast porosity of alginate substitutes to the control, alginate. The null hypothesis was that there are no significant differences between the alginate substitute materials and alginate for detail reproduction, tear strength, and cast porosity.

MATERIALS AND METHODS

Three alginate substitute materials (AlgiNot, Algin-X Ultra, and Position Penta Quick) and a traditional alginate (Jeltrate) were evaluated using three testing methodologies. The characteristics of the three alginate substitute materials and alginate (control) are described in Table 1.

Detail Reproduction

ANSI/ADA Specification No. 19 for elastomeric impression materials was used to evaluate the detail reproduction of the alginate substitute materials. Three specimens were produced for each material. The ANSI/ADA specified steel die with 20-μm, 50-μm, and 75-μm scribed lines was used for this test. Prior to testing, the die was heated to oral temperature (37.8°C) in a water bath (Hygrobath, Whip Mix, Louisville, KY, USA). A ring was then placed on top of the die, and impression material was loaded into the ring. A glass slab was placed on top of the ring and secured with a 1-kg weight. The entire assembly was transferred to a 37.8°C water bath for the manufacturers' in-mouth setting time (Table 1). The specimens were then carefully separated from the die (Figure 1) and examined with a Keyence Digital Microscope VHX600 (Keyence, Itasca, IL, USA) at 12× optical magnification. Specimens were determined to have either passed or failed the test based on their ability to capture the entire length of the 50-μm line.

Tear Strength

A plexiglass mold as described in a previous study[13] was employed for this test. Specimens had dimensions of 70 mm × 10 mm with a 0.1-mm thickness film in their center. Twelve tear strength specimens were prepared of each material. After a small amount of material was extruded and discarded to ensure proper mixing in the dispensing tip, the molds were filled with impression material. The cover of the mold was applied with finger pressure and secured to the base. All fabrication occurred at 24°C and 51% humidity before moving the specimens to a water bath (Hygrobath, Whip Mix) at 37.8°C for the manufacturers' set time (Table 1). After setting, the mold was removed from the incubator and the specimen removed from the mold. Any excess material from the edges of the specimen was trimmed using a razor blade, and benchmarks were drawn on the specimen 10 mm on either side of the center line with a digital caliper. The specimen was secured into a universal testing machine (Instron Corp, Canton, MA, USA) with a pneumatic clamp at the location of the previously applied benchmarks. Before the test began, the fixture was adjusted so that the specimen was neither in compression nor tension. Starting 2.5 minutes after the specimens were removed from the water bath, the specimens were loaded in tension until failure with a crosshead speed of 500 mm/min. The tear strength was calculated as tear strength = ultimate tensile strength / (10 mm × 0.1 mm).

Cast Porosity Test

A custom milled Teflon mold (Figure 2) was used to prepare specimens of each impression material. The

Table 1: *Materials Used in This Study*

Brand Name	Type	LOT/Exp	Manufacturer	Setting Speed	Working Time	Setting Time
Jeltrate (control)	Traditional alginate	1006091 2012-12	DENTSPLY/Caulk (Konstanz, Germany)	Fast set	1:30	2:30
AlgiNot	Alginate substitute	01142 2012-05	Kerr (Orange, CA, USA)	Fast set	1:00	2:30
Algin-X Ultra	Alginate substitute	100414 2012-04	DENTSPLY/Caulk (Konstanz, Germany)	Fast set	1:00	2:30
Position Penta Quick	Alginate substitute	401166 2012-05	3M ESPE (St Paul, MN, USA)	Fast set	1:10	2:40

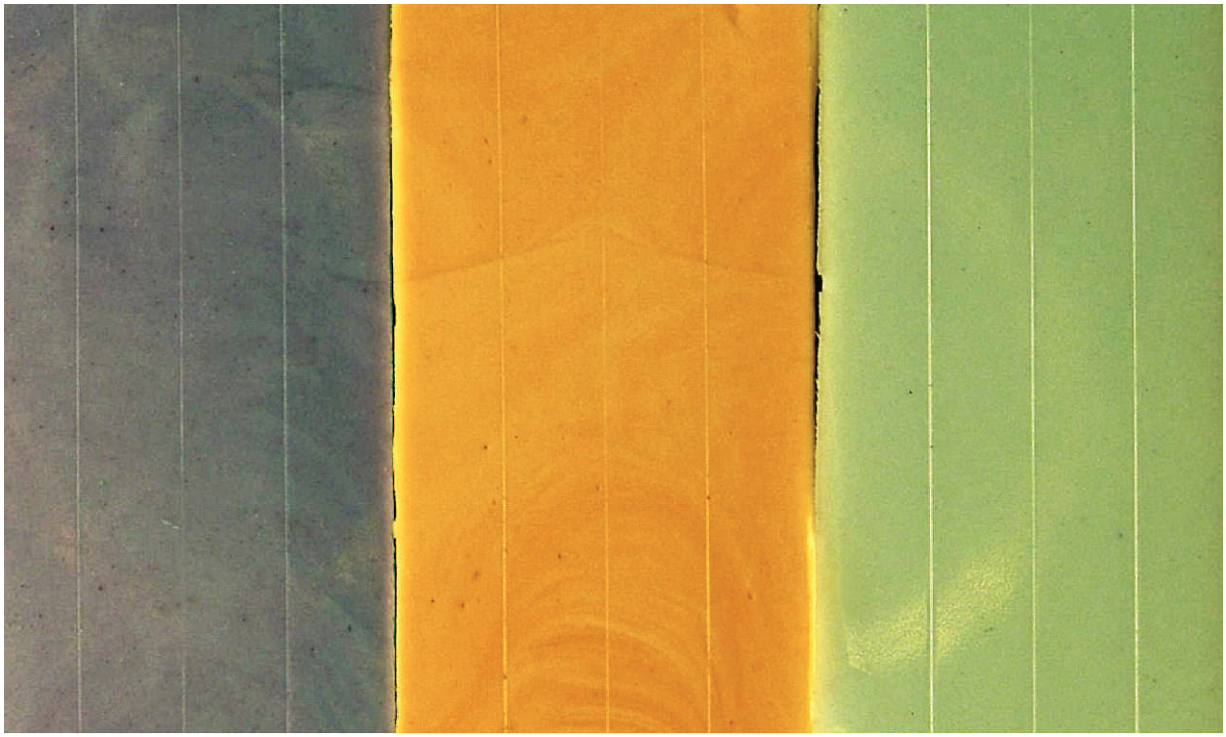

Figure 1. *Detail reproduction specimens. (Left to right: Position Penta Quick, AlgiNot, Algin-X Ultra)*

mold was designed so that its impression could retain dental stone independently and provide a uniform surface area to analyze porosities on the stone. Five impressions of the mold were taken with each impression material at each time period. A glass slab was placed over the mold, and the filled molds were placed into a water bath (37.8°C) for the in-mouth setting time (Table 1). Each impression specimen was

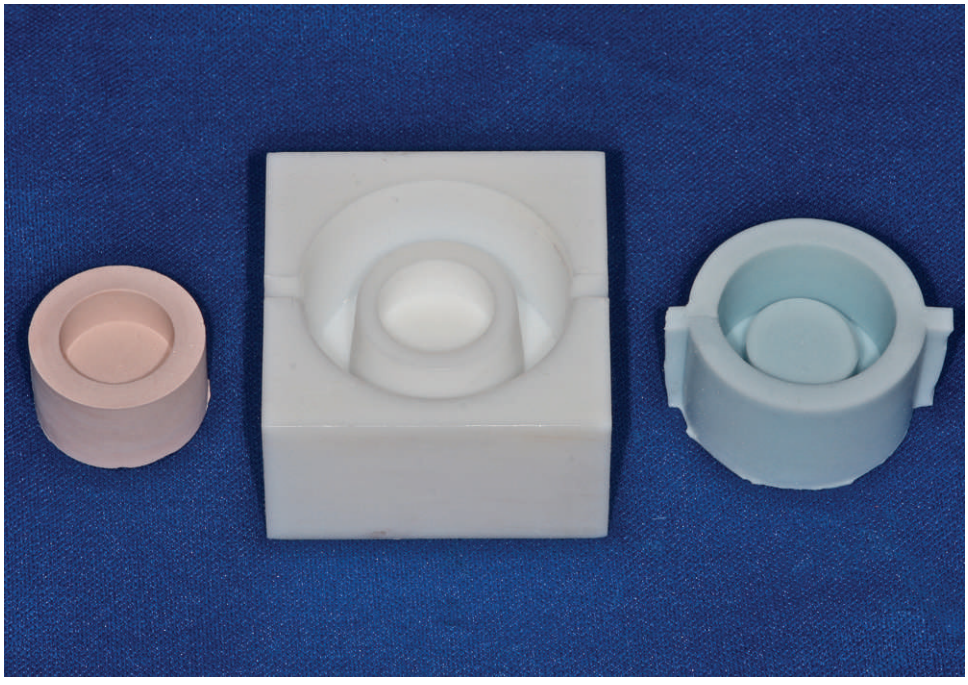

Figure 2. *Teflon mold used for cast porosity testing. (Left to right: cast, mold, impression)*

then filled with vacuum-mixed (−100 kPa) Silky Rock die stone (32 mL:140 g) (Whip Mix) at the appropriate interval (5, 10, 30, or 60 minutes after mixing the impression material). After setting, the surface of the stone casts that were in contact with impression material was analyzed using a Keyence Digital Microscope VHX600 at 5× magnification for surface porosity indicative of hydrogen gas release. The samples were each scored (1–4) against four representative samples with varying degrees of porosity/outgassing (Figure 3). The scores were given based upon the following criteria: 1: smooth, uniform surface; 2: slight outgassing, but uniform surface; 3: more outgassing, at irregular intervals; and 4: most outgassing at irregular intervals. The scores were averaged and used for statistical analysis.

Statistical Analysis

Tear strength data were analyzed by one-way analysis of variance (ANOVA), and significant differences between groups were examined with Tukey analysis (α=0.05). Results of the cast porosity testing were compared using a Kruskal-Wallis ANOVA (α=0.05) at each time interval. Alginate substitute materials were compared to each other and Jeltrate at each time point with the Mann-Whitney U test with a Bonferroni-adjusted alpha level of 0.0167. Each material was then compared for change in porosity over time with a Kruskal-Wallis ANOVA (α=0.05).

RESULTS

All alginate substitute impression materials exceeded the requirements of the ANSI/ADA Specification No. 19 detail reproduction test by reproducing not only the 50-μm line, but also the 20-μm line. The alginate control did not reproduce the 50-μm line, failing the detail reproduction test (Table 2).

During tear strength measurement, the alginate failed (tore) before testing. For this reason, alginate was excluded from the statistical analysis and assumed to have a significantly lower tear strength than all tested alginate substitute materials. The one-way ANOVA showed significant differences between the alginate substitute materials and the Tukey/Kramer test differentiated them into three statistically separate groups ($p<0.05$). Algin-X Ultra had the highest tear strength with a mean value of 5.48 ± 0.64 MPa. Position Penta Quick was intermediate, measuring 3.53 ± 0.56 MPa; and the lowest group was AlgiNot FS at 2.44 ± 0.19 MPa (Table 2).

Porosity values for casts made using three alginate substitute materials were analyzed at each time interval and a significant difference in cast porosity

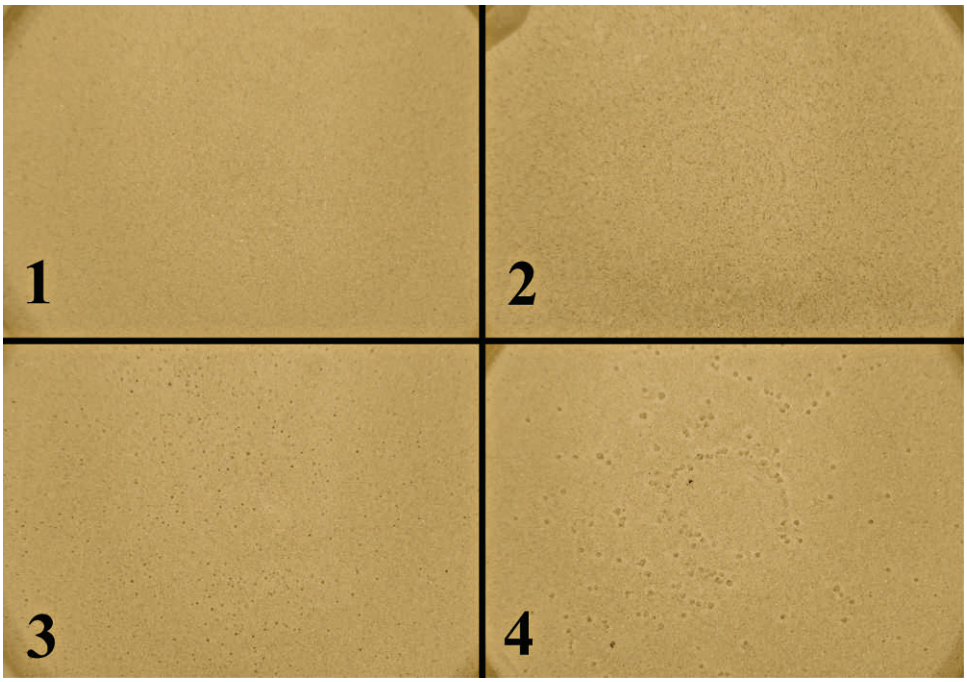

Figure 3. *Porosity/outgassing reference samples. (1: smooth, uniform surface; 2: slight outgassing, but uniform surface; 3: more outgassing, at irregular intervals; and 4: most outgassing at irregular intervals)*

Table 2: *Tested Properties of Alginate Substitute and Traditional Alginate Materials*

Material	Detail Reproduction	Average Cast Porosity Score				Tear Strength (Mean± SD), MPa
		5 min	10 min	30 min	60 min	
Jeltrate (control)	Failed	1 ± 0	1 ± 0	1 ± 0	1 ± 0	0
AlgiNot	Passed	1.8 ± 0.4	1.4 ± 0.5	1 ± 0	1 ± 0	3.53 ± 0.56
Algin-X Ultra	Passed	2.2 ± 0.4	2.2 ± 0.4	2.4 ± 0.5	1.8 ± 0.4	5.48 ± 0.64
Position Penta Quick	Passed	1.4 ± 0.5	1.6 ± 0.5	1.6 ± 0.5	1.2 ± 0.4	2.44 ± 0.19

among these groups was noted at 30 minutes (p=0.0074) and 60 minutes (p=0.0263). Cast porosity at 30-minute and 60-minute periods was then analyzed with the Mann-Whitney U test for independent samples with a Bonferroni-adjusted alpha level of 0.0167. At 30 and 60 minutes, Algin-X Ultra demonstrated significantly higher cast porosity than AlgiNot (p=0.0046 and p=0.0143, respectively).

Each alginate substitute was then compared to an alginate control using the Mann-Whitney U test for independent samples at each of the four time intervals (alpha=0.0167). Cast porosity in Algin-X Ultra samples was significantly elevated from casts poured in alginate at each time interval. In AlgiNot specimens, cast porosity was significantly higher than alginate only in the first time interval. Position Penta Quick demonstrated no significant difference from alginate at any time interval.

Each material was observed to produce less mean porosity at the 60-minute interval. When each material was analyzed using Kruskal-Wallis ANOVA for change in porosity over time, AlgiNot was seen to exhibit a significantly lower value from the initial time period. Algin-X Ultra and Position Penta Quick were not significantly reduced.

DISCUSSION

The results of this study indicate that alginate substitute materials have greater detail reproduction and tear strength than alginates, but some have an increase in cast porosity at certain pouring times. Therefore, the null hypothesis is rejected for all of the tests performed. Further analysis into each property will be discussed below.

All alginate substitute materials were able to accurately capture the 20-μm and 50-μm line, whereas the control alginate material was not able to reproduce the 50-μm line. The results of this test are within expected outcomes because PVS materials are considered to produce the greatest detail of all impression materials,[17] and a recent study of alginate substitutes found similar results.[16] Detail reproduction is a factor of the pressure exerted on an impression material and the material's wettability and rheological properties.[18] Equal pressure was exerted on all materials in this study, so differences between materials should be related to differences in their wetting and rheological properties. Wettability is the ability of a liquid to spread on the surface of a solid,[19] and it is related to the material's contact angle.[20] Alginates are recognized as having better wettability than PVS.[8] Rheological properties, on the other hand, refer to the material's viscosity and ability to flow. German and others[21] determined that surface detail reproduction was related to a material's tan delta (a measure of viscosity) and flow. Although the flow properties of alginates and PVS have not been directly compared, PVS has demonstrated exceptional flow properties.[22,23] The high level of detail reproduction produced by alginate substitutes in this study can therefore be credited to the flow properties of PVS.

The tear strength test demonstrates the ability of alginate substitute materials to reproduce thin intrasulcular and interproximal areas.[24] The control, alginate, failed before testing, supporting the clinical observation of torn alginate fragments remaining in interproximal areas of the mouth after making an impression. In the present study, the tear strength results may be compared to a similar study using the same split molds (0.1-mm thick) and testing conditions.[13] In that study, tear strengths of traditional PVS materials ranged from 4.71 MPa to 8.24 MPa, and the tear strength of the polyether material (Impregum) was 2.05 MPa. The tear strengths of the

alginate substitute materials in this study ranged from 2.44 MPa to 5.48 MPa. Comparing the data from the current study to the previous study, alginate substitutes produced lower tear strength values than traditional PVS materials, possibly due to modifications made to PVS to produce these cheaper alternatives. However, all alginate substitute materials tested in this study exhibited greater tear strength than Impregum, a polyether material used for fixed prosthodontics.

In this study, some alginate substitute materials showed significantly more cast porosity than a traditional alginate, particularly when poured soon after they were mixed. A study by McCrosson and others[7] measured hydrogen gas released from setting PVS materials by gas chromatography and compared it to the porosity observed on casts poured from the same materials. Similar to the present study, cast porosity was quantified by scoring casts by the number of defects on their surface. Scores ranged from 0 to 6, generally differentiated by differences of 25 defects. The study found a relationship between hydrogen gas release and cast porosity and recommended waiting the manufacturer's recommended pouring time (30–360 minutes) before pouring PVS materials. The results of the present study suggest that alginate substitute materials will produce similar cast porosity as alginate after waiting five minutes for Position Penta Quick and 10 minutes for AlgiNot. Algin-X Ultra had slight outgassing even after 60 minutes; however, it produced less cast porosity after 60 minutes than at earlier times. Clinically, it is important to determine the degree of cast surface integrity required when selecting an appropriate material and pouring time.

One of the major limitations of this study was that detail reproduction was only measured on a dry surface. As PVS materials are generally hydrophobic,[25] the measurement of surface detail on a wet or moist surface has shown to reduce its detail reproduction.[26,27] Additional properties of alginate substitutes, such as contact angle, elastic deformation, and recovery and flow measurement must be compared to traditional PVS materials in order to completely characterize these materials. Ultimately, clinical testing of these materials must be performed to determine the range of their clinical applications.

CONCLUSIONS

In conclusion, alginate substitute materials are superior to alginate in tear strength. The alginate substitute material with the highest tear strength was Algin-X Ultra. All three alginate substitute materials passed the detail reproduction test, while the control, alginate, did not.

These materials did exhibit more outgassing than the control, particularly when poured five minutes after mixing. Position Penta Quick was the only alginate substitute material that produced cast porosity similar to that of the control at all time points. All alginate substitute materials produced the least outgassing if poured 60 minutes after mixing.

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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Influence of Post-cure Time on the Microhardness of Self-Adhesive Resin Cements Inside the Root Canal

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Clinical Relevance

The microhardness of self-adhesive, dual-cure resin cements, when used to lute fiber posts, depends on the material brand, with higher values of microhardness verified at the coronal third. Because changes in microhardness were detected between 24 hours and 7 days after luting, clinicians should take this into account to prevent damage to the biomechanical bonding of the post cement-dentin immediately following cementation.

SUMMARY

Purpose: To compare the microhardness of several dual-cure, self-adhesive resin cements used to lute fiber posts at 24 hours and seven days after cementation.

Methods: Bovine incisors were selected to lute 15 fiber posts that were 12 mm long (FRC Postec Plus size 3, Ivoclar-Vivadent). Five resin cements were tested: Multilink Automix (Ivoclar-Vivadent), without light-curing, and the self-adhesive resin cements Maxcem Elite (Kerr), RelyX Unicem (3M ESPE), G-Cem (GC), and Smartcem 2 (Dentsply), which were light-cured for 40 seconds (LED Bluephase, Ivoclar-Vivadent). Each root was embedded in chemically cured acrylic resin and stored at 37°C for 24 hours. The roots were transversally sectioned into nine specimens that were each 1 mm thick, with three specimens corresponding to each root third. Indentations (100g, 30 seconds) were performed on each section in the resin cement, at 24 hours and seven days after cementation, using a Vickers digital microdurometer (Buehler). Data were analyzed by two-way analysis of variance, Stu-

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dent-Newman-Keuls test, and paired *t*-test ($p<0.05$).

Results: A significant influence was found ($p<0.05$) for the resin cement evaluated, the root third, and their interactions on microhardness values at 24 hours and seven days after post cementation. RelyX Unicem and G-Cem exhibited the highest microhardness values, whereas Multilink Automix presented the lowest. All resin cements suffered a decrease in microhardness according to root canal depth, with the exception of G-Cem and Multilink Automix at 24 hours and Smartcem 2 after seven days. After seven days, the evaluated resin cements showed a significant increase in microhardness values, with the exception of Maxcem Elite and Smartcem 2 at the coronal third.

Conclusions: Microhardness of the self-adhesive resin cements when used to lute fiber posts was material-dependent and higher values were obtained in the coronal third, revealing their sensitivity to light irradiation. More information regarding the polymerization reaction of these cements is warranted. According to the current results, microhardness values were significantly higher one week after post luting.

INTRODUCTION

The restoration of endodontically treated teeth with large coronal loss generally requires the use of intraradicular posts to hold a core and eventual coronal restoration.

Translucent fiber-reinforced composite posts are often the preferred option because they exhibit mechanical properties that are more similar to dentin than do metal or ceramic posts, reducing the stress inside the root canal and preventing the risk of radicular fracture.[1] The similar moduli of elasticity among dentin and the materials that constitute the core and the post would contribute to establish a monoblock restoration, with the ability of the restorative materials to bond strongly and mutually to root dentin.[2] However, these monoblock restorations, which create mechanically homogeneous units with root dentin, are difficult to attempt and would be compromised if the resin cement used does not reach an adequate degree of conversion. Translucent posts are suggested as a better option over opaque posts to improve the curing of resin cements and, consequently, the bond strength[3-5] and microhardness.[4-6] However, some widely used translucent posts have been shown to ineffectively transmit light to the apical region.[7]

To compensate for light attenuation by the post and root depth, a selected luting agent should be a dual-cure or self-cure resin material. Dual-cure resin cements possess a chemical-curing system that can achieve a more extensive polymerization in dark locations[8] while also providing a light-curing mechanism that allows for an extended working time and a rapid initial hardening of the resin cement to stabilize the restoration.[9] Both the light- and chemical-curing mechanisms are complementary and independent.[6] Although chemical curing is responsible for curing at sites not reached by light exposure, the chemical component in some dual-cure resin composites has been described as slower, less effective,[9-11] or virtually ineffective.[8,12,13]

Apart from their curing mode, resin cements have also been classified according to their mechanism of interaction with the smear layer. Therefore, resin cements require either the application of an etch-and-rinse adhesive system or of a self-etching primer.[5,14] Recently, a new subgroup was introduced into the self-etching category: self-adhesive resin cements. These new materials are applied to enamel and dentin without a previous application of an adhesive system.[9,14] The first self-adhesive cement was RelyX Unicem, which was launched into the market in 2002. Since then, new products have been constantly introduced. The keys to their clinical success are based on their ability to adequately bond to different substrates[11] and their reduced technique and operator sensitivity.[14] Self-adhesive resin cements are characterized as being dual-curing cements. However, their behavior as a group is not fully understood due to their complex and sometimes unknown compositions.[15]

Microhardness testing has been described as a valid indirect method to determine the degree of cure because it presents a good correlation with the spectroscopy approach.[16] Therefore, the aim of this current study was to evaluate the microhardness along the depth of the root canal post space of a dual-cure resin cement, which requires the application of an adhesive system, and four self-adhesive resin cements used to lute a fiber post at 24 hours and seven days after cementation. The research hypotheses were that the self-adhesive resin cements attain higher microhardness; their microhardness decreases through the root length; and their microhardness increases after seven days.

METHODS AND MATERIALS

Fifteen bovine teeth that had been stored at 4°C in thymol for a maximum of six months were selected to lute 15 glass fiber-reinforced posts.

Post Space Preparation

Roots were sectioned perpendicularly to the long axis of the tooth to a length of 16 mm from the apex, using a diamond bur under copious water cooling.

Root canals were manually instrumented using the step-back technique, and the master apical K-file used was an ISO 080 (Dentsply-Maillefer, Ballaigues, Switzerland), due to the anatomy of the bovine teeth. Each root canal was flushed with 2.5% sodium hypochlorite and dried with ISO-standardized paper points (Dentsply-Maillefer).

Post preparation was carried out with low-speed FRC Postec size 3 drills (Ivoclar-Vivadent, Schaan, Liechtenstein) under water cooling, creating a 12-mm-deep post space and leaving the remaining 4 mm to the apex untouched. Before cementing the posts, the external root surfaces were painted with black nail varnish to prevent external light from interfering with resin-cement curing.

FRC Postec Plus Cementation

Each post was cut to a length of 16 mm. Therefore, 12 mm of the post was inside the root canal and 4 mm out of the root canal, which was determined to be the amount needed to place the coronal core. Prior to luting, the posts were checked to fit in the post space and conditioned according to the manufacturer's instructions. The post surfaces were cleaned with 35% phosphoric acid for 60 seconds (Coltène Whaledent, OH, USA), washed, and dried. The posts were silanized with Monobond-S (Ivoclar-Vivadent) for 60 seconds and dried with compressed air.

Five resin cements were evaluated (Table 1). The self-cured resin cement Automix (Ivoclar-Vivadent) and the self-adhesive resin cements Maxcem Elite (Kerr Corp, Orange, CA, USA), RelyX Unicem (3M ESPE, St Paul, MN, USA), G-Cem (GC Corporation, Tokyo, Japan), and SmartCem 2 (Dentsply-Detrey, GmbH, Konstanz, Germany) were used to lute glass fiber-reinforced posts (FRC Postec Plus, size 3, Ivoclar-Vivadent) inside the root spaces. All resin cements were applied following the manufacturers' instructions and similar shades were selected, according to the available products (opaque or yellow shades). The self-adhesive resin cements were light-cured with the LED polymerization unit Bluephase (Ivoclar-Vivadent), set for the high curing program (1200 mW/cm^2). To standardize the curing procedure, cements were light-cured for 40 seconds by contacting the lamp with the external portion of the post. The output intensity of the LED light-curing unit was checked (>600 mW/cm^2) before every five luting procedures.

Specimen Preparation

Each root was embedded in a transparent chemically cured epoxy resin (Buehler, Lake Bluff, IL, USA) and stored for 24 hours at 37°C. Then, nine specimens of 1 mm width were obtained from each root through transverse sectioning (three from the coronal third, three from the middle third, and three from the apical third) using an Isomet 5000 with a diamond blade (Buehler).

Each specimen was sequentially polished with Beta Polisher (Buehler) using 320-, 800-, 1200-, and 4000-grit polishing disks at 300 rpm for 30 seconds. Afterward, specimens were stored at 37°C and 100% humidity in a light-free container.

Microhardness

Measurements were carried out at 24 hours and seven days after post cementation, applying a 100g load for 30 seconds with a Vickers digital microhardness tester (Buehler 2101). Indentations were performed on the resin cement avoiding artifacts due to luting procedures, such as voids. A minimum distance corresponding to the length of two indentations was maintained between indentations and between the indentations and the post or dentin.

Statistical Analysis

Mean and standard deviation values were determined for each experimental group. The influence of the independent variables, resin cement and root third, on the dependent variable, microhardness at 24 hours and seven days after luting, was evaluated by two-way analysis of variance. A *post hoc* test was performed using the Student-Newman-Keuls test.

Microhardness values of resin cements obtained at 24 hours were compared with those measured at seven days using a paired *t*-test. All statistical testing was performed at a preset α of 0.05 by means of IBM SPSS statistics software, version 19.0 (IBM, New York, USA).

RESULTS

Microhardness values (VHNs) obtained for the self-cured resin cement and the self-adhesive resin cements have been divided into three sections:

Table 1: Composition of the Resin Cements

Resin Cement	Manufacturer	Composition	
		Resin Matrix	Filler
MultilinkAutomix Shade: Yellow	Ivoclar-Vivadent, Schaan, Liechtenstein	DM, HEMA	Ba–glass, SiO_2, YF_3 40 vol.%, 68.5 wt.%
Maxcem Elite Shade: Yellow	Kerr Corp. Orange, CA, USA	GPDM, comonomers, mono-, di-, tri-functional methacrylate monomers	F–Al–Si–glass, Ba–glass, SiO_2 46 vol.%, 67 wt.%
RelyX Unicem Shade: AO3	3M ESPE St. Paul, MN, USA	PAE, TEGMA, BisGMA	SiO2, glass 54 vol.%, 72 wt.%
G- Cem Capsule Shade: AO3	GC Corporation Tokyo, Japan	UDMA, PAE, 4-META, DM	F–Al–Si–glass, SiO_2 56.6 vol.%, 71 wt.%
SmartCem 2 Shade: Opaque	Denstply-Detrey GmbH Konstanz, Germany	UDMA, Urethane Modified BisGMA, DM, DPP	Ba–B–F–Al–Si–glass, SiO_2 46 vol.%, 69 wt.%

DM: dimethacrylate, HEMA: hydroxyethyl methacrylate, GPDM: glyceroldimethacrylate dihydrogen, PAE: phosphoric acid ester monomer, TEGDMA: triethyleneglycol dimethacrylate, Bis-GMA: bisphenol A dimethacrylate, UDMA: urethane dimethacrylate, 4-META: 4-methacryloyloxyethyl trimellitate anhydride, DPP: dipentaerythritol pentaacrylate phosphate.

microhardness evaluated at 24 hours, microhardness evaluated at seven days, and microhardness variation according to time elapsed after post luting.

Microhardness Evaluated at 24 hours

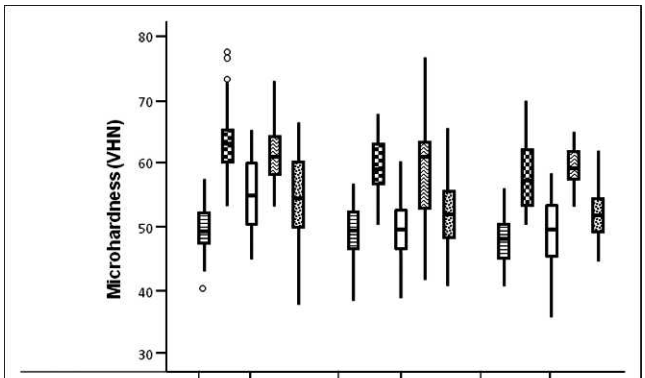
Figure 1 shows mean (SD) values obtained for each resin cement evaluated at the coronal, middle, and apical root thirds.

According to the statistical analysis, a significant influence on microhardness of the resin cement ($p<0.001$), root third ($p<0.001$), and their interaction ($p<0.01$) was detected.

At the coronal third, RelyX Unicem showed the highest microhardness values, followed by G-Cem. Multilink Automix, a self-cured resin cement, exhibited the lowest mean values. Intermediate and statistically similar mean values were obtained by Maxcem Elite and SmartCem 2.

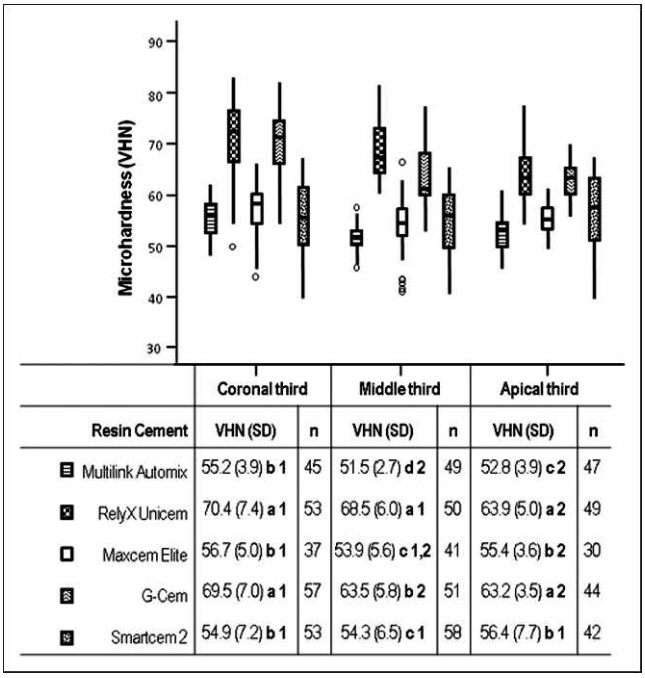
At the middle and apical thirds, RelyX Unicem and G-Cem showed the highest microhardness values, with no statistical differences. SmartCem 2 exhibited intermediate mean values that were significantly higher than those obtained for Maxcem Elite and Multilink Automix.

Regarding the variation of microhardness values of the resin cements evaluated according to the root third, G-Cem and Multilink Automix were the only



Resin Cement	Coronal third VHN (SD)	n	Middle third VHN (SD)	n	Apical third VHN (SD)	n
Multilink Automix	49.7 (3.8) **d 1**	45	49.3 (3.8) **c 1**	49	47.8 (4.0) **c 1**	47
RelyX Unicem	63.9 (5.5) **a 1**	53	59.5 (4.1) **a 2**	50	58.2 (5.5) **a 2**	49
Maxcem Elite	55.1 (6.0) **c 1**	37	49.6 (5.0) **c 2**	41	49.3 (4.9) **c 2**	30
G-Cem	61.5 (4.4) **b 1**	57	59.1 (7.4) **a 1**	51	59.4 (3.0) **a 1**	44
Smartcem 2	54.8 (6.0) **c 1**	53	51.9 (5.4) **b 2**	58	52.6 (4.6) **b 2**	42

Figure 1. *VHNs means and standard deviations (sd) of resin cements evaluated at 24 hours. Same letters in hte same column mean no statistically significant differences among cements at each root third ($p<0.05$). Same numbers in the same row mean no statistically significant differences among root thirds for each cement ($p<0.05$).*



Resin Cement	Coronal third VHN (SD)	n	Middle third VHN (SD)	n	Apical third VHN (SD)	n
Multilink Automix	55.2 (3.9) **b 1**	45	51.5 (2.7) **d 2**	49	52.8 (3.9) **c 2**	47
RelyX Unicem	70.4 (7.4) **a 1**	53	68.5 (6.0) **a 1**	50	63.9 (5.0) **a 2**	49
Maxcem Elite	56.7 (5.0) **b 1**	37	53.9 (5.6) **c 1,2**	41	55.4 (3.6) **b 2**	30
G-Cem	69.5 (7.0) **a 1**	57	63.5 (5.8) **b 2**	51	63.2 (3.5) **a 2**	44
Smartcem 2	54.9 (7.2) **b 1**	53	54.3 (6.5) **c 1**	58	56.4 (7.7) **b 1**	42

Figure 2. *VHNs means and standard deviations (sd) of resin cements evaluated at 7 days. Same letters in hte same column mean no statistically significant differences among cements at each root third (*$p<0.05$*). Same numbers in the same row mean no statistically significant differences among root thirds for each cement (*$p<0.05$*).*

ones that showed a homogeneous behavior among the root thirds, whereas the other cements suffered a significant decrease in microhardness from the coronal to the middle thirds.

Microhardness Evaluated at Seven Days

Figure 2 shows mean (SD) values obtained for each resin cement evaluated at coronal, middle, and apical root thirds.

According to the results, a significant influence on microhardness of the resin cement ($p<0.001$), root third ($p<0.001$), and their interaction ($p<0.001$) was detected.

At the coronal third, RelyX Unicem and G-Cem exhibited similar microhardness values, which were statistically higher than those determined for Multilink Automix, Maxcem Elite, and SmartCem 2, which were statistically similar.

At the middle third, RelyX Unicem exhibited the highest microhardness values, followed by G-Cem. Maxcem Elite and SmartCem 2 attained intermediate values with no statistical differences. The lowest microhardness values were determined for Multilink Automix.

At the apical third, RelyX Unicem and G-Cem obtained the highest microhardness values with no statistical differences. Maxcem Elite and SmartCem 2 obtained similar and intermediate microhardness values. The lowest values were attained with Multilink Automix.

The evaluation of microhardness along root thirds was also evaluated, showing that SmartCem 2 was the only resin cement that had microhardness values that were homogeneous through the root space. The other cements showed a decrease of microhardness when comparing the mean values at the coronal and middle thirds.

Microhardness Variation According to Time Elapsed From Post Cementation

Table 2 shows mean (SD) values obtained for each resin cement evaluated at the coronal, middle, and apical root thirds.

Table 2: *VHN Means and Standard Deviations (SD) of Resin Cements Evaluated at 24 Hours and Seven Days, Together With the Comparison Between Both Times*

Resin Cement	Coronal Third			Middle Third			Apical Third		
	24 h	7 d	24 h vs 7 d	24 h	7 d	24 h vs 7 d	24 h	7 d	24 h vs 7 d
Multilink Automix	49.7 (3.8)	55.2 (3.9)	$p<0.001$	49.3 (3.8)	51.5 (2.7)	$p<0.01$	47.8 (4.0)	52.8 (3.9)	$p<0.001$
RelyX Unicem	63.9 (5.5)	70.4 (7.4)	$p<0.001$	59.5 (4.1)	68.5 (6.0)	$p<0.001$	58.2 (5.5)	63.9 (5.1)	$p<0.001$
Maxcem Elite	55.1 (6.0)	56.7 (5.0)	$p=0.279$	49.6 (5.0)	53.9 (5.6)	$p<0.001$	49.3 (4.9)	55.4 (3.6)	$p<0.001$
G-Cem	61.5 (4.4)	69.5 (7.0)	$p<0.001$	59.1 (7.4)	63.5 (5.8)	$p<0.01$	59.4 (3.0)	63.2 (3.5)	$p<0.001$
Smartcem 2	54.8 (6.0)	54.9 (7.2)	$p=0.922$	51.9 (5.4)	54.3 (6.5)	$p<0.05$	52.6 (4.6)	56.4 (7.7)	$p<0.01$

All resin cements tested showed a significant increase in microhardness values after seven days, with the exception of Maxcem Elite and SmartCem 2 at the coronal third.

DISCUSSION

The results of the present study indicate that microhardness values depend on resin luting material brand, root third, and time elapsed from post cementation. Thus, the research hypotheses formulated must be accepted.

Self-adhesive resin cements possess a complex chemical composition. They contain conventional mono-, di-, and/or multi-methacrylate monomers, carboxylic or phosphoric acid-functionalized monomers, fillers, and photo-initiators. This unique composition allows them to combine a curing mechanism based on a free-radical redox polymerization and an acid-base reaction.[17] The acid-base reaction occurs between the acidic functionality on the monomers and the acid-soluble glass or the mineralized tooth surface. As this reaction proceeds, ionic cross-links that form between acid groups and calcium or aluminium ions cause the pH to rise.[17] This pH neutralization is a matter of relevance because redox initiators and photoinitiators have been described to be sensitive to acidic monomers.[18]

In the present study, two of the self-adhesive resin cements tested, RelyX Unicem and G-Cem, attained the highest microhardness values. Self-adhesive resin cements constitute an attractive alternative for post cementation because no dentin pretreatment is required. This lack of pretreatment simplifies the procedure and reduces technique and operator sensitivity.[5] Although there are a dozen self-adhesive resin cements available, the majority of the studies regarding these materials concern RelyX Unicem. This self-adhesive cement has been reported to produce an effective adhesion with dentin, despite its very superficial interaction with this tissue, exhibiting similar[19] or even higher bond-strength values to root dentin than conventional resin cements[20] and a better sealing ability.[21] The setting of RelyX Unicem is characterized by a very rapid rise in pH, probably related to the presence of calcium hydroxide in its composition, achieving neutrality only 15 minutes after mixing when used in the dual-curing mode.[22] This allows the cement to change from a hydrophilic paste into a hydrophobic mixture that exhibits better properties, such as limited post-cure swelling and material deterioration.[17] Additionally, filler weight and volume percentage significantly influence the mechanical properties of resin cements.[23] Accordingly, RelyX Unicem and G-Cem contain a higher filler content in comparison with the other resin cements (Table 1).

In contrast, Multilink Automix, a self-cured resin cement, consistently presents low microhardness values, which can be related to its reduced filler content. It is crucial to note that Multilink Automix is described by the manufacturer as a self-curing luting material with a light-curing option. However, several studies indicate that Multilink Automix behaves better when applied using the dual-curing mode instead of the self-curing mode. Vrochari and others,[24] in 2009, evaluated the degree of cure for Multilink Automix in the self-curing and dual-curing mode using micro-attenuated total reflectance (ATR) Fourier transform infrared spectroscopy (FTIR), obtaining a very low degree of conversion (14.47%) in the self-curing mode and almost a six times higher degree of conversion (61.36%) in the dual-curing mode. Accordingly, low Vickers microhardness values (5.79 VHN) were determined for Multilink Automix when this material was left to self-cure when compared with those obtained after 40 or 80 seconds of light irradiation through a 4-mm indirect composite restoration (16.75 VHN or 19.37 VHN, respectively).[25] Furthermore, Multilink Automix requires a self-etching adhesive containing acidic resin monomers. These acidic monomers may impair polymerization because they interact with the benzoyl peroxide of the dual-cure cement.[26] Therefore, given that the effectiveness of the Multilink Automix self-curable component does not appear to be very high, a dual-curing mode seems to be the preferred option, despite the manufacturer's description.

Regarding Maxcem Elite and SmartCem 2, both materials attained intermediate to low microhardness values, depending on the root third evaluated and time elapsed since cementation. Both self-adhesive resin cements were statistically softer than RelyX Unicem and G-Cem, according to a previous report.[25] The lower filler amount in Maxcem Elite and SmartCem 2, in comparison with RelyX Unicem and G-Cem, may explain their lower mechanical properties (Table 1). There is not much information regarding Maxcem Elite; however, the previous version, Maxcem, has been described in several studies to achieve deficient mechanical and physical properties when compared with other self-adhesive cements, like RelyX Unicem or G-Cem,[22,27,28] including a lower degree of cure.[24] SmartCem 2 has also shown poor mechanical properties, such as unfavorable bond strength.[27] It has been shown that

Maxcem and SmartCem 2 do not have an important acid-base reaction while setting, as do other self-adhesive cements like RelyX Unicem,[28] maintaining a low pH for a long time[22,28] that could adversely influence the adhesion to dentin and the formation of an optimal cross-linked polymer network.[29]

Although microhardness is an indirect method widely used and accepted to determine the degree of cure,[8,29] the data obtained cannot be linearly correlated if compared across different materials.[30] Other factors, such as the nature of the matrix, type of filler, filler load, the quantity of initiators, the amount of inhibitors, and the ratio between auto- and light-polymerizing components, strongly influence the final amount of reacted monomers.[8,18,30] Thus, only microhardness data from the same resin cement should be compared according to the root third or time elapsed from luting.

Regarding the variation of microhardness values with root canal depth, all of the cements evaluated achieved the highest values in the coronal third and decreased through the root canal space, with the exception of G-Cem and Multilink Automix at 24 hours and Smartcem 2 after seven days. Various studies agree that light attenuation along the root canal negatively affects the polymerization of resin cements[10,31,32] and that the capacity of the translucent posts to transmit light is insufficient for clinical luminous activation of resin cement at the apical or middle thirds.[33] Therefore, the proximity of the irradiation source is a determinant in the extent of polymerization, despite the dual-cure nature of a material. G-Cem showed a homogeneous microhardness at different root thirds. It is possible that light attenuation is compensated for in this material by its chemical-curing component, indicating that G-Cem might present a higher amount of chemical-curing initiator in its composition when compared with the rest of the evaluated cements. High microhardness values have been reported for G-Cem even without light irradiation when applied under a 4-mm-thick indirect composite restoration.[25] Moreover, Multilink Automix was expected to behave homogeneously along the different root thirds because no light irradiation was applied.

In the present study, all resin cements evaluated had a post-curing effect seven days after luting, showing statistically higher microhardness values, with the exceptions of SmartCem 2 and Maxcem Elite at the coronal third. These results are in disagreement with the study by Yan and others,[34] who did not find changes in microhardness values after 24 hours postirradiation or postmix. However, the polymerization reaction of dual-cured materials might be material specific,[35] and the resin cements tested in that study were not the same as those in the present study. In fact, no self-adhesive resin cement was evaluated in the former study and the luting of fiber posts inside the root canal was not simulated. Therefore, the curing mechanism of the resin cements was based only on a free-radical redox polymerization and no acid-base reaction was expected.

According to the current results, the degree of conversion was lower where the light did not reach, due to the dual-curing behavior. It has been reported that dual-cure resin cements are characterized by slow polymerization[10] and, specifically for RelyX Unicem, increasing microhardness values have been reported even three months post luting.[6] Only the microhardness values of SmartCem 2 and Maxcem Elite at the coronal third remained similar after one week. As explained above, the information regarding the curing mechanism of these self-adhesive resin cements is scarce, but it may be that their final curing mainly depends on the level of initial conversion obtained from light exposure at the coronal third.

This current study confirms that the microhardness of the self-adhesive resin cements, when used to lute fiber posts, is brand dependent. Moreover, this degree of conversion is affected by the proximity to the light irradiation source because the resin cements were significantly softer in the middle and apical thirds, regardless of their dual-cure nature. Nevertheless, the polymerization reactions seem to continue longer than 24 hours postmix, attaining a significantly higher microhardness one week later.

Although microhardness testing is a sensitive method to detect small changes during resin-cement setting, it is only one mechanical property and no value has been established to ensure clinical success. Further research is warranted to explain how the polymerization reaction of these self-adhesive cements occurs as a function of time, with other properties being considered, in order to estimate their clinical performance.

CONCLUSIONS

The studied resin cements showed statistically different microhardness values, with RelyX Unicem and G-Cem presenting the highest values. All resin cements suffered a decrease in microhardness from the coronal to middle thirds, except for G-Cem and Multilink Automix at 24 hours and Smartcem 2 at seven days. Additionally, microhardness values were

significantly higher seven days after post luting, with the exception of Maxcem Elite and Smartcem 2 at the coronal third, revealing a long postirradiation curing reaction.

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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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Erratum

The following authors also participated in the manuscript titled, "*Effect of Gender, Experience, and Value on Color Perception*", printed in 37(3) 228–233:

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Clinical Research

Repair of Dimethacrylate-Based Composite Restorations by a Silorane-Based Composite: A One-Year Randomized Clinical Trial

DAV Popoff • TTA Santa Rosa • RC Ferreira • CS Magalhães • AN Moreira • IA Mjör

Clinical Relevance

A one-year clinical trial showed that a low-shrinkage silorane-based composite exhibited a similar performance to conventional dimethacrylate-based composites when used to repair composite resin restorations. This corroborates in vitro studies suggesting that bonding of silorane-based composites to old dimethacrylate-based composites can be a viable clinical procedure.

DOI: 10.2341/11-121-C

Laboratory Research

Comparative Evaluation of Microleakage of Silorane-Based Composite and Nanohybrid Composite With or Without Polyethylene Fiber Inserts in Class II Restorations: An In Vitro Study

VS Agrawal • VV Parekh • NC Shah

Clinical Relevance

Fiber inserts incorporated at the gingival floor of class II composite restorations resulted in a highly significant reduction of microleakage. Also, silorane composites based on a ring-opening mechanism showed reduced microleakage.

DOI: 10.2341/11-353-L

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Repair of Dimethacrylate-Based Composite Restorations by a Silorane-Based Composite: A One-Year Randomized Clinical Trial

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Clinical Relevance

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DOI: 10.2341/11-121-C

SUMMARY

Purpose: **To investigate clinical performance of a low-shrinkage silorane-based composite resin when used for repairing conventional dimethacrylate-based composite restorations.**

Background: **Despite the continued development of resin-based materials, polymerization shrinkage and shrinkage stress still require improvement. A silorane-based monomer system was recently made available for dental restorations. This report refers to the use of this material for making repairs and evaluates the clinical performance of this alternative treatment.**

Materials and Methods: **One operator repaired the defective dimethacrylate-based composite resin restorations that were randomly assigned to one of two treatment groups: control (n=50) repair with Adper SE Plus (3M/ESPE) and Filtek P60 Posterior Restorative (3M/**

ESPE), and test (n=50) repair with P90 System Adhesive Self-Etch Primer and Bond (3M/ESPE) and Filtek P90 Low Shrink Posterior Restorative (3M/ESPE). After one week, restorations were finished and polished. Two calibrated examiners (Kw≥0.78) evaluated all repaired restorations, blindly and independently, at baseline and one year. The parameters examined were marginal adaptation, anatomic form, surface roughness, marginal discoloration, postoperative sensitivity, and secondary caries. The restorations were classified as Alpha, Bravo, or Charlie, according to modified US Public Health Service criteria. Mann-Whitney and Wilcoxon tests were used to compare the groups.

Results: **Of the 100 restorations repaired in this study, 93 were reexamined at baseline. Dropout from baseline to one-year recall was 11%. No statistically significant differences were found between the materials for all clinical criteria, at baseline or at one-year recall ($p>0.05$). No statistically significant differences were registered ($p>0.05$) for each material when compared for all clinical criteria at baseline and at one-year recall.**

Conclusions:**The hypothesis tested in this randomized controlled clinical trial was accepted. After the one-year evaluations, the silorane-based composite exhibited a similar performance compared with dimethacrylate-based composite when used to make repairs.**

INTRODUCTION

The demand for esthetic restorations, the development of new adhesives and curing systems, and improvement of material properties have made dental composites the most widely used direct restorative material today.[1–3] Despite such developments, two features still require improvement: polymerization shrinkage and the development of polymerization shrinkage stress.[4]

The intrinsic contraction of the composite remains a challenge, and changes in the monomer composition seem to be the most promising way to minimize the effects of shrinkage.[5–8] Clinically, the incremental insertion and control of polymerization rate are the main strategies used to control polymerization shrinkage.

Recently, an innovative monomer system was made available for dental restorations: silorane, obtained from the reaction of oxirane and siloxane molecules; oxiranes are known for their low shrinkage, while siloxanes are known for their hydrophobicity.[5,9] *In vitro* studies have compared the new system to dimethacrylate-based composites. The results show that silorane-based composites demonstrate the lowest polymerization shrinkage as well as more ambient light stability, contributing to the convenience of handling the composite material. The new system also has the lowest sorption and water solubility and a lower diffusion coefficient than conventional monomers. Other parameters such as tensile modulus, flexural strength, and biocompatibility in toxicology tests are comparable to dimethacrylate-based composite.[5,8,10–12]

Imperfect margins result in marginal discoloration and secondary caries lesions, the most important cause for the replacement of restorations.[4] Reducing shrinkage and the stress generated by polymerization may positively influence marginal integrity. Total replacement is the most common treatment adopted for restorations that are clinically diagnosed as defective. However, the assessment of the quality of restorations is made subjectively, and often minimum deviations from ideal determine the systematic replacement of restorations.[13,14]

With the exception of conditions in which there is a fracture of the resin restoration, staining of the entire resin-tooth interface, and secondary caries, total removal is considered undesirable and inappropriate.[3,15] Thus, keeping in mind the current trends toward minimally interventional procedures, several studies have suggested partial removal of the restoration.[14,16–21] This approach allows preservation of sound tooth structure.[22,23]

Clinical studies involving composite resin repairs have shown that when properly planned, the repairs may increase the clinical longevity of restorations, representing a conservative choice for treatment of restorations.[13,14,24]

Thus, once *in vitro* studies suggest that bonding of silorane-based composites to old dimethacrylate-based composites may be a viable clinical procedure,[25–27] it would be desirable to evaluate the clinical performance of this new system for making repairs. The hypothesis tested in this randomized controlled clinical trial was that low-shrinkage silorane-based composites exhibit a similar performance when compared with that of conventional dimethacrylate-based composites when repairing composite resin restorations.

MATERIALS AND METHODS

Study Design

This was a prospective randomized clinical trial. The observation unit was the restoration, and the dependent variable was qualitative categorical ordinal. Patients aged 18 to 56 years with 100 defective composite resin restorations participated in this study. They were routinely assigned for treatment at the operative dentistry clinic, School of Dentistry–Federal University of Minas Gerais, Belo Horizonte, Minas Gerais, Brazil.

The inclusion criteria were patients who were older than 18 years of age and signed a consent form approved by the Institutional Ethics Committee, patients with no contraindications for dental treatment, patients who had class I or class II composite resin restorations with occlusal defects and no diagnosis of caries according to clinical and bitewing radiographic exams, and patients who had restorations that scored at least Bravo according to Modified United States Public Health Service (USPHS) clinical criteria (Table 1). The exclusion criteria were patients with contraindications for regular dental treatment according to their medical history; patients with xerostomia, including those taking medications that are proven to significantly reduce salivary flow; patients with visible plaque index (VPI) >30%; and patients with defective restorations, unacceptable for repairs, that scored Charlie (modified USPHS clinical criteria).

This study was approved by the Institutional Ethics Committee (ETIC 0546.0.203.000–09). A written informed consent was obtained from all patients.

Study Methods

The restorations were examined one week after they were repaired for baseline assessment and at one year. Two examiners independently evaluated all repaired restorations by direct observation, using a plane buccal mirror and a WHO model explorer. A calibration exercise revealed an interexaminer agreement ratio $\geq$0.78. If there was disagreement on the rating, the clinicians reexamined the repaired restoration together and arrived at a joint final decision. The parameters examined were marginal adaptation, anatomic form, surface roughness, marginal discoloration, postoperative sensitivity, and secondary caries. The examiners classified all restoration as Alpha, Bravo, or Charlie, according to modified USPHS clinical criteria.

Treatment Groups

To minimize preparation variability, the same operator repaired all defective composite resin restorations. The defective surfaces of the restorations were explored using high-speed spherical diamond burs (KG Sorensen, São Paulo, SP, Brazil) compatible with the size of the defect in a hand piece with air-water coolant, beginning with the removal of the restorative material in the area of the defect as well as any stained and soft tooth tissues. The operator randomly assigned the restorations to one of two treatment groups: control group (n=50), repair with a self-etching primer (Adper SE Plus, 3M/ESPE, St Paul, MN) and a dimethcrylate-based composite (Filtek P60 Posterior Restorative, 3M/ESPE), and test group (n=50), repair with a self-etching primer (P90 System Adhesive Self-Etch Primer and Bond, 3M/ESPE) and a low-shrinking silorane-based composite (Filtek P90 Low Shrink Posterior Restorative, 3M/ESPE; Table 2).

Rubber dam isolation was used for the restorative procedures. The surfaces of restorations and enamel margins were etched with 37% phosphoric acid (Magic Acid Gel, VIGODENT/COLTENE, Rio de Janeiro, Brazil) before adhesive procedures. Materials were used according to the manufacturer's recommendations (Table 3).

Outcome Measurements and Statistical Analysis

At baseline and 12-month recall, all restorations received a clinical rating of Alpha, Bravo, or Charlie. The ordinal dependent variable was the percentage of Alpha, Bravo, or Charlie ratings.

Data management and analysis were done using a statistical analysis system (SPSS 15.0.1 for Windows, SPSS, Chicago, IL). Mann-Whitney test was used to assess differences between the materials tested and for all clinical criteria, at baseline and one-year recall examination (α=0.05). Wilcoxon test was used to compare each composite resin for all clinical criteria at baseline examinations and one-year recall (α=0.05).

RESULTS

In the present study, the main reasons for restorations being repaired were marginal defects (81%) and loss of anatomic form (19%). Of the 100 repaired restorations, 93 (50 for Filtek P60 and 43 for Filtek P90) were examined at baseline. From those, 83 were reexamined at the one-year recall (42 for Filtek P60 and 41 for Filtek P90). The flow of participants and

Table 1: *Modified US Public Health Service Clinical Criteria*

Category	Rating	Criteria Description
Marginal adaptation	Alfa (A)	Restoration adapts closely to the tooth structure; there is no visible crevice
	Bravo (B)	There is a visible crevice, the explorer will penetrate, without dentin exposure
	Charlie (C)	The explorer penetrates into crevice in which dentin or the base is exposed
Anatomic form	Alfa (A)	Anatomic form ideal
	Bravo (B)	Restoration is undercontoured, without dentin or base exposure;
	Charlie (C)	Restoration is undercontoured, with dentin or base exposure; anatomic form is unsatisfactory; restoration needs replacement
Marginal discoloration	Alfa (A)	No marginal discoloration
	Bravo (B)	Minor marginal discoloration without staining toward pulp, only visible using mirror and operating light
	Charlie (C)	Deep discoloration with staining toward pulp, visible at a speaking distance of 60 to 100 cm
Surface roughness	Alfa (A)	As smooth as the surrounding enamel
	Bravo (B)	Rougher than surrounding enamel; improvement by finishing is feasible
	Charlie (C)	Very rough, could become antiesthetic and/or retain biofilm; improvement by finishing is not feasible
Postoperative sensitivity	Alfa (A)	No postoperative sensitivity
	Bravo (B)	Short-term and tolerable postoperative sensitivity
	Charlie (C)	Long-term or intolerable postoperative sensitivity; restoration replacement is necessary
Secondary caries	Alfa (A)	No active caries present
	Charlie (C)	Active caries is present in contact with the restoration

the number of restorations through each examination period of the study are shown in Figure 1. Dropout in this study was about 11% from baseline to one-year recall.

Table 4 summarizes the comparison between the materials tested for all clinical criteria at one-year recall examination and baseline. Bravo ratings can be derived by subtraction, and no restoration received Charlie ratings. No statistically significant difference between the materials was found ($p>0.05$).

Table 5 shows the comparison between baseline and one-year recall examination for each material independently, for all clinical parameters. No statistically significant difference was found in any criteria between the examination periods ($p>0.05$).

DISCUSSION

Silorane is a nonmethacrylate-based resin that has been introduced to control polymerization shrinkage. The new monomer is obtained from the reaction of

Table 2: *Materials: Chemical Composition and Manufacturers*

Material	Chemical Composition	Manufacturer
Magic Acid Gel	37% Phosphoric acid	Vigodent/Coltene
Adper SE Plus Self-Etch Adhesive–Liquid A	Water, HEMA, surfactant, pink colorant	3M/ESPE
Adper SE Plus Self-Etch Adhesive–Liquid B	UDMA, TEGMA, TMPTMA, HEMA, MHP, Bonded zirconia nanofiller, initiator system based ond camphorquinone	3M/ESPE
Filtek P60 Posterior Restorative	Matrix: UDMA (urethane dimethacrylate, TEG-DMA, BIS-EMA; Filler: Silica/Zirconia; Initiator system: Camphorquinone	3M/ESPE
P90 System Adhesive Self-Etch Primer	Phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, water and ethanol, silane-treated silica, initiators and stabilizers	3M/ESPE
P90 System Adhesive Bond	3M/ESPE hydrophobic bifunctional monomer, acidic monomers, silane-treated sílica, initiators and stabilizers	3M/ESPE
Filtek P90 Low Shrink Posterior Restorative	Matrix: silorane; Filler: quartz, yttrium fluoride; Initiator system: camphorquinone, iodonium salts and electron donors; stabilizers and pigments	3M/ESPE

oxirane and siloxane molecules and was developed with the primary purpose of overcoming some drawbacks related to polymerization of dimethacrylate-based composites, such as radical oxygen inhibition, polymerization shrinkage, polymerization stress, water sorption, and instability of conventional monomers in aqueous systems. As a result, silorane has the ability to compensate shrinkage by opening the oxirane ring during polymerization, reducing volume shrinkage to 1% from 1.7% to 3.5% in dimethacrylate-based materials. Because of the presence of siloxane species, the hydrophobicity is also increased.[5,28–30]

Silorane-based composites have been thoroughly investigated by *in vitro* tests, and promising results have been obtained regarding biocompatibility and mechanical characteristics, including reduced polymerization shrinkage.[5,9,31] However, *in vitro* studies are limited in predicting short- and long-term clinical conditions, and laboratory findings should be substantiated by clinical investigations.

Dropout in this study was about 11% after one year. This response rate is in accordance with other similar clinical studies that had rates of 0% to 15% for the first year recall.[13,24,28,32,33] The dropout rates highlight part of the problems associated with long-term clinical studies and having multiple restorations in one patient.[28]

In the present study, the main reasons for repairing restorations were marginal defects and loss of anatomical form. Six modified USPHS criteria—marginal adaptation, anatomic form, surface roughness, marginal discoloration, postoperative sensitivity, and secondary caries—were used to verify the clinical performance of repairs performed on failed dimethacrylate-based composite restorations. No statistically significant differences between the groups were found for all clinical parameters tested at each time interval ($p>0.05$). The frequency of no change in ratings from one-year recall examinations compared with baseline was much higher than the frequency of downgrades from an Alpha to Bravo rating.

It is generally agreed that USPHS criteria may have a limited application since the information provided is too broad; the criteria may also lead to a misinterpretation as a good clinical performance since any changes over time are not easily detected by the limited sensitivity in short-term clinical investigation.[13,34] However, it is the most widely used method for clinical evaluations of restorations worldwide, and the main reason for adopting it relies on the fact that it can be compared with previous

Table 3: *Clinical Sequence of Repair Procedures*

Repair Procedure	Filtek P90/P90 System Adhesive	Filtek P60/ Adper SE Plus
Rubber dam	x	x
Etching of enamel with 37% phosphoric acid for 15 seconds	x	x
Rinse the acid with water and air dried	x	x
Removal of excess water with absorbent paper	x	x
Application of self-etching primer for 15 seconds	x	
Application of liquid A (Adper SE Plus) for 10 seconds		x
Light cured for 10 seconds	x	
Adhesive application with disposable brush	x	
Application of liquid B (Adper SE Plus) for 20 seconds		x
Application of hydrophobic layer		x
Light cured for 10 seconds	x	x
Insertion of 2 mm of maximum thickness horizontal increments and resin sculpture	x	
Insertion of 2 mm of maximum thickness oblique increments and resin sculpture		x
Light curing (600 mW/cm^2)	40 seconds	20 seconds
Removal of excess restorative material with a scalpel blade #15	x	x
Finishing with #9714FF bur (KG Sorensen, Rio de Janeiro, RJ, Brazil)	x	x
Polishing with Enhance System (Dentsply, Petrópolis, RJ, Brazil)	x	x

studies. In addition, this criterion involves visual inspection as well as the use of a dental explorer.[13]

Marginal Adaptation and Secondary Caries

In the current study, no statistically significant differences between the materials tested were found for marginal adaptation for the entire one-year follow-up. There are no results from clinical trials that have tested silorane-based composite as repair material available for comparison. However, a recent clinical trial investigated marginal adaptation of a low-shrinkage silorane-based composite and compared it with a dimethacrylate-based composite material across the same time interval.[28] Even though this study had outcomes related to total-replaced restorations, they disagree with the findings from the present study, since a better performance was found for the dimethacrylate-based composite material. Laboratory studies have shown lower values of polymerization shrinkage related to silorane-based composites, but it is difficult to show the effects in clinical studies, where so many factors influence the final result.[5,35,36]

In this study, no statistically significant differences have been found between the materials tested for secondary caries. Because secondary caries are

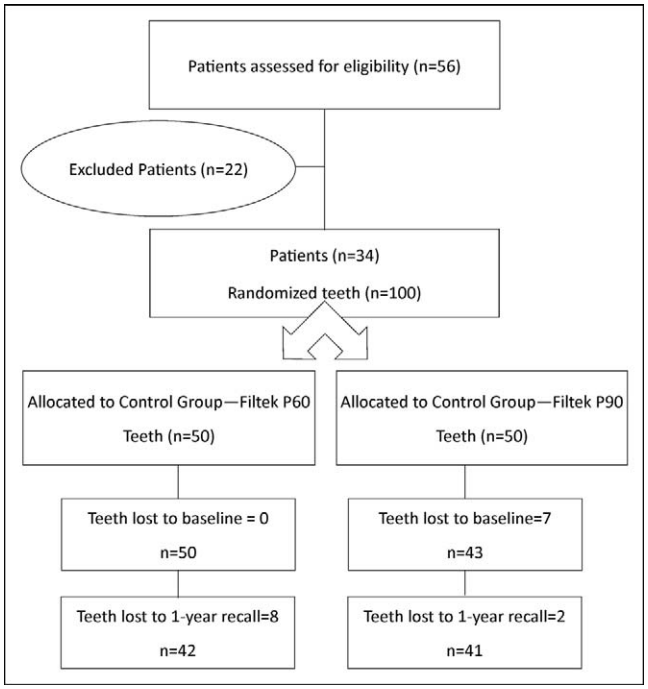

Figure 1. *Flowchart of patients and number of restorations through each stage of the study.*

usually associated with marginal integrity and marginal adaptation is usually associated with reduced polymerization shrinkage, we expected favorable results for a low-shrinkage resin-based composite.[5] On the other hand, the observation time reported in this study may not be considered long enough for the development of secondary caries. Furthermore, patients in the study did not develop carious lesions, most likely because those with inadequate oral hygiene (VPI>30%) and decreased salivary flow were excluded.

Anatomic Form

No statistically significant difference was found when each composite resin was evaluated independently at baseline and after one year. Regarding the difference of 10% in the baseline results between the two materials, this did not remain in the one-year results since some restorations scored better at follow-up than at baseline. It may reflect the difficulty of assessing some criteria clinically, even with an interexaminer agreement ratio $\geq$0.78. Moreover, if a study requires recording of minute detail, calibration becomes difficult with the concomitant risk of recording differences in clinical judgment between evaluators rather than between experimental and control groups.[35]

In addition, general practitioners in five European countries were asked to rate several handling criteria of the Filtek P90 on a five-point scale, in which rating 1 was assigned for an excellent performance and rating 5 for a poor performance. Regarding the criteria sculptability, the best score assigned for Filtek P90 was 3 (3M ESPE, Filtek P90 Technical Profile). This was also observed in the present study and could explain the percentage of Bravo ratings registered for anatomic form in the test group.

In general, restorations remained stable and unchanged over the first-year observation period. Previous studies that have investigated the longev-

Table 4: *Comparison Between the Materials Tested for All Clinical Criteria at Each Examination Period*

Frequency Number of Restorations Rated Alpha and Percentage (%)						
	Baseline			**One Year**		
	Filtek P60	**Filtek P90**	***p* Value**	**Filtek P60**	**Filtek P90**	***p* Value**
Marginal adaptation	47 (94.0)	43 (100.0)	0.104	40 (95.2)	40 (97.6)	0.573
Anatomic form	49 (98.0)	38 (88.4)	0.061	40 (95.2)	36 (87.8)	0.226
Surface roughness	40 (80.0)	28 (65.1)	0.108	30 (71.4)	26 (63.4)	0.439
Marginal discoloration	49 (98.0)	43 (100.0)	0.354	42 (100.0)	38 (92.7)	0.076
Postoperative sensitivity	50 (100.0)	41 (95.3)	0.125	42 (100.0)	41 (100.0)	1.00
Secondary caries	50 (100.0)	43 (100.0)	1.00	42 (100.0)	41 (100.0)	1.00

Table 5: *Comparison Between Each Material Independently for All Clinical Parameters, at Baseline and at One-Year Recall Examination*

Frequency Number of Restorations Rated Alpha and Percentage (%)							
		Marginal Adaptation	**Anatomic Form**	**Surface Roughness**	**Marginal Discoloration**	**Postoperative Sensitivity**	**Secondary Caries**
Filtek P60	Baseline	47 (94.0)	49 (98.0)	40 (80.0)	49 (98.0)	50 (100.0)	50 (100.0)
	12-month	40 (95.2)	40 (95.2)	30 (71.4)	42 (100.0)	42 (100.0)	42 (100.0)
p value		0.317	0.317	0.180	0.317	1.00	1.00
Filtek P90	Baseline	43 (100.0)	38 (88.4)	28 (65.1)	43 (100.0)	41 (95.3)	43 (100.0)
	12-month	40 (97.6)	36 (87.8)	26 (63.4)	38 (92.7)	41 (100.0)	41 (100.0)
p value		0.317	1.00	0.317	0.083	0.317	1.00

ity of dimethacrylate-based restoration by minimal intervention have found the same good performance when dimethacrylate-based composites were used as repair materials.[13,14,24]

Surface Roughness

The surface roughness property of any material is the result of the interaction of multiple factors. Some of them are related to the material itself, such as the filler (type, shape, size, and distribution of the particles), the type of resinous matrix as well as the ultimate degree of cure reached, and the bond efficiency at the filler-matrix interface.[37,38] In this context, a direct correlation was found between the hardness and surface roughness, indicating that a composite with a higher hardness value is usually associated with a higher surface roughness.[38,39]

In the current study, no statistically significant difference between the materials was found for surface roughness at any recall examination. However, there is a trend that indicates a problem, since there is a 15% difference in roughness between the materials at baseline and an 8% difference at follow-up, indicating a better performance to the methacrylate-based composite resin. A previous study has shown a higher Knoop hardness for Filtek P90 than for dimethacrylate-based composites due to its organic matrix composed mainly by silorane resin and inorganic particles as quartz and yttrium fluoride (76% by weight),[40] explaining the current findings for surface roughness.

Furthermore, the percentages of Bravo ratings found for both materials could be explained by the fact that assessment "alpha" is given to a surface as smooth as the surrounding enamel, and maybe the evaluators were very critical in their evaluation, since it is known that there is no material to replace all the qualities of the enamel, and this especially applies for its smooth, polished surface.[41]

Marginal Discoloration

For marginal discoloration, no statistically significant difference between the two materials was found at recall examinations. In a recent study related to the repair potential of composite resin materials, the highest bond strength when a dimethacrylate-based composite was used as substrate was when Filtek P90 was used as the repair material and the P90 System as the adhesive. Although it is customarily assumed that the bond between old and new composite is micromechanical, data from when Filtek P90 was the substrate suggest that there is a possibility of chemical bonding, most likely because products that contain a silane coupling agent have improved the wettability of the substrate surface and the ability to effect a chemical (siloxane) bind to inorganic filler particles; in Filtek P90, these are silanated ceramics.[27]

Postoperative Sensitivity

Initial postoperative sensitivity has been reported in clinical studies with resin-based composites, but the sensitivity generally decreases during the first

weeks after placement of restorations.[28,42] At baseline examination, the low incidence of restorations that received a Bravo rating can be explained by the use of a self-etching bonding system in both treatment groups. These systems make the smear layers part of the hybrid layer, providing better penetration of the monomers onto the collagen fibers of the demineralized dentin. At follow-up, the same good performance was observed for all composites, likely because resin-based agents may provide pulp protection as long as the dentin is sealed by hydrophilic resins.[28,42]

Thereby, the null hypothesis tested in this study was confirmed since the low-shrinkage silorane-based composites exhibited a similar clinical performance to the dimethacrylate-based composites when repairing dimethacrylate-based composite restorations after a one-year observation period. Nevertheless, it is appropriate to highlight that short-term results can provide only an early prediction of the material clinical performance and that no evidence of early failure or sudden change in the clinical characteristics occurred. Longer observation periods are thus necessary to confirm these findings.

CONCLUSIONS

This clinical trial shows that low-shrinkage silorane-based composites exhibited a similar performance to the conventional dimethacrylate-based composites when used to repair composite resin restorations.

Repairs with a different resin chemistry were successful as long as the bonding agent was of the new chemistry being used for the repair.

After one year, the reduced polymerization shrinkage assigned to silorane-based composites did not establish better clinical performance, indicating that laboratory findings should be substantiated by clinical investigations, and a long-term answer to the question should be determined after a longer recall period.

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Conflict of Interest

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

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Comparative Evaluation of Microleakage of Silorane-based Composite and Nanohybrid Composite With or Without Polyethylene Fiber Inserts in Class II Restorations: An In Vitro Study

VS Agrawal • VV Parekh • NC Shah

Clinical Relevance

Fiber inserts incorporated at the gingival floor of class II composite restorations resulted in a highly significant reduction of microleakage. Also, silorane composites based on a ring-opening mechanism showed reduced microleakage.

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SUMMARY

Aim: To evaluate microleakage between nanocomposite and silorane composite in class II restorations with or without a polyethylene fiber insert.

Methodology: Standardized class II cavities were prepared on extracted molars and randomly divided into 4 groups (n=20 each): group 1, Ceram X mono; group 2, Ceram X mono + Ribbond; group 3, Filtek P90; and Group 4, Filtek P90 + Ribbond. All specimens were subjected to a thermocycling regime, immersed in 2% methylene blue dye for 24 hours,

sectioned longitudinally, and examined under a stereomicroscope to assess dye penetration on a six-point scale. The score data were subjected to statistical analysis whereby Kruskal-Wallis analysis of variance was used for multiple group comparisons and the Mann-Whitney test for groupwise comparisons at a significance level of $p \leq 0.05$.

Results: A statistically significant decrease in microleakage was found when Ribbond fiber was used with nanoceramic and silorane composite. A highly significant decrease in microleakage scores was found in silorane composite when compared to nanoceramic composite.

Conclusion: Use of polyethylene fiber and silorane composite reduces microleakage in class II composite restorations with gingival margins below the cemento-enamel junction.

INTRODUCTION

Dental resin composites have become the most popular and widely used direct restorative material in today's clinical dentistry.[1,2] Although composites are now the material of choice for most restorations, the polymerization shrinkage of 2.6%-7.1% and, thus, microleakage is one of the most frequently encountered problems, especially at gingival margins placed apical to the cemento-enamel junction, as in deep class II cavities[3–5]

Microleakage has led to the development of recurrent caries, postoperative sensitivity, enamel fracture, marginal staining, and eventual failure of restorations.[6] Various studies have reported efforts to develop methods to decrease this problem by slowing down the composite polymerization rate,[7] using an incremental placement technique[8] or low modulus intermediate layer,[9] and reducing the C factor (the ratio of bonded to unbonded restoration surfaces).[10]

Ribbond fibers are bondable, reinforced, patented Leno Weave, ultrahigh strength polyethylene fibers.[11] A high modulus of elasticity and lower flexural modulus of the polyethylene fiber was reported to have a modifying effect on the interfacial stresses developed along the etched enamel-resin boundary. By embedding a polyethylene fiber into the bed of a composite before restoration, higher microtensile bond strength could be achieved in prepared cavities with a high C factor.[12] Also, it will reduce the total amount of resin matrix required to restore the cavity, and their strength will help decrease the shrinkage.[4,13]

Siloranes make up a new category of composite resin matrix based on ring-opening monomers obtained from the reaction of oxirane and siloxane molecules. The volumetric shrinkage of silorane-based composite was determined to be 0.99 vol % using Archimedes' method.[14] Reduced shrinkage in silorane is due to opening and extending the oxirane rings during polymerization, compensating for volume reduction.[1,14]

This study determined the effect of polyethylene fiber inserts and silorane composites on reducing the gingival microleakage in class II composite restorations placed apical to the cemento-enamel junction and comparing it with nanoceramic composite.

MATERIALS AND METHODS

Eighty extracted intact mandibular first and second molars were selected, cleaned with a periodontal scaler (Satelec, Gustave Eiffel, BP, France), and stored in 0.5% chloramine T solution for 1 month.

All the teeth were embedded in poly (vinyl) siloxane impression material such that it was 2 mm below the cemento-enamel junction, and standardized mesio-occlusal/disto-occlusal class II cavities were prepared using round bur No 4 and No. 245 straight fissure diamond burs (Mani Inc.,Utsunomiya, Tochigi, Japan) in a high-speed air-turbine hand piece (NSK, Tochigi-Ken, Japan) with copious water irrigation (burs were replaced after every eight preparations) to the following dimensions ($\pm$0.3 mm): 2.0-mm occlusal isthmus depth, 5.0-mm facio-lingual proximal box width occlusally and 5.5 mm gingivally, 2.5-mm pulpal-proximal box depth occlusally and 1.5 mm gingivally, and 6.0- to 8.0-mm proximal box height but always terminating 1.0 mm below the cemento-enamel junction.

The dimensions were verified with help of a UNC-15 periodontal probe. No bevels were placed at cavo-surface margins.

Prepared teeth were randomly divided into four groups (n=20) as described below. A universal Tofflemire retainer (API, Schweinfurt, Germany) with matrix band (Hahnenkratt, Benzstrasse, Germany) was placed around each prepared tooth and supported externally by applying low-fusing compound (DPI Manufacturers Inc, Mumbai, India).

Group 1

Teeth were restored with Prime & Bond NT and Ceram X (Dentsply DeTrey GmbH, Germany). After application of etching gel (Dentsply Caulk, GmbH, Germany) for 15 seconds, the cavity was blot dried,

leaving a moist surface. Prime & Bond NT was applied twice to thoroughly wet all the cavity surfaces for 20 seconds. The cavity was gently air dried for 5 seconds to evaporate solvent carrier followed by light curing for 10 seconds with a halogen light-curing unit (Unicorn Med., Korea). Ceram X was dispensed directly into the prepared cavity in 2-mm increments by the oblique layering method; starting in the proximal box, the first increment was placed at a 45-degree angle to the facio-gingivo-proximal line angle and cured for 40 seconds. Next, increments were placed and packed at the linguo-proximal box and finally in the occlusal portion of the box and the isthmus and cured for 40 seconds. After removal of the band, the composite was cured from all the sides again for 40 seconds.

Group 2

Teeth were restored with Prime & Bond NT, Ceram X, and Ribbond fibers (Ribbond, Inc, WA, USA). Acid etching and bonding was similarly carried out as in group 1. But before restoration with Ceram X, a <1-mm-thick amount of Ceram X was first placed on the gingival floor. Then one Ribbond fiber insert, approximately 1 mm less than the bucco-lingual dimension of the proximal box, was cut, impregnated with Prime & Bond NT, and condensed into the bed of −1mm composite resin and light cured for 40 seconds. Then Ceram X was dispensed into the remainder of the prepared cavity in 2-mm increments using the oblique layering technique as in group 1.

Group 3

Teeth were restored with P90 system adhesive self-etch primer + P90 system adhesive bond + Filtek P 90 (3M ESPE, St Paul, MN, USA). After each cavity was cleaned with a water spray and blot dried, P90 system adhesive self-etch primer was applied to the cavity surfaces for 15 seconds, followed by gentle air dispersion and light curing for 10 seconds. P90 system adhesive bond was applied to wet all the cavity surfaces, followed by gentle air dispersion and light curing for 10 seconds. Filtek P90 was dispensed directly into the prepared cavity in 2-mm increments by the oblique layering method similarly to that of Ceram X in group 1.

Group 4

Teeth were restored with P90 system adhesive self-etch primer + P90 system adhesive bond + Filtek P 90 + Ribbond Fibers. Acid etching and bonding was similarly carried out as in group 3. But before restoration with Filtek P90, one Ribbond fiber insert was placed at the gingival floor similar to that in group 2, and Filtek P90 was dispensed into the remainder of the prepared cavity in 2-mm increments using the oblique layering technique as in group 1.

A similar shade (B1) was used for all the materials. The intensity of the light-curing unit was measured as 800 mW/cm^2 using an intensity meter (Optilux radiometer, Kerr Corp, Sybron Dental Specialties, Orange, CA, USA).

All restorations were finished with a graded series of aluminum oxide discs (Sof-Lex TM, St Paul, 3M ESPE) according to manufacturer's instructions and were subjected to thermocycling according to the International Organization for Standardization (ISO) standard 11405[21] for 500 cycles at 5°C-55°C with a 30-second dwell time.

For microleakage evaluation, the apical 2 mm of each tooth was sectioned, retrograde cavity prepared, and sealed with resin-modified glass ionomer cement (GC Fuji II LC, GC Corp, Tokyo, Japan). Also, teeth were coated with two layers of nail varnish (Sunshine Cosmetics, Metoda, India) except for an area 1 mm around the gingival cavosurface margin of the restorations. Specimens were then immersed in 2% methylene blue dye buffered at pH=7 (Merck Specialties Private Ltd,, Mumbai, India) at 37°C for 24 hours, washed, and dried. All the teeth were mounted on acrylic blocks and longitudinally sectioned mesio-distally from the center of the restoration with a diamond disk (Sunshine Diamonds, Langenhagen, Germany) at a low speed and with continuous irrigation of water.

Dye penetration was evaluated at the gingival margin with a stereomicroscope (Motic Microscopes, China) at 40× magnification, and microleakage was scored according to the six-point scale used in the study by El-Mowafy and others[4] and as described in Table 1.

The median of the scores were subjected to statistical analysis using the nonparametric Kruskal-Wallis analysis of variance test and the Mann-Whitney test at a 95% significance level. Statistical analysis was done using the SPSS 11.0 program (SPSS Inc, Chicago, IL, USA).

RESULTS

Descriptive statistics, including the mean ranks and median leakage scores, are shown in Table 2. The Kruskal-Wallis test revealed highly significant dif-

Table 1: *Definition of Dye Penetration Scores*

Scores	Definition
0	No dye penetration
1	Dye penetration extending to the outer half of the gingival floor
2	Dye penetration extending to the inner half of the gingival floor
3	Dye penetration extending through the gingival floor up to one-third of the axial wall
4	Dye penetration extending through the gingival floor up to two-thirds of the axial wall
5	Dye penetration extending through the gingival floor up to full length of the axial wall

ferences in microleakage scores among the groups (p=0.000).

The Mann-Whitney U-test was used to make a pairwise comparison between the four studied groups, and it showed a significant decrease in microleakage scores when a Ribbond fiber insert was used; that is, group 2 showed a significant decrease in microleakage (p=0.020) when compared to group 1, and group 4 showed a significant decrease in microleakage (p=0.014) when compared to group 3 (Table 3).

Also, the Mann-Whitney U-test showed a highly significant decrease in microleakage scores in the silorane composite groups compared to the nano-ceramic composite groups; that is, groups 3 and 4 showed a highly significant decrease in microleakage ($p<0.01$) when compared to groups 1 and 2 (Table 3). Referring to mean rank values (Table 1), we can also conclude that group 4 had the least microleakage and that group 1 has the maximum microleakage scores. Figure 1 shows microleakage scores in representative specimens of test groups under a stereomicroscope.

DISCUSSION

Microleakage of composite restorations occurs because of stresses placed along the tooth/restoration interface from polymerization shrinkage, temperature fluctuations in the oral environment, and mechanical fatigue through repetitive masticatory loading.[15] Previous studies reported that composite restorations showed relatively greater microleakage at gingival rather than occlusal margins.[3,4] The most likely cause for this phenomenon is polymerization contraction, which is toward the "stronger" enamel-composite joint and the light source.[4,10]

The incremental oblique layering placement of light-activated composites has been recommended to decrease overall contraction and residual stresses at the tooth/restoration interface and thus to decrease microleakage by reducing the bulk of material cured at one time.[8,13] In the present study, the oblique layering technique for incremental placement of composite described by Tjan and others[8] was used for placing the restorations.

The various methods to detect microleakage include the dye leakage method, the use of color-producing microorganisms, radioactive isotopes, the air pressure method, neutron activation analysis, electrochemical studies, scanning electron microscopy, thermal and mechanical cycling, and chemical tracers.[16] Since there is no gold standard method for

Table 2: *Descriptive Statistics of Kruskal-Wallis Test*

Groups	N	Mean Rank	Chi-Square	Degree of Freedom	*p*-Value	Significant Difference?
Group 1 (Ceram X)	20	33.40	30.765	3	0.000	Yes
Group 2 (Ceram X + Ribbond)	20	26.60				
Group 3 (Filtek P90)	20	14.50				
Group 4 (Filtek P90 + Ribbond)	20	7.50				
Total	80					

Table 3: *Mann-Whitney U-Test Exhibits Significant Difference Between Groups*

Group	Group	*U*-Value	*p*-Value (Two Tailed)	Significant Difference?
Group 1 (Ceram X)	Group 2 (Ceram X + Ribbond)	20.500	0.020	Yes
Group 1 (Ceram X)	Group 3 (Filtek P90)	0.500	0.000	Yes
Group 1 (Ceram X)	Group 4 (Filtek P90 + Ribbond)	0.000	0.000	Yes
Group 2 (Ceram X + Ribbond)	Group 3 (Filtek P90)	8.500	0.001	Yes
Group 2 (Ceram X + Ribbond)	Group 4 (Filtek P90 + Ribbond)	1.000	0.000	Yes
Group 3 (Filtek P90)	Group 4 (Filtek P90 + Ribbond)	19.000	0.014	Yes

microleakage evaluation, we used the dye leakage method because it did not require the use of complex laboratory equipment and because it is nondestructive, thus allowing the longitudinal study of restorative margins.[17] However, concerns have been raised, as there is lack of evidence supporting any correlation between clinical testing and in vitro dye penetration testing.[18]

Researchers suggested the use of a dye with a particle diameter equal to the bacterial size or somewhat smaller (around 2 μm).[19] For this reason, we used in our study a 2% solution of the methylene blue molecule (one methylene blue molecule = 1.2 $nm^2 = 120A^{0.2}$), of which the particle size is less than that of the bacterial one.[19] Also, methylene blue dye provides an excellent contrast with the surrounding environment along with a perfect and easy visualization of the prepared cavity in the digital images, which gives the evaluators a clear reference point from which to score. Moreover, as methylene blue is normally acidic (pH=3), it may demineralize enamel and dentin, generating more microleakage; hence, the solution was buffered to pH=7. Storage time for dye penetration varies from 10 seconds to 180 days. An extended penetration time might become a problem in more hydrophilic, self-etching adhesives, as they might absorb water and dye to a higher extent than conventional etch and rinse adhesives. Ernst and others[20] showed that a 30-minute dye penetration time seems to be suitable for marking marginal gaps. Nevertheless, a penetration time of 24 hours is mostly used in the literature for in-depth determination of marginal gaps.[2,20] As methylene blue is a delicate dye substance that is not stable in the presence of acidic and basic substances as well as when it is exposed to ambient light, in this study care was taken to ensure that the results were not influenced by the reaction of acidic or basic monomers that are in the adhesive systems by buffering the solution to pH=7 and penetration time of 24 hours.

For thermocycling, the temperature range of 5°C-55°C with a dwell time of 30 seconds for 500 cycles was used according to the ISO TR11405 standard,[21] and this is the estimate of the range that has been reported on the surfaces of molar teeth in the mouth of the patient.

The results showed a significant decrease in microleakage at the gingival margin when Ribbond

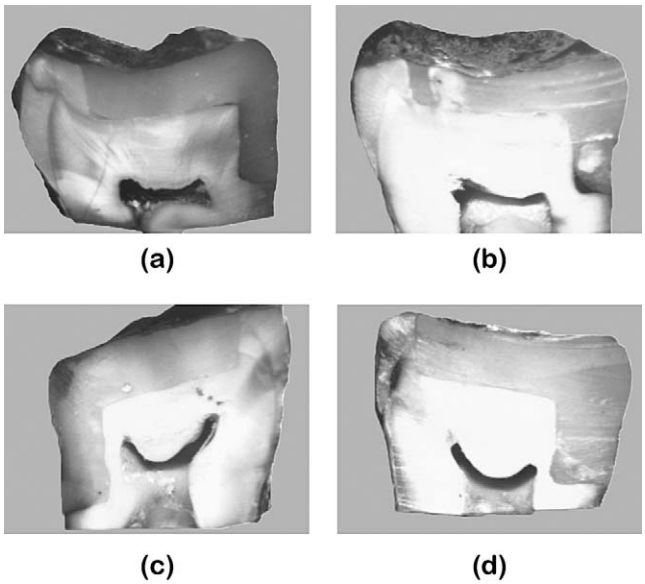

Figure 1. *(a) Representative specimen from Group 1 showing score 5 (b) Representative specimen from Group 2 showing score 3 (c) Representative specimen from Group 3 showing score 2 (d) Representative specimen from Group 4 showing score 0.*

fibers were incorporated. Placement of a fiber insert reduces gingival microleakage, as it replace part of the composite increment at this location, resulting in a decrease in overall volumetric polymerization contraction.[4,13] Also, fibers assist the initial increment of composite in resisting pull-away from the margins toward the curing light.[4,13] The fibers also have the strengthening effect of a composite margin, increasing resistance to the dimensional change or deformation that occurs during thermal and mechanical loading and hence improving marginal adaptation.[22] Studies conducted by El-Mowafy and others[4] and Ozel and Soyman [13] also showed that insertion of fiber insert reduces the gingival microleakage in class II composite restorations. In contrast, Belli and others[9] showed that there is a reduction in microleakage only at the occlusal margins and not at the gingival margins after placing the fiber inserts. One reason for such a contrasting result might be that all the restorations placed in this study were in bulk rather than incrementally placed.

The results of our study also showed a highly significant decrease in microleakage score found in cavities restored with silorane composite compared to nanoceramic composite. This might be attributed to the inherent ring-opening polymerization of silorane monomers, which can compensate for volume reduction as molecules come closer to each other, compared to radical polymerization of other composites, which is linear polymerization, manifested as a reduction in polymerization shrinkage stress at the tooth/restoration interface.[23,24] Studies conducted by Bagis,[23] Al-Boni and Raja,[24] and Yamazaki[25] also proved that microleakage with silorane is lower than that of methacrylate-based composites. These results contrast with those of Ernst,[20] who proved that microleakage of teeth restored with silorane is similar to that restored with methacrylate composites. The reason for this could be that the author had used an all-in-one (seventh generation) experimental bonding agent of silorane previously produced by the company, and in the present study, the new bonding agent produced with silorane, which is a two-step, two-component bond (sixth generation) is used, thereby giving different results.

The modulus of elasticity of the restorative material can also be considered one of the causes of marginal microleakage, thus the importance of applying an intermediary layer with a low elasticity module or a stress breaker layer.[9] This layer would then provide enough flexibility to compensate for the tension generated by polymerization shrinkage. Thus, a higher modulus of elasticity and lower flexural modulus of the polyethylene fiber might have a modifying effect on interfacial stresses and result in decreased microleakage.[9]

Although these are *in vitro* results, they are of significance because these factors cannot easily be quantitatively determined *in vivo*. Nevertheless, further clinical studies are necessary to confirm these results and evaluate their relevance to treatment outcome.

CONCLUSION

Within the limitations of this study, it can be concluded that the use of polyethylene fiber inserts and silorane composite significantly reduces microleakage in class II resin composite restorations with gingival margins below the cemento-enamel junction compared to the methacrylate-based nanoceramic composite.

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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