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Clinical Technique

Accomplishing Esthetics Using Enamel Microabrasion and Bleaching—A Case Report

RH Sundfeld • LM Franco • RS Gonçalves
RS de Alexandre • LS Machado • DS Neto

Clinical Relevance

This article discusses indications and limitations of enamel microabrasion treatment and indicates that this technique may be a promising alternative to restoring severe stains.

SUMMARY

This case report describes the sequential steps that were used to treat unesthetic, white, hard-texture enamel stains of unknown etiology. A tapered fine diamond bur was used to remove superficial enamel followed by the use of an enamel microabrasion compound Opalustre (Ultradent Products Inc). This technique removed the stains and was followed by polishing with a fluoride paste to restore the enamel to a smooth finish. The teeth were subsequently bleached with carbamide peroxide (Opalescence 10%, Ultradent Products), which achieved the patient's desired esthetic results.

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INTRODUCTION

Since the study by Croll and Cavanaugh in 1986,[1] the application of enamel microabrasion to remove hard, superficial enamel stains has routinely been used with considerable success.[1-4] Extensive studies have been performed to develop enamel microabrasive products that ensure adequate safety for oral tissues while allowing for ease of use.[5,6] Enamel microabrasive materials are currently composed of low concentration hydrochloric acid and silica carbide microparticles, which offer a good margin of safety during application to the operator and the patient.[3,5]

Several publications have reported on techniques to reduce the time needed for enamel microabrasion.[3,7,8] It has been suggested that clinicians begin the treatment by performing enamel macroreduction

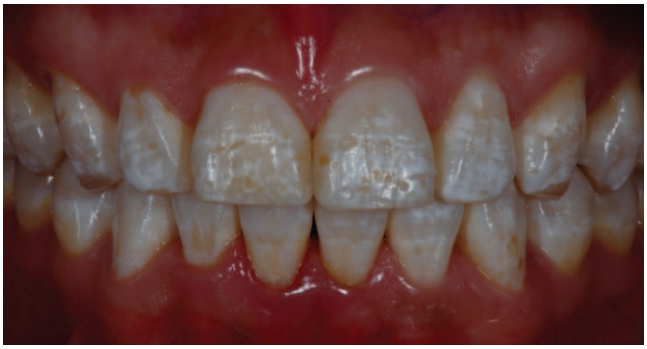

Figure 1. *A 28-year-old man with intrinsic white stains that have a hard texture, located on the buccal and lingual enamel surface of all maxillary incisors, canines, and premolars.*

using a tapered fine diamond bur to lightly abrade the affected area. This is followed by application of an enamel microabrasive paste to complete the removal of remaining stains and to smooth the enamel surface that was ground by the diamond bur.[3,4]

The enamel microabrasion technique causes a negligible loss of enamel[5,9]; however, teeth may acquire a darker and yellower hue in areas where the enamel has been reduced to remove the stains, thus revealing the underlying dentin. In these cases, a color correction may be accomplished using carbamide peroxide or hydrogen peroxide.[3] The case reported here presents a protocol for an enamel microabrasion technique that removes stains from the buccal enamel surface and is followed by bleaching with carbamide peroxide to acquire the desired esthetic results.

CLINICAL CASE

A 28-year-old patient presented at Araçatuba Dental School—UNESP, Araçatuba, Brazil, complaining about white enamel stains and some eroded areas on his upper and lower teeth (Figure 1). A clinical examination determined the white stains to be intrinsic and of a hard texture (Figure 2). The patient was offered the option of an enamel microabrasion treatment on the buccal surfaces of incisors, canines, and premolars of the maxillary and mandibular teeth, followed by bleaching. The steps involved in this proposed treatment were explained. After consenting to the treatment, and in preparation for the bleaching phase, alginate impressions were made, poured in stone, and used to fabricate custom bleaching trays.

After a dental prophylaxis with pumice and water, a high-speed tapered fine diamond bur (No. 3195 FF, KG Sorensen Indústria e Comércio Ltda, Barueri, São Paulo, Brazil) was used under copious irrigation, and the superficial layer of the stained enamel was removed (macroreduction) (Figure 3). A layer of petroleum jelly was applied to the gingival tissues, followed by the placement of a rubber dam, to protect the gingiva from the microabrasion compound (Figure 4). The patient, assistants, and operator all wore eye protection during the procedures.

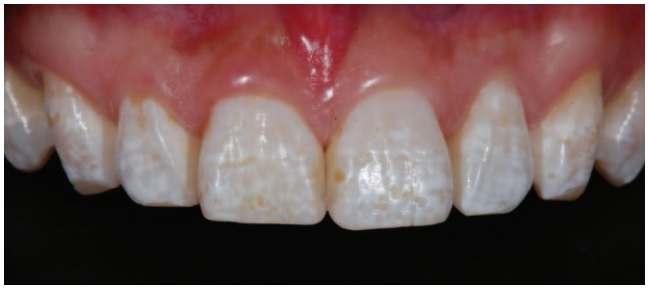

Figure 2. *Intrinsic white stains with a hard texture located on the buccal enamel surface of all maxillary incisors, canines, and premolars.*

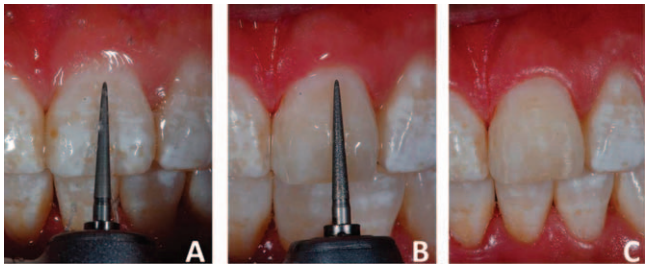

Figure 3. *The use of a tapered fine diamond bur No. 3195 FF on the buccal surfaces of the maxillary incisors, canines, and premolars.*

An enamel microabrasive product containing a mild concentration of hydrochloric acid (6%) and a fine-grit silicon carbide abrasive in a water-soluble gel (Opalustre, Ultradent Products Inc, South Jordan, Utah, USA) was subsequently used to remove the remaining stains (Figure 5). This compound was applied using a specially designed rubber cup for a low speed hand piece under firm pressure. The

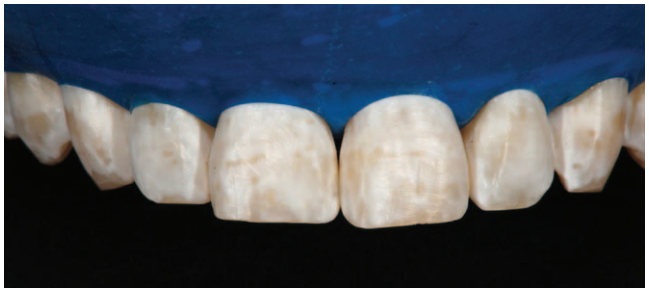

Figure 4. *Total isolation with a rubber dam.*

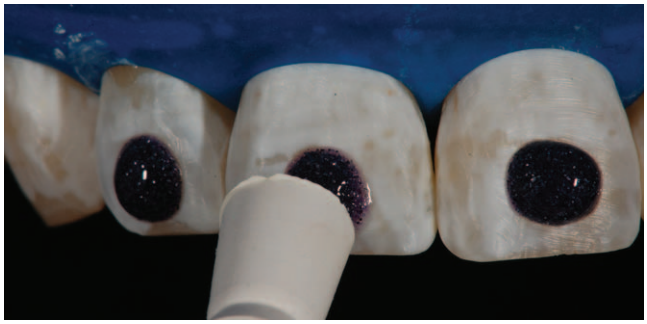

Figure 5. *Application of Opalustre (Ultradent Products) for 1 minute.*

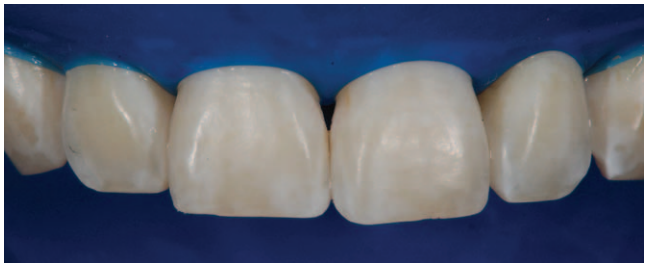

Figure 6. *Appearance immediately after performing enamel microabrasion.*

abrasive compound was applied in 3 applications of 1 minute each, with irrigation between each application (Figure 6).

After the abrasion compound treatment, the surfaces were polished with a 1200-ppm fluoride paste (Herjos, Vigodent SA Indústria e Comércio, Rio de Janeiro, Brazil) (Figure 7). A 2% neutral-pH sodium fluoride gel was applied to the enamel surfaces for 4 minutes (Figure 8). After removal of the rubber dam, the patient was advised not to ingest solids or liquids for at least 30 minutes (Figure 9).

After completion of the enamel microabrasion, the patient was instructed in the use of the bleaching trays and supplied with 10% carbamide peroxide (Opalescence, Ultradent Products Inc, South Jordan, Utah, USA)[4] (Figure 10), in an application of a

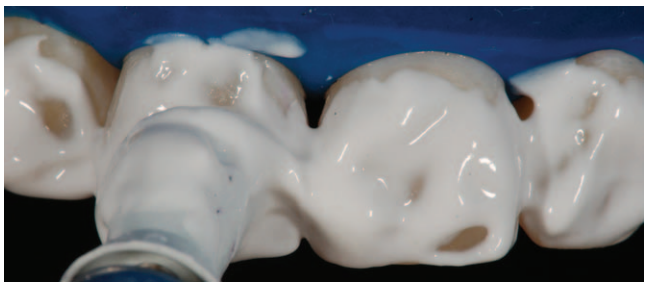

Figure 7. *Polishing with fluoride paste.*

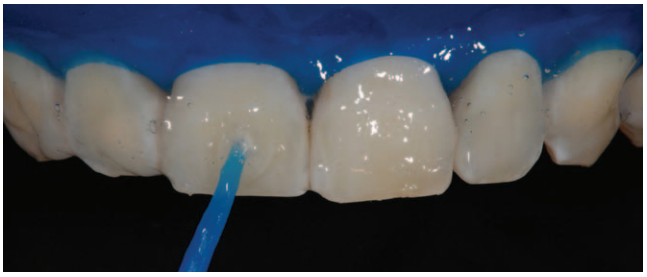

Figure 8. *Application of 2% neutral-pH sodium fluoride gel.*

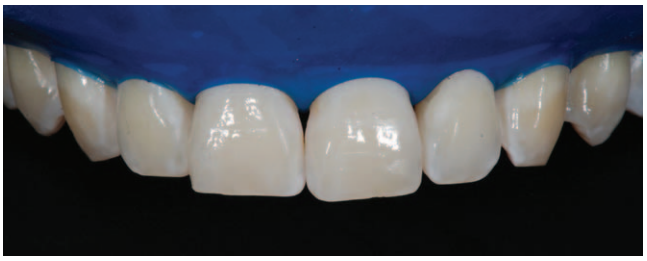

Figure 9. *Appearance immediately after microabrasion, polishing, and application of topical fluoride paste.*

dentist-supervised home bleaching protocol following the manufacturer's instructions. The patient placed a small amount of bleaching gel in each tooth indentation, inserted the tray and wore it overnight. Initially, the patient wore the tray for 4 weeks; however, the patient was required to use the bleaching trays for 2 additional weeks to achieve the desired esthetic results. After reaching the esthetic end result (Figures 11 and 12), the patient received topical applications of 2% neutral-pH sodium fluoride gel for 4 minutes each day for 1 week at the office.[3]

DISCUSSION

Undesirable esthetics of enamel and dentin can be resolved with enamel microabrasion and bleaching techniques. This current clinical case addressed esthetic concerns and is in agreement with reports

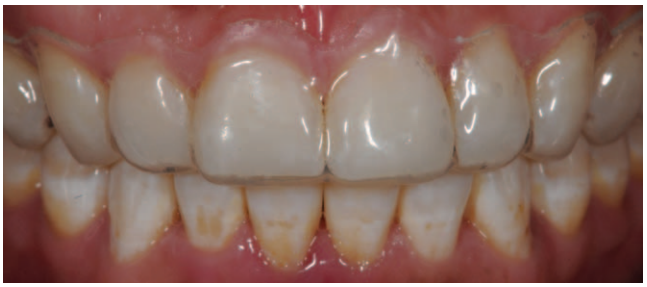

Figure 10. *Dental bleaching with carbamide peroxide (Opalescence 10%, Ultradent Products).*

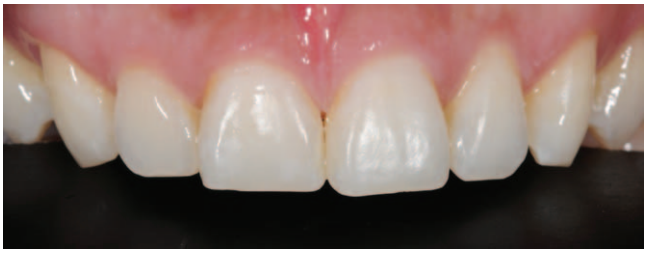

Figure 11. *Final esthetic results after completion of the treatment.*

from other authors.[2-4,10-13] The most likely diagnosis of the white enamel stains in the present case was dental fluorosis. This was based on the fact that the teeth showed opaque white spots on the enamel with hypoplastic areas and mild erosion. Dental fluorosis is caused by a change in enamel from excessive ingestion of fluoride during tooth development. Fluoride intake over and above what is present in fluoridated water, fluoride toothpastes, and fluoride supplements is considered a risk factor for dental fluorosis.[14] However, in this clinical case and based on the clinical history, excessive intake of fluoride at the time of tooth development was not considered a factor. Killian in 1993[10] and Croll in 1998[2] used the terms "fluorosis-type stains" and "fluorosis-like dysmineralization," respectively, to describe the chromatic alterations in the enamel surface resulting from a disturbance in the process of enamel mineralization. It has been reported[3,4] that the texture of the intrinsic white stain (ie, a stain of hard texture and of any color presenting on the buccal surface of the anterior and premolar teeth), and not its etiology, is the main indicator for the microabrasive procedure.

The microabrasion procedure to remove enamel stains causes minimal loss of enamel when related to the total amount of remaining enamel,[9] and clinically over time,[3] promotes the achievement of an enamel surface with considerable uniformity, smoothness, and luster, which is known as the "abrosion effect". This, according to Croll in 1991,[5] Donly and others in 1992,[15] and Segura in 1993,[16] is the result of the deposition of mineral substances from the abrasive and erosive action of the microabrasive compound.

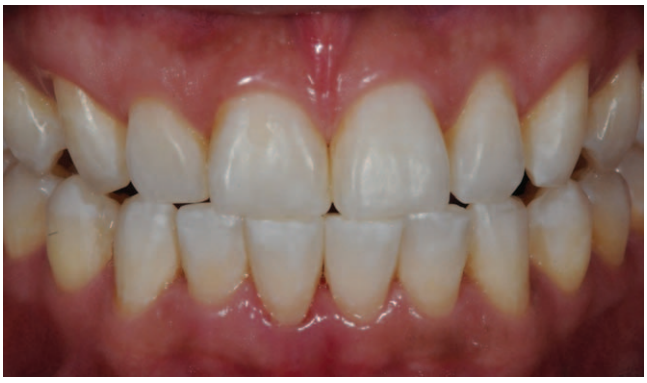

Figure 12. *Final esthetic appearance.*

Based on years of experience in clinical practice,[3,4,7,17] it has also been observed that using a tapered, fine diamond bur as a first step results in less clinical time for the procedure. Using two or three applications of microabrasion, at intervals of 1 minute each, allows the microabrasive product to produce the desired esthetic appearance. Other than the advantages stated earlier, the stains in the presented case were removed and did not return.

In some cases, the patient will present with lip incompetence. This clinical condition greatly hinders the formation of moistened film on enamel surfaces[4] and routine follow-up with the dentist is important to ensure that the stains are not returning. However, it is recommended that patients who present with lip incompetence be referred to an orthodontist who can correct the labial positioning before beginning the microabrasion procedure.[3]

Bleaching with carbamide peroxide can result in considerable improvement of esthetics that becomes more apparent after the removal of enamel stains. Bleaching should be performed only under professional supervision and preferably on patients who do not present with exposed dentin, as this may cause dentin sensitivity during bleaching. Over time, the sensitivity disappears without the need for intervention.

CONCLUSION

Enamel microabrasion, followed by tooth bleaching, can result in a satisfactory improvement of the esthetics of a patient.

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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.

(Accepted 10 April 2013)

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Clinical Research

Clinical Performance of One-step Self-etch Adhesives Applied Actively in Cervical Lesions: 24-month Clinical Trial

C Zander-Grande • R C Amaral • AD Loguercio
LP Barroso • A Reis

Clinical Relevance

The active application mode seems to be an easy and effective clinical alternative to improve the retention rates and reduce marginal staining of one-step self-etch adhesives in noncarious cervical lesions.

SUMMARY

Objectives: To evaluate the clinical performance of two one-step self-etch adhesives in noncarious cervical lesions (NCCL) under active or passive application mode.

Methods: Thirty-one patients with four NCCL were enrolled in this study. One hundred and twenty-four restorations were placed according to one of the following conditions: 1) Adper Prompt L-Pop (AP), active application (APA); 2) AP, passive application (APP); 3) Xeno III (XE), active application (XEA), or 4) XE, passive application (XEP). The restorations were evaluated by the FDI World Dental Federation criteria at baseline and after six, 12, and 24 months of clinical service. The effects of adhesive, mode of application, and recall period were assessed via mixed generalized linear model (α=0.05).

Results: The adhesive AP and the passive application mode showed significantly higher marginal staining than did XE and active application, respectively ($p<0.05$). With regard to the retention rates, the active application mode yielded higher retention rates at the 24-month recall compared to the passive application, regardless of the material. The individual

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retention rates (95% confidence interval) of both adhesives in the active application mode were the same, 96.8% (83.8-99.4%), while in the passive application rates were 87.1% (71.2-94.9%) and 80.7% (63.7-90.8%) for XE and AP, respectively.

Conclusions: The active application improved the retention rates of both adhesives after 24 months and minimized the marginal staining at enamel margins.

INTRODUCTION

The etiology of noncarious cervical lesions (NCCL) is quite variable, but their prevalence is increasing as the adult population continues to age.[1,2] Often, these lesions need to be restored as a result of sensitivity or for esthetic reasons or in such a way as to prevent further loss of dental structure. In general, the retention of resin-based composites in NCCL has shown considerable increase;[3,4] however, as reported by Van Dijken,[3] there is a vast range of adhesives available from which to choose, including the older three-step version to the most recent one-step self-etch systems.

One-step self-etch adhesive systems require shorter clinical application times and they are less technique sensitive.[5,6] The elimination of separate etching and rinsing steps has simplified the bonding technique and has been responsible for the increased popularity of these systems in daily practice. These systems do not require removal of the smear layer and smear plugs, as they are incorporated into the hybrid layer complex.[4]

Unfortunately, different studies[7,8] have shown that some one-step self-etch adhesives produce relatively low bond strength values and inferior marginal adaptation to both enamel and dentin when compared to two-step self-etch or etch-and-rinse systems, findings confirmed in a recent systematic review of clinical studies.[9] The authors have reported that the clinical effectiveness of one-step self-etch adhesives was the least efficient among all classes of available adhesives. This may be partially attributed to the acidity of the adhesive system, since the interaction of the material with the underlying substrate may be quite superficial[10] for low acidic materials, which may preclude an adequate retention of the restorative resin-based composite.

In vitro studies have reported that the bond strength of self-etch adhesives to enamel[11-13] and the dentin[12,14-16] can be improved by vigorous agitation. This clinical approach was shown[15,17] to increase the durability of adhesive interfaces produced with one-step self-etch adhesives when applied to dentin. It has been suggested[8,18] that the active primer application may improve smear layer dissolution and improve micromechanical interlocking and chemical interaction with dentin.

Despite the favorable laboratory findings with this technique, only one study[19] evaluated this approach clinically, but a conventional etch-and-rinse system was employed. To the extent of the authors' knowledge, no study has so far addressed the benefits of active application under a clinical scenario using self-etch adhesives. Though *in vitro* testing methodology with aging protocols tends to predict clinical performance,[20,21] clinical trials remain necessary to evaluate the ultimate clinical efficacy of adhesives and/or clinical techniques. Thus, the aim of this randomized clinical trial was to evaluate the influence of the application method of two one-step self-etch adhesives placed in NCCL after 24 months of clinical service. The null hypothesis tested was that the retention rates of both materials will be similar after 24 months of clinical service, regardless of the application mode.

MATERIALS AND METHODS

Study Design and Participant Selection

The study was reported following the CONSORT statement.[22] This was a randomized, double-blind clinical trial. The local Ethics Committee on Investigations Involving Human Subjects reviewed and approved the protocol and consent form for this study (protocol 6291/06).

Inclusion and Exclusion Criteria

A total of 48 participants were examined to determine if they met the inclusion and exclusion criteria (described below) (Figure 1) by three calibrated dental students. The evaluations were performed using a mouth mirror, an explorer, and a periodontal probe. Participants had to be healthy and at least 18 years old. They had to have an acceptable oral hygiene level and present with at least 20 teeth under occlusion. They were required to have at least four NCCL to be restored in four different teeth. These lesions had to be noncarious and nonretentive (greater than 1 mm deep) and involve both the enamel and dentin of vital teeth without mobility. The cavo-surface margin could not involve more that 50% of the enamel.[4]

All patients were given oral hygiene instructions before operative treatment was performed. Patients

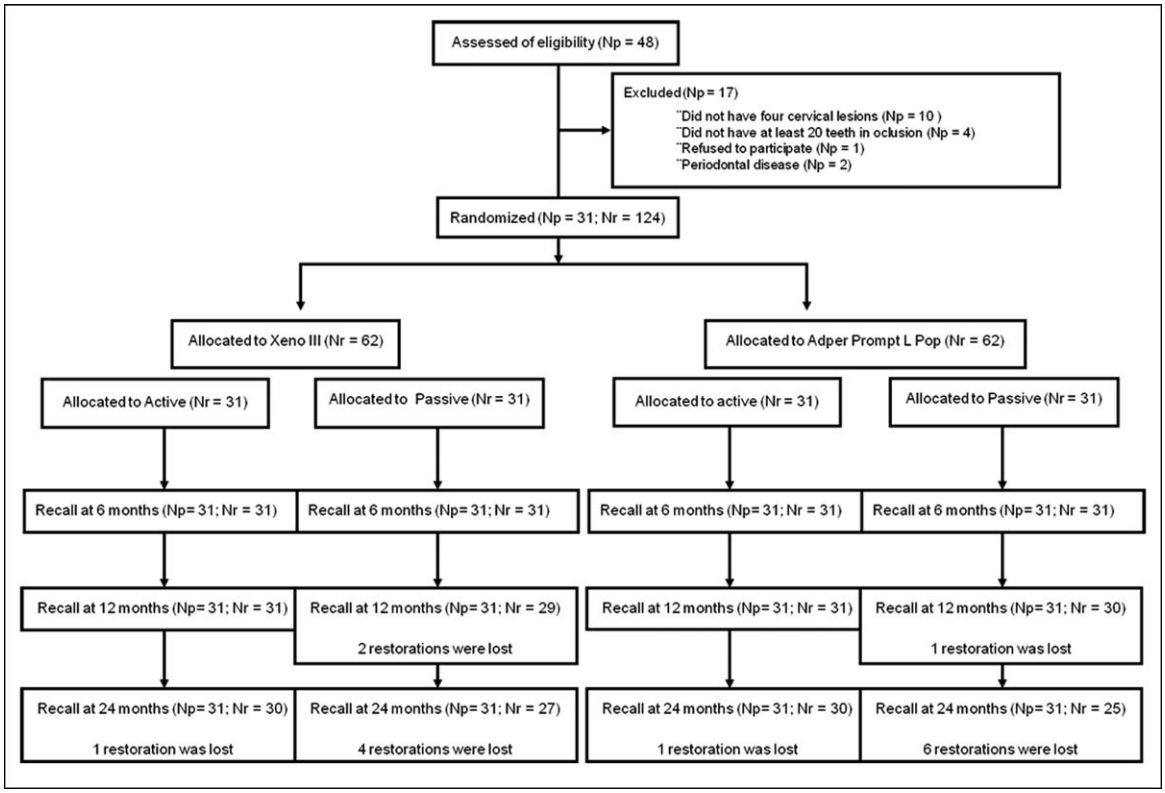

Figure 1. *Flow diagram. Np, number of patients; Nr, number of restorations.*

with extremely poor oral hygiene, severe or chronic periodontitis, or heavy bruxism habits were excluded from the study.

Interventions: Restorative Procedure

Two weeks before the restorative procedures, all of the volunteers received a dental screening and a dental prophylaxis with pumice and water in a rubber cup and signed an informed consent form.

Before restoration placement, some features of the NCCL were evaluated. The degree of sclerotic dentin was measured according to the criteria described by Swift and others[23] (Table 1). The cavity dimensions in millimeters (height, width, and depth) and the geometry of the cavity (evaluated by photograph profile and labeled at $<45°$, 45-90°, 90-135°, and $>135°$) were also recorded. Other features, such as the presence of antagonist and attrition facet, were observed and recorded. These features were recorded to allow comparison of the baseline features of the dentin cavities among experimental groups.

The previously calibrated dental students who participated in the patient screening selection restored all teeth under the supervision of an experienced clinician.[19] All participants received four restorations, one of each experimental group in different teeth with similar characteristic, such as depth, shape, dentin sclerosis, and others.

The operator classified the order of the teeth to be restored. After that, the groups were described in

Table 1: *Dentin Sclerosis Scale*[a]

Category	Criteria
1	No sclerosis present; dentin is light yellowish or whitish, with little discoloration; dentin is opaque, with little translucency or transparency
2	More sclerosis than in category 1 but less than halfway between categories 1 and 4
3	Less sclerosis than in category 4 but more than halfway between categories 1 and 4
4	Significant sclerosis present; dentin is dark yellow or even discolored (brownish); glassy appearance, with significant translucency or transparency evident

[a] *Adapted from Swift and others.*

Table 2: *Adhesive Systems: Composition and Application Mode*

Adhesive Systems	Composition	Mode of Application	Application Mode[a]
Adper Prompt L-Pop (AD; 3M ESPE, St Paul, MN, USA)	Liquid 1 (red blister): methacrylated phosphoric esters, bis-GMA, initiators based on camphorquinone and stabilizers Liquid 2 (yellow blister): water, HEMA, polyalkenoic acid and stabilizers	Active (manufacturer's recommendation)	a, b1, c, b1, c, d
		Passive	a, b2, c, b2, c, d
Xeno III (XE; Dentsply Caulk, Milford, DE, USA)	Liquid A (green cap): HEMA, purified water, ethanol, UDMA, BHT, highly dispersed silicon dioxide Liquid B (black cap): phosphoric acid modified polymethacrylate resin, PEM-F, modified methacrylate resin, UDMA, camphorquinone, ethyl-4-dimethylaminobenzoate	Active	a, b3, c, d
		Passive (manufacturer's recommendation)	a, b4, c, d

Abbreviations: BHT, butylated hydroxy toluene; Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; PEM-F, pentamethacryloyloxyethylcyclohexaphosphazene monofluoride; UDMA, urethane dimethacrylate.
[a] a: Dispense equal amounts of Liquid A or 1 and Liquid B or 2 and mix them in mixing well thoroughly (five seconds); b1: Apply one coat with agitation for 15-20 seconds; b2: Apply one coat passively and leave undisturbed for at least 15-20 seconds; b3: Apply one coat with agitation for 20 seconds; b4: Apply one coat passively and leave undisturbed for at least 20 seconds; c: air-dry for 10 seconds at 20 cm; d: light-cure (10 seconds, 600 mW/cm^2).

opaque papers that were folded and randomly selected by a third person not involved in the research protocol. The allocation assignment of the four groups was revealed by opening these folded papers on the day of the restorative procedure. The operator was not blinded to group assignment when administering interventions; however, participants were blinded to the group assignment.

Before rubber dam placement, the operators anesthetized the teeth (Mepisv 3%, NovaDFL, Rio de Janeiro, RJ, Brazil) and cleaned all lesions with pumice and water in a rubber cup (reference #8040RA and #8045RA, KG Sorensen, Barueri, SP, Brazil); this step was followed by rinsing and drying. With a shade selection guide, the proper shade of the composite was determined. According to the American Dental Association (ADA) guidelines,[24] the operators did not prepare any additional retention or bevel.

Next, the cavities received the self-etch adhesive systems Adper Prompt L-Pop (AP; 3M ESPE, St Paul, MN, USA) or Xeno III (XE; Dentsply Caulk, Milford, DE, USA) applied under active or passive application. Their composition, application mode, and batch number are described in Table 2.

1. Passive application (P): In these groups, the adhesive was only spread over the entire surface for approximately three to five seconds and was left undisturbed for 15 to 20 seconds (Table 2). Then an airstream was applied for 10 seconds at a distance of 20 cm. The air-dry pressure used was 40 psi (0.27 MPa).
2. Active application (A): The adhesive was rigorously agitated on the entire dentin surface for approximately 15 to 20 seconds (Table 2). The microbrush was scrubbed on the dentin surface under manual pressure (equivalent to approximately 34.5 $\pm$ 6.9 *g*, tested in an analytical balance before the beginning of the clinical trial). An airstream was applied for 10 seconds at a distance of 20 cm. The air-dry pressure used was 40 psi (0.27 MPa).

The resin-based composites Esthet X (Dentsply Caulk) and Filtek Z250 (3M ESPE) were used in combination with XE or AP, respectively. The cavities were restored in three increments, and each increment was light-cured for 40 seconds (VIP light-curing unit, Bisco Inc, Schaumburg, IL, USA; 600 mW/cm^2). The restorations were finished with fine-grit diamond burs, and the polishing procedure was performed with abrasive discs (Sof-Lex Pop-On discs, 3M ESPE) one week after placement of the restorations.

Sample Size Calculation

The retention rate of the antecessor of AP, commercially available as Prompt L-Pop, was reported[25] to be 69% after 12 months of clinical service. With an α of 0.05, a power of 80%, and a two-sided test, the minimal sample size was 31 restorations in each group in order to detect a difference of 25% among the tested groups.[26]

Clinical Evaluation

Two experienced and calibrated examiners who were not involved in the placement of the restorations and who were therefore blinded to the group assignment performed the evaluation. For training purposes, the examiners observed 10 photographs that were representative of each score for each criterion. They

Table 3: *FDI Criteria Used for Clinical Evaluation (Hickel and others[28,29])*

	Esthetic Property	Functional Properties		Biological Properties	
	1. Staining Margin	**2. Fractures and Retention**	**3. Marginal Adaptation**	**4. Postoperative Sensitivity**	**5. Secondary Caries**
1. Clinically very good	1.1. No marginal staining	2.1. Restoration retained, no fractures/cracks	3.1. Harmonious outline, no gaps, no discoloration	4.1. No hypersensitivity	5.1. No secondary or primary caries
2. Clinically good (after correction very good	1.2. Minor marginal staining, easily removable by polishing	2.2. Small hairline crack	3.2.1. Marginal gap (50 μm) 3.2.2 Small marginal fracture removable by polishing	4.2. Low hypersensitivity for a limited period of time	5.2. Very small and localized demineralization. No operative treatment required
3. Clinically sufficient/ satisfactory (minor shortcomings with no adverse effects but not adjustable without damage to the tooth)	1.3. Moderate marginal staining, not esthetically unacceptable	2.3. Two or more or larger hairline cracks and/or chipping (not affecting the marginal integrity)	3.3.1. Gap <150 μm not removable 3.3.2. Several small enamel or dentin fractures	4.3.1. Premature/ slightly more intense 11.3.2. Delayed/ weak sensitivity; no subjective complaints, no treatment needed..	5.3. Larger areas of demineralization, but only preventive measures necessary (dentine not exposed)
4. Clinically unsatisfactory (repair for prophylactic reasons)	1.4. Pronounced marginal staining; major intervention necessary for improvement	2.4. Chipping fractures that damage marginal quality; bulk fractures with or without partial loss (less than half of the restoration)	3.4.1. Gap >250 μm or dentine/base exposed 3.4.2. Chip fracture damaging margins 3.4.3. Notable enamel or dentine wall fracture	4.4.1. Premature/ very intense 4.4.2. Extremely delayed/weak with subjective complaints 4.4.3. Negative sensitivity; intervention necessary but not replacement	5.4. Caries with cavitation (localized and accessible and can be repaired)
5. Clinically poor (replacement necessary)	1.5. Deep marginal staining not accessible for intervention	2.5. (Partial or complete) loss of restoration	3.5. Filling is loose but *in situ*	4.5. Very intense, acute pulpitis or nonvital Endodontic treatment is necessary and restoration has to be replaced	5.5. Deep secondary caries or exposed dentine that is not accessible for repair of restoration
Acceptable or not acceptable (n, %, and reasons)	Esthetic criteria	Functional criteria		Biological criteria	

evaluated 10 to 15 teeth in two different clinical appointments to allow intraexaminer agreement. The intraexaminer and interexaminer agreement of at least 85% was necessary before the beginning of the evaluation.[27] The evaluation paper form of each recall was not available for the next evaluation, so that evaluators were blinded to group assignment during follow-up recalls.

The restorations were evaluated by the FDI World Dental Federation criteria[28,29] at baseline and after six, 12, and 24 months of clinical service. Only the most relevant items for testing the adhesive performance were selected (Table 3). The primary measurable variable was restoration retention/fractures, but the following secondary measurable variables were also evaluated: marginal staining, marginal adaptation, postoperative sensitivity, and recurrence of caries. Those measurable variables were ranked with the following scores: clinically very good, clinically good, clinically sufficient/satisfactory, clinically unsatisfactory, and clinically poor. Both examiners evaluated all the restorations once and independently. When disagreements occurred during the evaluations, the examiners had to reach a consensus before the participant was dismissed. The restoration retention rates were calculated according to the ADA guidelines.[24]

Statistical Analysis

Descriptive statistics were used to describe the distributions of the evaluated criteria. Statistical analysis was performed for each item as well as for

each property. The effects of adhesive, mode of application, and time were assessed via mixed generalized linear model[30] associated to a link function. This was required because the items marginal adaptation and caries recurrence had only two responses, and, thus, a binary model should be used for this analysis. For all others items, a multinomial model was used. As the four groups were always placed in the same patient, the patient was considered the repeated measure.

Two different statistical analyses were run. One followed the intention-to-treat protocol, which included all teeth in their originally randomized groups, even those that were not able to be analyzed during the scheduled recall visits. In this case, we filled in the missing data by carrying the last observed value of such teeth. The second approach followed the per-protocol or on-treatment approach, in which the participants with missing data were excluded from the statistical analysis. The first approach is more conservative and less open to bias than the second one and is recommended by the CONSORT statement.[22]

RESULTS

The restorative procedures were implemented exactly as planned and no modification was performed. No subgroup analysis was done. Seventeen out of 48 participants initially screened were excluded from the study because they did not fulfill the inclusion criteria. Thus, a total of 31 subjects (12 men and 19 women), with a mean age of 48 years, were enrolled. One hundred and twenty-four restorations were placed, 31 for each group (Figure 1). All details regarding the research subjects and characteristics of the restored lesions are presented in Table 4.

Both statistical analyses (intention-to-treat and per-protocol) led to similar conclusions (data not shown), and the *p*-values reported in this section are from the intention-to-treat analysis. All research subjects were evaluated at baseline and in the six-, 12-, and 24-month recalls. When a restoration was lost, the other criteria could not be evaluated as the restoration was no longer in place. In the intention-to-treat protocol, we filled in this missing data by carrying the last observed value of such teeth.

Esthetic Properties

With regard to the marginal staining, only the main factors of adhesive ($p=0.011$), application mode ($p=0.008$), and recall period ($p<0.001$) were statistically significant. At the 24-month recall, four

Table 4: *Demographic Characteristic of Research Subject and Features of Noncarious Cervical Lesions (NCCL)*

Characteristics	Number of Lesions			
Research subjects				
Gender distribution				
Male	48			
Female	76			
Age distribution, y				
20-29	04			
30-39	36			
39-49	40			
>49	44			
	XEA	XEP	APA	APP
NCCL				
Shape, degree of angle				
<45	01	01	01	01
45-90	08	08	09	10
90-135	12	14	10	12
>135	10	08	11	08
Cervico-incisal height, mm				
<1.5	02	02	02	02
1.5-2.5	09	03	09	06
>2.5	20	26	20	23
Degree of sclerotic dentin				
1	10	09	11	08
2	18	19	17	20
3	02	02	01	01
4	01	01	02	02
Presence of antagonist				
Yes	29	29	30	28
No	02	02	01	03
Attrition facet				
Yes	26	26	25	25
No	05	05	06	06
Preoperative sensitivity, spontaneous				
Yes	15	15	17	15
No	16	16	14	16
Tooth distribution				
Incisors	02	02	02	02
Canines	05	06	08	09
Premolars	21	20	20	19
Molars	03	03	01	01
Arch distribution				
Maxillary	15	15	13	13
Mandibular	16	16	18	18

Abbreviations: APA, Adper Prompt L-Pop, active application; APP, Adper Prompt L-Pop, passive application; XEA, Xeno III, active application; XEP, Xeno III, passive application.

Table 5: *Number of Restorations for Each Experimental Group Classified According to the FDI Criteria (Hickel and others[28,29])*

Hickel Criteria [a]		Baseline				Six Months				12 Months				24 Months			
		XEA	XEP	APA	APP	XEA	XEP	APA	APP	XEA	XEP	APA	APP	XEA	XEP	APA	APP
1. Marginal staining	VG	31	31	31	31	31	31	31	31	28	23	24	20	26	18	22	12
	GO	—	—	—	—	—	—	—	—	03	06	07	09	04	08	08	12
	SS	—	—	—	—	—	—	—	—	—	—	—	—	—	01	—	01
	UN	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	PO	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
2. Fractures and retention	VG	31	31	31	31	31	31	31	31	31	27	30	26	28	19	26	15
	GO	—	—	—	—	—	—	—	—	—	02	01	03	02	06	04	08
	SS	—	—	—	—	—	—	—	—	—	—	—	—	—	02	—	02
	UN	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	PO	—	—	—	—	—	—	—	—	—	02	—	02	01	04	01	06
3. Marginal adaptation	VG	31	31	31	31	31	31	31	31	27	27	28	23	24	19	23	16
	GO	—	—	—	—	—	—	—	—	04	02	03	06	06	08	07	09
	SS	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	UN	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	PO	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
4. Postoperative sensitivity	VG	29	31	30	30	31	31	31	31	31	29	31	29	30	27	30	25
	GO	02	—	01	01	—	—	—	—	—	—	—	—	—	—	—	—
	SS	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	UN	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	PO	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
5. Secondary caries	VG	31	31	31	31	31	31	31	31	29	31	27	28	28	26	29	22
	GO	—	—	—	—	—	—	—	—	02	—	01	01	02	01	02	03
	SS	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	UN	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
	PO	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—

Abbreviations: APA, Adper Prompt L-Pop, active application; APP, Adper Prompt L-Pop, passive application; XEA, Xeno III, active application; XEP, Xeno III, passive application.
[a] *VG, clinically very good; GO, clinically good; SS, clinically sufficient/satisfactory; UN, clinically unsatisfactory; and PO, clinically poor.*

restorations from XEA, nine from XEP, eight from ADA, and 13 from APP showed marginal staining (Table 5). The adhesive AP and the passive application mode showed significantly higher marginal staining than did XE and active application, respectively. The overall marginal staining in the active and passive groups was 19.4% (95% confidence interval [CI], 11.4-30.9%) and 35.5% (95% CI, 24.7-47.9%) (Figure 2). The marginal staining was statistically higher in the 12- and 24-month recall in comparison with the baseline and six-month findings.

Functional Properties

For marginal adaptation, only the main factor of recall period was statistically significant ($p<0.001$), meaning that lack of marginal adaptation was more often observed in the 12- and 24-month recalls compared to baseline and six-month findings. Although at the 24-month recall a total of 30 restorations showed a lack of marginal adaptation in the enamel margins, they were all considered clinically good (Table 5).

With regard to the retention rates, only the main factors of application mode ($p<0.0001$) and recall

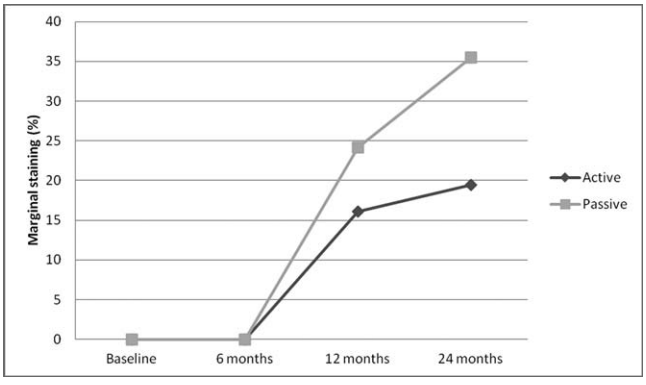

Figure 2. *Overall marginal staining for active and passive application groups.*

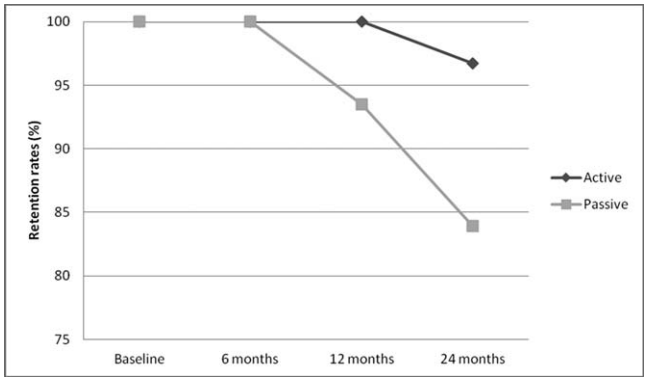

Figure 3. *Overall retention rates for active and passive application groups.*

period ($p<0.0001$) were statistically significant. The active application mode yielded higher retention rates at the 24-month recall compared to the passive application, regardless of the material. A total of two and 10 restorations from the active and passive application modes, respectively, were lost and considered clinically not acceptable (Table 5). The overall retention rates for the active and passive groups were 96.8% (95% CI, 89.0-99.1%) and 83.9% (95% CI, 72.8-91.0%), respectively. The individual retention rates of XE and AD in the active application mode were the same, 96.8% (95% CI, 83.8-99.4%). In the passive application, the rates were 87.1% (95% CI, 71.2-94.9%) and 80.7% (95% CI, 63.7-90.8%) for XE and AD, respectively (Table 5; Figure 3).

For the item fracture, the main factors of adhesive ($p<0.01$) and recall period ($p<0.001$) were statistically significant. More fractures were observed for the passive application group (eight for XEP and 10 for APP) than for the passive application (two for XEA and four for APA). The adhesive AP showed significantly more fractures than did XE, and this was shown to increase in the latter recall periods (12- and 24-month recall vs baseline and six-month recall). These 24 small fractures detected at the 24-month recall were all considered clinically good (Table 5).

Biological Properties

For the item secondary caries only the main factor of recall time ($p=0.003$) was statistically significant. After 24 months, a total of eight restorations showed signs of secondary caries, but they were all ranked as clinically good. With regard to postoperative sensitivity, no difference was detected among groups since no report of postoperative sensitivity was recorded throughout the study period.

Overall Analysis

The overall analysis of the study results (Table 6) demonstrated that only the lack of retention of the clinical restorations were considered clinically unacceptable, and this loss of Class V restorations could be significantly improved by the active adhesive application.

DISCUSSION

Although one-step self-etch adhesive systems are marketed as simplified materials, a more complex chemistry is required to blend hydrophilic and hydrophobic monomers, water, solvents, and additives.[5,31] Water is essential, because it provides the ionization medium for the self-etch activity. Other solvents, such as acetone and ethanol, are necessary to dissolve both hydrophilic and hydrophobic monomers into one single phase[32] and to assist water to evaporate upon completion of the self-etch process. Because of these features, one-step self-etch adhesives are reported[33,34] to behave as permeable membranes after polymerization, allowing the diffusion of water from the hybridized dentin to the adhesive surfaces.[35] Altogether these factors have been blamed for the lower retention rates of one-step

Table 6: *Restorations Acceptable or Not Acceptable According to the FDI Criteria at the 24-Month Recall (Hickel and others[28,29])*

	Esthetic				Functional								Biological							
	1. Staining Margin				2. Fractures and Retention				3. Marginal Adaptation				4. Postoperative (Hyper-)Sensitivity				5. Recurrence of Caries			
	XEA	XEP	APA	APP	XEA	XEP	APA	APP	XEA	XEP	APA	APP	XEA	XEP	APA	APP	XEA	XEP	APA	APP
Acceptable	31	31	31	31	30	27	30	25	31	31	31	31	31	31	31	31	31	31	31	31
Not acceptable	00	00	00	00	01	04	01	06	00	00	00	00	00	00	00	00	00	00	00	00
Reasons					Total loss of the restorations															

Abbreviations: APA, Adper Prompt L-Pop, active application; APP, Adper Prompt L-Pop, passive application; XEA, Xeno III, active application; XEP, Xeno III, passive application.

self-etch systems in clinical trials when compared to the two-step version.[9,36,37]

In the present investigation, the retention rates of both adhesives investigated were increased by active adhesive application, and this led us to reject the anticipated null hypothesis. Retention rates of 96.8% were observed for both adhesives under active application. Other studies[25,38-42] reported retention rates varying from 75% to 90% for AP after one to two years of clinical service and rates varying from 90% to 93% for XE (Dentsply). Compared with existing literature, the retention rates of XE and AP applied actively in the present study were the highest already reported after 24 months of clinical service, resembling those achieved with three-step etch-and-rinse systems.[9,43]

The active application may accelerate solvent evaporation and increase the diffusion rate of monomer inside the smear layer, carrying fresh acidic resin monomers to the basal part of the etched dentin. This may produce a more aggressive demineralization[44] and promote a better interaction with the smear layer and underlying dentin.[16,18] This method can also increase the moieties' kinetics and allow for better monomer diffusion inward, while solvents are diffusing outward. Laboratory tests investigating the active application of the etch-and-rinse adhesives[45,46] and the self-etch adhesives[12,14-16] have also reflected good results. This was also confirmed in *in vitro* longevity studies for the one-step self-etch[15,17] and in one recent clinical trial[19] that employed an acetone-based two-step etch-and-rinse adhesive system.

With regard to the marginal staining, this began to be observed in the present study after 12 months, mainly for the passive application groups. Marginal staining is primarily the result of infiltration of colored molecules into the interface and/or inside the adhesive layer. Simplified one-step self-etch adhesives are much more hydrophilic than their two-step counterparts.[47] The hydrophilic nature of methacrylate copolymers allows increased water sorption,[47-49] and this is a time-driven process, meaning that the more the contact of the adhesive with water, the higher the water sorption ratio.[47-49]

As a consequence of polymer swelling caused by water, the frictional forces between the polymer chains are reduced,[50] leading to solubility of residual monomers and oligomers, which makes the adhesive interface even more porous and prone to marginal staining over time. The prevalence of marginal staining was similar to that described in previous clinical studies[19,41,51-53] and in most of the cases would be easily solved by re-polishing the restoration margins.[40] The lower marginal staining rates in the active application groups may be attributed to the improved quality of the polymer. This could be achieved as a result of the higher water/solvent evaporation and also as a result of deeper etching of the enamel margins and, consequently, better resin infiltration.[54]

In terms of marginal adaptation, all groups were similar to one another, but lack of marginal adaptation was increasingly seen in the later recall periods. These findings can also justify the increase of the marginal staining, as it has already been demonstrated[39,55] that marginal adaptation is usually correlated with marginal staining.

The intervention and control groups were implemented in participants of both sexes who were older than 18 years of age. Therefore, the results of the present trial apply for an adult population having NCCL with similar features to the ones selected to be included in this clinical trial. Further long-term evaluations should be carried out in order to detect if the positive findings of the active application still remain after a longer clinical service period.

CONCLUSIONS

Within the limitations of the present study, we can conclude that the active application of one-step self-etch systems improved the retention rates of Class V restorations and minimized the marginal staining in the enamel margins. As long as one-step self-etch systems are employed actively, retention rates similar to those reported in the literature for three-step etch-and-rinse systems can be achieved.

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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 in any product, service, and/or company that is presented in this article.

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Assessment of the Effect of Casein Phosphopeptide–amorphous Calcium Phosphate on Postoperative Sensitivity Associated With In-office Vital Tooth Whitening

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Clinical Relevance

Tooth sensitivity is the most frequent adverse effect of in-office vital tooth whitening. The use of a desensitizing gel after tooth whitening may reduce the incidence and severity of in-office vital tooth-whitening sensitivity.

SUMMARY

The aim of this study was to evaluate the efficacy of tooth mousse containing 10% casein phosphopeptide-amorphous calcium phosphate (CPP-ACP) in reducing tooth sensitivity associated with in-office vital tooth whitening. In-office tooth whitening was performed for 51 participants using 35% hydrogen peroxide gel in a single visit. After the procedure, each participant was randomly assigned to one of three groups: gel without desensitizing agent (n=17), gel with 2% sodium fluoride (n=17), gel with 10% CPP-ACP (n=17). A small amount of the desensitizing gel assigned for each participant was applied directly on the labial surfaces of teeth and left undisturbed for three minutes. The participants were asked to apply the gel assigned to them for three minutes twice daily after brushing their teeth, and to continue this for 14 days. The participants were asked to return for follow-up visits after 24 hours and on days 3, 7, and 14, at which time teeth shade changes were assessed by one evaluator using a value-oriented Vita classic shade guide. The incidence, duration, and intensity of tooth sensitivity experienced was

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self-assessed on a daily basis for the 14-day study period using a visual analog scale (VAS). The effect of the three gels on tooth sensitivity was assessed using one-way analysis of variance and a χ^2 test (α=0.05). The general linear model was used to compare intensity-level differences in the three studied groups and for shade stability over the follow-up period. The results of this study showed that all three gels decreased the intensity of sensitivity associated with tooth whitening. The intensity of sensitivity was lower in the fluoride group than in the other two groups; however, it was not statistically significant (p=0.112 and p=0.532 on day 1 and day 2, respectively). The average shade change was 6.8. None of the tested materials affected the efficacy of tooth whitening, but the shade change among the fluoride group showed more color stability than that of the other two groups. This study suggested that using a gel after tooth whitening can reduce the intensity of tooth sensitivity associated with in-office whitening procedures without affecting the efficiency of tooth whitening. However, it failed to demonstrate that using a 10% CPP-ACP could provide additional therapeutic benefits.

INTRODUCTION

Tooth discoloration is a frequent dental finding, associated with clinical and esthetic problems. Invasive esthetic restorative dentistry includes many treatment modalities, which range from the routine placement of composite resin restorations to porcelain veneers, all-ceramic full and partial coverage restorations, and porcelain-metal restorations. Veneers and crowns are relatively difficult to make and are technique sensitive. Consequently, simpler, faster, and successful conservative whitening procedures have gained wider acceptance.[1,2]

Discoloration arises from a variety of causes, discolored teeth differ widely in appearance, severity, and modes of treatment. Detailed clinical examination and a review of the patient's oral hygiene practices; dietary habits; and history of exposure to chemicals, trauma, and infection are essential in making a final diagnosis of the cause of tooth discoloration and consequently the type of tooth whitening.[3] Vital tooth whitening is typically classified into three types: The first type is in-office procedures in which high concentrations of hydrogen peroxide are used to achieve an immediate result. The second type is tray whitening, an outgrowth of in-office procedures, in which carbamide peroxide is used as the whitening agent. The third type is over-the-counter products such as whitening strips and whitening toothpastes that contain low concentrations of hydrogen peroxide.[4,5]

Whitening is an oxidation reaction. Contemporary whitening systems are based primarily on hydrogen peroxide or one of its precursors, notably carbamide peroxide, and these are often used in combination with an activating agent such as heat or light.[6] Whether a tooth-whitening system contains carbamide peroxide or hydrogen peroxide, the mechanism of action of these systems involves hydrogen peroxide. Carbamide peroxide is broken down by salivary enzymes to release hydrogen peroxide and urea. Hydrogen peroxide converts colored materials into noncolored materials by oxidizing organic compounds within enamel and dentin.[7]

Tooth-whitening sensitivity is the most frequent adverse effect of vital tooth whitening.[8] It presents as a generalized hypersensitivity but may also occur as a spontaneous, sharp, shooting or tingling pain or zinger limited to one or a few teeth.[9] The incidence of tooth-whitening sensitivity can be as high as 60%.[10] The greatest sensitivity generally occurs with in-office tooth whitening because of the high concentrations of peroxide applied. The sensitivity can be so painful that some dentists premedicate their patients with nonsteroidal anti-inflammatory drugs to minimize it.[11] Numerous studies have examined various agents in an attempt to reduce the incidence and intensity of whitening sensitivity. A number of desensitizing agents have proven effective in reducing whitening sensitivity, but others only reduced the intensity of pain.[12,13] Although a large number of treatment modalities have been introduced for night guard–associated whitening, few studies have examined in-office whitening.[8]

Tooth mousse is a sugar-free water-based cream that contains 10% casein phosphopeptide-amorphous calcium phosphate (CPP-ACP) and has a polishing, cleaning, and dentinal tubule sealing effect.[14] It has been proposed that CPP-ACP can exert a rapid desensitizing effect through immediate protein binding followed by the deposition of calcium and phosphate compounds within exposed dentin tubules.[15] Adding CPP-ACP to home whitening agents resulted in a significant reduction of whitening sensitivity during and after treatment.[16] On the other hand, Tang and Millar[17] failed to prove a therapeutic benefit of Recaldent chewing gum (0.6% CPP-ACP) in reducing sensitivity of vital in-office

Table 1: *Inclusion and Exclusion Criteria*

Inclusion Criteria	Exclusion Criteria
Eight maxillary anterior teeth were present	Active caries and/or periodontal diseases (gingivitis and recession) or wasting diseases
The selected teeth had a mean shade of C2 or darker (Vitapan Classical, Vita-Zahnfabrik, Säckingen, Germany)	Smoker
	Previous whitening procedures
No restorations or carious lesions on the buccal surfaces of the anterior teeth to be whitened	Moderate or severe tetracycline stains or fluorosis
No history of tooth sensitivity	Pregnant or lactating
No use of a desensitizing agent or desensitizing toothpaste in the past six months	No schedule availability

tooth whitening. This outcome may be related to the low concentration of CPP-ACP and the short exposure time between teeth and CPP-ACP.

Therefore, the main aim of this study was to evaluate the efficacy of 10% CPP-ACP in reducing sensitivity associated with in-office vital tooth whitening as there is no consensus regarding its effectiveness. In addition, this study aimed to compare the effectiveness of 10% CPP-ACP with fluoride gel and a gel without desensitizing agent with regard to incidence, intensity, and duration of tooth sensitivity and, finally, to investigate how these agents affect tooth-shade stability.

METHODS AND MATERIALS

This study protocol was approved by the Institutional Review Board of Jordan University of Science and Technology (JUST). The volunteers were students, staff, and patients who worked on or attended the dental clinics at the faculty of dentistry at JUST and who expressed an interest in whitening their teeth. Fifty-one of the volunteers who met the inclusion and exclusion criteria (Table 1) signed a consent form containing all the information regarding the risks and benefits of the treatment and completed the study to the end. Before dental examination, a case sheet and a history form were completed for each participant to record all information regarding age, sex, medical and dental history, dentifrice being used, history of any habits (especially smoking), history of sensitivity or previous whitening, and presence of wasting disease. All participants received a complete oral prophylaxis by a dentist two weeks before the whitening appointment. Participants were randomly assigned to one of three study groups (Table 2).

Tooth shade evaluation was performed using the Vitapan classical shade guide (Vita Zahnfabrik, Bad Sackingen, Germany) by matching the middle third of the maxillary anterior teeth. The 16 shade tabs in the shade guide were numbered from 1 (highest/ lightest value, B1) to 16 (lowest/ darkest value, C4), and the total tab change was calculated by subtracting the tab number corresponding to the baseline shade from that the tab number of the shade at each evaluation. One evaluator recorded the shade of the participants' teeth at baseline (before whitening), directly after whitening, and at days 3, 7, and 14 after whitening. Photographs were taken using a digital camera (Camedia C-5060 5.1 MP Digital Camera w/4× Optical Zoom, Olympus, Tokyo, Japan) immediately after the whitening and at each shade evaluation to document results.

A lip and cheek retractor was placed. The gingival tissues of the teeth to be whitened were isolated using a light-cured resin dam (Top Dam, FGM, Joinville, Brazil) to prevent the whitening gel from coming into contact with the gingival tissue. Participants' lips were painted with vaseline, and protective eyewear was used. To aid in the isolation process, a plastic suction tip with high suction power was used.

A whitening agent with 35% hydrogen peroxide (Whiteness HP Maxx, FGM, Joinvile, Brazil) was applied on the labial surfaces of the upper teeth selected for whitening using an applicator. The whitening agent was washed away and refreshed every 15 minutes during the 45-minute application period, as instructed by the manufacturer. After

Table 2: *Experimental Groups*

Material	Description and Manufacturer
Gel without desensitizing agent	Experimental
Gel with 2% sodium fluoride	Frutti Flúor Gel 214826 and Frutti Fluor Gel Neutro, , Biodinamica, Ibipora, Brazil
Gel with 10% CPP-ACP	GC Tooth Mousse Recaldent (CPP-ACP), GC Corporation, Tokyo, Japan

completion, the whitening agent was removed with cotton rolls and then the gingival barrier was removed. Teeth were rinsed thoroughly, and the post-whitening shade was determined using the Vita oriented shade guide tab (Vitapan Classical, Vita-Zahnfabrik, Säckengin, Germany). Participants were scheduled for lower arch whitening after 14 days.

A small amount of the gel assigned for each participant was applied directly on the labial surface of each tooth and left undisturbed for three minutes; care was taken to cover the entire labial surface, and the gel was carried interproximally as much as possible. Participants were asked to expectorate thoroughly and avoid rinsing, eating, or drinking for half an hour after gel application. Each participant was given the assigned gel in an unmarked syringe and was instructed to use the gel in a similar manner twice daily, once in the morning and once at night, after brushing their teeth with the supplied dentifrice (Colgate toothpaste maximum cavity protection 100 mL, Colgate-Palmolive Arabia, Dammam, Saudi Arabia) for 14 days.

Post-whitening sensitivity was evaluated by relying on patient's feeling of pain. All participants were given a sensitivity sheet to record the post-whitening sensitivity of the whitened teeth on daily basis according to the visual analog scale of pain (VAS). The VAS is a horizontal line containing 10 numbers from 0 to 10; zero indicates no pain and 10 indicates the worst pain. Participants were asked to mark the number that indicated their level of pain. The participants were also asked to record the stimulus that caused sensitivity, including hot, cold, or other. The duration of pain, whether seconds, minutes, or hours, was also recorded for the 14-day follow-up period. For descriptive analysis purposes, the intensity results were categorized according to the following scale: 0 = none, 1-3 = mild, 4-6 = moderate, and 7-10 = severe.

Data analysis was carried out using the Statistical Package for Social Science (SPSS version 15, SPSS Inc. Chicago, IL). Descriptive analysis of the demographic data, the incidence, the stimulus and the duration of tooth sensitivity for each study group was calculated. Means and standard deviation for each group were also calculated with regard to the intensity levels of tooth sensitivity as well as tooth shade stability during the follow-up period.

One-way analysis of variance (ANOVA) was used for statistical analysis. A χ^2 test was used to find the effect of gender and age as well as the effect of different desensitizing agents on the intensity of sensitivity. The probability value was set at ($p \leq 0.05$). The general linear model was used to compare intensity level differences among the three studied groups and for shade stability over the follow-up period.

RESULTS

Fifteen men nd 36 women, ranging in age from 18 to 38 years old participated in this study. Mean age was 23.1 years.

Incidence of Tooth Sensitivity

Tooth sensitivity occurred in 50 participants on the first day after whitening. Sensitivity was reported by 22 participants on day 2 and 9 participants on day 3. During the 14-day follow-up period, the sensitivity among the fluoride gel group lasted up to day 10 after whitening. In the CPP-ACP gel group, sensitivity was reported by participants up to day 4 after whitening. In the gel without desensitizing agent group, some days the participants experienced sensitivity and sometimes they did not over the 14 day period.

Intensity

To statistically compare the intensity of pain among the tested groups, only the data collected from the first two days of the follow-up period were taken into consideration because of the larger number of patients who reported sensitivity in that specific period compared with the remaining follow-up days. Despite the fact that the participants in the fluoride gel group experienced lower intensity of sensitivity than those in the gel without desensitizing agent group or the CPP-ACP gel group, these differences were not statistically significant (Table 3). The predominant level of sensitivity among participants on day 1 was moderate in all tested groups, but the level of sensitivity was mild on day 2 and day 3. The sensitivity that persisted after post-whitening day 3 was mild or nonexistent (Figure 1).

Table 3: *Means and Standard Deviations and p Values of Intensity Levels on Day 1 and Day 2*

Day	Gel Without Desensitizing Agent (Mean ± SD)	Gel With 2% Sodium Fluoride (Mean ± SD)	Gel With 10% CPP-ACP (Mean ± SD)	*p*
1	5.47 ± 2.2	4.24 ± 1.8	5.76 ± 2.5	0.112
2	1.24 ± 1.6	0.71 ± 1.4	1.29 ± 2.0	0.532

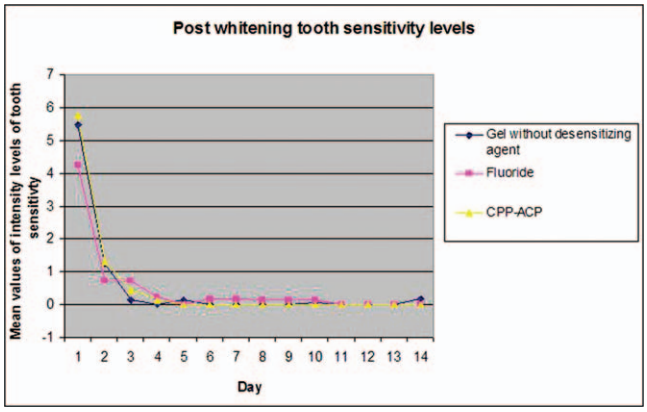

Figure 1. *Post-whitening tooth sensitivity levels in the three study groups during the study period.*

Stimulus of Sensitivity

The sensitivity reported by participants in all groups was mainly spontaneous; there was no provoking stimulus on day 1. Although most participants reported no pain on day 1, the remaining reported pain that was either spontaneous or provoked by a stimulus, as shown in Table 4.

Duration of Tooth Sensitivity

On day 1, nine participants of the gel without desensitizing agent group reported that the duration of pain was seconds, four reported minutes, and the remaining four reported hours. The experienced pain durations with the fluoride group were 10 participants having a pain episode that lasted seconds, three for minutes, and three for hours; one participant had no pain. In the CPP-ACP group, nine participants reported pain that lasted for seconds, six reported duration of minutes, and two reported pain duration of hours.

On day 2, among the gel without desensitizing agent group, four participants had pain that lasted for seconds, two for minutes, and one for hours; 10 had no pain. In the fluoride group, four participants experienced pain that lasted for seconds but one had pain lasting minutes, and 12 had no pain. Among the CPP-ACP group, six participants had pain lasting seconds, one for minutes, and one for hours; nine had no pain**.** The duration of sensitivity in all the groups throughout the 14-day period is shown in Table 5.

Tooth Shade Stability

The recorded shades using the Vitapan shade guide showed that the smallest number of shade tab changes was three tabs, and the largest was 11 tabs. None of the tested materials affected the efficacy of tooth whitening, but the shades among the fluoride group showed more color stability than that of the gel without desensitizing agent and the CPP-ACP groups. The mean and standard deviation of shade unit difference on days 1, 3, 7, and 14 are presented in Table 6. There was an improvement in the shade on day 3, with relapse on day 7 and day 14, but on day 14 the shade was the same as on day 1 (Figure 2).

DISCUSSION

Faster tooth whitening is increasingly required; hence, higher concentrations of whitening agents and in-office tooth-whitening regimens are used.[18,19] Although rapid lightening is the main advantage of in-office tooth whitening,[20] rapid relapse also occurs with most in-office products. Moreover, transient and sometimes intolerable sensitivity is the main drawback associated with in-office tooth whitening.[4,21] As the concentration of the peroxide increases, greater insult to the pulp occurs; hence, the incidence and severity of sensitivity increase.[22] To overcome this drawback of tooth whitening, several investigators have conducted studies on different

Table 4: *Stimulus of Sensitivity Reported in the Three Study Groups on Day 1 and Day 2*

Stimulus	Gel Without Desensitizing Agent n (%)		Gel With 2% Sodium Fluoride n (%)		Gel With 10% CPP-ACP n (%)	
	Day 1	Day 2	Day 1	Day 2	Day 1	Day 2
No pain	0 (0.0%)	10 (41.2%)	1 (5.9%)	13 (76.5%)	0 (0.0%)	9 (52.9%)
Spontaneous pain	14 (82.4%)	3 (17.6%)	12 (70.6%)	2 (11.8%)	9 (52.9%)	3 (17.6%)
Hot	0 (0.0%)	0 (0.0%)	1 (5.9%)	0 (0.0%)	1 (5.9%)	1 (5.9%)
Cold	3 (17.6%)	3 (17.6%)	1 (5.9%)	0 (0.0%)	4 (23.5%)	3 (17.6%)
Hot and cold	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	1 (5.9%)	0 (0.0%)
Cold and spontaneous pain	0 (0.0%)	1 (5.9%)	1 (5.9%)	1 (5.9%)	0 (0.0%)	0 (0.0%)
Hot and spontaneous pain	0 (0.0%)	0 (0.0%)	1 (5.9%)	0 (0.0%)	0 (0.0%)	0 (0.0%)
All stimuli	0 (0.0%)	0 (0.0%)	0 (0.0%)	1 (5.9%)	2 (11.8%)	1 (5.9%)

Table 5: *Duration of Sensitivity Among Participants Over the 14-day Follow-up Period*

Group	Gel Without Desensitizing Agent (n=17)				Gel With 2% Sodium Fluoride (n=17)				Gel With 10% CPP-ACP (n=17)			
	No Pain	S	Min	H	No Pain	S	Min	H	No Pain	S	Min	H
Day 1	0	9	4	4	1	10	3	3	0	9	6	2
Day 2	10	4	2	1	12	4	1	0	9	6	1	1
Day 3	15	2	0	0	14	2	1	0	14	3	0	0
Day 4	17	0	0	0	10	0	1	0	16	1	0	0
Day 5	15	2	0	0	16	1	0	0	17	0	0	0
Day 6	17	0	0	0	16	1	0	0	17	0	0	0
Day 7	17	0	0	0	16	1	0	0	17	0	0	0
Day8	17	0	0	0	16	1	0	0	17	0	0	0
Day 9	17	0	0	0	16	1	0	0	17	0	0	0
Day 10	16	1	0	0	16	1	0	0	17	0	0	0
Day 11	17	0	0	0	17	0	0	0	17	0	0	0
Day 12	17	0	0	0	17	0	0	0	17	0	0	0
Day 13	17	0	0	0	17	0	0	0	17	0	0	0
Day 14	16	1	0	0	17	0	0	0	17	0	0	0

Abbreviations: S, Seconds; Min, Minutes; H, Hours.

desensitizing agents in an attempt to prevent, or at least reduce, the intensity to an acceptable level for patients.[13,16,23]

Tooth Sensitivity Evaluation

Incidence of Tooth Sensitivity—Most of the participants (98.0%) were able to complete the treatment, although one participant had two applications instead of three because of intolerable pain. Almost all participants experienced sensitivity (98.0%) after tooth whitening. Our results are in agreement with those of other studies in that the occurrence of sensitivity is usually high after concentrated peroxide-based whitening. Tang and Millar[17] reported that 85.2% of their study participants had sensitivity after in-office whitening using 15% hydrogen peroxide. Tay and others[8] used the same whitening product used in the current study, with the same concentration, and reported that 66.8% of their participants experienced sensitivity.

The present study recorded sensitivity occurrence until day 14 after whitening. The results showed that in the three groups, most participants experienced sensitivity during the first two days. Few participants in the fluoride group experienced sensitivity that lasted up to day 10, whereas in the gel without desensitizing agent group, one or two participants experienced sensitivity on some days and sometimes did not up to day14. Several studies reported that in-office tooth-whitening sensitivity usually lasts up to one day after the procedure; for instance, Tang and Millar[17] reported that no patients had lingering pain after 24 hours. Reported sensitivity was similar in the study by Tay and others,[8] where most of the participants had sensitivity on the first day and about 15% had pain that continued until the second day.

When comparing the sensitivity among the fluoride group and the gel without desensitizing agent group, there was no statistically significant difference in the incidence of pain. The lack of difference in the incidence of pain in the current study is in agreement with Armênio and others,[13] where the use of 1.23% sodium fluoride for 4 minutes after the application of 16% carbamide peroxide did not reduce or eliminate the incidence of sensitivity compared with the placebo group. Moreover, similar findings were reported by Jorgensen and Carroll,[24]

Table 6: *Vita Classic Tab Values (Means and Standard Deviations) at Each Evaluation*

Group	Day 1 (Mean ± SD)	Day 3 (Mean ± SD)	Day 7 (Mean ± SD)	Day 14 (Mean ± SD)
Gel without desensitizing agent	6.59 ±1.7	7.06 ±1.1	7.00 ±1.2	6.65 ±1.5
Gel with 2% fluoride	7.29 ±1.4	7.94 ±1.5	7.76 ±1.4	7.53 ±1.5
Gel with 10% CPP-ACP	6.47 ±1.7	7.29 ±1.7	6.76 ±1.4	6.53 ±1.4

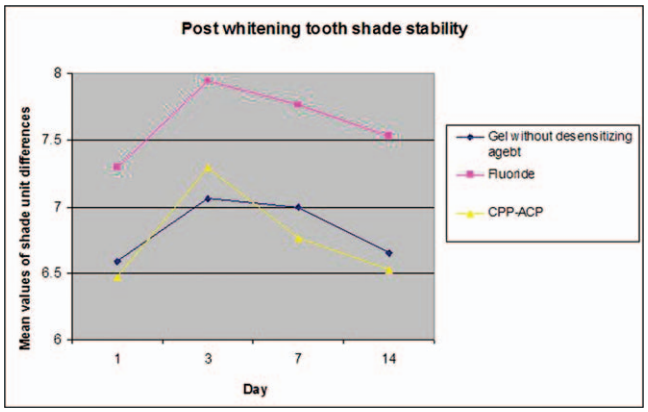

Figure 2. *Post-whitening tooth-shade unit differences in the three study groups over the study period.*

where patients using 15% carbamide peroxide with 0.11% fluoride had similar incidence levels as the placebo group.[24]

In the current study, there was no statistically significant difference in the incidence of tooth sensitivity between men and women. Most studies did not point out the relation between men and women, perhaps because of the lack of a significant difference, although it has been reported that women seem to have a higher incidence of sensitivity.[10]

Intensity—The intensity of sensitivity was lower in participants using the fluoride gel than those in the gel without desensitizing agent group and the CPP-ACP group. However, the difference was not statistically significant on day 1 or day 2 (p=0.112 and p=0.532, respectively). The results of this study are in agreement with the findings of Tang and Millar,[17] who studied the effect of using a sugar-free chewing gum containing 0.6% CPP-ACP on the intensity of sensitivity reported by participants. The results showed that chewing sugar-free gum can help reduce tooth sensitivity regardless of the presence of CPP-ACP; in contrast, not using a post-whitening agent was accompanied by a significantly higher intensity of sensitivity compared with the other groups. The results of our study are also in agreement with Jorgensen and Carroll,[24] where participants using 15% carbamide peroxide with 0.11% fluoride had no significant difference in sensitivity levels compared with participants who used a placebo gel; both groups reported mild sensitivity. Although the gel without desensitizing agent that was used in the current study had no active desensitizing agent, it may have stimulated salivary flow, which might have reduced the sensitivity given that dehydration is one of the causes of sensitivity.[25]

However, Tay and others[8] found that the intensity of sensitivity was significantly higher in the placebo gel group than in a group of participants who used fluoride/potassium nitrate gel. This might be attributed to the fact that the desensitizing gel was applied for 10 minutes before the whitening procedure; in addition, there was a possible dual effect of using both fluoride and potassium nitrate. The results of the present study contrast with the findings of Armênio and others,[13] where 1.23% sodium fluoride had a significant effect in lowering sensitivity intensity compared with the placebo group when applied in the tray for four minutes after 16% carbamide peroxide whitening. This difference may be due to the higher concentration (35% hydrogen peroxide) of the whitening agent used in our study, in addition to the time-release approach of carbamide peroxide, which is essentially associated with lower pain intensity than hydrogen peroxide.[22]

Giniger and others[16] found that the use of 0.5% CPP-ACP with 16% carbamide peroxide mixed immediately before application proved to be significantly effective in reducing the intensity of tooth sensitivity during treatment and five days after treatment. Another study, done by Borges and others,[23] where 22% carbamide peroxide with 10% CPP-ACP was used, showed reduction in sensitivity levels compared with the placebo group; however, this was based on clinical reports where only five patients were enrolled. This result could be attributed to the lower concentration and type of the whitening agent used compared with the in-office agents.

Stimulus of Sensitivity—In this study, pain was mostly spontaneous, which is comparable with reports that sensitivity actually does occur in healthy intact teeth without any provoking stimulus.[27] On the other hand, the study of Browning and others[26] reported that cold stimulus was the most painful stimulus, followed by gingival sensitivity and then sensitivity due to hot stimuli in patients using 10% carbamide peroxide with potassium nitrate and sodium fluoride as desensitizing agents.

Duration of Tooth Sensitivity—It has been reported that sensitivity associated with in-office tooth whitening usually peaks within one to six hours after whitening.[11] In the current study, it was found that in all test groups more than half of the participants had sensitivity for only seconds on day 1. Furthermore, equal numbers of subjects had pain for minutes and hours in the gel without desensitizing agent group and the fluoride groups, whereas in

the CPP-ACP group, the number of participants who had pain for minutes was higher than those who experienced pain for hours.

Browning and others[26] found that sensitivity on average was short lived when using 10% carbamide peroxide with potassium nitrate and sodium fluoride. Tang and Millar[17] found that the duration of sensitivity varies greatly from participant to participant; however, overall it was short-lived, with a mean value of 4.9 hours and a range of 0 to 12 hours. In our study, sensitivity was mainly reported in the first 48 hours, and the tingling sensation lasted mainly for seconds.

Tooth Shade Stability

Participants in this study had a significant shade improvement: an average of 6.8 units of shade change. It was found that participants in the fluoride gel group had a better shade stability compared with the CPP-ACP group and the gel without desensitizing agent groups. It was observed that the shade improved on day 3 among all groups, probably because of the temporary dehydration effect, which lasts up to 72 hours.[28] This was followed by minor relapse on day 14 in the gel without desensitizing agent group and the CPP-ACP group; the shade relapse was less noticeable in the fluoride group. This could be due to the teeth taking water back from saliva, and thus the shade becomes darker again,[28] which in this study occurred on day 7. It has been shown that fluoride increases enamel hardness and renders it less permeable,[29] which can explain why the participants in the fluoride group had a better shade stability.

The results of the current study indicate that application of a 2% sodium fluoride or 10% CPP-ACP or a gel without desensitizing agent does not hinder the whitening efficacy of 35% hydrogen peroxide, as the shade recorded on day 1 was approximately the same as the one recorded on day 14. This is in agreement with previous studies that used fluoride,[8] CPP-ACP materials,[16] or potassium nitrate and sodium fluoride.[25]

CONCLUSION

Under the imitations of this study, the following conclusions can be drawn:

1. There was no statistically significant difference among the gel without desensitizing agent, 2% sodium fluoride, and 10% CPP-ACP on reducing the intensity of tooth sensitivity associated with in-office tooth whitening. All three gels reduced sensitivity to a moderate level. This indicates that the use of 10% CPP-ACP may reduce sensitivity associated with in-office tooth whitening to a level comparable with that of fluoride, which is a well-established therapeutic agent used to minimize tooth sensitivity.
2. Using sodium fluoride was accompanied by better shade stability compared with the gel without desensitizing agent and the 10% CPP-ACP gel.

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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Three-year Clinical Evaluation of Different Restorative Resins in Class I Restorations

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Clinical Relevance

Except for the better marginal adaptation that packable resin composite showed, silorane-based restorative, nanofilled resin, and packable resin composite resulted in similar clinical performance in restoring Class I cavities after 3 years.

SUMMARY

The aim of the present study was to evaluate the three-year clinical performance of a nanofilled resin composite, a packable resin composite, and silorane-based resin restorations in Class I occlusal cavities. Twenty-eight patients with at least three similar-sized occlusal lesions in molar teeth participated in the study. A total of 84 Class I occlusal restorations were placed: 28 with nanofilled resin composite (Filtek Supreme), 28 with packable resin composite (P60), and 28 with silorane-based resin (Filtek Silorane). Filtek Supreme and P60 were used with their respective etch-and-rinse adhesive system, Adper Single Bond 2, and Filtek Silorane was used with its respective self-etch adhesive, Filtek Silorane Adhesive. All restorations were placed by the same operator. The restorations were evaluated at baseline, at six months, and annually for three years according to modified US Public Health Service criteria by two calibrated examiners who did not know which restorative resin had been used. The three restorative materials for each category were compared using the χ^2 test at a significance level of 0.05. Cochran's Q test was used to compare the changes across the five time points for each restorative material. McNemar's test followed by Bonferroni adjustment was used when significance differences were found. At the end of the three years, 60 restorations were evaluated in 20 patients, with a recall rate of 71.4%. The retention rate was 100% for all restorative resins. Eight restorations from the P60 group, ten from the Filtek Supreme group, and nine from the Filtek Silorane group were rated Bravo for marginal discoloration. For marginal adapta-

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tion, three P60, five Filtek Supreme, and 11 Filtek Silorane restorations were rated Bravo. No statistically significant differences in overall clinical performance were found between the restorative materials except for marginal adaptation. P60 showed the best marginal adaptation at the end of the three years. No differences were observed between the restorative resins for any of the evaluation criteria tested ($p>0.05$). None of the restorations showed postoperative sensitivity, secondary caries, or loss of anatomic form. All restorative resins performed equally well in clinical conditions during the three-year evaluation, and no significant differences were found among them, except for marginal adaptation, in which P60 showed superior results.

INTRODUCTION

Patients' increasing demand for esthetic restorations, even in posterior regions, has led to improvements in the mechanical and physical properties of composite restorative materials. Currently, composite resins have been diversified according to chemical composition and particle filler size, with the objective of finding better ones.

Difficulty in restoration placement and establishment of appropriate proximal contacts as well as the sticky form of conventional composite resins prompted producers to introduce packable or condensable composite resins to overcome these handicaps.[1] Packable resin composites are claimed to be suitable for stress-bearing posterior restorations. Although packable resins were developed for better clinical performance, some studies showed that the properties of packable composites are not superior to those of conventional resin composites. [2-4]

A new type of resin composite, so-called nanocomposite, has been developed recently to improve the performance of resins. The use of nanotechnology in resin composite systems offers high translucency and high polish, similar to that of microfilled composites, while maintaining physical properties and wear equivalent to several hybrid composites.[5] However, shrinkage during polymerization is still a major challenge. Different techniques and restoratives have been proposed to overcome this phenomenon. Low-shrinkage silorane-based resins such as Filtek Silorane have been suggested as a means to reduce shrinkage and cuspal deflection.[6] During silorane-based resin composite polymerization, ring-opening monomers connect by opening, flattening, and extending toward each other. This mechanism results in less volumetric shrinkage compared with methacrylate-based composites. As methacrylate-based composites cure, the molecules of these "linear monomers" connect by actually shifting closer together in a linear response that causes a loss of volume.[7]

Many laboratory studies have addressed bond strength of silorane-based restorative on tooth structure; however, it is of paramount relevance to study the clinical behavior of silorane restorative resin, as information about the clinical performance of this restorative is very limited.[8-12] Moreover, the results of these *in vitro* studies cannot always be directly extrapolated to the performance of restorative materials.

The aim of this clinical study was to investigate whether a low-shrinkage silorane-based restorative resin improves the clinical performance of restorations in Class I cavities. We tested the hypothesis that a low-shrinkage silorane-based restorative resin would show better clinical results than the packable and nanofilled resin composites because of its lower polymerization shrinkage.

METHODS AND MATERIALS

Selection Criteria

Approval for this clinical study was obtained from the Human Ethics Committee of Hacettepe University. The subjects were recruited from patients seeking routine dental care at the Department of Restorative Dentistry, Hacettepe University.

For inclusion in the study, the teeth to be restored had to be vital and without pulpal or periodontal disease, pain, and preoperative sensitivity. Teeth with an existing occlusal contact and at least one neighboring tooth were required. Patients with poor oral hygiene, serious health problems, heavy bruxism, or a known allergy to the substances used in the study were not included. Before participating in the study, all patients signed a written consent form after receiving a full explanation of the treatment procedure.

Of the 50 patients evaluated, only the 28 (17 women and 11 men) who had three primary carious lesions that required Class I restorations in their molars (first or second molars) were enrolled in the study. The 28 subjects had a mean age of 29.3 (range = 18-52 years).

Restorative Procedures

Bitewing radiographs of the teeth to be restored were taken preoperatively. The lesion depth was the

middle or beyond the middle-third of the dentin. The teeth to be restored were first cleaned with a nonfluoridated prophylaxis paste on a slowly rotating rubber cup and then washed and dried. Isolation was accomplished using cotton rolls. The cavity was prepared using a diamond bur (Diatech, Heerbrugg, Switzerland) with a high-speed handpiece. Carious tissue was removed using a slow-speed steel round bur and excavator. Removal of carious tissue was checked with visual and tactile feedback from an explorer. The teeth to be restored were randomly assigned according to a table of random numbers for restoration with a packable resin composite, P60 (3M, ESPE, St Paul, MN, USA); a nanofilled resin composite, Filtek Supreme (3M); or a low-shrinkage silorane-based restorative resin, Filtek Silorane (3M). Filtek Supreme and P60 were used with their respective etch-and-rinse adhesive system, Adper Single Bond 2 (3M), and Filtek Silorane was used with its respective adhesive, Filtek Silorane Adhesive (3M). Enamel and dentin were etched for 30 and 15 seconds, respectively, with 37% phosphoric acid gel (3M) for the P60 and Filtek Supreme groups. The adhesive systems were applied strictly according to the manufacturers' instructions (Table 1). The restorative resins were placed in increments not exceeding 2 mm in thickness, and each increment was cured for 40 seconds with a quartz-tungsten-halogen curing unit (Benlioglu, Ankara, Turkey) at 600 mW/cm^2. Finishing was accomplished using finishing diamond burs (Diatech) at high speed, and polishing was carried out with silicon points and flexible discs (SwissFlex, Diatech). They were all used under constant water cooling. Occlusion was checked with articulating paper and adjusted. Clinical photographs at 1:1 magnification were also taken before and after surgery, at baseline, and at each recall with a digital camera (Canon, PowerShot 300HS, Melville, NY, USA).

Evaluation Criteria and Procedures

Patients were recalled at baseline (one week after placement), at six months after placement, and at one, two, and three years after placement. Two calibrated examiners, other than the operator, evaluated each restoration. Modified US Public Health Service criteria described by Cvar and Ryge were used to evaluate the following characteristics: retention, marginal discoloration, marginal adaptation, color match, surface texture, anatomic form, and secondary caries (Table 2).[13] Postoperative sensitivity was assessed by air and/or tactile contact and was recorded as absent, mild, or severe. Sensitivity to air was assessed by blowing a stream of compressed air for five seconds while shielding the neighboring teeth with the fingers. Sensitivity to tactile contact was assessed by moving a probe over the restored tooth surface. Subjects were also questioned regarding sensitivity to cold/hot or other stimuli.

The examiners were calibrated to a predetermined level of inter- and intraexaminer agreement of at least 95% per criterion. The calibrated examiners were blinded to the restorative material used. Any discrepancy in evaluation between the two evaluators was immediately resolved at chair-side. The restorations were scored as follows: Alpha represented the ideal clinical situation, Bravo was clinically acceptable, and Charlie represented a clinically unacceptable situation.

Table 1. *Restorative Resins and Adhesive Systems Used in the Study*

Product	Composition
Adper Single Bond 2, 3M, ESPE, St Paul, MN, USA Batch # 51202	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid, copolymer, initiators, water, ethanol
Filtek Silorane Adhesive Primer, 3M, ESPE, St Paul, MN, USA Batch # N132529	Phosphorylated methacrylates, Bis-GMA, HEMA, water, ethanol, silane-treated silica filler
Filtek Silorane Adhesive Bond, 3M, ESPE, St Paul, MN, USA Batch # N132530	Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers
P60, 3M, ESPE, St Paul, MN, USA, Batch # N319331	Zirconia/silica, Bis-GMA, UDMA, Bis-EMA
Filtek Silorane, 3M, ESPE, St Paul, MN, USA Batch # N 128819	Silorane resin, camphorquinone, iodonium salt, electron donor, quartz filler, yttrium fluoride, stabilizers, pigments
Filtek Supreme, 3M, ESPE, St Paul, MN, USA Batch # 44-0023-3903-2AC	Bis-GMA, UDMA, TEGDMA, Bis-EMA

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

Table 2. *Modified US Public Health Service Evaluation Criteria*[13]

Characteristic	Evaluation Criteria
Retention	Alpha: The restoration is present.
	Charlie: The restoration is absent.
Marginal discoloration	Alpha: There is no visual evidence of marginal discoloration different from the color of the restorative material and from the color of the adjacent tooth structure.
	Bravo: There is visual evidence of marginal discoloration at the junction of the tooth structure and the restoration that has not penetrated along the restoration in a pulpal direction.
	Charlie: There is visual evidence of marginal discoloration at the junction of the tooth structure and the restoration but the discoloration has penetrated along the restoration in a pulpal direction.
Marginal adaptation	Alpha: Restoration is closely adapted to the tooth. The explorer does not catch when drawn across the surface of the restoration toward the tooth structure, or if the explorer does catch there is no visible crevice along the periphery of the restoration.
	Bravo: The explorer catches and there is visible evidence of a crevice, which the explorer penetrates, indicating that the edge of the restoration does not adapt closely to the tooth structure. The dentin and/or the base are not exposed and the restoration is not mobile.
	Charlie: The explorer penetrates a crevice defect that extends to the dentino-enamel junction.
Color match	Alpha: Restoration matches the shade and translucency of adjacent tooth structure.
	Bravo: Restoration does not match the shade and translucency of adjacent tooth structure but the mismatch is within the normal range of tooth shades.
	Charlie: Restoration does not match the shade and translucency of adjacent tooth structure and the mismatch is outside the normal range of tooth shades and translucency.
Surface texture	Alpha: Surface texture is similar to polished enamel as determined by means of a sharp explorer.
	Bravo: Surface texture is gritty or similar to a surface subject to a white stone or rougher than the adjacent tooth structure.
	Charlie: Surface pitting is sufficiently coarse to inhibit the continuous movement of an explorer across the surface.
Anatomic form	Alpha: Restoration is continuous with existing anatomic form.
	Bravo: Restoration is discontinuous with existing anatomic form but missing material is not sufficient to expose dentin or base.
	Charlie: Sufficient material is lost to expose dentin or base.
Secondary caries	Alpha: No caries is present.
	Charlie: Caries is present.

Statistical Analysis

The three restorative materials for each category were compared using a χ^2 test at a significance level of 0.05. Cochran's Q test was used to compare the changes across the five time points for each restorative material. McNemar's test followed by Bonferroni adjustment was used when significant differences were found.

RESULTS

Three restorations were placed in each subject, resulting in a total of 84 restorations in 28 patients. The recall rate was 100% at one year. At two years, one patient had moved away and three restorations could not be evaluated (recall rate = 96.4%). At the end of three years, 20 patients (60 of 84 restorations) were evaluated (recall rate = 71.4%).

Table 3 presents the data for retention, marginal discoloration, marginal adaptation, color match, and surface texture. As no loss of restorations occurred, a 100% retention rate was recorded for all three restoratives at the three-year recall. At the six-month recall, no statistically significant differences were found between the restorative materials for any of the evaluation criteria tested ($p>0.05$).

There were no significant differences among the restorative groups in terms of marginal discoloration. After three years, 10 Filtek Silorane restorations, 9 Filtek Supreme restorations, and 8 P60 restorations received Bravo scores. These discolorations occurred at the enamel margin.

For marginal adaptation, there was a significant difference only between P60 and Filtek Silorane at the end of one year. At the two-year recall, Filtek Silorane showed worse marginal adaptation than P60 and Filtek Supreme. Eleven restorations from Filtek Silorane, five from Filtek Supreme, and only three from P60 received Bravo scores for marginal adaptation at the three-year recall; the difference between Filtek Silorane and P60 was significant ($p<0.05$).

With regard to color match, two teeth, one restored with P60 and the other with Filtek Silorane, received Bravo scores at the end of two years and three years. All restorations from the Filtek Supreme group showed Alpha scores.

In terms of surface texture, only one restoration from the P60 group and one from the Filtek Supreme group received Bravo scores, whereas all restorations from the Filtek Silorane group received Alpha scores at the end of three years. No significant differences were observed in color match or surface texture between the restorative materials. All the restorations were scored as Alpha for anatomic form. None of the teeth showed secondary caries or pulp inflammation. None of the subjects reported postoperative sensitivity.

The results for intragroup comparisons between baseline and each evaluation period were as follows. Regarding marginal discoloration, there was only a significant difference between baseline and three-year results for P60. For Filtek Silorane, statistically significant differences were observed in marginal discoloration between baseline vs two years, baseline vs three years, and six months vs three years. For Filtek Supreme, significant differences were only observed between baseline vs three years and six months vs three years.

In marginal adaptation, there were no statistical differences for any evaluation-period comparisons for P60 and Filtek Supreme. Marginal adaptation significantly worsened over time compared to baseline for Filtek Silorane (baseline vs one year, two years, and three years; and six months vs two years and three years) ($p<0.05$).

DISCUSSION

A recent innovation in posterior resin restoratives is the introduction of silorane restorative resin. Many *in vitro* studies have been published regarding silorane's performance. In a recent *in vitro* study, the bonding effectiveness and marginal integrity of a silorane restorative system to dentin were compared with those of a methacrylate-based one, P60/Adper Easy Bond. No difference was noted between the silorane-based system and the methacrylate-based system.[14] The durability of the bond to dentin of the new silorane-based bonding agent, Filtek Silorane System Adhesive, was found to be as good as that of the methacrylate-based composite resin-bonding agent, Clearfil SE Bond.[15] Similar results were obtained in a study that compared the microtensile bond strength of methacrylate resin systems to a silorane-based restorative system on dentin. The silorane-based system performed similarly to that of methacrylate-based materials on dentin.[16] On the other hand, in another *in vitro* study evaluating the marginal adaptation of a methacrylate-based composite and a silorane-based composite, the silorane-based composite exhibited significantly lower shrinkage forces and better marginal adaptation than did the methacrylate-based composite, Filtek Supreme XT.[17] Although the results of those *in vitro* studies can help to predict clinical effectiveness of restorative resins, the best way is to conduct clinical trials. In the present study, the three-year clinical performances of nanofilled, packable, and silorane restorative resins were compared.

In terms of marginal adaptation, the results of this clinical study do not appear to confirm the hypothesis that a low-shrinkage restorative resin could show better performance than packable and nanofilled resin composite. Eleven restorations from the silorane group received Bravo scores for marginal adaption at the end of three years. However, significant differences were only seen between P60 and silorane at the end of three years. The reason is probably related to the adhesive system used. Although Filtek Supreme and P60 restorations were performed with the use of an etch-and-rinse adhesive, Adper Single Bond 2, silorane was used with its respective self-etch adhesive system. With a pH of about 2.7, Silorane System Adhesive Self-Etch Primer provides rather ultra-mild etching and demineralization of the tooth structure and, therefore, might produce less pronounced etching patterns than those achieved with phosphoric acid etching.[18] Baracco and others[10] evaluated the one-year clinical performance in Class I and II cavities of three different restoratives: Filtek Silorane with its respective adhesive and Filtek Z250 used either with an etch-and-rinse adhesive or a two-step self-etch adhesive. Although Filtek Silorane showed acceptable clinical performance, these restorations had no advantage over those with methacrylate-based composite combined with etch-and-rinse adhesive. Moreover, Silorane restorations tended to degrade in terms of marginal adaptation compared with baseline values. In their two-year follow-up, the three restorative systems showed statistically similar clinical performances.[12] The same results were obtained in the present study. Although there were no differences between baseline and three-year recall results in terms of marginal adaptation in P60 and Filtek Silorane restorations, silorane restorations worsened over three years.

Table 3: *Results for Different Parameters Evaluated in the Study*[a]

Parameter Rating	Baseline			6 Mo			1 Y		
	Filtek P60	Filtek Silorane	Filtek Supreme	Filtek P60	Filtek Silorane	Filtek Supreme	Filtek P60	Filtek Silorane	Filtek Supreme
Retention									
Alpha	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)
Marginal discoloration									
Alpha	28/28 (100)	28/28 (100)	28/28 (100)	28/27 (96)	28/26 (93)	28/27 (96)	28/25 (89)	28/23 (82)	28/24 (86)
Bravo	28/0 (0)	28/0 (0)	28/0 (0)	28/1 (4)	28/2 (7)	28/1 (4)	28/3 (11)	28/5 (18)	28/4 (14)
Marginal adaptation									
Alpha	28/28 (100)	28/28 (100)	28/28 (100)	28/27 (96)	28/24 (86)	28/26 (93)	28/27 (96)	28/19 (68)	28/25 (89)
Bravo	28/0 (0)	28/0 (0)	28/0 (0)	28/1 (4)	28/4 (14)	28/2 (7)	28/1 (4)	28/9 (32)	28/3 (11)
Color match									
Alpha	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)
Bravo	28/0 (0)	28/0 (0)	28/0 (0)	28/0 (0)	28/0 (0)	28/0 (0)	28/0 (0)	28/0 (0)	28/0 (0)
Surface texture									
Alpha	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/28 (100)	28/27 (96)	28/28 (100)	28/28 (100)
Bravo	28/0 (0)	28/0 (0)	28/0 (0)	28/0 (0)	28/0 (0)	28/0 (0)	28/1 (4)	28/0 (0)	28/0 (0)

[a] *Data shown are number of examined restorations/number of ratings (% of ratings). There were no Charlie ratings.*

On the other hand, Burke and others[8] evaluated the clinical performance of 100 restorations (30 Class I and 70 Class II) placed with Filtek Silorane. Satisfactory clinical performance was reported with a rate of 84% optimal for marginal integrity and 77% for marginal discoloration at the end of two years.[8] At the two-year and three-year recalls, respectively, Filtek Silorane received 73% and 50% Alpha scores in marginal discoloration and 56% and 45% Alpha scores in terms of marginal adaptation.

In a short-term clinical study, Goncalves and others[11] compared the proximal contact of a silorane-based resin composite with a conventional methacrylate-based resin composite, P60, in Class II restorations. Both restorative resins gave satisfactory results. However, these results were obtained after six months of clinical service. The follow-up period seems very short and differences could develop over longer periods of use; therefore, these results cannot be directly compared with our study's results.

Only three P60 restorations showed Bravo scores in terms of marginal adaptation at the end of three years. The superior behavior of P60 has been demonstrated in a study conducted by Kiremitci and others[19] In their clinical study, the clinical performance of P60 was acceptable after six years of service. Regarding marginal adaptation and discoloration, 95% and 91% of P60 restorations, respectively, received an Alpha rating. In another study, the clinical performance of two adhesive restorative systems (Single Bond/Filtek P-60 and Single Bond/Filtek Z-250) was assessed in posterior teeth.[20] Marginal integrity for P-60 was scored as 94.3% and 91.4% Alpha at six and 12 months, respectively. All restorations were found to be clinically satisfactory.

In a recent study, the clinical performance of Filtek Supreme used with either etch-and-rinse or self-etch adhesive in Class I cavities was evaluated.[21] At the end of one year, composite resin restorations using either adhesive system showed equally satisfactory results. In another study, the performance of Filtek Supreme was found to be satisfactory even when used with a self-etch adhesive, Adper Prompt-L-Pop, over 15 months of clinical service.[22]

In a one-year clinical study, the performance of a nanofilled resin composite for posterior restorations was compared with that of two microhybrid composites and one packable composite.[23] Similar to our findings, the nanofilled resin composite showed a performance similar to that of the other packable and microhybrid resin composites. Sadeghi and others[24] evaluated the clinical performance of microhybrid, packable, and nanofilled resin composite restorations placed in Class I cavities in molar teeth and found no difference between the restorations over 18 months.

In the present study, no significant differences were found between the restorative materials in terms of marginal discoloration. It is known that if the bond between the resin and tooth structure is

Table 3: *Results for Different Parameters Evaluated in the Study (ext.)*

Parameter Rating	2 Y			3 Y		
	Filtek P60	Filtek Silorane	Filtek Supreme	Filtek P60	Filtek Silorane	Filtek Supreme
Retention						
Alpha	27/27 (100)	27/27 (100)	27/27 (100)	20/20 (100)	20/20 (100)	20/20 (100)
Marginal discoloration						
Alpha	27/22 (81)	27/19 (73)	27/22 (81)	20/12 (60)	20/10 (50)	20/11 (55)
Bravo	27/5 (19)	27/8 (27)	27/5 (19)	20/8 (40)	20/10 (50)	20/9 (45)
Marginal adaptation						
Alpha	27/22 (81)	27/15 (56)	27/22 (81)	20/17 (85)	20/9 (45)	20/15 (75)
Bravo	27/5 (19)	27/12 (44)	27/5 (19)	20/3 (15)	20/11 (55)	20/5 (25)
Color match						
Alpha	27/26 (96)	27/26 (96)	27/27 (100)	20/19 (95)	20/19 (95)	20/20 (100)
Bravo	27/1 (4)	27/1 (4)	27/0 (0)	20/1 (5)	20/1 (5)	20/0 (0)
Surface texture						
Alpha	27/26 (96)	27/27 (100)	27/26 (96)	20/19 (95)	20/20 (100)	20/19 (95)
Bravo	27/1 (4)	27/0 (0)	27/1 (4)	20/1 (5)	20/0 (0)	20/1 (5)

less than the polymerization shrinkage force, microleakage and marginal failure will occur.[1] Marginal discoloration is generally related to microleakage. All the restorations showed quite similar results in terms of marginal discoloration despite having different thermal expansion coefficients and polymerization shrinkage rates. These discolorations were superficial and localized. Although silorane system adhesive is self-etching, chemical bonding to the hydroxyapatite crystals occurs with the silorane's primer.[25] Moreover, the primer and adhesive resin of this system are separately cured, which means a twofold bonding layer[25] that might also act as an elastic buffer.[26] This might be a reason for the similar results obtained with nanofilled and packable resin composite restorations.

Postoperative sensitivity is one of the most common complaints of patients with posterior resin restoratives. Class I resin composite restorations are more prone to postoperative sensitivity and marginal failure because of the higher cavity configuration factor (C=5). However, in the present study, none of the subjects reported postoperative sensitivity. This might have been related to the insertion of resin. As incremental placement was used, polymerization shrinkage stress might have been decreased. Other evaluation criteria, such as anatomic form, color match, and surface texture remained optimal over the three-year period.

In the present study, no restoration loss was observed during the three years of clinical functioning. All the restorative resins presented an almost excellent clinical performance with a 100% retention rate and no statistically significant difference between them except in marginal adaptation. However, this clinical trial was conducted in Class I cavities with a retentive cavity shape and margins lying on enamel. Different cavity types, such as Class II extending to the dentin, might have yielded significant differences. Therefore, further clinical studies addressing different cavity types should be conducted.

Conclusion

Within the limitations of this clinical study, it is concluded that low shrinkage silorane-based restorative, packable, and nanofilled composite resin showed similar clinical results with the exception of packable resin composite, which showed a better performance in terms of marginal adaptation.

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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

Efficacy of Four Lining Materials in Sandwich Technique to Reduce Microleakage in Class II Composite Resin Restorations

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Clinical Relevance

The sandwich technique results in more microleakage than classically bonded composite resin restorations.

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SUMMARY

Objectives: **The aim of the present study was to evaluate the effect of four different sandwich techniques on gingival microleakage of Class II direct composite resin restorations.**

Materials and Methods: **Fifty sound human premolars were selected and randomly divided into five groups (n=10). Class II box only cavities were prepared in one of the proximal surfaces of each tooth with a gingival margin located approximately 0.5 mm below the cemento-enamel junction. Group A (control) was restored incrementally with composite resin (Tetric Ceram). Groups B, C, D, and E were restored with the sandwich technique using a compomer (Compoglass F), flowable composite resin (Tetric Flow), self-cure composite resin (Degufill SC), or resin modified glass ionomer (Fuji II LC), respectively. After thermal-load cycling, the specimens were immersed in 0.5% basic fuschin for 24 hours. Dye penetration (10^{-1} mm) was detected using a sectioning technique. Data were analyzed with repeated measurements and Duncan test at α=0.05.**

Results: **The least amount of microleakage was detected in the incremental group (1.28 ±**

0.98). The sandwich technique using resin modified glass ionomer (7.99 ± 9.57) or compomer (4.36 ± 1.78) resulted in significantly more leakage than did the sandwich technique using flowable (1.50 ± 1.97) or self-cure composite (2.26 ± 1.52).

Conclusion: **According to the results of this study, none of the four sandwich technique composite resin restorations used in this study could reduce gingival microleakage to a greater degree than the incremental technique.**

INTRODUCTION

Although composite resins are the most commonly used tooth-colored restorative materials, they still present several difficulties when used as posterior restorations. These materials undergo contraction during curing, and the resulting shrinkage causes debonding of the restoration from the tooth structure.[1,2] Therefore, marginal adaptation remains a problem for composite, especially at the dentinal gingival floor of Class II restorations.[3] Poor marginal sealing is associated with bacterial, liquid, and molecular penetration through the cavity-material interface, resulting in marginal staining, postoperative sensitivity, secondary caries, pulp pathosis, and, finally, restoration failure.[4]

These related problems have led to the development of open sandwich techniques. Crim and Chapman[6] and Aboushala and others[5] demonstrated that glass ionomer liners reduced marginal microleakage. The benefits of glass ionomers include similar thermal expansion to those of dental structures, bacteriostatic function, molecular bonding to dentin and enamel, and low setting shrinkage.[7]

Sandwich restorations with glass ionomer are less technique sensitive than is composite restoration.[8] However, despite good short-term clinical results, the problems associated with these materials over time comprise a noticeable concern.[9]

The authors of some studies[10,11] have suggested the use of a flowable composite between the floor of the box and the restorative material. These materials may exhibit a stress-reduction property.[11,12] Their lower modulus of elasticity can reduce marginal microleakage, which is thought to compensate for the polymerization contraction stresses of the final restoration.[13-15]

On the other hand, Fusayama[16] and Truffier-Boutry and others[17] have suggested the use of self-cured composites for deeper cavities or dentinal gingival margins because of their slower rate of polymerization, a lower polymerization contraction stress, and consequent lower microleakage.

The use of compomer material that has more mechanical strength and simplified usage than do resin modified glass ionomers (RMGIs) is considered in the sandwich technique.[18] Studies[19-23] pertaining to the microleakage associated with these techniques have given controversial results. The purposes of the present *in vitro* study were as follows:

1. To evaluate gingival microleakage below the cemento-enamel junction (CEJ) in Class II composite resin restorations with four different sandwich techniques using compomer, flowable composite, self-cure composite, or RMGI and to compare them with the microleakage associated with the incremental technique.
2. To determine if there was any difference in microleakage between the lateral and medial areas of the gingival floor.

MATERIALS AND METHODS

Fifty freshly extracted sound human premolars with similar size, stored in 0.2% sodium azide solution, were randomly divided into five groups of 10 samples each. The teeth were vertically embedded 2 mm below the CEJ in a cylindrical auto-polymerizing acrylic resin (Neocryl™, Bosworth Co., Skokie, Illinois, USA). A box cavity was prepared in one of the proximal areas of each tooth. The dimensions of the cavities measured 4 mm buccolingual in width and 2 mm in axial depth at the axiopulpal line angle. The gingival margin was located 0.5 mm below the CEJ with nearly 1.0 mm in depth. All preparations were accomplished with a new high-speed handpiece and #57 fissure burs (Brasseler USA Dental, Savannah, Georgia, USA) that were changed for each 10 cavity preparations. Tofflemire matrix and matrix holder were applied, and teeth in each group were restored as described in Table 1.

Group A (Control)

In this group, after washing and drying the cavity, dentin bonding agent (Syntac Single Component, Ivoclar/Vivadent, Schaan, Liechtenstein) was applied according to manufacturer's instructions (Table 1). The cavity was restored with a light-cured composite resin (Tetric Ceram, Ivoclar/Vivadent) by the gingivocclusal incremental technique. While the first increment was 1 mm thick, the others were placed parallel to the gingival floor up to 2 mm in thickness. Each increment was light-cured from the

Table 1: *Experimental Groups, Material, and the Restorative Procedures*

Group	Bonding System	Lining Material	Restoration Material	Restorative Procedures[a]
A	Syntac Single Component	No lining material (control)	Tetric Ceram	a, b, d
B	Syntac Single Component	Compoglass F	Tetric Ceram	a, b, c, d
C	Syntac Single Component	Tetric Flow	Tetric Ceram	a, b, c, d
D	Syntac Single Component	Degufill SC	Tetric Ceram	a, b, c, d
E	Syntac Single Component	Fuji II LC	Tetric Ceram	c, a, b, d

[a] *Procedure codes: a, etching with 35% phosphoric acid was done 30 seconds for enamel and 15 seconds for dentin, rinsed with water for 20 seconds, then air-dried gently to keep dentin slightly moist; b, bonding system was applied according to manufacturers' recommendations; c, liner material was placed onto the cavity floor and was light-cured per manufacturers' recommendations. Light-curing was not done for group D; d, composite resin was placed up to 2 mm and cured for 40 seconds at 400 mW/cm^2 incrementally.*

occlusal aspect for 40 seconds with a new QTH light-curing unit with 400 mW/cm^2 (Coltlux 50, Coltene/Whaledent AG, Altstatten, Switzerland). Soon after the removal of the metal matrix, the restorations received further light-curing from the buccal and lingual sides, each for 40 seconds. The accuracy of the light-cure unit was monitored with a radiometer (Radiometer, Coltene/Whaledent AG) after each five restorations.

The bonding step and the process for restoring the whole remaining mass of the cavities in groups B, C, D, and E are the same as described for group A, with the following sandwich material exceptions (Table 1).

Group B

A compomer (Compoglass F, Ivoclar/Vivadent), measuring 1 mm in thickness, was placed and light-cured from the occlusal for 40 seconds.

Group C

A flowable resin composite (Tetric Flow, Ivoclar/Vivadent), also 1 mm in thickness, was placed and light-cured from the occlusal for 40 seconds.

Group D

A self-cure composite resin (Degufill SC, Degussa AG, Hanau, Germany), measuring 1 mm in thickness, was placed and was given four minutes for initial setting.

Group E

RMGI (Fuji II LC Improved, GC Corporation, Tokyo, Japan), measuring 1 mm in thickness, was placed and light-cured for 20 seconds. Bonding system was applied and the rest of the cavity was restored as described for group A (Table 1).

All restorations were finished with diamond finishing burs (Drendel & Zweiling, Diamant GmbH, Kalletal, Germany) and polished with polishing disks (Soft-lex, 3M ESPE, St.Paul, Minnesota, USA). Then thermocycling was conducted between 5°C and 55°C (±2°C) for 1000 cycles. The immersion time for each bath and the interval time were 30 seconds. Then load cycling was done with a load of 14 N, which lasted 0.2 seconds at a frequency of 3 Hz for 250,000 cycles. The teeth were then coated with two coats of nail varnish 1 mm beyond the margins of the restorations and soaked in a solution of 0.5% basic fuchsin dye at 37°C for 24 hours. Roots were cut and the crowns were embedded in slow-cure epoxy resin (Araldit® , Ciba-Geigy AG, Aarberg, Switzerland). Two cuts were made in a mesio-distal direction along the long axes of the teeth with a 1.0-mm–thickness diamond disc cooled with water (Leitz 1600, Leitz Wetzlar GmbH, Wetzlar, Germany). The cuts were 0.5 mm away from the buccal and lingual sides of the gingival floor. Therefore, two cuts per tooth provided three sections with four surfaces for gingival microleakage evaluation (Figure 1).

Dye penetration was evaluated using a stereomicroscope (Zoom, Blue Light Industry USA Inc., La Habra, USA) at 40× magnification. For each surface, the dye penetration, measured in 10^{-1} mm, was recorded at the gingival floor by Asus Digital VCR software (Figure 2). Finally, the data were analyzed by repeated measures and Duncan (α=0.05) tests.

RESULTS

The results for dye penetration are presented in Table 2. All testing groups showed some degree of microleakage (10^{-1} mm) (Figures 3 and 4). Group E (Fuji II LC/Tetric Ceram) showed the highest amount of microleakage (7.99 ± 9.57), and group A (Tetric Ceram) revealed the lowest amount of microleakage (1.28 ± 0.98).

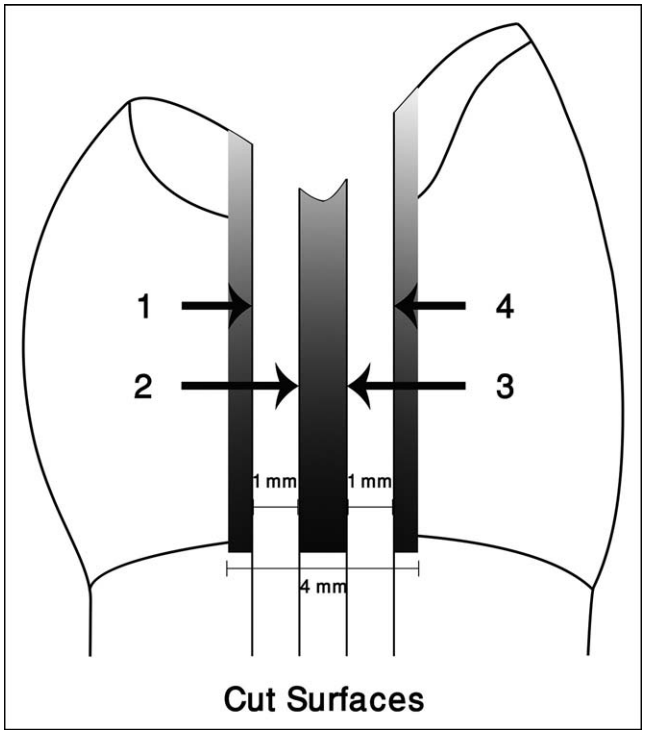

Figure 1. *Schematic illustration of two sectioning cuts made on four cut surfaces (1, 2, 3, and 4).*

There was a statistically significant difference in terms of total microleakage between the groups ($p<0.05$). However, group E (Fuji II LC/Tetric Ceram) showed significantly higher amounts of dye penetration compared to groups A (Tetric Ceram), C (Tetric Flow/Tetric Ceram), and D (Degufill SC/ Tetric Ceram).

Repeated measurements were performed to detect the probable pattern of microleakage in the gingival floor of different cut surfaces in each group. There was no significant difference between the four cut surfaces of the restorations in each group ($p>0.05$).

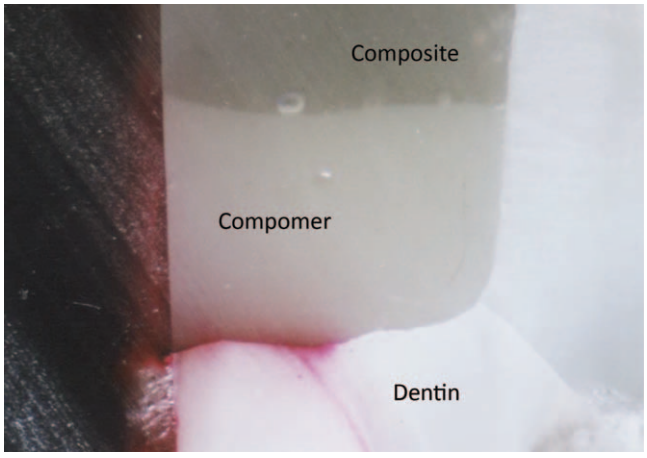

Figure 2. *Die penetration in one sample of group B (Compoglass F) at cut surface 2.*

Table 2: *Mean of Microleakage for Testing Groups*

Group	Lining Material	N	Mean ± SD,[a] 10^{-1} mm
A	No lining material (control)	10	1.28 ± 0.98 A
B	Compoglass F	10	4.36 ± 5.64 A,B
C	Tetric Flow	10	1.50 ± 1.97 A
D	Degufill SC	10	2.26 ± 1.52 A
E	Fuji II LC	10	7.99 ± 9.57 B

Abbreviation: SD, standard deviation.
[a] *Groups with different letters indicate statistical difference at $\alpha=0.05$.*

DISCUSSION

Microleakage has been a major concern in operative dentistry. A variety of techniques have been described to evaluate microleakage and the sealing properties of restorations, such as air pressure, bacterial assessment, radioisotope studies, scanning electron microscopy, chemical identifiers, electrochemical studies, and measurement of dye penetration. Some studies have reported that different methods of microleakage evaluation do not differ in terms of the final results. Because of its long-term presence in the literature,[15,24,25] the dye penetration method, which is a semiquantitative method, was chosen in this study.

Gale and others[26] clearly demonstrated that microleakage is a three-dimensional phenomenon. Raskin and others[27] performed a study on three different sites of gingival wall to test how the number of sections affected the detection of the maximum depth of tracer penetration, and they inspected the influence of the number of sections on the reliability of *in vitro* microleakage evaluations. They found that the correlation coefficient increased as the number of sections increased up to three.[27] In the present study, two cuts per restoration allowed us to measure dye penetration in four locations at the gingival floor in order to find a pattern for gingival microleakage in each group (Figure 1).

It can be assumed that in light-cure dependent lining materials (groups A, B, and C) there would be better polymerization in the middle part of the gingival floor because of the conic pattern of light transmission. Therefore, less microleakage is expected to be observed in this part. On the other hand, in groups D and E, which are self- and dual-cured, no difference is expected among the four

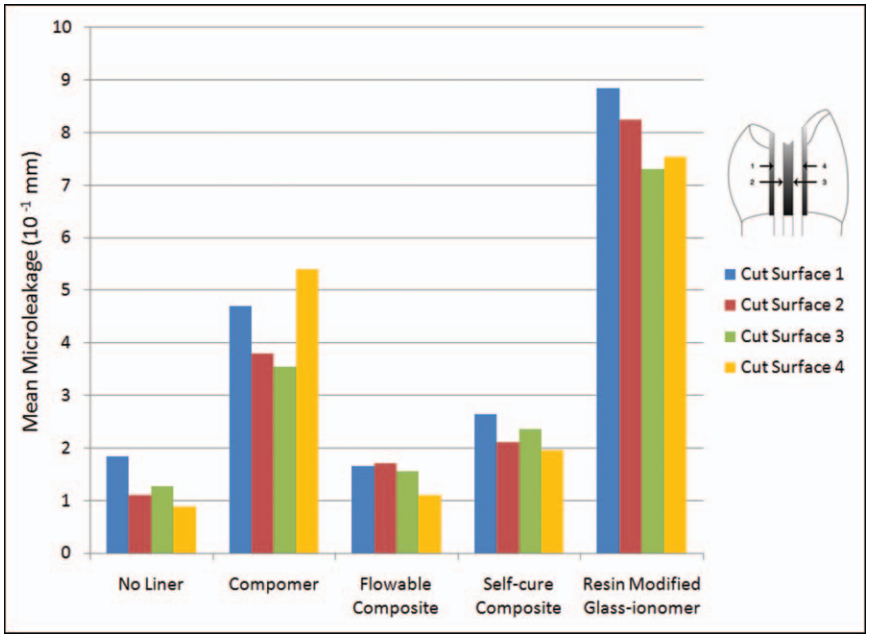

Figure 3. *Mean microleakage of each cut surface in different groups.*

surfaces. We believe that using four cut surfaces in this study in order to find the probable gingival floor microleakage pattern renders the dye penetration evaluation more quantitative in nature. However, this study does not reveal any significant difference in the microleakage values between different cut surfaces in each group ($p>0.05$).

In our study all testing groups showed some degree of microleakage, and there was no significant difference between groups A, B, C, and D. However, groups B and E showed significantly higher amounts of dye penetration or microleakage compared to groups A, C, and D (Table 2).

Some studies[11,12,28] have indicated that the use of flowable composite lining in Class II composite restorations reduced the gingival microleakage. However, other studies[29-31] indicate that it could not reduce microleakage.

The ability of flowable materials to improve marginal seal may be attributed to their composition and

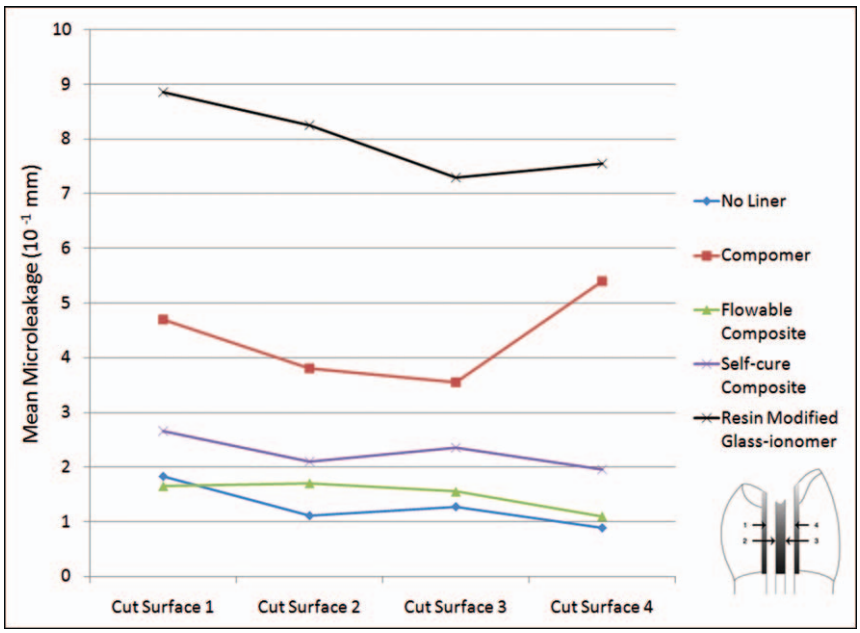

Figure 4. *Mean microleakage of each group in different cut surfaces.*

injectability. Flowable composite materials shrink more than traditional composites because of low filler loading. On the other hand, flowable composites exhibit a lower modulus of elasticity, resulting in more stress-buffering capacity than is offered by hybrid composite resins.[32] The interaction between parameters of polymerization contraction and modulus of elasticity consequently leads to an improved marginal seal of flowable composites. Chuang and others[12] demonstrated that it was advantageous to apply flowable composite in thinner layers.

In this study the microleakage of flowable composite-lined restorations was comparable to that of group A, which may be the result of the mentioned properties.[13,33]

Restorations lined with RMGI cement material presented inferior marginal sealing in group E. This is not in agreement with the findings of Chuang and others,[34] Dietrich and others,[35] and Kasraei and others,[36] who demonstrated that RMGIs improved the marginal seal and adaptation of direct Class II restorations with a sandwich technique. The controversy over sandwich restorations with glass ionomer can be related to the viscosity and application technique. An injection technique combined with a low-modulus material produced a more homogeneous restoration than resulted from use of a high-viscosity material.[37] It was demonstrated[38] that the injection technique, vs probe placement, could significantly reduce gingival microleakage in both opened and closed sandwich techniques with RMGI. The RMGI with higher viscosity and the probe placement technique used in this study could provide an explanation for the poor results noted in group E.

The thermal and load simulation of the clinical situation in the present study could be another reason for the inferior results associated with group E. Indeed, the better results for RMGI in other studies may be due to the fact that they use injection placement and low-viscosity glass ionomer with no simulation of thermal and mechanical loading. Therefore, when samples are load-cycled to simulate the clinical situation, there is no longer an advantage to glass ionomer.

Andersson-Wenckert and others[39] indicated that a noticeable distortion of RMGI occurred after years. Opdam and others[23] reported that posterior composite restorations (PCRs) with RMGI lining showed more frequent failure than did PCRs placed with the total bond technique.

The use of compomer materials for sandwich restorations has been investigated by some researchers,[35,40] and they showed that it can reduce microleakage. This finding is not in agreement with the results of this study, in which relatively higher amounts of microleakage were observed in group B (Figure 3). This finding is confirmed with another study,[41] in which Compoglass F was demonstrated to have the greatest overall microleakage. Our finding may be the result of the lower modulus of elasticity and dentinal bond strength than are associated with flowable composites.[18]

The gingival floor of Class II cavity preparations yields the greatest distance to the light source, which could decrease the degree of polymerization, leading to greater leakage values. Therefore, the use of a material with which curing is not dependent on light may be beneficial in deep cavity areas, which are far from the light source. A layer of chemically cured resin composite for the gingival floor of a proximal cavity has been suggested in order to solve this problem and improve marginal adaptation.[16,22,42,43]

The other rationales for suggesting the usage of a self-cured composite in the proximal box are its potential tendency to shrink toward the center of the mass. The mode of curing also has vectors directed toward the cavity walls. This curing "toward the tooth" is enhanced by the tendency of chemically cured composites to begin polymerizing in the warmest area of the preparation.[43] Therefore, we assume that the vectors toward the tooth could neutralize the vectors toward the mass, to some extent. In addition, the rate of polymerization allows better chances for bond formation. Some degree of stress relief could be attributed to the free surfaces offered by the porosity of chemically cured composites.[44] This can justify the lack of any significant difference between groups A and D.

The adhesive used in the present study was Syntac Single Component, which contains acidic elements, and this may explain the relatively greater-than-expected microleakage. Adverse chemical interaction between acidic resin monomers from the oxygen inhibition layer of polymerized adhesive and the tertiary amine catalyst in the self-cured composite were thought to be responsible for the incompatibility.[45,46] The other reason for incompatibility is the increase in permeability with the polymerized adhesive layer that allows time-dependent water movement through the polymerized adhesive. This can result in water blisters along the adhesive/composite interface, which is adversely affected by such an adhesive.[47-49] We used extracted teeth that had no pulpal pressure or dentinal fluid; therefore, the formation of water blisters was improbable.

The reliability of *in vitro* research depends on the extent of simulation of the oral cavity conditions. The current study used thermocycling associated with mechanical loading to offer more simulation that can accelerate adhesive and/or cohesive failures. It can increase interface gap formation and dye penetration, which means we have greater certainty in applying the results to clinical conditions.[18]

CONCLUSION

According to the results of this study, none of the four sandwich techniques used could reduce gingival microleakage more than the incremental technique.

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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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Characterization of Water Sorption, Solubility, and Roughness of Silorane- and Methacrylate-based Composite Resins

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LC Cidreira Boaro • RR Braga

Clinical Relevance

This study showed that silorane-based composites demonstrated acceptable performance in all parameters studied (water sorption, solubility, and roughness), supporting their use as an alternative restorative material.

SUMMARY

Objective: The objective of this study was to evaluate the surface roughness (SR), water sorption (WS), and solubility (SO) of four composite resins after finishing/polishing and after one year of water storage.

Materials and Methods: Two low-shrinkage composites (Filtek Silorane [3M ESPE] and Aelite LS [Bisco Inc]) and two composites of conventional formulations (Heliomolar and Tetric N-Ceram [Ivoclar Vivadent]) were tested. Their respective finishing and polishing systems (Sof-Lex Discs, 3M ESPE; Finishing Discs Kit, Bisco Inc; and Astropol F, P, HP, Ivoclar Vivadent) were used according to the manufacturers' instructions. Ten disc-shaped specimens of each composite resin were made for each evaluation. Polished surfaces were analyzed using a profilometer after 24 hours and one year. For the WS and SO, the discs were stored in desiccators until constant mass was achieved. Specimens were then stored in water for seven days or one year, at which time the mass of each specimen was measured. The specimens were dried again and dried specimen mass determined. The WS and SO were calculated from these measurements. Data were analyzed by two-way analysis of variance and Tukey post hoc test (α=0.05).

Results: Filtek Silorane showed the lowest SR, WS, and SO means. Water storage for one year increased the WS means for all composite resins tested.

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Conclusions: The silorane-based composite resin results were better than those obtained for methacrylate-based resins. One-year water storage did not change the SR and SO properties in any of the composite resins.

INTRODUCTION

Restorative composite resins consist of a polymerizable resin matrix and filler particles that are chemically bonded by silane coupling agents. In recent years, the clinical use of resin composites has considerably increased because of the advances in composite technology and the efficacy of bonding agents.[1-3]

The polymerization shrinkage of composite resins has been reduced, but it remains a concern in the restorative dentistry field. Its effects are related to marginal gap formation by interface debonding, microcracking, or fracture of the thin surrounding walls of the dental preparations.[4-7] Considerable effort has been invested in minimizing shrinkage in order to reduce the stress that stems from polymerization of composite materials. Different strategies are used to reduce polymerization shrinkage: changing the monomeric matrix or increasing the filler load.[8] Low-shrinkage composites are more beneficial when used to restore Class I and II cavity preparations because of the geometry of the preparations.

Resistance to degradation in the oral environment is essential to the longevity of composite resin restorations.[9] Some properties of composites, such as surface roughness (SR), water sorption (WS), and solubility (SO), are important parameters with which to predict the behavior of composite restorations. WS by composite resins is a diffusion-controlled process that may cause chemical degradation of the material, leading to several drawbacks, such as filler-polymeric matrix debonding[10] and residual monomer release. This process can decrease the mechanical properties of the material[10] and reduce the longevity of resin composite restorations. The SO of resin composites is reflected by the amount of leached unreacted monomers and filler particle loss.

SR is an important property that affects the appearance of the composite material. A smooth surface improves esthetics and reduces plaque retention, surface discoloration, and tissue inflammation.[11-15] It also increases the patient's comfort.[16] SR, WS, and SO properties depend on the formulation of each material, including filler content, size, shape, interparticle spacing, the monomer type, degree of curing, and the efficiency of the filler-matrix bonding.[14,17]

The aim of this study was to investigate the SR, WS, and SO of two low-shrinkage composites (Filtek Silorane and Aelite LS) and two regular composites (Heliomolar and Tetric N-Ceram) after finishing and polishing and after one year of water storage. The null hypothesis tested was that low-shrinkage composites would not present the same SR, WS, and SO when compared to conventional restorative resin-based materials.

MATERIALS AND METHODS

Four commercial composite resins were selected: two low-shrinkage composites (Filtek Silorane [3M ESPE, St Paul, MN, USA] and Aelite LS [Bisco Inc, Schaumburg, IL, USA]) and two regular composites (Heliomolar and Tetric N-Ceram [Ivoclar Vivadent, Schaan, Liechtenstein]). The respective finishing and polishing systems were also used: Sof-Lex Finishing and Polishing Discs (3M ESPE), Finishing Discs Kit (Bisco Inc), and Astropol F, P, AH (Ivoclar Vivadent). The compositions and lot numbers of the composite resins and the finishing and polishing systems are listed in Tables 1 and 2, respectively.

Surface Roughness and Scanning Electron Microscopy (SEM) Observations

For evaluating the SR, 80 cylindrical specimens of composite resins (n=10) with diameters of 2.0 mm and 6.0 mm were prepared in Teflon molds (Figure 1). The composites were inserted into the mold incrementally using a composite placement instrument (Suprafill Plastic Filling, Duflex SS White, Juiz de Fora, MG, Brazil). Two layers were enough to fill the cavity, and each increment was cured for 40 seconds with a visible light-curing unit (Demetron Optilux 501, Kerr Corp, Orange, CA, USA). The second layer was covered with a polyester Mylar strip (Dentsply, Petrópolis, RJ, Brazil), pressed with a glass slide (Glassteìcnica Import Com de Vidros Ltd, São Paulo, SP, Brazil), and light-cured.[1,18] All specimens were stored in distilled water at 37°C for 24 hours.

The specimens were finished and polished by a single investigator, and the cup and disc polishers were applied using a low-speed hand-piece (Intramatic ES, Kavo do Brazil, Joinville, SC, Brazil) for 15 seconds at a speed of 10,000 rpm. The sequence and time of application of the finishing systems are described in Table 2.

Half of the specimens of each composite were stored in water for 24 hours at 37°C (baseline SR),

Table 1: *Composition of the Composite Resins Tested in this Study*

Material (Manufacturer)	Resin Monomer	Filler Type	Filler Volume, %	Particle Size, μm	Batch Number
Filtek Silorane (3M ESPE, St Paul, MN, USA)	Silorane	Silanized quartz, yttrium fluoride	55	0.1-2	N205711
Aelite LS (Bisco Inc, Schaumburg, IL, USA)	Bis-GMA, Bis-EMA, TEGDMA	Glass filler, amorphous silica	74	~1.1	0900005990
Heliomolar (IvoclarVivadent, Schaan, Liechtenstein)	Bis-GMA, UDMA	Silicon dioxide, ytterbium difluoride	46	0.04-0.2	K35053
Tetric N-Ceram (IvoclarVivadent, Schaan, Liechtenstein)	Bis-GMA, Bis-EMA, TEGDMA	Barium glass, ytterbium difluoride	56	0.04-3	L48183

Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, Bis-phenol A diglycidylmethacrylate; Silorane, Bis-3,4-epoxycyclohexylethyl-phenylmethylsilane, 3,4-epoxycyclohexylcyclopolymethylsiloxane silanized; TEGDMA, triethylene glycol-dimethacrylate; UDMA, urethane dimethacrylate.

and the other half were stored in 10 mL of distilled water in Eppendorfs for one year before testing. To measure the SR of the specimens, a profilometer (Surfcorder SE 1700, Kosaka Laboratory Ltd, Tokyo, Japan) with a speed of 0.05 mm/s (0.25-mm cutoff) was used. Three measurements taken in different directions were recorded for all specimens to obtain the SR average (Ra) for each specimen. Two-way analysis of variance (ANOVA) was used to evaluate the data from the profilometric experiment. To identify significant differences, a Tukey test at a 5% level of significance was used (MINITAB 15, State College, PA, USA).

Three specimens per group were randomly assigned for observation using SEM (JSM 5600, Jeol Inc, Peabody, MA, USA). Specimens were sputter-coated with gold to a thickness of approximately 50 Å in a vacuum evaporator (SCD 050, Bal-Tec AG, Balzers, Liechtenstein) and photomicrographs of a representative area of the surfaces were taken at 200×.

Water Sorption and Solubility

The WS and SO analyses were assessed following short-term (seven-day) and long-term (one-year) immersion periods (n=10). The specimens of each composite resin were prepared using Teflon molds (2.0 mm in thickness and 6.0 mm in diameter) (Figure 1). After filling the mold, the composite resin surface was covered with a polyester strip (Dentsply, Petrópolis) and glass slide (Glassteìcnica Import Com de Vidros Ltd). The resulting samples were then compressed to avoid porosity and to remove the excess. Specimens were light-cured from the surface with a halogen light-curing unit (Optilux 501; Demetron/Kerr Corp).

The resin discs were stored in a desiccator (Pyrex, São Paulo, SP, Brazil) at 37°C for 22 hours until constant mass was achieved (m1). The masses of these completely dried specimens were recorded (Chyo Balance JK 180; Chyo Corp, Tokyo, Japan). Specimens were then stored for seven days or one year in water at 37°C, and the water-saturated mass was measured (m2). Finally, the specimens were dried again in the desiccator until constant mass was obtained, and their masses were once again determined (m3). The difference in mass between the initial dry and final dry mass represented the amount of SO (m1 – m3/volume of specimen), which was analyzed by two-way ANOVA and Tukey post hoc tests (α=0.05). The difference in mass between the saturated and final dry specimens provided WS values (m2 – m3/volume of specimen), which were analyzed by two-way ANOVA and Tukey post hoc test (α=0.05).

RESULTS

Surface Roughness and SEM Observations

Two-way ANOVA indicated that the factor "composite resin" ($p<0.0001$) significantly influenced SR

Table 2: *Mean Surface Roughness (SR) Produced by the Finishing Instruments Initially and After One Year of Water Storage*[a]

Composite Resins/Finishing Systems	Baseline, μm	One Year, μm
Filtek Silorane/Sof-Lex Discs	0.15 ± 0.01 Aa	0.17 ± 0.05 Aa
Aelite LS/Finishing Disc Kit	0.28 ± 0.02 Ba	0.24 ± 0.06 Ba
Heliomolar/Astropol F, P, HP	0.20 ± 0.02 Ba	0.27 ± 0.09 Ba
Tetric N-Ceram/Astropol F, P, HP	0.25 ± 0.04 Ba	0.27 ± 0.10 Ba

[a] *Groups with different uppercase (column: comparison among composite/polishing agent within the same evaluation time) and lowercase (row: comparison among the evaluation time within the same composite/polishing agent) letters are significantly different.*

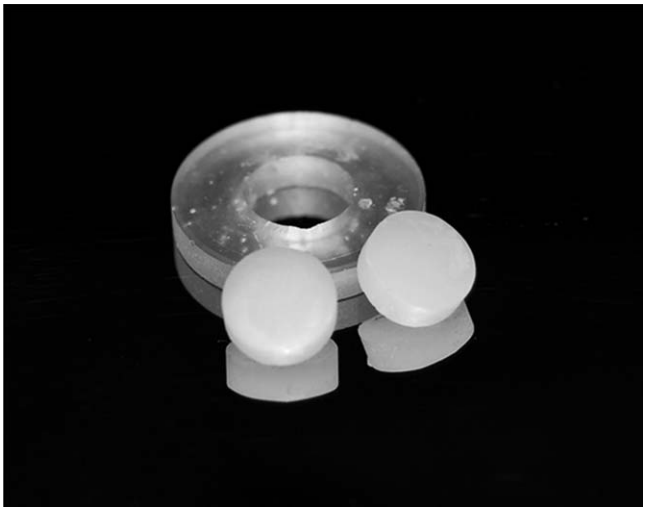

Figure 1. *Picture of cylindrical specimens prepared for testing SR, WS, and SO.*

results. Conversely, the statistical analysis revealed no significant differences for the factor "evaluation time" (p=0.167) or for interaction between factors (p=0.223). A summary of the SR means for the composite resins is shown in Table 2 and Figure 2. Analysis of data with respect to differences in composite resins showed the lowest SR means for Filtek Silorane (p<0.05). Heliomolar, Tetric N-Ceram, and Aelite LS did not differ significantly among themselves (p>0.05). The evaluation time did not influence the SR results (p>0.05).

Representative photomicrographs of the polished specimens are shown in Figures 3 through 6. After storage for one year, some porosity could be observed on the surfaces of the Aelite LS (Figure 3) and Heliomolar (Figure 4) composite resins. Nevertheless, Filtek Silorane (Figure 5) and Tetric N-Ceram (Figure 6) showed the smoothest surfaces, with similar characteristics before and after water storage for one year.

Water Sorption and Solubility

Two-way ANOVA indicated that the factors "composite resin" (p=0.023) and "evaluation time" (p=0.030) significantly influenced results. No interaction between factors (p=0.165) was identified. A summary of the WS means for the composite resins is shown in Table 3. Analysis of the data with respect to differences in composite resins showed the lowest WS means for Filtek Silorane (p<0.05). The composites differed significantly among themselves (p>0.05). Heliomolar presented the highest WS (p>0.05). The WS of all composites increased after the storage of specimens for one year in water (p>0.05).

A summary of the SO means for the composite resins is shown in Table 3 and Figure 7. Two-way

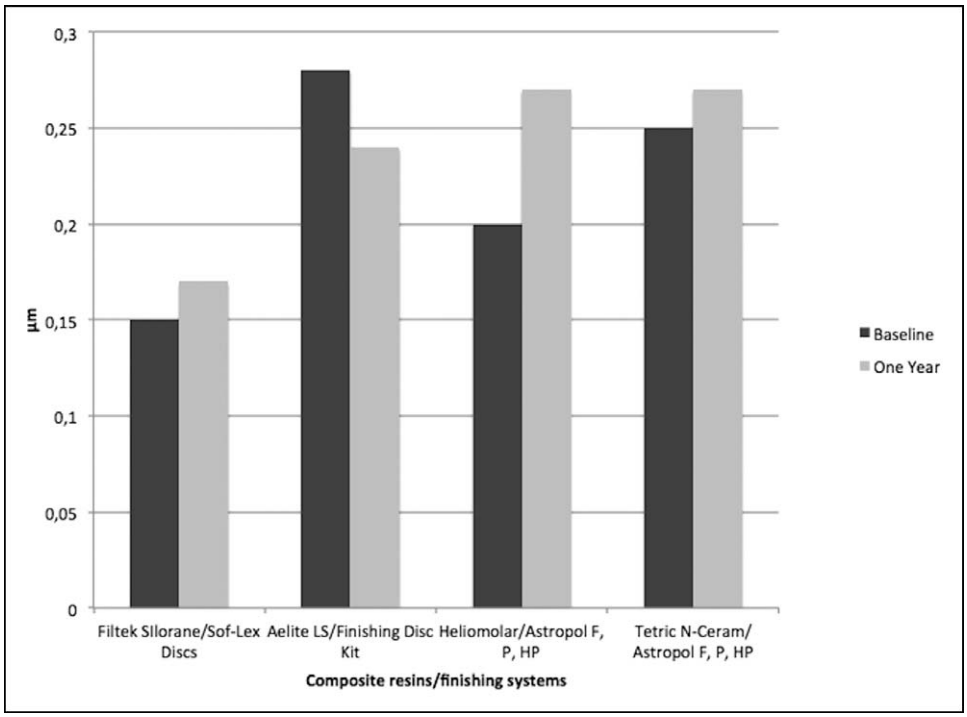

Figure 2. *Mean of the surface roughness (µm) determined 24 hours and one year after finishing and polishing.*

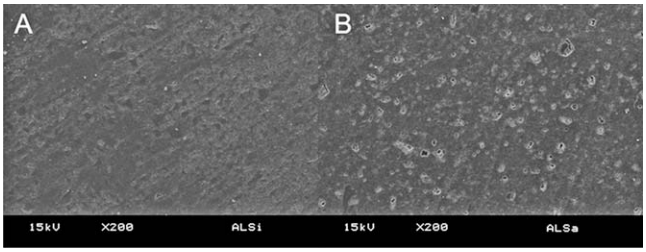

Figure 3. *SEM photograph of Aelite LS low-shrinkage composite resin surface after finishing and polishing with Finishing Disc Kit (A) and after one year of storage in water (B) (magnification 200×).*

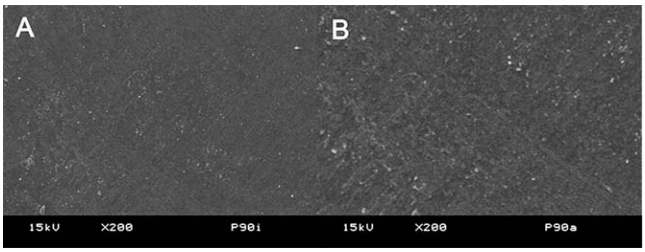

Figure 5. *SEM photograph of Filtek Silorane low-shrinkage composite resin surface after finishing and polishing with Sof-Lex Discs (A) and after one year of storage in water (B) (magnification 200×).*

ANOVA indicated that the factor "composite resin" ($p=0.001$) significantly influenced SO results. Conversely, the statistical analysis revealed no significant differences for the factor "evaluation time" ($p=0.114$) or for interaction between factors ($p=0.175$). Analysis of the data with respect to differences in composite resins showed the lowest SO means also for Filtek Silorane ($p<0.05$). The evaluation time did not influence the SO results ($p>0.05$). Tetric N-Ceram yielded significantly lower SO means than did Aelite LS ($p<0.05$). The storage time did not influence the SO results ($p>0.05$).

DISCUSSION

An important factor in determining the SR is the intrinsic roughness of a composite material, which is determined by the size, shape, and quantity of the filler particles.[19] Filtek Silorane contains quartz and yttrium fluoride as filler particles. Its average particle size is 0.1-2 μm and the filler volume is 55% according to the manufacturer's information. The lowest SR was observed for Filtek Silorane, whereas other materials did not differ among themselves. In addition, the surfaces analyzed by SEM did not change after storage for one year. Thus, the null hypothesis tested against SR was rejected.

The WS and SO means were also the lowest for Filtek Silorane, which contains silorane monomers and is a unique material that does not contain methacrylated monomers. For the properties investigated above, the null hypothesis was also rejected.

The finishing and polishing systems influence the SR, the gloss, and the maintenance of the color of restorations.[20-22] Most of the published data show that existing polishing systems provide sufficiently smooth surfaces, with Ra values ranging from 0.02 μm to 0.56 μm.[20,23,24] All means obtained in this study for the Filtek Silorane, Heliomolar, Tetric N-Ceram, and Aelite LS composites are included in this range. Other studies[25-29] have shown that when the finishing/polishing system and composite material are from the same manufacturer, their compatibility and polishing results are significantly better. Polishing particles must be harder than the filler particles to ensure that the removal of the resin matrix and the fillers would be accomplished in the same way during the polishing.[25,27,29] Sof-Lex discs are composed of aluminum oxide, which is able to cut the filler particles and the polymerized matrix in almost the same way, which explains the lower values of SR and the smooth surface for Filtek Silorane.

The photomicrographs showed that after finishing and polishing the surfaces were initially smooth, but after a year of water storage, the surfaces of Heliomolar and Aelite LS showed some irregularities and porosity. Heliomolar has the lowest filler loading (46% by volume), whereas Aelite LS presents the

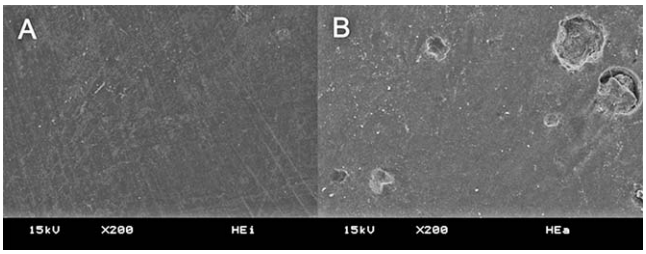

Figure 4. *SEM photograph of Heliomolar composite resin surface after finishing and polishing with Astropol F, P, HP (A) and after one year of storage in water (B) (magnification 200×).*

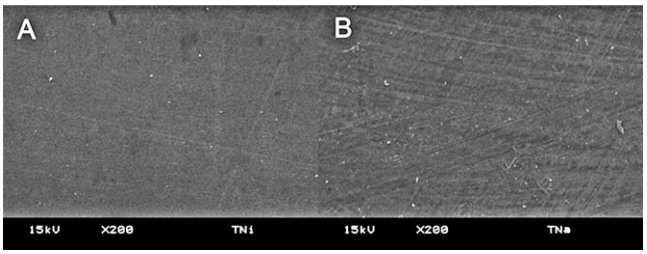

Figure 6. *SEM photograph of Tetric N-Ceram composite resin surface after finishing and polishing with Astropol F, P, HP (A) and after one year of storage in water (B) (magnification 200×).*

Table 3: *Mean Water Sorption (WS) and Solubility (SO) for Each Sample Group*[a]

Composite	Sorption, μg/mm³		Solubility, μg/mm³	
	Baseline	One Year	Baseline	One Year
Filtek Silorane	9.3 ± 1.9 Da	12.4 ± 0.6 Db	−1.6 ± 0.7 Ca	−1.7 ± 0.5 Ca
Aelite LS	17.6 ± 2.0 Ca	18.1 ± 1.6 Cb	10.6 ± 2.1 Aa	12.2 ± 0.8 Aa
Heliomolar	22.3 ± 3.4 Aa	28.1 ± 1.7 Ab	7.6 ± 1.1 ABa	10.5 ± 0.5 ABa
Tetric N-Ceram	19.2 ± 1.9 Ba	24.1 ± 1.0 Bb	4.7 ± 1.3 Ba	6.1 ± 0.5 Ba

[a] *Groups with different uppercase (column: comparison among composite within the same evaluation time) and lowercase (row: comparison among the evaluation time within the same composite) letters are significantly different.*

highest filler content (74%). Conversely, Heliomolar has a higher monomeric content than does Aelite LS. One of the reasons for the change in SR for Heliomolar is that the water-exposed polymerized organic matrix may be degraded or dissolved, although no alterations were observed for SO analysis. On the other hand, the finishing and polishing for Aelite is more difficult as a result of the high amount of filler particles, which can be seen in the exposed composite surface after storage for one year (Figure 3B).

The ISO 4049 standard established that the maximum WS value is 40 μg/mm³, whereas the maximum SO value is <7.5 μg/mm³. No composite exceeded the maximum WS value, even after one year of storage in water, at which point the means increased significantly. The WS for the composite resins tested in this study ranged from 9.3 to 28.1 μg/mm³. For SO, the Filtek Silorane and Tetric N-Ceram showed lower means than those established by ISO 4049. The SO for the composite resins tested in this study ranged from −1.6 to 12.2 μg/mm³.

Toledano and others[30] (2003) reported that WS and SO mainly depend on the resin compositions. Statistically speaking, the silorane-based composite Filtek Silorane had the lowest values of WS and SO. Silorane is a monomer, with a combination of hydrophobic siloxane and low-shrinkage ring-opening oxirane.[8] Its cationic photo-initiated polymerization reduces the polymerization shrinkage and increases the degree of conversion.[8,31,32] Thus, the WS of Filtek Silorane is expected to be low. These

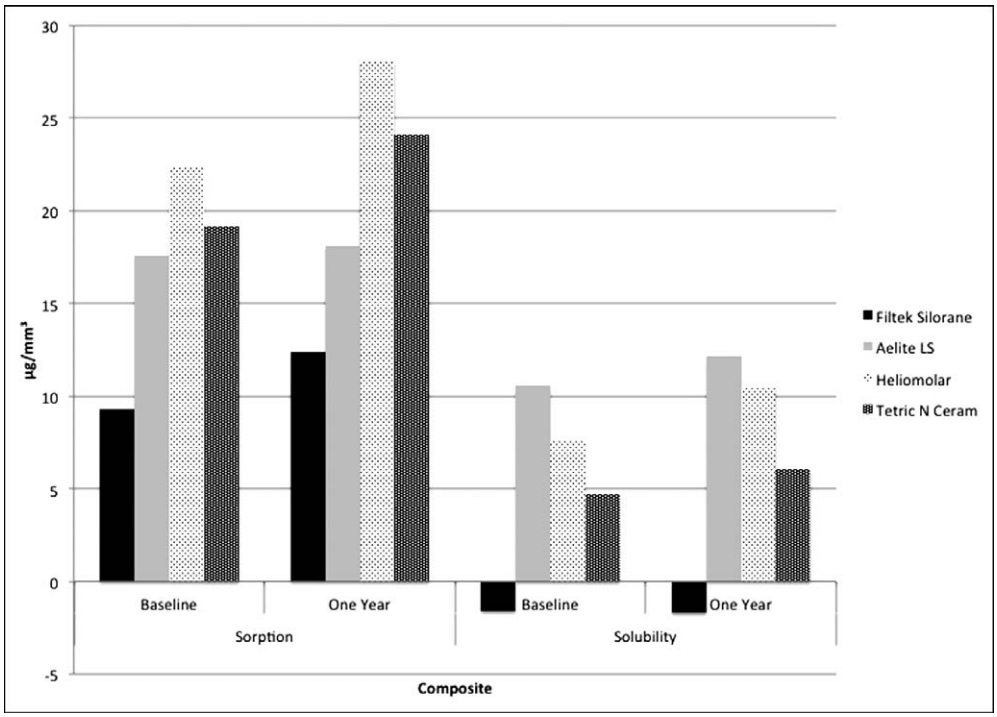

Figure 7. *Mean of the water sorption and solubility (μg/mm³) determined after short-term (sevenday) and long-term (one-year) immersion periods.*

findings are in accordance with the literature,[33,34] in which it is reported that silorane-based composites exhibit greater hydrophobic behavior than do methacrylate-based composite resins.

Some hydrophilic constituents, such as urethane dimethacrylate or resin molecules that contain hydrophilic moieties, increase WS,[17] as observed in Heliomolar, which showed the highest WS means. Tetric N-Ceram showed more WS than did Aelite LS, which is a low-shrinkage composite. Although the Tetric N-Ceram and Aelite LS composites contain the same monomeric composition (Bis-phenol A diglycidylmethacrylate, ethoxylated bisphenol A dimethacrylate, and triethylene glycol-dimethacrylate resins), there is a significant difference when the filler particle content was compared (56% and 74% by volume, respectively). The high volume of filler and consequently low monomeric content resulted in reduction of WS, because the WS is mainly promoted by the organic matrix.[35]

Some factors affect the SO of composites, such as the number and the size of leachable species, the type of monomers, the quality of resin-filler adhesion, the solvents, immersion time, and temperature.[30,36] The mass of the components eluted from the composite is found through the water SO data.

The siloxane compound presented in the Filtek Silorane sample clearly provided a material with much lower SO than the other samples tested in this study due to their hydrophobicity.[37] This confirmed previous findings,[34-37] which have indicated that this material is stable in aqueous environments. The value of SO was negative for Filtek Silorane. Berger and others[38] found that methacrylate-based composite also demonstrated a negative value. The negative values were obtained because the m3 (mass after storage) was higher than the m1 (mass after specimen preparation). One possible explanation for the negative values is that the water absorbed during storage may be trapped and included as part of the polymeric structure of the composite material.

The high filler-loaded Aelite LS composite presented the highest mean of SO after one year. It is possible that the water that is in contact with silica surface breaks the siloxane bonds, forming silanol groups, which in turn facilitates particle debonding. Because the hydrolytic stability of coupling agents can vary according to the type of filler particles,[39] no conclusive evidence can be provided to indicate that hydrolytic degradation of the fillers affects the SO behavior of dental composites.[40]

In this study, the length of storage time only influenced the WS, which was higher after one year than during the baseline measurements. For the SO, the storage time did not change the values, although the means tended to increase after long-term water storage. Several investigations have analyzed the WS and SO of resin-based materials; however, it is difficult to compare the data because the studies have used different storage periods, expression units,[35] and sample dimensions.[30] Regarding the properties studied, silorane-based composites are shown to be a better alternative for low-shrinkage restorative materials (as compared to increasing the filler content of composite resins for the purpose of decreasing the polymerization shrinkage).

CONCLUSIONS

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

1. The silorane-based material Filtek Silorane showed lower SR, WS, and SO results than were obtained with methacrylate resin-based materials.
2. The storage in water for one year increased the WS for all composite resins tested, while no changes were observed for SO.

Acknowledgement

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Conflict of Interest

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

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Effects of Pulsed Nd:YAG Laser on Tensile Bond Strength and Caries Resistance of Human Enamel

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Clinical Relevance

Pulsed Nd:YAG laser may contribute to the tensile bond strength of resin to enamel and increase the acid resistance of enamel surfaces. It can be a clinical alternative for pretreating enamel surfaces when direct bonding orthodontic attachments or adhesive restorations.

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SUMMARY

This study aims to evaluate the effects of pulsed Nd:YAG laser on the tensile bond strength (TBS) of resin to human enamel and caries resistance of human enamel. A total of 201 human premolars were used in this *in vitro* study. A flat enamel surface greater than 4 × 4 mm in area was prepared on each specimen using a low-speed cutting machine under a water coolant. Twenty-one specimens were divided into seven groups for morphology observations with no treatment, 35% phosphoric acid etching (30 seconds), and laser irradiation (30 seconds) of pulsed Nd:YAG laser with five different laser-parameter combinations. Another 100 specimens were used for TBS testing. They were embedded in self-cured acrylic resin and randomly divided into 10 groups. After enamel surface pretreatments according to the group design, resin was applied. The TBS values were tested using a universal testing machine. The other 80 specimens were randomly divided into eight groups for acid resistance evaluation. Scanning electron microscope (SEM) results

showed that the enamel surfaces treated with 1.5 W/20 Hz and 2.0 W/20 Hz showed more etching-like appearance than those with other laser-parameter combinations. The laser-parameter combinations of 1.5 W/15 Hz and 1.5 W/20 Hz were found to be efficient for the TBS test. The mean TBS value of 14.45 ± 1.67 MPa in the laser irradiated group was significantly higher than that in the untreated group (3.48 ± 0.35 MPa) but lower than that in the 35% phosphoric acid group (21.50 ± 3.02 MPa). The highest mean TBS value of 26.64 ± 5.22 MPa was identified in the combination group (laser irradiation and then acid etching). Acid resistance evaluation showed that the pulsed Nd:YAG laser was efficient in preventing enamel demineralization. The SEM results of the fractured enamel surfaces, resin/enamel interfaces, and demineralization depths were consistent with those of the TBS test and the acid resistance evaluation. Pulsed Nd:YAG laser as an enamel surface pretreatment method presents a potential clinical application, especially for the caries-susceptible population or individuals with recently bleached teeth.

INTRODUCTION

The field of adhesive dentistry has achieved remarkable progress over the past decades. This development is largely attributed to the major advancements in enamel surface pretreatments, which roughen the enamel surface and offer more microspace for resin monomers to penetrate and form resin tags. The microspace significantly increases the bond strength of resin to the enamel.[1,2] The acid-etching technique that usually uses 35% phosphoric acid gel is one of the most common enamel surface pretreatments for resin bonding.[3] However, the possibility of decalcification that leaves the enamel at the resin restoration margin or around orthodontic attachments more susceptible to caries attack cannot be ignored because it ultimately leads to treatment failures.[4-6] Therefore, enamel surface pretreatment needs to be improved to maintain clinically useful bond strengths while minimizing enamel loss; enamel surface pretreatment also needs to be simplified to reduce the bonding steps.[7,8]

Various lasers have recently elicited considerable interest in the field of dentistry because of their function in the detection and removal of enamel caries, diagnosis of dental caries, cavity preparation, etching of teeth for resin bonding, demineralization prevention, root canal sterilization, and so on.[9-11] Lasers have received significant attention in resin bonding, especially in terms of their roughness and demineralization prevention effect on dental hard tissues.[11,12] Laser etching eliminates the need for separate steps of water spraying and air drying, thereby reducing procedural errors and saving time.

Pulsed Nd:YAG laser emits a wavelength of 1.064 μm. It is capable of promoting the fusion and recrystallization of dental hard tissue surfaces and causing an irregular honeycomb or crater morphology, which increases surface microhardness and contributes to resin bonding.[12,13] In our previous studies, pulsed Nd:YAG laser was found to contribute significantly to root canal sterilization and to enhance the bond strength/microleakage of resin to human dentin.[14,15] However, the enamel is less pigmented than the dentin, and the Nd:YAG laser wavelength is preferentially absorbed in pigmented tissues. Therefore, the effects of pulsed Nd:YAG laser on the bond strength of human enamel may differ from its influence on human dentin.

This study aims to investigate the effects of pulsed Nd:YAG laser on the bond strength and acid resistance of human enamel to provide a potential enamel surface pretreatment for resin bonding.

METHODS AND MATERIALS

Tooth Selection and Preparation

A total of 201 human premolars that are caries-free and freshly extracted for orthodontic treatment were selected after approval from the Institutional Review Board. From the 201 human premolars, 21 teeth were used for morphology observations, 100 for tensile bond strength (TBS) testing, and 80 for acid resistance evaluation. The roots were removed and the buccal surfaces were ground using a low-speed cutting machine (MF-PERFECTA, Bürmoos, Austria) under a water coolant. A flat surface more than 4×4 mm in enamel was prepared and polished with 200-, 400-, and 600-grit silicon carbide abrasive papers.

Enamel Surface Treatments

The prepared specimens were conditioned with the treatments according to the different experiments in this study. The surface-conditioning methods used in this study are presented in Table 1.

Morphology Observations

The 21 prepared specimens were divided into seven groups (three specimens for each group). They

Table 1: *Enamel Surface Conditioning Methods Used in This Study*

Surface Conditioning Methods	Details for Each Method
Untreated	Enamel surfaces were left no treatment (as control).
Acid etching	Enamel surface were etched with 35% phosphoric acid gel for 30 s, then washed, and dried.
Laser irradiation	Enamel surface were irradiated by pulsed Nd:YAG laser (Friendly A4.0, Milan, Italy) for 30 s in a freehand scanning mode, and the laser optic fiber (320 mm) was used in the standard position, ie, perpendicular to and 1 mm from the enamel surfaces. Black ink was used to initiate and enhance absorption.

received no treatment, acid etching, or laser irradiation with five different laser-parameter combinations (Table 2). The treated specimens were cleaned with distilled water, dehydrated in a graded series of alcohol solutions (70%, 90%, and 100%) for 10 minutes at each concentration, and then sputter-coated with gold. The morphologic changes were examined using a scanning electron microscope (SEM) (Jeol JMS 5200, Tokyo, Japan).

TBS Test and Fractured Surface Examination

The 100 prepared specimens were randomly divided into 10 groups (n=10). Five groups were used to optimize laser-parameter combinations, whereas the other five were utilized to compare pulsed Nd:YAG laser with 35% phosphoric acid gel (Table 2).

The specimens were embedded in self-cured acrylic resin (Shanghai Dental Factory, Shanghai, China). The prepared buccal enamel surfaces were maintained in a face-up position and about 1 mm higher than acrylic resin to keep them unaffected. A thin paper with a 4 × 4 mm hole was fixed on the enamel surface of each specimen. The treatments were performed as shown in Table 2. A two-step etch-and-rinse adhesive system (Adper Single Bond 2, 3M/ESPE, Irvine, CA, USA) was used according to the manufacturer's instructions. The adhesive was applied with disposable microbrush tips and light-cured for 10 seconds with a halogen curing light (Elipar 2500, 3M ESPE, St Paul, MN, USA) under a light intensity of 800 mW/cm^2 after the excess solvent was evaporated with a gentle air stream for 5 seconds. A 4-mm high composite buildup that entirely covered the prepared enamel surface was made with a micro-hybrid resin composite (Filtek Z250, A2, 3M/ESPE) and light-cured for 80 seconds (20 seconds for each side). All specimens were immersed in distilled water at 37°C for 24 hours. The TBS values were tested using a universal testing machine (WDW-10, Panasonic, Osaka, Japan). Each specimen was mounted on the machine, after which tensile force was applied at a constant speed of 0.5 mm/min through the self-made clamps. The force values (N) at the failure point were recorded and converted to MPa by dividing them with the exposed enamel surface (16 mm^2).

The fractured surfaces of the specimens were examined under stereomicroscope (10×) and SEM. The fracture patterns were assessed by adhesive remnant index (ARI), which was based on the remaining resin amount on the enamel surface (ARI score: 0, no resin left; 1, little resin left; 2, resin covering less than half of the bonding surface; 3, resin covering more than half of the bonding

Table 2: *Tensile Bond Strength (TBS) Means in the Ten Groups With Different Treatments (n=10)*†

Groups	Enamel Surface Treatments	Tensile Bond Strengths, MPa
1	Laser irradiation (1.0 W/15 Hz)	5.99 ± 0.56*
2	Laser irradiation (1.0 W/20 Hz)	9.15 ± 0.51**
3	Laser irradiation (1.5 W/15 Hz)	10.43 ± 1.48**
4	Laser irradiation (1.5 W/20 Hz)	13.87 ± 1.91***
5	Laser irradiation (2.0 W/20 Hz)	5.14 ± 1.04*
A	Untreated	3.48 ± 0.35^d
B	Acid etching	21.50 ± 3.02^b
C	Laser irradiation (optimized)	14.45 ± 1.67^c
D	Laser irradiation then acid etching	26.64 ± 5.22^a
E	Acid etching then laser irradiation	12.42 ± 0.39^c

† *Labeled groups showed statistical significance (p<0.05). In groups 1-5 and A-E, superscripts indicate statistically similar groups.*

Table 3: *Ca^{2+} Concentrations in the Eight Groups With Different Treatments (n=10)†*

Groups	Enamel Surface Treatments	Ca^{2+} Concentration, ppm
I	Laser irradiation (1.0 W/15 Hz)	116.69 ± 7.47**
II	Laser irradiation (1.0 W/20 Hz)	105.40 ± 7.82**
III	Laser irradiation (1.5 W/15 Hz)	80.57 ± 6.26*
IV	Laser irradiation (1.5 W/20 Hz)	82.32 ± 8.31*
V	Laser irradiation (2.0 W/20 Hz)	124.40 ± 8.40***
a	Untreated	247.54 ± 6.80[b]
b	Acid etching	319.84 ± 6.16[c]
c	Laser irradiation (optimized)	84.81 ± 9.57[a]

† *Labeled groups showed statistical significance ($p<0.05$). In groups I-V and a-c, superscripts indicate statistically similar groups.*

surface; and 4, resin covering the whole bonding surface).[16]

Acid Resistance Evaluation

The 80 prepared specimens were randomly divided into eight groups (n=10). Before undergoing the treatments listed in Table 3, the specimens were coated by an acid-resistant varnish (nail polish) with a 4-mm square window on the enamel surface left uncoated. The specimens were then subjected to an artificial demineralization solution (0.1 M lactic acid) for 48 hours, as described in a previous study.[17] The demineralization solutions were analyzed using a calcium-selective electrolyte analyzer (ISE-trol AVL 9180, AVL Scientific Corp, Roswell, GA, USA) to evaluate the concentration of calcium ion (Ca^{2+}) dissolved from the treated enamel surfaces in each group. The fully dried specimens were longitudinally sectioned and examined by SEM. An electron probe microanalyzer was used to determine the mean demineralization depths in the enamel surfaces of each group.

Statistical Analysis

The data were analyzed with SPSS 13.0 software. One-way analysis of variance (ANOVA) was used to determine whether a significant difference existed between the experimental groups on TBS, Ca^{2+} concentrations, and artificial caries depths. Pearson chi-square test was used to analyze the frequencies of the specimens in each ARI score.

RESULTS

Effect of Pulsed Nd:YAG Laser on Human Enamel Morphology

SEM images showed that the untreated enamel surfaces (Figure 1A) were coated with a smear layer that was replaced by a typical acid-etched pattern with a regular rough surface after 35% phosphoric acid gel treatment (Figure 1B). The laser irradiation caused uneven surface melting and resolidification in the enamel surface and the formation of numerous irregular honeycombs or craters (Figure 1C through G). The honeycombs or craters surrounded by abundant bubble-like cavities and numerous micropores were scattered on the enamel surface irradiated with low frequency (15 Hz) (Figure 1C,E), but became relatively equally distributed with an increase in frequency (20 Hz) (Figure 1D,F,G). The diameter of the honeycombs or craters was enlarged with a change in power from 1.0 W to 2.0 W. The enamel surfaces treated with 1.5 W/20 Hz and 2.0 W/20 Hz showed more etch-like appearance than those with other laser-parameter combinations.

Effect of Pulsed Nd:YAG Laser on Resin to Human Enamel TBS

The mean TBS values are shown in Table 2. In the five laser irradiation groups, TBS values increased and subsequently decreased with an increase in laser output energy. The mean TBS values significantly increased ($p<0.01$) when the power increased from 1.0 W to 1.5 W, and reached the highest value of 13.87 ± 1.91 MPa at 1.5 W/20 Hz. However, the values significantly decreased at a power of 2.0 W. The data indicate that the optimal laser-parameter combination for enamel pretreatment before bonding is 1.5 W/20 Hz.

Compared with the 35% phosphoric acid group whose TBS mean was 21.50 ± 3.02 MPa in this study (Table 2), the pulsed Nd:YAG laser group with optimized laser-parameter combination showed less efficient effects on TBS value, though its TBS mean (14.45 ± 1.67 MPa) was significantly higher than that of the untreated group (3.48 ± 0.35 MPa) ($p<0.01$). The combination of acid etching and laser irradiation (12.42 ± 0.39 MPa) showed no significant decrease compared with the laser group ($p>0.05$), whereas the combination of laser irradiation and

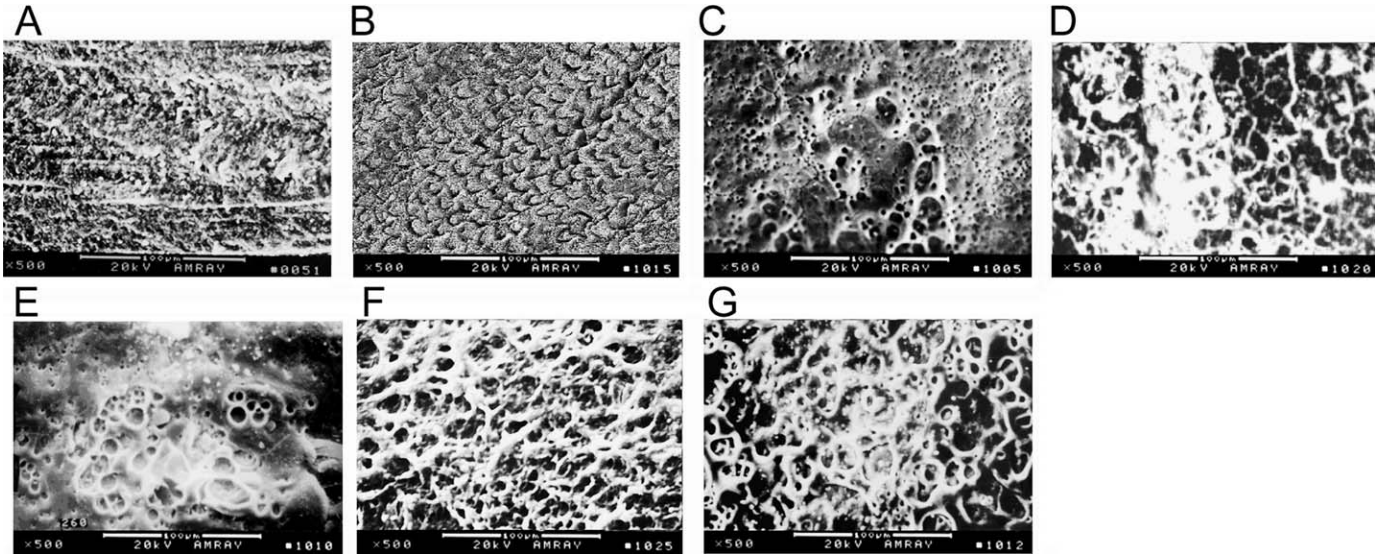

Figure 1. *SEM images of enamel surfaces with different treatments. (A): Enamel surface (untreated) coated with a smear layer. (B): Enamel surface (acid etching) showing a uniform honeycomb appearance. (C-G) Images of enamel surfaces with 1.0 W/15 Hz, 1.0 W/20 Hz, 1.5 W/15 Hz, 1.5 W/20 Hz, and 2.0 W/20 Hz laser parameters, respectively. The enamel surface irradiated with low frequency (C and E) showed scattered honeycombs or craters, whereas the enamel surface irradiated with high frequency showed an etching-like appearance (D, F, and G).*

acid etching significantly increased the TBS value. The TBS mean of the latter combined methods (26.64 ± 5.22 MPa) was significantly higher than that of the acid etching group ($p<0.01$), indicating that the irradiation of pulsed Nd:YAG laser before etching significantly contributed to the TBS of resin to human enamel.

The frequencies of the specimens in each ARI column are shown in Table 4. The ARI scores for each group are calculated as follows: 34, group D; 30, group B; 23, group C; 20, group E; and 7, group A. These scores were positively related to the TBS values. The SEM images of the fractured enamel surfaces showed that the remnant resin covered almost the whole bonding surface in most specimens of groups B and D (Figure 2B,D). By contrast, only a small amount of remnant resin was observed in group A (Figure 2A). The remnant resin covered more than half of the bonding surface in most specimens in groups B and E (Figure 2C,E). The SEM images of resin/enamel interfaces showed the thinnest gaps in groups B (Figure 3A) and D (data not shown), followed by those in groups C (Figure 3B) and E (data not shown). A clear gap was observed in group A (Figure 3C). The morphologic data enhanced understanding of the TBS results.

Table 4: *Frequencies of Specimens in Each Adhesive Remnant Index (ARI) Column for the Five Experimental Groups (n=10)*

Groups	ARI				
	0	**1**	**2**	**3**	**4**
A	6	2	1	1	0
B	0	0	3	4	3
C	0	2	4	3	1
D	0	0	1	4	5
E	0	4	3	2	1

Effect of Pulsed Nd:YAG Laser on the Acid Resistance of Human Enamel

The effects of laser-parameter settings on enamel surface demineralization were tested (Table 3). Group III showed the lowest Ca^{2+} concentration dissolved from enamel surfaces, followed by group IV. No significant difference was observed between the two groups ($p>0.05$), but they were significantly lower than those of the other three groups ($p<0.01$). The Ca^{2+} concentration in group V was significantly higher than those in all other groups ($p<0.05$). The data showed that the moderate laser-parameter combinations of 1.5 W/15 Hz and 1.5 W/20 Hz significantly contributed to enamel acid resistance.

In the comparison experiments, the pulsed Nd:YAG laser showed the lowest Ca^{2+} concentration (Table 3), whereas the 35% phosphoric acid showed the highest Ca^{2+} level dissolved from enamel surfaces, which was even significantly higher than that in the untreated group ($p<0.05$). SEM investi-

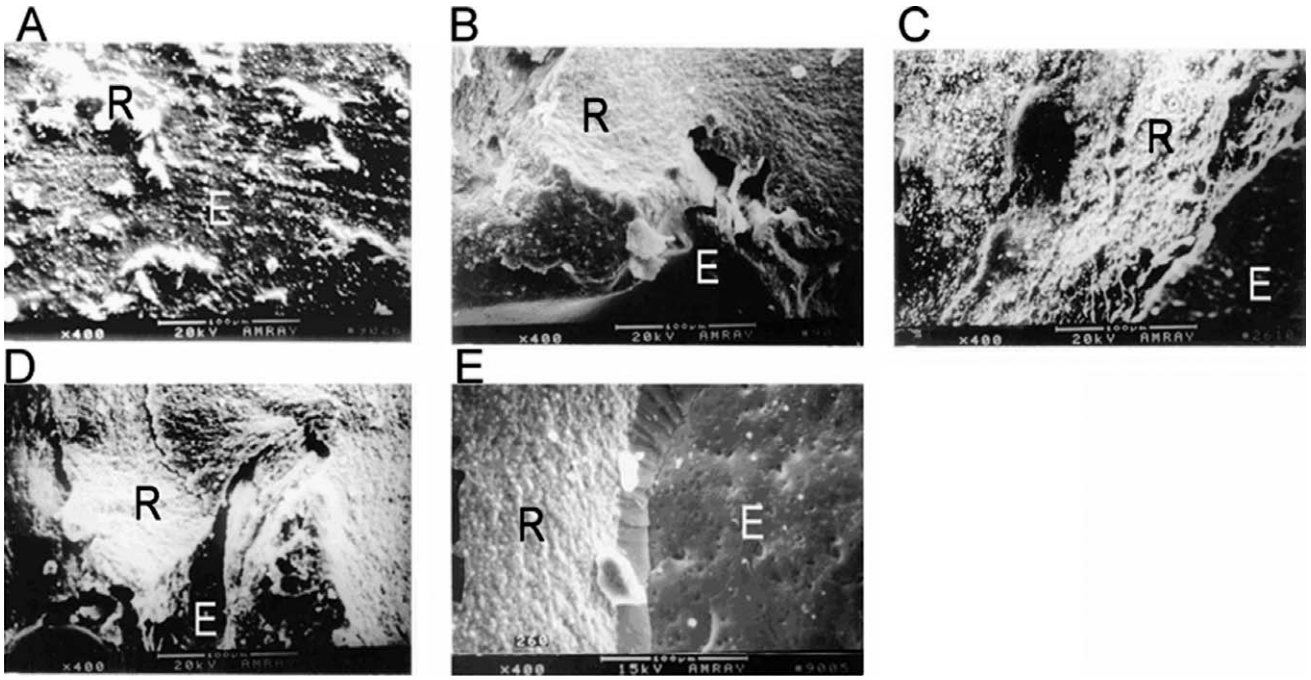

Figure 2. *SEM images of fractured enamel surfaces after TBS tests. A small amount of resin was left on the bonding surface of the untreated group (A). Remnant resin almost covered the whole bonding surface in most specimens of both acid etching (B) and laser/etching groups (D), more than half of the specimens in the laser irradiation group (C), and approximately half in the etching/laser group (E).*

gations showed that several shallow demineralization islands (Figure 4A) were found in the enamel surfaces of the laser irradiation group, whereas whole demineralization defects were found in the acid-etching (Figure 4B) and untreated groups (Figure 4C). Demineralization depth measurements (Figure 5) showed that the mean demineralization depth was only 47.78 ± 6.31 μm in the laser irradiation group, whereas it was 236.42 ± 11.88 μm in the acid etching group. This value was also significantly higher than that in the untreated group (179.06 ± 13.25 μm) ($p<0.05$). The data indicate that enamel acid resistance was significantly enhanced by the pulsed Nd:YAG laser, but adversely affected by 35% phosphoric acid.

DISCUSSION

Enamel pretreatment is critical for the application of composite resin in the direct bonding of orthodontic attachments or the esthetic restoration of anterior tooth defects.[18,19] Evidence has shown that the Nd:YAG laser can cause an irregular honeycomb or crater morphology on the enamel surface by irradiation, and thus, presents a potential use in enamel pretreatment.[13] To establish an alternative enamel

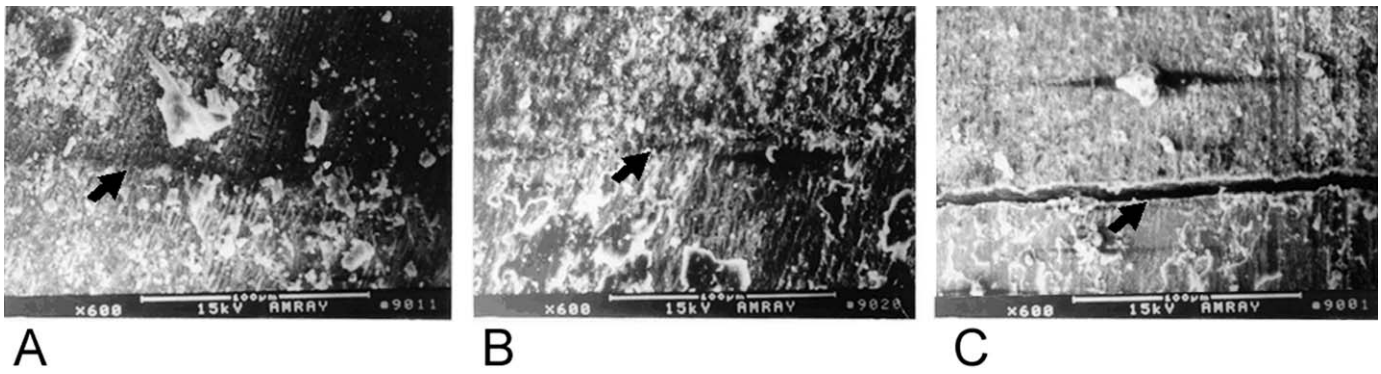

Figure 3. *SEM images of resin/enamel interfaces. The untreated group (A) showed a clear gap between the resin and enamel. A similar thin gap was observed in the acid etching (B) and laser irradiation groups (C).*

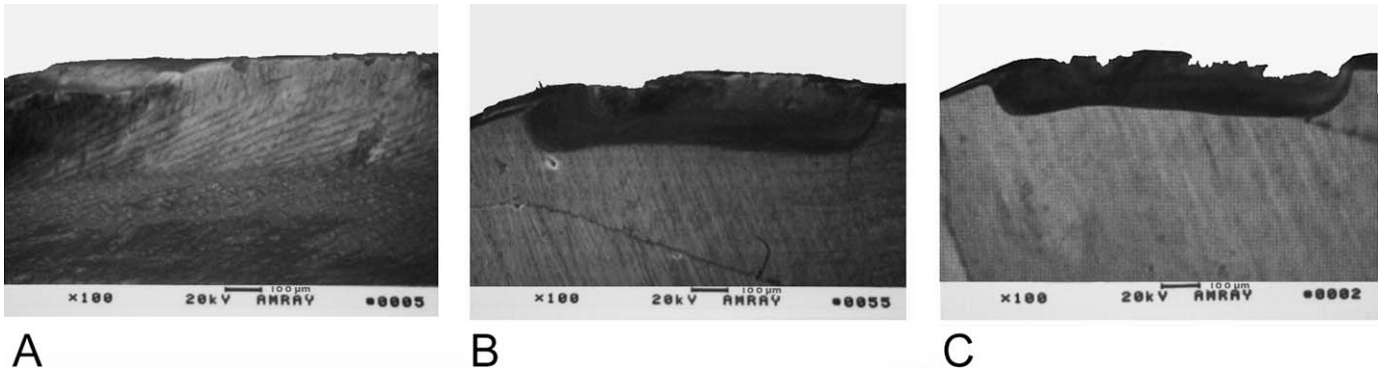

Figure 4. *SEM images of demineralized enamel interfaces. Whole demineralization defects were found in the untreated (A) and acid etching (B) groups, whereas several shallow demineralization islands were observed in the laser irradiation group (C).*

pretreatment method, we investigated the effects of pulsed Nd:YAG laser on the TBS and caries resistance of human enamel, given that acid etching increases the risk of caries.

Previous studies have indicated that lasers produce morphologic modifications on dental hard tissue by melting and solidification.[20,21] These alterations include honeycombs or craters and numerous microcavities or micropores at their periphery, which may offer microspace for the sealant or composite anchorage.[22-24] However, previous studies have shown conflicting results because of the high number of variables involved in the lasing process, such as power, pulse frequency, and duration of irradiation.[25] Laser-parameter combinations should be optimized to maximize the potential of pulsed Nd:YAG laser. We found that laser output energy level is crucial in the effects of pulsed Nd:YAG laser on enamel morphologic modifications. Low power and frequency were insufficient to alter enamel surface, and the formed honeycombs or craters were scattered. An increase in laser output energy resulted in relatively equally distributed honeycombs or craters and etching-like appearances on the enamel surfaces. This finding was consistent with the report of Bedini and others[26] that a high-energy level of Nd:YAG laser creates a retentive surface suitable for sealant or composite anchorage.

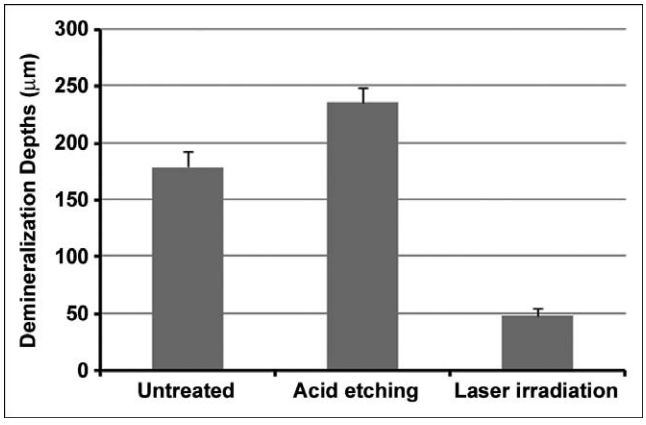

Figure 5. *Mean demineralization depths for the three experimental groups. A significant difference was observed among the groups ($p<0.05$).*

The Nd:YAG laser is less efficient in ablating the dental hard tissues compared with the Er:YAG laser, but it is superior in inducing morphologic alterations that are essential in providing microspaces for resin monomer penetration and in increasing enamel acid resistance.[27] In this study, we investigated the different output energy levels of pulsed Nd:YAG laser and found that neither low nor high laser-parameter combinations contributed to the TBS of resin to enamel. On one hand, significantly low laser-parameter combinations were proven inadequate in altering enamel surfaces and in offering sufficient microspace for resin monomers to penetrate. On the other hand, significantly high laser-parameter combinations induce excessive melting and recrystallization, as well as decrease the enamel surface free energy and microhardness that seriously damage the bond strength of resin to enamel. Moderate energy levels are more efficient in increasing the bond strength of resin to enamel. These findings were inconsistent with the study of Kwon and others[24] who reported that a higher laser power produces higher bond strength than a lower laser power. This phenomenon is possibly caused by the different laser scan speeds and irradiation times used in this study, which resulted in the different energy levels in the unit area.

Although considerable TBS values were achieved with pulsed Nd:YAG laser treatment, they remained significantly lower than that of 35% phosphoric acid treatment, as presented in previous reports.[21,28]

However, the difference in the current study is that laser irradiation, followed by etching, significantly increased the TBS of resin to enamel. We speculate that the subsequent treatment of 35% phosphoric acid further roughened the lased enamel surfaces and simultaneously eliminated the weak structures caused by the Nd:YAG laser.

Among the physical and chemical alterations of lasers on enamel surfaces, the enhancement of acid resistance has immense advantages in adhesive dentistry. A number of studies have demonstrated that lasers significantly alter the permeability, crystallinity, and acid-solubility of the enamel surface, thus promoting its resistance to demineralization.[29-31] In addition, lasers promote fluoride uptake and prolong the fluoride-releasing time in an oral environment.[32] However, the influence of laser output energy parameters on enamel acid resistance has not been fully discussed. In this study, the effect of pulsed Nd:YAG laser on Ca^{2+} concentration dissolved from human enamel surfaces was significantly affected by the laser-parameter combinations. Moderate, not low or high, laser-parameter combinations were proven efficient in preventing enamel surface demineralization. Previous studies have indicated that the thermal effect of a laser is the main factor that prevents the demineralization of enamel.[33,34] A lased enamel surface is subjected to water loss from 80°C to 120°C, to decomposition of organic substance at 350°C, to initial loss of carbonate hydroxyapatite from 400°C to 660°C, and to enamel melting at more than 800°C to 1000°C with the formation of β- and α-TCP (tri-calcium-phosphate), which is potentially soluble in an acid environment.[30,33] The foregoing are the reasons why the moderate laser-parameter combinations were tested for efficiency in the acid resistance of enamel surface in this study. Low laser-parameter combinations were insufficient to induce melting and recrystallization, which are essential factors in the acid resistance of enamel surfaces. However, high laser-parameter combinations led to the formation of acid-soluble compounds, such as β- and α-TCP, which increased the demineralization of enamel surfaces.

Unlike laser ablation, phosphoric acid etching modifies enamel surface morphology by the selective removal of interprismatic mineral structure; the organic materials are less affected.[35] This regularly rough and extensively microfissured structure is very useful in increasing the retention of resin composites for adhesion but is very vulnerable to acid attack. Acid etching does not improve the crystalline structure and block the ion diffusion channel, but it removes the acid-protecting superficial enamel layer.[36] In this study, 35% phosphoric acid significantly increased the mean Ca^{2+} concentration dissolved from enamel surfaces and the demineralization depth. Data and SEM observations suggest that the pulsed Nd:YAG laser-treated enamel subsurface presents fewer soluble compounds and may be more acid-resistant during pH cycling.

Previous studies have reported that a minimum bond strength of 5.9 MPa to 7.9 MPa could result in successful clinical bonding.[37,38] The mean TBS value of 14.45 ± 1.67 MPa in the pulsed Nd:YAG laser group in this study is adequate to meet the clinical need for dental adhesion, although this value is significantly lower than that in the 35% phosphoric acid group. Pulsed Nd:YAG laser has numerous advantages when used as an enamel surface pretreatment method. Pulsed Nd:YAG laser can eliminate the need for the separate steps of water spraying and air drying, which are essential in the acid-etching technique. In contrast to acid-etching that increased the risk of caries attack, pulsed Nd:YAG laser enhanced the acid resistance of enamel surfaces in this study. In addition, the Nd:YAG laser has been reported to be more efficient than the acid-etching technique in reinforcing the decreased bond strength of resin composite to recently bleached enamel.[39,40] Therefore, the pulsed Nd:YAG laser can be used as an alternative enamel pretreatment method, especially in the caries-susceptible population or in individuals who recently underwent 35% hydrogen peroxide bleaching of the teeth. However, the proposed method needs further investigation in our future studies.

CONCLUSIONS

The effects of pulsed Nd:YAG laser on morphologic modification, TBS, and acid resistance of human enamel were influenced by laser output energy parameters. The moderate laser parameters significantly enhanced the TBS and acid resistance of human enamel, although high-energy level laser parameters were proven efficient in altering the enamel surface. The pulsed Nd:YAG laser was less efficient in increasing the TBS of resin to enamel compared with the acid-etching technique, but the proposed method significantly contributed to the acid resistance of human enamel. The combinations of pulsed Nd:YAG laser and 35% phosphoric acid showed the highest mean TBS value in this study.

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Conflict of Interest

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

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In Vitro Evaluation of Benzalkonium Chloride in the Preservation of Adhesive Interfaces

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Clinical Relevance

Benzalkonium chloride preserves resin-dentin bonds by reducing collagen solubilization. When incorporated into adhesive blends, benzalkonium chloride provides comparable bond stability to other inhibitors and application protocols without adding more steps to the bonding sequence.

SUMMARY

Inhibition of endogenous dentin matrix metalloproteinases (MMPs) by benzalkonium chloride (BAC) decreases collagen solubilization and may help improve resin-dentin bond stability. Objective: This study evaluated the resin-dentin bond stability of experimental adhesive blends containing BAC and the stability of dentin matrices by assessing the mass loss and collagen solubilization from dentin beams pretreated with BAC.

Materials and Methods: Twenty-five healthy molars were used for the bond strength evaluation of a two-step etch-and-rinse adhesive (Adper Single Bond Plus, SB) modified with BAC or not. The following groups were tested: 1) SB with no inhibitor (control); 2) topical 2.0% chlorhexidine + SB; 3) 1.0% BAC etchant + SB; 4) 0.5% BAC-SB; and 5) 1.0% BAC-SB. Microtensile bond strength (μTBS) and failure mode distribution under standard error of the mean were evaluated after 24 hours and six months of storage in artificial saliva (AS). A two-way analysis of variance and Tukey test with a significance level of $p<0.05$ was used for data analysis. In addition, 30 completely demineralized dentin beams from human molars were either dipped in deionized water (DW, control) or dipped in 0.5% and 1.0% BAC for 60 seconds, and then incubated in AS. Collagen solubilization was assessed by evaluating the dry mass loss and quantifying the amount of hydroxyproline (HYP) released from hydrolyzed specimens after four weeks of incubation.

Results: The control group demonstrated lower μTBS than some of the experimental groups containing BAC at 24 hours and six months ($p<0.05$). When BAC was incorporated into the adhesive blend in concentrations of 0.5% and

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1.0%, no reduction in dentin bond strength was observed after six months ($p<0.05$). Less mass loss and HYP release was seen for dentin matrices pretreated with BAC relative to the control pretreated with DW ($p<0.05$).

Conclusion: This *in vitro* study demonstrates that BAC contributes to the preservation of resin-dentin bonds by reducing collagen degradation.

INTRODUCTION

Resin-dentin bonds created by contemporary dentin adhesive systems still deteriorate over time.[1] The ionic and hydrophilic nature of current adhesives yields permeable hybrid layers that are susceptible to hydrolytic degradation.[1] Collagenolytic activity by host-derived matrix metalloproteinases (MMPs) has also been shown to contribute to the degradation of resin-dentin bonds.[2,3] MMPs, a group of calcium- and zinc-dependent endopeptidases that remain trapped in the dentin matrix during tooth development[4] are known to contribute to the collagenolytic activity in areas of suboptimally infiltrated collagen[3] once they have been activated by exposure to an acidic environment such as the one created by the application of acidic adhesive resins.[5]

Dentin treatment with protease inhibitors has been proposed to prolong the durability of the bonds. Chlorhexidine (CHX), a potent cationic antimicrobial agent[6] and nonspecific dentin MMP inhibitor has been extensively tested for its antiproteolytic effects with good results.[7] However, the large CHX molecule is water soluble and may leach out of the hybrid layer, which limits its long-term antiproteolytic benefits. Another group of antimicrobial agents, the quaternary ammonium compounds (QACs), also display positively charged molecules that bind to negatively charged phosphate and carboxylic groups in hydroxyapatite and collagen respectively. While CHX has two fixed charges, most QACs have only one positive charge.[8] Because QACs are also cationic molecules with antimicrobial properties, it has been speculated that they may display similar anti-MMP properties to CHX, while allowing easier stabilization of the compound within the hybrid layer because of their smaller size. A number of QACs have been recently investigated for their inhibitory properties in dentin MMPs with encouraging results.[8]

Benzalkonium chloride (BAC), a nitrogenous agent containing a quaternary ammonium group, has recently demonstrated effective dentin MMP inhibition.[9] For several years, a phosphoric acid etchant containing 1.0% wt BAC (Etch-37 w/BAC, Bisco Inc, Schaumburg, IL, USA) has been commercially available for its antibacterial properties and has shown no adverse effect on the immediate bond strengths.[10] The additional antiproteolytic benefits that may be derived from the use of BAC have gained attention only recently. Moreover, the issue of the most effective vehicle for the delivery of BAC has also been raised as rinsing the etchant may displace some of the BAC, perhaps limiting the amount that remains viable in the hybrid layer and, thus, its antiproteolytic benefits. A more effective delivery system may incorporate BAC into the primer and/or adhesive formulation. We speculate that incorporating the BAC into the adhesive may yield deeper infiltration of the agent into the demineralized collagen mesh, thus yielding greater antiproteolytic benefits while allowing a simplified clinical application technique.

Therefore, the purpose of this study was to investigate the efficacy of BAC as an inhibitor of dentin MMP activity by quantitatively assessing changes in bond degradation and collagen solubilization. Specific aims included the following: 1) evaluate the resin-dentin bonds created with a commercially available adhesive modified with BAC by means of microtensile bond strength (μTBS) at 24 hours and six months compared with the use of topical 2.0% CHX and BAC-modified phosophoric acid; 2) evaluate the collagen solubilization by assessing the dry mass loss and amount of hydroxyproline released from hydrolyzed specimens after four weeks of incubation dentin matrices pretreated with BAC. The null hypothesis was that dentin treatment with BAC would have no effect on the bond degradation, mass loss and hydroxyproline (HYP) release over time.

MATERIALS AND METHODS

Microtensile Bond Strength (μTBS)

Twenty-five recently extracted, noncarious human molars were used to obtain dentin substrate for bonding. The teeth were obtained under a protocol approved by the State University of New York's Institutional Review Board. A flat, transversely cut surface of superficial/middle dentin was obtained by means of a water-cooled slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA), and a smear layer was created with 600-grit silicon carbide abrasive paper (SiC paper, Buehler). The teeth were equally and randomly assigned to five study groups (Table 1) with five teeth in each group as follows: group 1, phosphoric acid treatment followed by

Table 1: *Study Groups, Composition and Application Protocol as per Manufacturer Recommendations*

Group	Code	Description	Adhesive (Composition / Application protocol)
1	Control	PA followed by conventional adhesive	**ADPER SINGLE BOND PLUS (SB**/ 3M ESPE, Lot# 9XT, Saint Paul, MN, USA) Composition (% by wt): Ethyl alcohol (25-35%); silane treated silica nanofiller (10-20%); bisphenol A diglycidyl ether dimethacrylate (BisGMA) (10-20%); 2-Hydroxyethyl methacrylate (HEMA) (5-15%); glycerol 1,3-dimethacrylate (5-10%); copolymer of acrylic and itaconic acids (5-10%); water (<5%); diurethane dimethacrylate (1-5%) Application protocol: • Etch (15 sec). Rinse (10 sec) • Blot dry. Leave surface slightly moist • Apply adhesive (2-3 coats). Scrub (15 sec) • Gently air thin to evaporate solvents (5 sec) • Light cure (10 sec)
2	2.0%CHX	PA followed by 2.0% CHX & conventional adhesive	
3	BAC-PA	1.0% BAC-PA followed by conventional adhesive	
4	0.5% BAC	PA followed by 0.5% BAC-adhesive	
5	1.0% BAC	PA followed by 1.0% BAC-adhesive	

Abbreviations: BAC, benzalkonium chloride; CHX, chlorhexidine; PA, phosphoric acid; SB, adper single bond plus. Conventional adhesive denotes adhesive that was not modified with inhibitor.

conventional adhesive (control); group 2, phosphoric acid treatment followed by topical 2% CHX and conventional adhesive (2.0% CHX); group 3, 1.0% BAC-modified phosphoric acid treatment followed by conventional adhesive (BAC-PA); group 4, phosphoric acid treatment followed by 0.5% BAC-containing adhesive (0.5% BAC); group 5, phosphoric acid treatment followed by 1.0% BAC- containing adhesive (1.0% BAC).

A commercially available aqueous solution of 2.0% CHX digluconate (Consepsis, Ultradent, South Jordan, UT, USA) was used for rewetting dentin in group 2. All groups were treated with 35% phosphoric acid (Ultra-Etch, Ultradent) with exception of group 3, which was etched with phosphoric acid containing 1.0% wt BAC (Etch-37 w/BAC, Lot 1100004919, Bisco). The adhesive used in this study was a two-step etch-and-rinse system (Adper Single Bond Plus, Lot 9XT, 3M ESPE, St Paul, MN, USA). Its composition and application protocol as described by the manufacturer are summarized in Table 1. Benzalkonium chloride was admixed into the primer/adhesive blend in concentrations of 0.5% and 1.0% (wt/vol) for treatment of groups 4 and 5, respectively. The adhesive was applied to the moist dentin surfaces according to the wet-bonding technique and polymerized according to manufacturer's instructions with a light-emitting diode light-curing unit (Bluephase 16i, Ivoclar-Vivadent, Amherst, NY, USA) with a power density of 1,600 mW/cm^2. Composite build-ups were fabricated with resin composite (Filtek Z100, Lot N372074, 3M ESPE) in shade A2 according to a standardized protocol by applying two increments no greater than 2 mm, each of which was polymerized for 40 seconds.

The restored teeth were stored in distilled water and placed in an incubator at 37°C for 24 hours to ensure adequate polymerization. After 24 hours, all teeth were sectioned, and dentin beams with a cross-sectional area of 0.9 ± 0.1 mm^2 were obtained according to the nontrimming technique.[11] The beams were divided into two equal groups for microtensile bond strength evaluation at 24 hours and after six months of storage at 37°C in an artificial saliva solution containing 0.02% sodium azide. The storage media was replaced with fresh solution once a month to ensure that the preservatives were fresh.

Beams were stressed to failure with a universal testing machine at a crosshead speed of 1 mm/min (Bisco). Failure modes were analyzed by observation by a single examiner (CS) with a stereomicroscope (Nikon SMZ-U, Melville, NY, USA) at a magnification of 50×. The fractured surfaces were classified as follows: 1) cohesive in dentin, 2) adhesive, 3) cohesive in composite, and 4) mixed failure, which was defined as the combination of different failure modes resulting from failure across the interfacial layers.

Because the data were normally distributed (Kolmogorov-Smirnov test), a two-way analysis of variance (ANOVA) was used to analyze the effect of the variables "treatment group" and "storage time" on μTBS. A post-hoc Tukey test was used for pairwise multiple comparisons between group means. A significance level of $p<0.05$ was used for all tests. All statistical analyses were performed with Statistical Package for Social Sciences (SPSS) version 16.0 (SPSS Inc, Chicago, IL, USA).

Collagen Solubilization and Mass Loss

Beam Demineralization and Incubation—Fifteen healthy human molars were obtained under a protocol approved by the State University of New York's Institutional Review Board and stored in a 0.9% NaCl solution containing 0.02% sodium azide at 4°C for no more than three months after extraction. The crowns were separated from the roots at the cementoenamel junction with a slow-speed diamond saw (Isomet, Buehler) and the crowns debrided from enamel, cementum, and pulpal tissue by means of a diamond bur in high-speed handpiece under air-water spray. Midcoronal occusal dentin was exposed by removing the enamel and superficial dentin using a slow-speed diamond saw under water cooling. One dentin disk (1 mm thick) was obtained from each tooth. A total of 30 dentin beams of standardized dimensions ($2.0 \times 1.0 \times 6.0$ mm) were obtained from the dentin disks.

All dentin beams were completely demineralized by immersion in 10 wt% liquid phosphoric acid (pH 1.0, Sigma Aldrich, St Louis, MO, USA) for 18 hours at 25°C under constant stirring, and then rinsed in deionized water (DW) for two hours at 4°C. To determine initial dry mass, the beams were desiccated over anhydrous calcium sulfate for 48 hours and weighed to a constant dry weight in an analytical balance (AG204, Mettler Toledo, LLC, Columbus, OH, USA). After rehydrating the beams in DW for one hour, they were equally and randomly divided in three groups (n=10) and treated with either 0.5% BAC, 1.0% BAC, or DW (control) for 60 seconds, blotted to remove excess, and then incubated in 1 mL of artificial saliva (AS, composed of 12.9 mM KCl, 1.9 mM KSCN, 2.4 mM $Na_2SO_4 \cdot 10\ H_2O$, 3.3 mM NH_4Cl, 1.5 mM $CaCl_2 \cdot 2H_2O$, 7.5 mM $NaHCO_3$, 0.02 mM $ZnCl_2$, and 5 mM HEPES buffer, pH 7.4). The beams were separately incubated in individual screw-capped tubes in a water shaker bath at 37°C. After four weeks, the AS media was analyzed for collagen solubilization by means of hydroxyproline (HYP) release, and the dry mass loss of the beams was determined. To determine dry mass, the beams were dried and weighed as described earlier.

Collagen Solubilization—Quantification of the amount of HYP in the hydrolysates has been proposed as an indirect assessment of collagen degradation as HYP is a relatively unique aminoacid to type I collagen.[12] Its presence in the storage media indicates that collagen peptides from the dentin beams were solubilized over time.[9] An aliquot of 200 μL of the AS solution was mixed with an equal volume of 12 N HCl, and its contents were hydrolyzed to aminoacids in an oil bath at 120°C for 18 hours. The content of each tube was allowed to evaporate to dryness for one week in a large desiccator under a vacuum containing sodium hydroxide and anhydrous calcium sulfate. Hydroxyproline standards were prepared from a 100 μg/mL HYP stock solution in 50% isopropanol. Concentrations of HYP in the standard solution were 0, 5, 10, 15, 20, 25, 30, 35, and 40 μg/mL. Aliquots of 200 μL of each of these standards were hydrolyzed and allowed to evaporate to dryness the same as unknowns. After one week, all residues of dry hydrolysates were evaluated for collagenolytic activity by quantifying the HYP content in the hydrolyzed specimens as per the colorimetric assay by Jamall and others.[13] Briefly, residues of dried hydrolysates, both unknowns and HYP standards, were resolubilized with 1.2 mL of 50% isopropanol and 0.2 mL of chloramine-T solution (1.1 mL of chloramine-T stock solution and 18.9 mL of acetate citrate buffer, pH 6.0). After 10 minutes, 1.0 mL of Ehrlich stock solution (10 g of 4-dimethylaminobenzaldehyde and 11 mL of 60% perchloric acid) in 100% isopropanol was added to give a final volume of 2.4 mL. The specimens were incubated at 50°C for 90 minutes to develop the chromophore. The absorbance values of all specimens' hydrolysates were measured in spectrophotometer (DU 800, Beckman Coulter Inc, Brea, California, USA) at 558 nm against a blank.

A standard curve was created by plotting the absorbance values of the HYP standards against the concentration of HYP in these standards ($R^2 = 0.99$). A regression equation generated by the standard curve was used to calculate the amount of HYP in the unknown samples based on their absorbance values. The resulting amount of HYP, expressed in micrograms, was divided by the original dry mass of each beam yielding the amount of HYP released per milligram of dentin (μg HYP/mg dentin). The data were normally distributed (Kolmogorov-Smirnov test). A one-way ANOVA was used to analyze the "treatment group" effect in both mass loss and HYP release. Post-hoc Student-Newman-Keuls was used for pairwise multiple comparisons between group means. A significance level of $p<0.05$ was used for all tests.

RESULTS

Microtensile Bond Strength (μTBS)

Two-way ANOVA demonstrated a significant effect of the main variables "treatment group" ($p<0.001$) and "time" ($p=0.007$), as well as their interactions

Table 2: *Mean Microtensile Bond Strength (μTBS) Results and Failure Mode Distribution for the Five Study Groups at 24 h and Six mo of Storage (n=10)*[a]

	Control (Group 1)	2.0%CHX (Group 2)	BAC-PA (Group 3)	0.5%BAC (Group 4)	1.0%BAC (Group 5)
24 h μTBS (MPa ± SD)	34.3 ± 7.8 [A,c]	38.3 ± 10.3 [A,b,c]	43.0 ± 11.8 [A,b]	36.4 ± 8.4 [A,b,c]	51.4 ± 7.9 [A,a]
Failure mode A/D/R/M	3 / 0 / 3 / 4	2 / 2 / 1 / 5	2 / 0 / 2 / 6	1 / 2 / 1 / 6	0 / 0 / 2 / 8
6 mo μTBS (MPa ± SD)	27.4 ± 6.2 [B,c]	34.3 ± 5.2 [A,b]	35.1 ± 6.5 [B,b]	36.6 ± 6.2 [A,b]	53.9 ± 6.9 [A,a]
Failure mode A/D/R/M	5 / 1 / 3 / 1	1 / 1/ 3 / 5	2 / 1 / 1 / 6	0 / 1 / 1 / 8	0 / 1 / 2 / 7

Abbreviations: A, adhesive; D, cohesive in dentin; R, cohesive in resin; M, mixed.
[a] *Same superscript letter indicates no significant differences between groups per the results of pairwise multiple comparisons Tukey test (p<0.05). Upper case denotes differences in μTBS values between 24 hours and six months for each of the individual groups (vertical). Lowercase letter denotes differences among treatment groups for each testing time (horizontal).*

(p=0.023) on the bond strength. Table 2 summarizes the mean μTBS values and failure mode distribution for all study groups at 24 hours and six months of storage. At 24 hours, all experimental groups demonstrated higher bond strength than the control group, but only BAC-containing etchant and 1.0% BAC-containing adhesive were significantly higher than the control (p=0.01 and p<0.001 respectively). When bond strength was evaluated at six months, significant differences were observed between the control and all the experimental groups, and the control group showed significantly lower bond strength than all experimental groups (p<0.05). The group treated with 1.0% BAC-containing adhesive demonstrated significantly higher bond strength than all other groups, and the groups treated with 2.0% CHX, BAC-PA, and 0.5% BAC were not significantly different from each other. With exception of the groups treated with BAC-containing adhesive (groups 4 and 5), all groups demonstrated a decrease in bond strength after six months of storage. This decrease was significant for the control group and the group treated with BAC-containing etchant (p<0.05) but not for the group treated with 2.0% CHX. The most prevalent failure modes observed were adhesive and mixed.

Collagen Solubilization and Mass Loss

When the MMP inhibitory properties of BAC were evaluated by quantifying the amount of HYP present in the AS incubation media after four weeks, a significant effect of the "treatment group" (p<0.001) was demonstrated. After four weeks of incubation, the storage media derived from the beams pretreated with 0.5% BAC and 1.0% BAC demonstrated significant less release of HYP than the media derived from the control beams pretreated with DW (p=0.003 and p<0.001, respectively). The amount of HYP in the media from beams pretreated with either 0.5% BAC or 1.0% BAC was not significantly different from each other (Figure 1).

Evaluating BAC MMP inhibitory properties by assessing the dry mass loss from dentin beams after four weeks of incubation also yielded a significant effect of the treatment group (p=0.018). After four weeks, the loss of dry mass was 15.7%, 11.8%, and 6.8% for beams pretreated with DW, 0.5% BAC, and 1.0% BAC, respectively. Only the beams pretreated with 1.0% BAC yielded significantly less mass loss than the control group (p=0.014). No significant differences were seen between beams pretreated with DW and 0.5% BAC, or 0.5% BAC and 1.0% BAC (Figure 2).

DISCUSSION

The present study evaluated changes in bond degradation and collagen solubilization from dentin matrices treated with BAC as an indirect assessment of its efficacy as an inhibitor of dentin MMP activity.

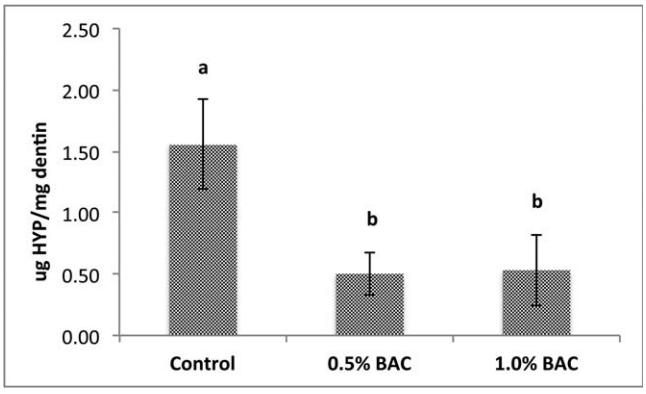

Figure 1. *HYP release from dentin collagen matrices after four weeks of incubation in AS (n=10). Control beams were pretreated with DW for 60 seconds and then incubated in AS. Experimental beams were pretreated with BAC in either 0.5% or 1.0% for 60 seconds. and then incubated in AS. Bars represent the mean values; brackets indicate the standard deviation values. Groups identified by different letters are significantly different (Student-Newman Keuls;* p < *0.05).*

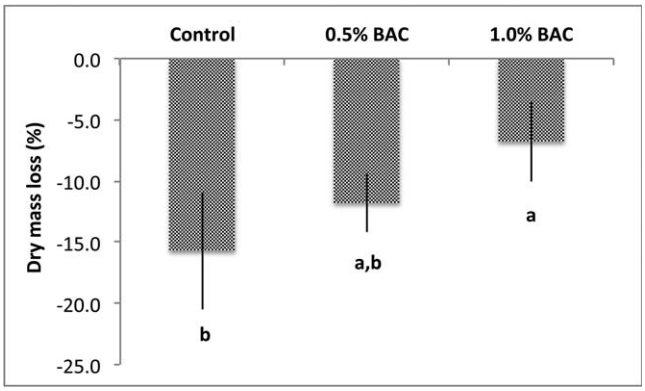

Figure 2. *Percent total dry mass loss of completely demineralized human dentin beams after four weeks of incubation in AS (n=10). Control beams were pretreated with DW for 60 seconds and then incubated in AS. Experimental beams were pretreated with BAC in either 0.5% or 1.0% for 60 seconds and then incubated in AS. Bars represent the mean values; brackets indicate the standard deviation values. Groups identified by different letters are significantly different (Student-Newman-Keuls;* $p < 0.05$*).*

The null hypothesis was rejected as BAC demonstrated both decreased bond degradation after six months and less collagen solubilization after four weeks of incubation. Our results confirm the previously demonstrated effectiveness of BAC as an MMP inhibitor.[9] Incorporatimg BAC, in concentrations of 0.5% and 1.0%, into a commercially available adhesive blend, Single Bond, yielded no bond degradation after six months of storage in AS. A decrease in bond strength after six months was demonstrated for all other groups, which was significant for the control group with no inhibitor and the group treated with BAC-containing etchant, but not for the group treated with 2.0% CHX. Although our results showed stability of the resin-dentin bonds after six months with the use of a BAC-modified adhesive, it is possible that this positive effect may have been the result of an incubation time of only six months; longer incubation periods may yield different results after BAC is allowed to leach out of the hybrid layer. Nonpolymerizable MMP inhibitors such as CHX and BAC are known to bind to dentin electrostatically.[14] Noncovalently bound molecules may leach out of the hybrid layer, compromising its long-term antiproteolytic benefits, and consequently only delaying but not preventing the degradation of adhesive interfaces.[15] Because of the weak electrostatic bonds between BAC and dentin, longer incubation periods may be necessary to evaluate its long-term antiproteolytic benefits. Our results confirm the findings of previous studies, which have shown that BAC used in combination with etch-and-rinse adhesives had no effect on the immediate bond strength.[16-18] These findings suggest that the antimicrobial can be safely combined with the resin monomers present in Single Bond without a compromise to its initial bond strength. Moreover, increased bond strength values were seen for BAC-treated groups relative to the control; the effect was shown to be dose-dependent, with mean bond strength values of 36.4 and 51.4 MPa for 0.5% and 1.0% BAC, respectively, at 24 hours, and 36.6 and 53.9 MPa for 0.5% and 1.0% BAC, respectively, at six months.

BAC, in concentrations of 0.5% and 1.0%, was also evaluated by colorimetric assay to determine collagen solubilization. In this model, demineralized dentin beams were pretreated with 0.5% or 10% BAC to allow the BAC to diffuse into the water-filled spaces between the collagen fibrils and within the dentinal tubules for 60 seconds, and then were incubated in BAC-free AS media. Significantly less release of HYP was seen when dentin beams were pretreated with 0.5% and 1.0% BAC. This finding confirms the results of a recent study that showed that BAC in concentrations of 0.5% or greater can inhibit MMPs.[9] Evaluation of the dry mass loss of the dentin beams after pretreatment with DW, 0.5% BAC, or 1.0% BAC also revealed differences; beams pretreated with 1.0% BAC showed significantly less mass loss than the beams pretreated with DW (control). There were no detectable differences between 0.5% and 1.0% BAC for HYP release or mass loss. Less solubilization of collagen is expected when BAC binds to collagen and MMPs bind to collagen as these molecular interactions are known to dissociate the enzyme's tertiary structure.[19] Nevertheless, a recent study investigating the binding ability of BAC to demineralized dentin found that not all the BAC binds to collagen. The BAC that is not bound but remains trapped in the water of the interfibrilar spaces can be easily removed by water rinsing as evidenced in that study by the loss of 50% of the BAC after water rinse.[9] Clinically, this indicates that phosphoric acid etchant may not be the most effective vehicle for the delivery of BAC. A significant 18.2% decrease in bond strength after six months when BAC was delivered into the etchant confirms this notion.

Protease inhibitors are commonly delivered either topically before the application of the adhesive or in conjunction with the phosphoric acid etchant. This, aside from introducing an additional step to the bonding sequence, may compromise the amount of agent that remains viable in the hybrid layer after rinsing the etchant. Attempts to incorporate

these agents into primers/adhesive blends have shown encouraging findings, and our results support this concept. Reduction in bond degradation has been observed when CHX was incorporated into the primer of two-step self-etch adhesives.[20,21] To date, no studies have evaluated the stability of adhesive interfaces after treatment with adhesive blends modified with BAC, and thus a direct comparison of our results with those from other studies is not possible. We can speculate, based on our results, that treatment of the acid-etched dentin with a therapeutic primer/adhesive blend containing BAC may allow greater diffusion of the agent into the water-filled spaces between the collagen fibers and dentinal tubules. We can further speculate that incorporating BAC into the primer/adhesive blend may prolong its availability within the hybrid layer with the assumption that the resin matrix may act as a reservoir for its slow release over time. BAC is known to be soluble in both ethanol and acetone, and its activity not greatly affected by pH, which may suggest that its antiproteolytic benefits may be safely extrapolated to adhesives of different composition. However, no assumptions can be made, and studies should be conducted to understand specific interactions between BAC and adhesives of different acidity, hydrophilicity, and monomeric composition as well as to determine its effect on the properties of the polymerized resin matrix.

Efforts continue toward gaining a better understanding of the many aspects that play a role in the long-term success of adhesive restorations. Other approaches also known to reduce collagen degradation and preserve dentin-resin interfaces are being investigated. Increasing the extent of collagen cross-linking before adhesive application,[22] and use of polymerizable acrylate or methacrylate groups, which can be stabilized within the hybrid layer, are examples of these. These interventions, however, still fail to address the critical issue of the water that remains entrapped within the collagen intrafibrilar compartments, weakening the interface and providing the functional medium for MMP activity. In this regard, molecular immobilization of MMPs through remineralization of water-rich collagen fibrils within the hybrid layer may represent a more permanent strategy to prevent organic matrix degradation.[23] However, despite the many aspects involved in the degradation of adhesive interfaces, our study clearly shows the importance of dentin MMP inhibition in the preservation of hybrid layers over time.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following can be concluded:

- An improved stability of the resin-dentin bonds after six months was demonstrated with the use of BAC-containing adhesives relative to the control group with no inhibitor.
- Beam treatment with 0.5% BAC and 1.0% BAC for 60 seconds yielded significantly less HYP release from dentin matrices, as determined by colorimetric assay indicating that BAC prevents collagen solubilization. This effect was shown to be dose-dependent.
- Beam treatment with 0.5% BAC and 1.0% BAC for 60 seconds yielded significantly less dry mass loss after four weeks, indicating that BAC prevents collagen solubilization.

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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Bonding of Y-TZP to Dentin: Effects of Y-TZP Surface Conditioning, Resin Cement Type, and Aging

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Clinical Relevance

The application of the low-fusing glaze porcelain followed by hydrofluoric acid etching and silanization and tribochemical silicatization generates strong bonds between resin cement and zirconia. Panavia generates stronger bonds than does Clearfil.

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SUMMARY

Purpose: **To evaluate the effects of two surface treatments, aging, and two resin cements on shear bond strength between dentin and yttrium-stabilized tetragonal zirconia polycrystal ceramic (Y-TZP).**

Materials and Methods: **Eighty human molars were embedded in acrylic resin and sectioned 3 mm below the occlusal plane. These teeth and 80 cylindrical Y-TZP specimens (height, 4 mm; diameter, 3.4 mm) were divided into eight groups (n=10) using the following factors: Y-TZP surface treatment (Vi: low-fusing porcelain [vitrification] + hydrofluoric acid etching + silanization or Si: tribochemical silicatization); cementation strategies (PF: Panavia or CC: Clearfil); and storage (nonaging or aging). Bonding surfaces of 40 Y-TZP specimens received Vi treatment, and the rest received Si treatment. Half of the ceramic-tooth assemblies were cemented with Panavia, the rest with Clearfil. Shear tests were executed using 0.4-mm–thick wire at 0.5 mm/min. Data were analyzed by three-way analysis of variance and Tukey test (α=0.05). Fractures were analyzed.**

Results: **Y-TZP surface treatments did not affect bond strength (p=0.762, Vi = Si), while resin cements (p<0.001, Panavia > Clearfil) and aging (p=0.006, nonaging > aging) showed a significant effect. Most failures were in adhesive at dentin-cement interfaces; no failure occurred between zirconia and cement.**

Conclusion: **When Y-TZP ceramic is bonded to dentin, the weakest interface is that between dentin and resin cement. The resin cement/Y-TZP interface was less susceptible to failures, owing to Y-TZP surface treatments.**

INTRODUCTION

Zirconia-based dental ceramics have better mechanical properties than do other commercially available ceramic materials.[1] However, their bond strength to resin cement has been reported[2] to be weak because zirconia-based ceramics have a large crystalline phase, rendering conventional hydrofluoric acid etching treatment impossible. Therefore, in the last few years, many researchers[2-6] have studied alternative methods of improving the adhesion between zirconia and resin cements.

Among the possible treatments being investigated to improve the adhesion, air-particle abrasion protocols have attracted much interest because of their simplicity. These methods are called tribochemical silicatization and involve air abrasion of the ceramic surface with alumina or with silica-modified alumina particles (30 μm), followed by the application of a silane coupling agent.[1,7-9] The alumina- or silica-modified alumina particles produce microroughness on the zirconia surface, while the silane coupling agent acts as a link between the sandblasted surface and the cement matrix.[8-13]

In addition to the air-abrasion protocols, resin cements containing phosphate ester monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP) have been shown to form strong bonds with zirconia. These cements react with oxides, creating a good interaction with the zirconia surface.[8,10-13] However, a study[10] has showed that the use of cements with MDP alone is not sufficient for creating a stable union between the resin cement and zirconia. Therefore, air-abrasion protocols[14] are also required to be a part of the bonding technique. On the other hand, airborne particle abrasion procedures can structurally damage the zirconia surface, decreasing the mechanical properties of this ceramic[15]; hence, the use of these procedures remains controversial.

As a new alternative to the surface treatment of zirconia, some researchers[4,16-18] have evaluated the application of a low-fusing porcelain material (glaze) on the yttrium-doped tetragonal zirconia (Y-TZP) intaglio surface. This technique aims to create a surface that can be etched by hydrofluoric acid, similar to feldspathic ceramics. Although this is a promising technique, it has been introduced very recently, and data on resin bond strength and bond durability as a result of using this method are scarce.

In fixed dental prostheses (FDPs), in which mechanical retention plays an important role, the mechanism responsible for adhesion with dentin appears to be less important.[19] On the other hand, in bonded FDPs for which mechanical retention is not the primary retention mechanism the bond strength between the resin cement and dentin is essential to the success of the treatment.[20,21] Hence, evaluation of the adhesion between Y-TZP, resin cements, and dentin is important.[5,22]

Chai and others[5] reported that studies that evaluate simply the bond strength between resin cements and restorative materials are limited in scope from a clinical perspective, as FDP restorations are normally cemented to an enamel/dentin substrate. When zirconia specimens are cemented onto a dental substrate, it is possible to assess both interfaces, such as the cement-ceramic and cement-dentin interfaces, which leads to better evaluation of adhesion, from the clinical point of view.

Therefore, the aim of the present study is to evaluate two cementation strategies (involving the use of the resin cements Panavia F and Clearfil SA Cement, respectively), two Y-TZP surface treatment techniques (involving the application of a low-fusing porcelain and silicatization, respectively), and the effect of thermocycling aging on the shear bond strength between zirconia and dentin. The following hypotheses were tested: 1) the application of a low-fusing glaze porcelain application and silanization as Y-TZP surface treatments will not influence the bond strength; 2) the cementation strategies will not influence the bond strength; and 3) aging will decrease the bond strength.

MATERIALS AND METHODS

The product names, manufacturers, chemical compositions, and batch numbers of the materials used in the study are listed in Table 1.

Selection of Teeth

Eighty human molars were selected, cleaned with periodontal curettes, and stored in distilled water

Table 1: *Material, Manufacturer, Chemical Composition, and Batch Number of the Products Used in the Study*

Material	Manufacturer	Chemical Composition	Batch No.
Rocatec Plus	3M ESPE, Seefeld, Germany	Silicatized aluminum oxide particles (30 μm)	1036301855
Condac 37	FGM, Joinvile, SC, Brazil	37% Phosphoric acid	140111
Porcelain Conditioner	Dentstply, Petrópolis, RJ, Brazil	10% Hydrofluoric acid	229431B
Vita Akzent Glaze Spray	Vita Zahnfabrick, Germany	Not available	21790
Clearfill SA Cement	Kuraray Medical Inc, Japan	Bis-GMA, TEGDMA, MDP, hydrophobic aromatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, di-camphorquinone, benzoyl peroxide, initiator, hydrophobic aliphatic dimethacrylate, silanated, surface-treated sodium fluoride, accelerators, pigments, 45vol% is inorganic fillers	023AAB
Monobond S	Ivoclar Vivadent, Schaan, Liechtenstein	Alcohol solution of silane metacrylate	532888
Panavia F–Adhesive Primer A	Kuraray Medical Inc, Japan	2-Hydroxyethyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, *N*-methacryloyl-5-aminosalicylic acid, water, accelerators	00282A
Panavia F–Adhesive Primer B	Kuraray Medical Inc, Japan	*N*-methacryloyl-5-aminosalicylic acid, water, catalysts, accelerators	00157A
Panavia F–Cement Paste A	Kuraray Medical Inc, Japan	10-Methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, DL-camphorquinone, catalysts, initiators, others	00251B
Panavia F–Cement Paste B	Kuraray Medical Inc, Japan	Sodium fluoride, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments, others	00029A
Vita In Ceram YZ	Vita Zahnfabrick, Germany	91% Zirconium oxide (ZrO_2), 5% yttrium oxide (Y_2O_3), 3% hafnium oxide (HfO_2), small amounts (<1%) of aluminum oxide (Al_2O_3) and silicon oxide (SiO_2)	28070

Abbreviations: Bis-GMA: bisphenol A-glycidyl methacrylate;TEGDMA: triethyleneglycol-dimethacrylate; MDP: methacryloyloxydecyl dihydrogen phosphate.

(4°C) until needed. With the assistance of a cylindrical metallic mold (diameter, 20 mm; height, 15 mm), each tooth was embedded 2 mm apical to the cemento-enamel junction with a self-curing acrylic resin (JET, Artigos Odontológicos Classico Ltda, Sao Paulo, Brazil).

Then, each tooth was sectioned 3 mm below the occlusal surface using a low-speed diamond cutting saw (Labcut 1010, Extec, Enfield, CT, USA) with extensive water cooling in order to expose the superficial coronal dentin surface. These surfaces were then wet-ground with 600-grit silicon paper for 60 seconds using a polishing machine (PSK-2V, Skill TEC, Sao Paulo, Brazil).

Before the cementation procedures, the teeth were numbered from 1 to 80, and eight random sequences consisting of 10 numbers each were generated using the computer program Random Allocator (developed by M Saghaei, Dept of Anesthesia, Isfahan University of Medical Sciences, Isfahan, Iran). This procedure was performed to homogenize the groups and randomize the allocation of the specimens to the eight groups.[23] Next, each of the eight groups, which comprised 10 samples each, was assigned to one of the following categories: Y-TZP surface treatment (Vi: application of the low-fusing glaze porcelain [vitrification] + hydrofluoric acid etching + silanization or Si: tribochemical silicatization), cementation strategies (PF: Panavia or CC: Clearfil), and storage condition (nonaging or aging): Si + PF; Si + PF + aging; Si + CC; Si + CC + aging; Vi + PF; Vi + PF + aging; Vi + CC; and Vi + CC + aging.

Preparation of the Y-TZP Specimens

First, blocks of Vita In Ceram YZ 2000 (Vita Zahnfabrik, Bad Säckingen, Germany) were sectioned using a diamond saw (Labcut 1010, Extec) to produce smaller cubes ($5\times15\times20$ mm^3). A drill-type trephine was used to perforate these cubes perpendicular to the surface (with the aid of a preparation device) to produce presintered zirconia cylinders (diameter, 4.5 mm; height, 5 mm) that were then

sintered, as recommended by the manufacturer, in an oven (Vita Zyrcomat, Vita Zahnfabrik). The final dimensions of the ceramic cylinders were 3.4 mm in diameter and 4 mm in height.

The surface of each cylinder that had to undergo cementation was polished with 800-, 1000-, and 1200-grit silicon carbide paper, under water cooling, for 60 seconds each using a polishing machine (PSK-2V, Skill TEC). After polishing, the cylinders were cleaned ultrasonically for five minutes in isopropyl alcohol.

Conditioning of the Y-TZP Surfaces

For 50% of the zirconia cylinders (N=40), a low-fusing porcelain glaze (Vita Akzent Glaze Spray, Vita Zahnfabrik) was applied for one to two seconds on the cementation surface at a 10-mm distance. The conditioned specimen was then sintered (VACUMAT 40T, Vita Zahnfabrik) according to the manufacturer's instructions. Then the glaze-coated surfaces were treated with 10% hydrofluoric acid gel (Porcelain Conditioner, Dentsply, Petropolis, RJ, Brazil) for 60 seconds, washed for 15 seconds, dried, and silanized with a methacryloxypropyltrimethoxysilane (MPS)–based silane coupling (Monobond S, Ivoclar Vivadent, Schaan, Liechtenstein). The silanized samples were kept aside for 60 seconds to let the solvent evaporate.

The remaining zirconia cylinders (N=40) were treated using the tribochemical silicatization method. First, the surfaces of the cylinders were air-abraded using 30 μm silica-coated alumina particles (Rocatec Soft, 3M ESPE, Seefeld, Germany) from a distance of 10 mm and with a pressure of 2.8 bar. Subsequently, the MPS-based silane coupling agent (Monobond S, Ivoclar Vivadent) was applied in the manner described above.

Cementation Procedures

The cementation surface of the specimens was defined by an adhesive tape (Scotch, 3M, Ribeirão Preto, Brazil) with a 3.4-mm–diameter hole, aiming to standardize the cementation area and prevent the overflow of the resin cement.

For the PF samples, the dentinal surface treatment was performed as follows. Equal amounts of Primers A and B were mixed, and this mixture was applied on the dentinal surface with a microbrush. This was followed by spraying the surface gently with air and letting it stand for one minute to allow the reaction to take place. For CC samples, dentinal surface treatment was not required, as the Clearfil SA Cement is self-adhesive.

The two resin cements were manipulated as recommended by the manufacturer and were applied on the conditioned surfaces of the zirconia cylinders. The cylinders were placed on top of the area bounded by the adhesive tape, and a load of 750g was applied on the cylinders for 60 seconds. Any excess cement was removed, and all of the surfaces (vestibular, mesial, distal, and lingual) were photoactivated using an LED (1200 mW/cm^2) (Radii Cal, SDI, Australia) for 20 seconds.

Storage Conditions

All the specimens were stored for 24 hours in distilled water at 37°C. Half of the specimens were submitted to the shear bond strength test and the other half were aged before testing using a thermocycling protocol that involved 5000 cycles of alternate immersion in baths at 5°C and 55°C for 30 seconds each with intervals of two seconds between the immersions.

Shear Bond Strength Test

The test was conducted using a universal testing machine (EMIC DL 1000, Emic, São José dos Pinhais, PR, Brazil) with a crosshead speed of 0.5 mm/min and a steel wire with a thickness of 0.4 mm. The test cylinder was aligned with the load cell, and the wire loop was positioned as close as possible to the ceramic/dentin interface and parallel to the direction of the load cell (50 Kgf). The steel wire was then pulled using the universal machine in order to perform the shear bond strength test.

The bond strength was calculated using the formula $R = F/A$, where R is the bond strength (MPa), F is the load required for rupture of the specimen (N), and A is the bonded cross-sectional area of the specimen (mm^2). The bonded cross-sectional area was calculated using the formula for the area of a circle, which is given by $A = \pi \times r^2$, where $\pi = 3.14$ and $r = 1.7$ mm (half of the diameter of the cylinder). Using this formula, the bonded cross-sectional area was found to be 9.07 mm^2.

Fracture Analysis

The surfaces of the fractured specimens were examined using an optical stereomicroscope (Discovery V20; Carl Zeiss, Gottingen, Germany) and a scanning electron microscope (SEM; Inspect S50, FEI, Oregon, USA). For the SEM observations, all of the specimens were gold-sputtered under vacuum.

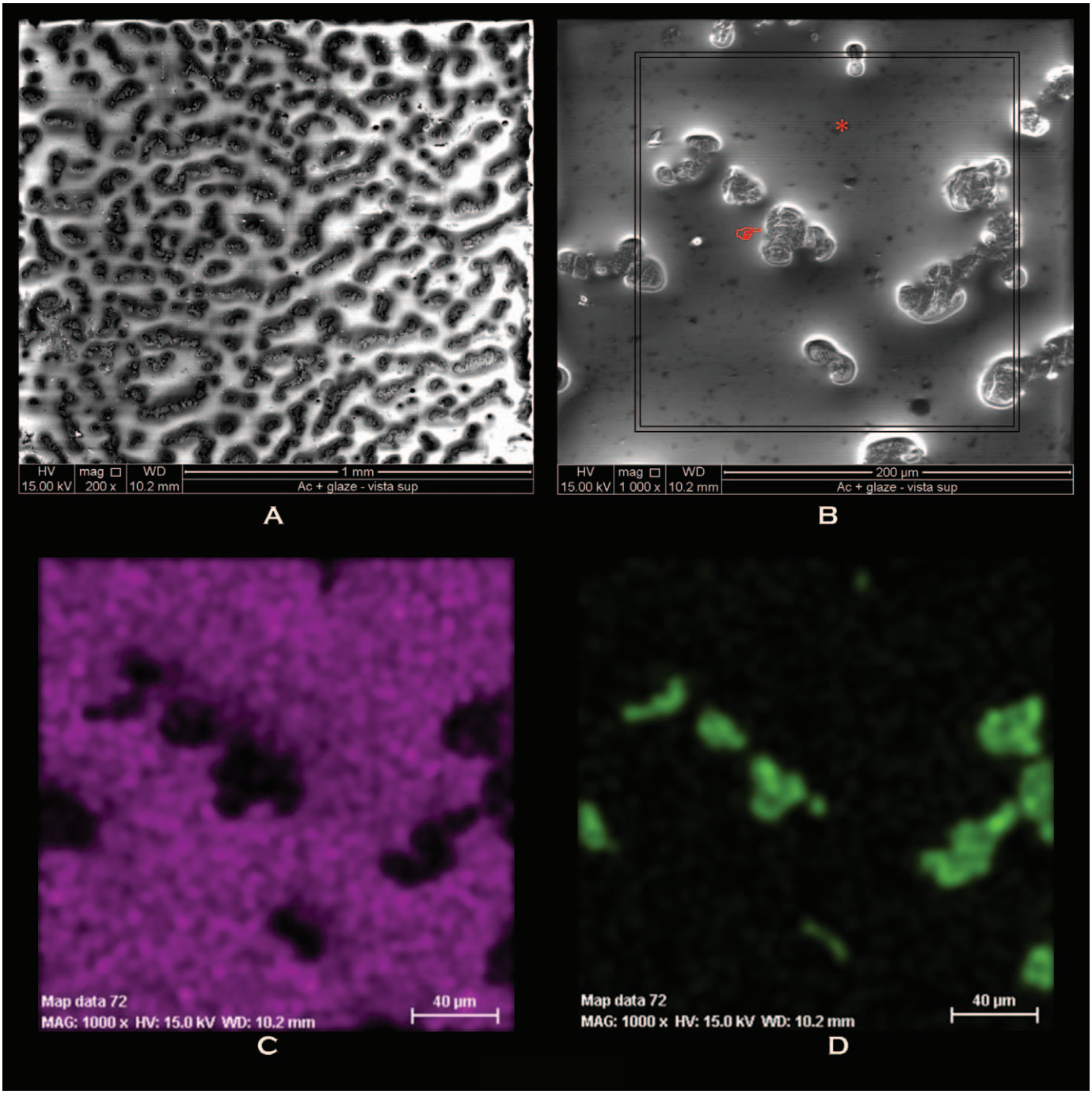

Figure 1. *(A) Low-fusing porcelain glaze film distribution on zirconia surface at 200×; (B) Glaze distribution at 1000×. The red pointer indicates the area of the zirconia surface that was not covered by glaze film, while the asterisk indicates the silicon surface created by vitrification (the square is the area of Energy Dispesive Scanning [EDS] analysis); (C) Representative* EDS element *maps acquired (the purple area represents the silicon on the zirconia surface); (D) EDS element maps acquired (the green area represents the zirconium surface that was not covered by silicon).*

Fracture analysis was performed for several specimens in order to identify the fracture origin and mode of fracture. Stereomicroscope examinations were performed using various lighting configurations to identify the fracture pattern. The identified fractures were classified on the basis of the following scheme: score A = a detachment of the resin cement from the dentin; score B = a detachment of the ceramic from the resin cement; score C = a fracture of ceramic without an adhesive failure; score D = a

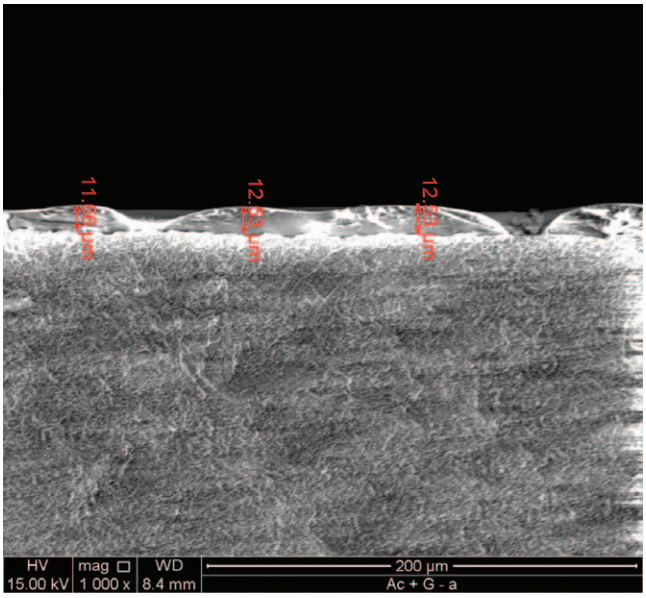

Figure 2. *Low-fusing porcelain glaze thickness measured on zirconia surface under SEM.*

fracture of dentin, without an adhesive failure; and score E = an area of resin cement fracture bigger than an area of adhesive failure.

Measurement of Glaze Thickness

A pilot study was conducted to measure the thickness of the glaze obtained by the vitrification process. To measure the thickness of the glaze layer, three zirconia beams (10×2×2 mm) were sprayed with the glaze, sintered, and broken into two pieces so that the glaze thickness could be measured by SEM at a magnification of 1000×. As can be noted in Figure 1, the applied glaze is homogeneously distributed on the zirconia surface. In addition, the thickness of the glaze layer was also found to be of an acceptable level, with a mean value of 12 ± 0.3 µm (Figure 2).

Data Analysis

The bond strength data were analyzed using three-way analysis of variance (ANOVA) and Tukey test (α=0.05) with the software Minitab 16.1.0. The specimens with pretest failures (during the aging process) were included in the statistical analysis and were conferred a bond strength value of 0 MPa.

RESULTS

The factors resin cement ($p<0.001$; Panavia> Clearfil) and storage condition ($p=0.006$; nonaging> aging) statistically influenced the bond strength values, while the factor surface treatment ($p=0.762$; Vi = Si) had no effect. According to the result of the Tukey test, the groups Si + CC + aging (4.0 ± 3.4 MPa) and Vi + CC + aging (3.9 ± 3.8 MPa) exhibited the lowest bond strength values, while the Vi + PF group (17.2 ± 10.1 MPa) showed the highest values of bond strength but was not statistically different than Vi + PF + TC (14.7 ± 6.0 MPa), Si + PF (14.6 ± 8.4 MPa), Si + PF + TC (11.6 ± 6.2 MPa), and Si + CC (10.8 ± 3.8 MPa). The group Vi + CC (7.0 ± 3.4 MPa) showed intermediate values. When subjected to the Si surface treatment, the zirconia cylinders cemented with Clearfil cement showed a significant decrease in bond strength values after aging.

Fracture analysis revealed that fractures occurred predominantly in the adhesive at the resin cement/

Table 2: *Shear bond strength (MPa) and incidence of failure type data.*

Surface Treatment	Cement	Thermo-cycling	Group Abbreviations	Mean[a] (SD)	Scores for Failure Type[b]				
					Score A	Score B	Score C	Score D	Score E
Tribosilicatization	Panavia F	No	Si + PF	14.6 (8.4) AB	6	0	0	0	4
		Yes	Si + PF + TC	11.6 (6.2) ABC	4	0	0	0	6
	Clearfill SE	No	Si + CC	10.8 (3.8) ABC	6	0	0	0	4
		Yes	Si + CC + TC	4.0 (3.4) C	7	0	0	0	3
Glassy application	Panavia F	No	Vi + PF	17.3 (10.1) A	6	1	0	0	3
		Yes	Vi + PF + TC	14.7 (6.0) AB	8	0	0	0	2
	Clearfill SE	No	Vi + CC	7.0 (3.4) BC	8	0	0	0	2
		Yes	Vi + CC + TC	3.9 (3.8) C	7	1	0	0	2
					52 (65%)	2 (2.5%)	0	0	26 (32.5%)

[a] *Means and standard deviations (SDs) of shear bond data (MPa) and Tukey test (α=0.05). Letters denote statistically similar treatment groups.*
[b] *The incidence of each failure type and percentage (%) after the shear bond strength test. Score A: adhesive failure between resin cement and dentin; Score B: adhesive failure between resin cement and ceramic; Score C: cohesive failure of ceramic; Score D: cohesive failure of dentin; Score E: cohesive failure of cement.*

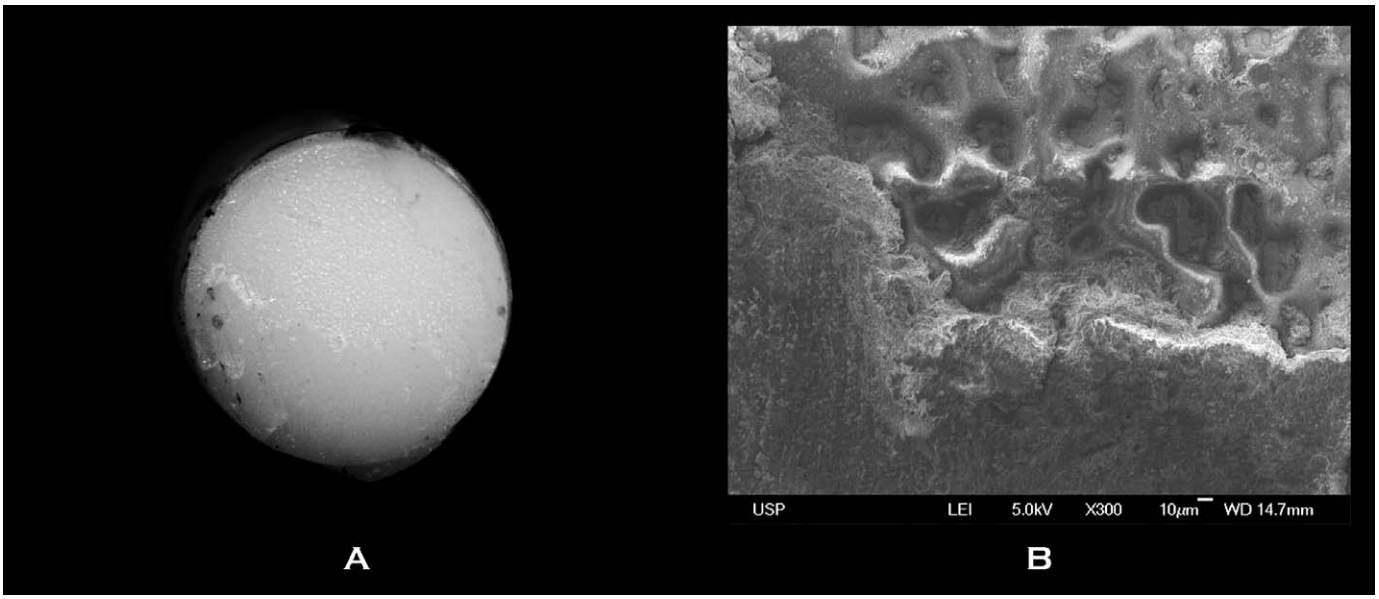

Figure 3. *(A) Representative image of a cohesive failure. (B) Detailed view of the small black square in (A). Fracture surface topography revealed the configuration of the glaze film on the zirconia surface, which is associated with exposure of zirconium oxides.*

dentin interface (65% of the failures were scored type A), followed by cohesive failures in the resin cement (32.5% were scored type E) and in the adhesive at the resin cement and ceramic interface (2.5% were scored type B) (Table 2). Representative images of the different failure modes are shown in Figures 3 and 4. For the groups Si + CC + aging and Vi + CC + aging, three specimens each failed during aging; for the group Si + PF + aging, one specimen failed during aging; and in the group Vi + PF + aging, there were no specimen failures during the aging process. All of the premature failures that occurred during aging were at the resin cement and dentin interface (scored type A).

DISCUSSION

Different surface treatments have been tested to improve the bond strength between resin cement and zirconia. Nowadays, special attention has been given to the treatment of the zirconia intaglio

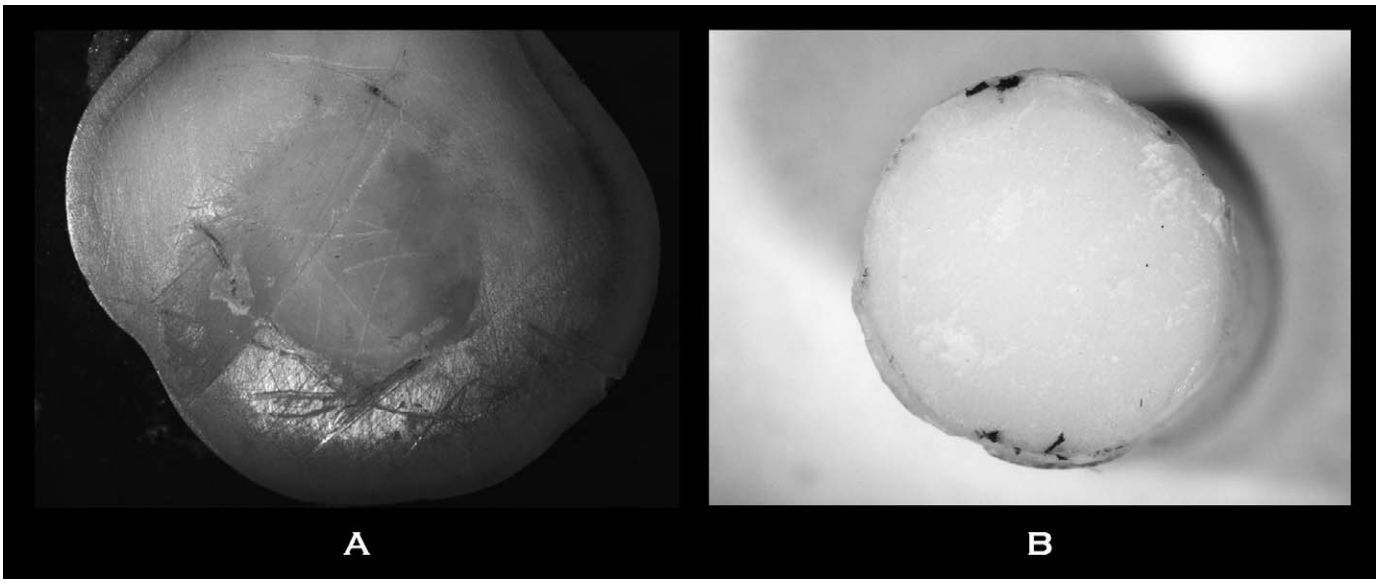

Figure 4. *Representative image of a failure at the cement/dentin interface.*

surface with low-fusing porcelain glazes (produced by vitrification), which would create an etchable layer on the acid-resistant material, creating a scenario similar to that found in silica-based ceramics. For studies on zirconia to have clinical relevance, it is important to consider the dentin surface as well.[5]

The three-way ANOVA showed that the zirconia surface treatments resulted in similar bond strengths, confirming the first hypothesis of the study and representing a good pattern of conditioning promoted by Y-TZP surface conditioning approaches. The silicatization after air-abrasion by silica-modified aluminum oxide particles is considered a prerequisite for achieving good adhesion between the resin cement and zirconia surfaces[10] and leads to the generation of bond strength values that are higher than those obtained from other surface treatments.[1,5,7,10] This method creates hydroxyl groups and enhances the micromechanical retention of the resins on the zirconia surface. In addition, the application of a silane coupling agent after the silica coating generates a siloxane network that improves the bond between the resin cement and zirconia.[10] Chai and others[5] employed an experimental design similar to that used in our study and observed that the tribochemical silica coating of the zirconia surface generated statistically higher values for the bond strength between the zirconia cylinders and the dentin surface. The vitrification method creates an etchable glassy thin film on the acid-resistant zirconia surface. This surface can then be etched by hydrofluoric acid and silanized using an MPS-based primer. In addition, only two specimens subjected to the vitrification process exhibited adhesive failure at the resin cement/zirconia interface. This result corroborates the findings of Cura and others.[4] On the other hand, in contrast to the results obtained by us, Everson and others,[16] Valentino and others,[17] and Ntala and others[18] observed statistically higher values of bond strength for vitrification techniques than for the tribochemical silica-coating approach. This may be because these studies did not use dentin as an adhesion substrate. We utilized dentin as an adhesion substrate for the zirconia samples, and the majority of the failures occurred at the dentin/resin cement interface, preventing a real evaluation of the adhesion of the resin cement to the treated zirconia surface (comparison between the Y-TZP surface conditioning).

The second hypothesis of the study was rejected, as the bond strength values are statistically affected by the resin cements (Panavia F: 14.5 MPa; Clearfil SE: 6.4 MPa). In this study, the resin cement Panavia F showed statistically higher values of shear bond strength than did the self-adhesive resin cement Clearfil SA Cement (Table 2).

According to previously reported studies,[24-26] the lower values of bond strength obtained for Clearfil SA Cement can be explained by the inability of this cement to remove the smear layer on the dentin surface, as is the case with other self-adhesive resin cements. This leads to the formation of a poor hybridization layer between the resin cement and dentin. Despite the poor interaction of the self-adhesive resin cement with the dentin, it is important to note that this cement showed a good interaction with the zirconia surfaces that had been subjected to either of the two surface treatments. This can be confirmed by the absence of adhesive failures between the self-adhesive resin cement and the ceramic. However, this finding should be further investigated by an experiment designed to evaluate the adhesion between this self-adhesive resin cement and zirconia alone without involving the dentin substrate.

Panavia F showed higher values of shear bond strength, and these could be related to a good dentin hybridization generated by the self-etching adhesive. This adhesive is considered to be a "mild" self-etchant and can remove the smear layer and expose the dentinal tubules. In addition, this adhesive contains the phosphate-based functional monomer 10-MDP, which interacts with collagen and hydroxyapatite components in dentin, resulting in the formation of a strong and stable bond between the resin cement and coronal dentin.[27-29]

Regardless of the resin cements and surface treatments used in the study, the aging process decreased the bond strength. This could be due to the use of the dentin substrate in the study, as it appears to be more susceptible to the hydrolysis. In contrast, even after the thermo-cycling aging process, the interfaces between the resin cements and the zirconia surfaces showed fewer adhesive failures. This is evidence that this interface exhibited stable bonding even after the aging process.

The thickness of the glassy film was found to be approximately 12 μm (Figure 2). It may be assumed that the film would have no effect on the seating of Y-TZP restorations and their marginal fit.[30,31] However, future studies should evaluate the real impact of this glassy film on the misfit of Y-TZP frameworks. In addition, the different methods of

applying the porcelain glaze and zirconia surface treatments before glaze application should also be investigated further. The fracture analysis provided important information about the system behavior. The cement/dentin interface was weaker than the cement/zirconia interface for all experimental groups. Chai and others[5] cemented zirconia cylinders that had undergone different surface treatments onto a dentin surface and observed a large number of adhesive failures between the cement and dentin. Their results, which are in accordance with the results obtained in this study, highlight the importance of including the dentin substrate in studies on bond strength so that they are more relevant clinically.

CONCLUSION

Considering the experimental design and the results obtained, we can draw the following conclusions:

1. The bond strength between the resin cements and the zirconia substrate was stronger than that between dentin and the cements, as a function of the efficient conditioning methods performed on the Y-TZP surface, even after lengthy storage.
2. The conventional resin cement containing MDP monomers showed better bond performance than did the self-adhesive resin cement.
3. The dentin/cement interface appears to be the more critical zone in this system.
4. The application of a thin film of low-fusing glaze porcelain on the zirconia surface followed by hydrofluoric acid etching and silanization appears to offer a promising surface treatment method with which to improve the adhesion between zirconia and resin cement. However, more studies should be performed to confirm this.

Conflict of Interest

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

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Influence of an Intermediary Base on the Microleakage of Simulated Class II Composite Resin Restorations

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Clinical Relevance

The use of resin-modified glass ionomer cement as an intermediary base provides lower microleakage, indicating better sealing of the tooth-restoration interface.

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SUMMARY

The aim of this *in vitro* study was to qualitatively and quantitatively evaluate the microleakage of Class II cavities restored with a methacrylate-based composite (Filtek Z250, 3M ESPE) or silorane-based composite (Filtek LS, 3M ESPE), varying the application of an intermediary base, using a low-viscosity composite resin (Filtek Z350 Flow, 3M ESPE) or resin-modified glass ionomer cement (RMGIC) (Vitrebond, 3M ESPE) and no intermediary base (control groups). Sixty cavities were prepared on the proximal surfaces of bovine teeth and were randomly divided according to the experimental groups (n=10). Following the restorative procedures and thermocycling, the samples were immersed in methylene blue for two hours. The qualitative evaluation was made using a stereomicroscope, whereby two observers analyzed the infiltration level of the dye within the tooth/filling. Microleakage scores among the groups were compared using the Kruskal-Wallis test followed by the Mann-Whitney test ($p \leq 0.05$). The samples were then

ground and the powder was prepared for quantitative analysis in an absorbance spectrophotometer. The results were statistically analyzed by analysis of variance and the Tukey test ($p \leq 0.05$). Results from the quantitative analysis showed that LS presented higher values of microleakage than did Z250. There was a significant difference between both composites concerning the intermediary materials, with the lowest values obtained using RMGIC as an intermediary base. Results from the qualitative analysis showed that there were no statistically significant differences between composites; however, there were significant differences for both composites concerning the intermediary materials, with the lowest values obtained using RMGIC as an intermediary. It is possible to conclude that using RMGIC as an intermediary base provided lower microleakage, indicating better sealing of the tooth-restoration interface.

INTRODUCTION

The polymerization reaction for methacrylate resin-based composites occurs through the conversion of monomer molecules into a structure of cross-linked polymers.[1] The main adverse effect of this material is volumetric shrinkage, which contributes to stress formation along the bonded interfaces of restorations[2] as well as to the formation of gaps in the composite-dentin interface, initiating the microleakage process.[3] Microleakage is defined as the passage of bacteria, fluids, or molecules between a cavity wall and the restorative material applied to it, and microleakage may cause hypersensitivity, recurrent caries, and pulpal pathoses.[4,5]

To reduce the side effects caused by polymerization shrinkage stress, an intermediary layer between the composite resin and the tooth has been proposed[3] as an alternative to creating a tooth-restoration interface without the presence of gaps. Studies have suggested using materials, such as flowable composite resins,[6] to act as a stress-absorbing layer, relieving the stress generated during polymerization shrinkage and promoting more effective sealing to the tooth structure. Resin-modified glass ionomer cements (RMGICs) have also been recommended as a base material, because they replace some of the composite volume and reduce the side effects of polymerization shrinkage.[7]

Recently, a new silorane-based composite resin has been introduced that has a distinctive polymerization characteristic that reduces polymerization shrinkage.[8] Silorane was so named by the manufacturer to indicate a hybrid compound of siloxane and oxirane functional moieties.[2] The silorane matrix is formed by a cationic ring opening of the silorane monomers during polymerization instead of free-radical polymerization of methacrylate monomers.[9] Therefore, a significantly lower polymerization shrinkage and lower stress development occur.[10] However, little is known about the effect associating an intermediate material and a silorane-based composite.

This current study compared the microleakage of Class II restorations restored with methacrylate- or silorane-based resins and different intermediary base materials. The null hypotheses were 1) there is no difference between marginal infiltration of methacrylate- and silorane-based restorations; and 2) there is no difference between the marginal infiltration of composite restorations when using different base materials.

MATERIALS AND METHODS

Sixty extracted bovine incisors were collected, cleaned with a periodontal curette, polished with a Robinson brush and pumice paste under water, and then stored in distilled water until they were used. The tooth had part of its root embedded in cold-cure polystyrene resin (Piraglass, Piracicaba, SP, Brazil). The specimens were then split obliquely 6 mm from the dentino-enamel proximal junction using a double-faced diamond disc (KG Sorensen, Barueri, SP, Brazil). After cutting, the incisal surfaces were finished with 600-grit water-abrasive papers to obtain a smooth surface.

Specimen Preparation

Cavities were made using a diamond tip #3146 (KG Sorensen) coupled to a cavity preparation unit on the flattest proximal surface, simulating a Class II restoration, measuring 6 mm high, 4 mm wide, and 1.5 mm deep, under irrigation with an air/water jet. The cervical limit of the proximal box was 1 mm above the cemento-enamel junction. The burs were replaced after every five preparations. The cavities were randomly restored, with one of the following six techniques.

Group 1—Filtek Z250 (3M ESPE, St Paul, MN, USA): The cavities were etched for 30 seconds in enamel and 15 seconds in dentin, using 37% phosphoric acid (3M ESPE), washed for 30 seconds, and gently dried with filter paper to prevent excessive dentin drying. The adhesive system Adper

Single Bond 2 (3M ESPE) was applied in two consecutive coats for 15 seconds with gentle agitation, gently air-dried for 10 seconds, and then light-cured for 10 seconds. The composite was inserted incrementally (2-mm horizontal increments), and each increment was light-cured for 20 seconds.

Group 2—Filtek LS (3M ESPE): The self-etching primer, LS Primer (3M ESPE), was applied in one coat for 15 seconds with gentle agitation, gently air-dried for 10 seconds, and then light-cured for 10 seconds. LS Bond was applied, gently air-dried for 10 seconds, and light-cured for 10 seconds. The composite was inserted incrementally (2-mm horizontal increments), and each increment was light-cured for 20 seconds.

Group 3—Filtek Z350 flow (3M ESPE)/Filtek Z250: The same restorative protocol described for group 1 was performed; however, the low-viscosity version of the resin was applied on the gingival wall up to the dento-enamel junction with a 2-mm thickness before inserting Filtek Z250 and light-curing for 20 seconds.

Group 4—Filtek 350 flow/Filtek LS: The same restorative protocol described for group 2 was performed; however, the low-viscosity version of the resin was applied on the gingival wall up to the dento-enamel junction with a 2-mm thickness prior to the application of LS Bond and light-cured for 20 seconds.

Group 5—Vitrebond (3M ESPE)/Filtek Z250: A RMGIC was applied on the gingival wall up to the dento-enamel junction, in a 2-mm thickness, and was light-cured for 40 seconds. After that, the same restorative protocol described for group 1 was performed.

Group 6—Vitrebond/Filtek LS: A RMGIC was applied on the gingival wall up to the dento-enamel junction, in a 2-mm thickness, and was light-cured for 40 seconds. After that, the same restorative protocol described for group 2 was performed.

A Bluephase 16i light-curing unit (Vivadent, Bürs, Austria) with an irradiance of 1100 mW/cm^2 was used to photoactivate all groups. The tip of the light-curing unit was positioned perpendicular to the incisal surface of the tooth without touching it.

After 24 hours of storage at 37°C the restorations were finished and polished with Sof-Lex Pop-on aluminum oxide disks (3M ESPE) in decreasing order of granulation. The samples were thermocycled 1000 times (5±2°C, 37±2°C, and 55±2°C) with a dwell time of one minute each at each temperature and a transfer interval of five seconds (MSCT, 3 PLUS, São Carlos, SP, Brazil).

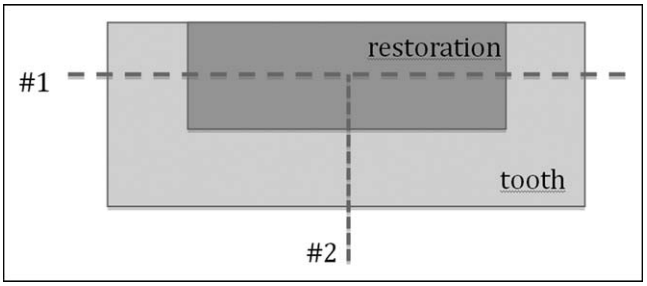

Figure 1. *The fragments for qualitative analysis were obtained by the sectioning of the tooth-restoration block in three parts. The first cut (#1) was made in the center of the tooth-restoration block in the mesio-distal direction, resulting in two segments (upper and lower). After that, the lower segment was also sectioned in half, now in the gingival-occlusal direction (#2). Obtaining fragments with approximate areas of microleakage was possible because of the prior standardization of the dimensions of the cavity.*

Dye Immersion

After these procedures, the entire sample (except for the cervical interface between the restoration and the tooth) was protected with two layers of fast-setting Superbonder cyanoacrylate-based adhesive (Henkel Loctite Adesivos, Itapevi, SP, Brazil). Before dye immersion, a 1-mm strip of adhesive tape (Fix-Baby, Embalando, Fitas Adesivas Ltda, Arujá, SP, Brazil) was placed around the area that was infiltrated, and two layers of nail varnish were applied. Then the tape was removed, the interface was cleaned with sterile gauze, and the specimens were totally immersed in 2% neutral methylene blue solution for two hours. The blocks were then removed from the dye solution, washed under running water, and dried. The nail varnish was removed using a periodontal curette, and the dye on the restoration was removed up to 0.05 mm from the surface, as controlled by a caliper.

Fragments

Three fragments were obtained for qualitative analysis, named "left wall," "gingival wall," and "right wall" (Figure 1).

Qualitative Analysis

The qualitative evaluation was performed by two previously calibrated examiners under a stereomicroscope at 40× magnification. The specimens were evaluated using the following microleakage scores: 0 = no infiltration; 1 = infiltration within tooth/material interface, beyond half of the gingival wall; 2 = infiltration within tooth/material interface,

beyond half of the gingival wall, but not reaching the axial wall; and 3 = infiltration within tooth/material interface up to or beyond axial wall (ISO 11405:2003).

Quantitative Analysis: Sample Trituration

To take a reading of the infiltrated dye color, specimens (dental block + restoration) were weighed. After weighing, the specimens were triturated in a hard fabric grinder (MA-475, Marconi Equip Ltda, Piracicaba, SP, Brazil) in order to obtain a powder composed of the tooth/restoration and were then weighed again. If the difference between the initial and final weights was greater than 10%, the specimen was discarded. In this present study, no specimens were discarded.

Dissolution

After trituration, the powder obtained from each sample was separately immersed in a test tube containing 4 mL of absolute alcohol PA for 24 hours to dissolve the dye that leaked through the tooth/restoration interface. The solution obtained was centrifuged at 3000 rpm for three minutes so that the powder and other elements separated. The supernatant of the centrifuged solution was submitted to quantitative analysis of the dye present in the solution with a spectrophotometry unit (Beckman DU-65 Instruments Inc, Fullerton, CA, USA) using absorbance reading.

The absorbance reading was taken in an adjusted unit at a wavelength of 668 nm, corresponding to the maximum absorbance of methylene blue dye. Prior to the readings, the spectrophotometry unit had been adjusted by spectral reading with pattern solutions at concentrations of 0.1; 0.2; 0.3; 0.5; 1; 2; 4; and 6 μg/mL to obtain the maximum spectral absorbance wavelength. Readings of the solutions were made using the wavelength value to find the maximum value of spectral absorbance. By using the ABS-concentration system, the r^2 value (0.9999) is obtained and the equation of the line is (y=a+bx). The following regression was obtained: absorbance = 0.22759 × (dye concentration) + 0.0011. From this regression, dye concentration can be calculated. A graph of lines in a Cartesian system of axes was drawn, using the values of dye concentration in micrograms per milliliters on the axis of the abscissas and the optical density obtained on the axes of the ordinates. The linear regression was obtained from Y as a function of X to determine the equation of the line, from which the concentration of dye was calculated.

Table 1: *Results of Quantitative Microleakage (Standard Deviation [SD])*[a]

	Composite	
	LS Mean (SD)	Z250 Mean (SD)
No intermediary base	0.103 (0.221) Aa	0.021 (0.020) Ba
RMGIC	0.015 (0.024) Ab	0.007 (0.007) Bb
Flow	0.034 (0.023) Aa	0.018 (0.016) Ba

Abbreviation: RMGIC, resin-modified glass ionomer cement.
[a] *Means followed by distinct letters (capital letters in the horizontal and lowercase letters in the vertical) differ between them (p<0.05).*

The microleakage data of experimental groups was submitted to three-way analysis of variance and Tukey test for quantitative analysis. For qualitative analyses, the Kruskal-Wallis test was used to assess the differences within the groups. The Mann-Whitney *U*-test was used to investigate the pairwise differences among the different groups. The level of statistical significance was set at 5%.

RESULTS

Results from the quantitative analysis are presented in Table 1 and Figure 2. Filtek Z250 showed significantly lower (69.8%) microleakage means when compared to Filtek LS. For both composite resins, the groups with RMGIC used as a base showed significantly lower (82.3% for no intermediary base; 57.7% for the flow) means than the other groups.

Results from the qualitative analysis are shown in Table 2. There was no statistically significant difference between composites. For both composites, there was a significant difference among the intermediary materials, with the lowest values obtained with the RMGIC base. Table 3 and Figure 3 present results from the quantitative analysis discriminated

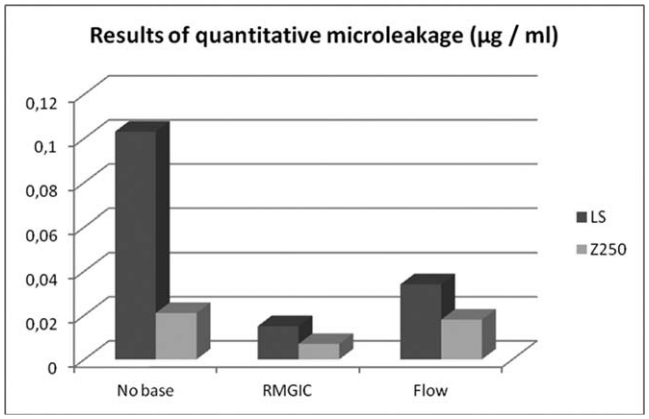

Figure 2. *Results of quantitative microleakage.*

Table 2: *Results of Qualitative Microleakage*[a]

	Composite	
	LS Median	**Z250 Median**
No intermediary base	3.00 Aa	2.00 Ab
RMGIC	1.00 Ab	1.00 Ab
Flow	3.00 Aa	3.00 Aa

Abbreviation: RMGIC, resin-modified glass ionomer cement.
[a] *Median followed by distinct letters (capital letters in the horizontal compare composites within intermediary base; lowercase letters in the vertical compare intermediary base within composite) differ between them ($p<0.05$).*

by walls. There was no statistical difference between the composite resins in the buccal and gingival walls. In the lingual wall, Filtek Z250 showed lower microleakage than did Filtek LS only for the RMGIC group. When comparing the intermediary base, RMGIC showed lower microleakage than did other groups for all walls in the group LS and for the lingual wall in group Z250.

DISCUSSION

This *in vitro* study qualitatively and quantitatively evaluated the microleakage of Class II restorations restored with methacrylate- and silorane-based composites using a low-viscosity resin or RMGIC as an intermediary base.

The first null hypothesis was partially rejected, since there was a difference found with the quantitative analysis when using different restorative materials. The silorane-based composite showed more microleakage than did the methacrylate-based composite. The second null hypothesis was rejected, since there was a difference in microleakage when different intermediary materials were used.

Table 3: *Qualitative Microleakage Discriminated by Walls*[a]

Wall	Composite	
	LS Median	**Z250 Median**
Buccal		
No intermediary base	3.00 Aa	1.00 Aa
RMGIC	0.00 Ab	0.50 Aa
Flow	3.00 Aa	1.00 Aa
No intermediary base	3.00 Aa	2.00 Aa
Gingival		
RMGIC	1.00 Ab	1.00 Aa
Flow	3.00 Aa	2.00 Aa
No intermediary base	3.00 Aa	1.00 Aa
Lingual		
RMGIC	1.00 Ab	0.00 Bb
Flow	3.00 Aab	2.00 Aa

Abbreviation: RMGIC, resin-modified glass ionomer cement.
[a] *Median followed by distinct letters (capital letters in the horizontal compare composites within intermediary base; lowercase letters in the vertical compare intermediary base within composite) differ between them ($p<0.05$).*

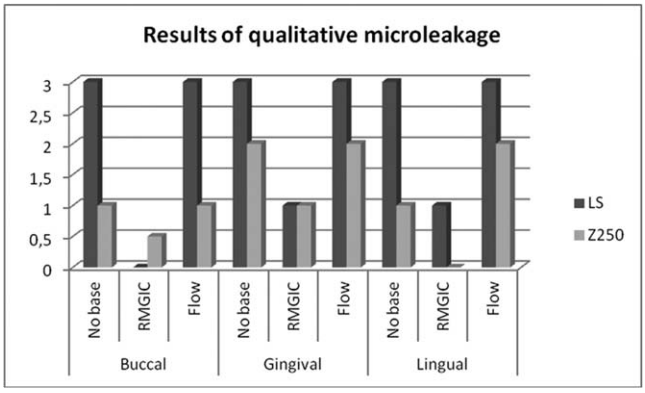

Figure 3. *Results of qualitative microleakage discriminated by walls.*

In the quantitative analysis, LS showed the highest values of microleakage, with a significant statistical difference from Z250. In the qualitative analysis, there were no differences between the restorative materials. These results indicate that both the methacrylate-based and silorane-based composites showed similar behavior only in the qualitative analysis of dye penetration. These findings indicate that low-shrinkage composites did not necessarily reduce the polymerization shrinkage stress at the tooth-restoration interface.[11] Microleakage is influenced by numerous factors related to the materials, technique, and cavity preparation, and the interaction between these factors dictates the exact manifestation of shrinkage for a given restoration.[3]

Several studies[8,9,12] have reported that silorane-based composites exhibit lower shrinkage rates and lower shrinkage volumes than do methacrylate-based composites because the polymerization reaction takes place by the opening and cleavage of a cationic ring structure.[12] However, a lower shrinkage rate does not necessarily indicate lower polymerization stress, especially if an incremental filling technique is used.[13]

Another factor to be considered is the possibility of the silorane-based composite being more susceptible to reduced curing of the bottom surface of the increment.[13] This composite exhibits a polymerization reaction with a slower onset because more energy density is required to form sufficient cations and initiate polymerization.[14,15] The irradiance that reaches the bottom surface of the cervical increment

is affected by the distance between the resin composite and tip of the light-curing unit and by the thickness of the resin composite.[16] Consequently, a low degree of conversion of silorane-based composite has been associated with a decreased mechanical strength of adhesives.[13]

The higher microleakage means of LS obtained using the quantitative analysis could also be explained by the adhesive systems. Restoration with Filtek Z250 was performed using the total etch technique and a single bottle adhesive, whereas Filtek LS required its own self-etch bonding system, which may explain the results. It has been well documented[17] that self-etch systems do not perform as well in enamel as total-etch systems. Additionally, the adhesive system of Filtek LS showed a bond mechanism similar to that of a one-step self-etching adhesive, since adhesion to the tooth surface is realized using the "ultra-mild" (pH>2.5) Primer LS, which superficially interacts with the smear layer.[13] One-step self-etching adhesives combine conditioning, priming, and application of the adhesive resin and do not require mixing. Consequently, they are considered to be more user friendly. However, they present major shortcomings, such as reduced immediate bond strength when compared to multi-step adhesives[18] and increased interfacial nano-leakage.[19]

The results for the qualitative technique showed no differences between the composites. The qualitative microleakage evaluation takes into account only a few points, whereas quantitative analysis evaluates the entire interface. It is possible to infer that this total evaluation could not detect differences between the restorative systems. Additionally, the quantitative method seems to be more suitable for detecting differences, since the entire interface is evaluated and considered in the final result of the sample. As a disadvantage, the quantitative technique is more time consuming and specific equipment is required. The results of this current study disagree with those of another study[20] that reported that different methods of microleakage evaluation do not differ in the final results.

On the other hand, the results of qualitative analysis showed no differences between the restorative systems. A possible explanation could be related to the higher failure of the LS adhesive, which was detected mainly by quantitative analysis.

In the quantitative and qualitative analyses, both composites showed statistically significant differences when considering the intermediary materials. The lowest results of marginal infiltration were obtained with RMGIC. The use of RMGICs as an intermediate layer for restorative materials has been suggested and largely applied. Previous studies[21-23] showed its benefits in lowering microleakage due to its thermal expansion, which is similar to that of dental structures; its bacteriostatic function; molecular bonding to dentin and enamel; and a low setting shrinkage.[23] The other intermediate material used in this current study was a flowable composite, which has been purported[24] to be able to diminish the stress during the polymerization shrinkage and to promote better sealing to dental structure as a result of its lower elastic modulus. However, the present study did not find this absorbing behavior, and the microleakage values found were similar to those of the control group. Flowable composite presents lower filler content and higher resin matrix than do traditional composites[22] and generally presents with a higher polymerization shrinkage volume, which has ranged from 3.6% a 6%, whereas traditional composites shrink around 1.9% and 2.3%. Increased volumetric shrinkage may indicate the potential for higher contraction stress at the restorative interface as well as the likelihood of bond failure.[22] These characteristics may explain the results obtained in this present study, which are in agreement with those of Oliveira and others.[25]

Finally, a qualitative analysis was performed that discriminated the microleakage by wall (Table 3). In general, the patterns of microleakage were similar on all of the walls, showing that stress distribution is concentrated in both the gingival and surrounding walls, independent of the composite resin used. The results of the present study corroborate with the study by Oliveira and others,[25] which demonstrated a high stress concentration in the gingival wall, surround- gingival angle, and the surrounding wall close to the angle, in a photoelastic model study. The association between the high concentration shrinkage stress and the inadequate polymerization at the bottom surface of the composite resin is a crucial factor for increasing the microleakage in this area.

CONCLUSION

Based on the experimental results from this study:

1. The silorane-based composite was not able to reduce marginal infiltration.
2. Regardless of restorative material, a RMGIC intermediary base showed lower results of marginal infiltration.

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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Accuracy of Ceramic Restorations Made Using an In-office Optical Scanning Technique: An *In Vitro* Study

P Tidehag • K Ottosson • G Sjögren

Clinical Relevance

The findings in the present study suggest that in-office digital impressions and subsequent computer-aided design/computer-aided manufacturing (CAD/CAM) fabrication of ceramic crowns could result in marginal and internal fit similar to that of hot-pressed all-ceramic crowns.

SUMMARY

The present in vitro study concerns determination of the pre-cementation gap width of all-ceramic crowns made using an in-office digital-impression technique and subsequent computer-aided design/computer-aided manufacturing (CAD/CAM) production. Two chairside video camera systems were used: the Lava Oral scanner and Cadent's iTero scanner. Digital scans were made of a first molar typodont tooth that was suitably prepared for an all-ceramic crown. The digital impressions were sent via the Internet to commercial dental laboratories, where the crowns were made. Also, an impression of the typodont tooth was made, poured, and scanned in order to evaluate the pre-cementation gap of crowns produced from scanning stone dies. These methods and systems were evaluated by creating replicas of the intermediate space using an addition-cured silicone, and the gap widths were determined using a measuring microscope. Hot-pressed leucite-reinforced glass-ceramic crowns were selected as a reference. The mean value for the marginal measuring points of the control was 170 μm, and the values for all the evaluated crowns ranged from 107 to 128 μm. Corresponding figures for the internal measuring points were 141-210 μm and 115-237 μm, respectively. Based on the findings in the present study, an in-office digital-impression technique can be used to fabricate CAD/CAM ceramic single crowns with a marginal and internal accuracy that is on the same level as that of a conventional hot-pressed glass-ceramic crown. In the pre-

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sent study, however, slight differences could be seen between the two types of ceramic crowns studied with respect to the internal fit obtained.

INTRODUCTION

The marginal and internal fit of dental restorations, such as inlays and crowns, is crucial for the clinical outcome of dental restorations. During the past few decades various impression and die materials have been used in dentistry,[1-3] and marginal and internal fit has been determined using a variety of measuring techniques: cementing on extracted teeth or replicas and subsequent slicing of the restorations and the teeth/replicas,[4-6] direct measurement with light microscope,[7,8] or replica technique with polyvinylsiloxane impression materials.[9-10]

Currently, most impression materials have to be sent by mail and/or courier to dental laboratories despite the possible risk of the impressions being disease carriers as disinfection of impression materials is problematic.[11,12] To avoid the use of impression materials, attempts to determine the shape of preparations for dental restorations using various mechanical and/or optical systems were presented in the 1970s,[13] but as far as we know, they were not routinely used in clinical practice. Later, around the middle of the 1980s, a system for electronically designing and milling ceramic restorations was introduced in dentistry.[14] This system used a three-dimensional (3D) miniature video camera to stereo-photogrammetrically scan the cavity preparation and make a 3D pattern of the tooth preparation, which could then be stored in a chairside microprocessor unit and, in combination with a numerically controlled miniature three-axis milling device, the restorations could be milled from prefabricated ceramic blocks.[14] Because it was a completely new way of producing dental restorations, the precision of the final restorations was questioned and was of particular interest. Thus, several studies addressing the internal and marginal fit of these restorations were presented; these stated, among other things, that the properties of the luting agents used, the shape of the preparation and the restoration, and the location of the measuring points could influence the outcome.[6,8-14] Recently, a number of improved versions of 3D miniature video cameras, in combination with improved microprocessors, have been introduced in dentistry. Examples of such systems are the Cerec AC (Sirona Dental Systems GmbH, Bensheim, Germany), E4D CADCAM system (Ivoclar AG, Schaan, Lichtenstein), Cadent's iTero (Cadent Inc, Carlstadt, NJ, USA), and Lava Chairside Oral Scanner C.O.S. (3M ESPE, St Paul, MN, USA).[15]

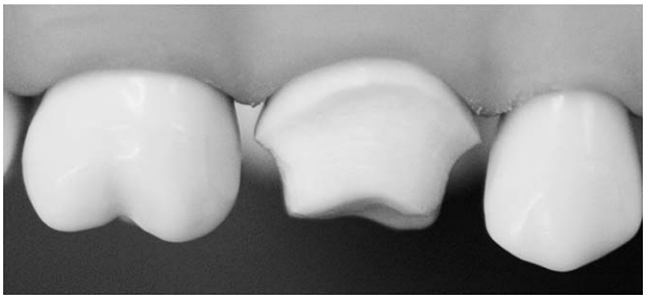

Figure 1. *Shape of the prepared maxillary right first phantom Frasaco molar with a ~1.5-mm chamfer preparation placed in a Frasaco phantom model.*

These systems are said to be improvements on the earlier types of equipment intended for digitalizing dental preparations[15] and should thus produce better internal and marginal fit for the restorations. The aim of the present work was, therefore, to determine the marginal and internal pre-cementation space in ceramic crowns made using two of the recently introduced systems and to compare the results with ceramic crowns made after taking impressions with an addition-cured silicone and using conventional die stone models, wax copings, and subsequent press casting.

METHODS AND MATERIALS

The internal and marginal fit was determined for two different types of all-ceramic crowns, which were made using two types of digital impression systems: the Lava Chairside Oral Scanner C.O.S. and Cadent's iTero systems.

A ~1.5-mm chamfer preparation was made for an all-ceramic crown on a maxillary right first phantom Frasaco molar (Frasaco GmbH, Tettnang, Germany) placed in a maxillary Frasaco model (Figure 1). The maxillary Frasaco model was placed in a Frasaco phantom head (Figure 2), and digital impressions, using the Lava or the Cadents iTero scanning systems, were made of the prepared phantom molar and the surrounding area and the opposing teeth, in accordance with the manufacturer's instructions. The digital impressions were then sent via the Internet to two different commercial dental laboratories experienced in fabricating the relevant types of crowns. One laboratory produced nine iTero all-ceramic crowns, and the other laboratory produced nine Lava all-ceramic crowns. In the following text

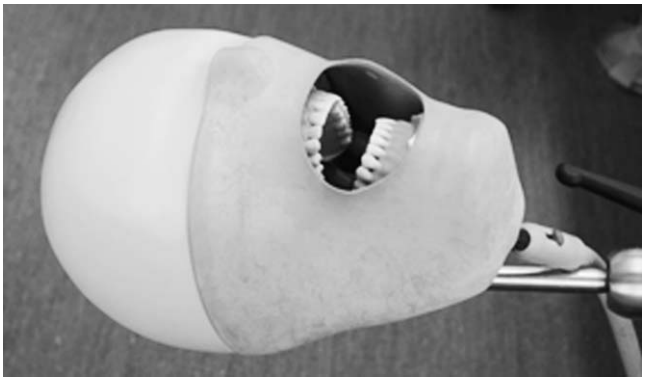

Figure 2. *A phantom Frasaco model placed in a Frasaco phantom head.*

those crowns are referred to as ”iTero Oral” and ”Lava Oral,” respectively.

In addition, to study the effect on the marginal and internal gap width of producing Lava and iTero crowns after scanning die stone models made from impressions of the prepared molar and the surrounding and opposing areas, addition-cured silicone impressions were made (Provil Novo Light body, batch number 310309, and Provil Novo Medium, batch number 320079, Heraeus Kulzer GmbH, Hanau, Germany). These silicone impressions were then sent to two different commercial dental laboratories experienced in the technique. Thereafter, one dental laboratory did the scans for the Lava crowns and another for the iTero crowns. The scanned files were used to produce nine Lava and nine iTero crowns, respectively. In the following text these crowns are designated “iTero Die Stone” and “Lava Die Stone”.

Hot-pressed leucite-reinforced glass-ceramic crowns (Empress, Ivoclar Vivadent AG, Schaan, Lichtenstein) were selected as the reference. After taking impressions of the prepared molar and the surrounding and opposing areas with an addition-cured silicone (Provil Novo Light body, batch number 310309, and Provil Novo Medium, batch number 320079), nine Empress crowns were made by one of the authors (KO) and a dental technician at the Department of Odontology, Umeå University, who was specially trained in the Empress technique using conventional die stone models (Vel-Mix Die Stone, Type IV, Kerr Dental Laboratory Products, Orange, CA, USA), wax copings, and subsequent press casting.

After all the crowns were produced, replicas of the intermediate space between the inner surface of the crowns and the prepared Frasaco tooth surface were

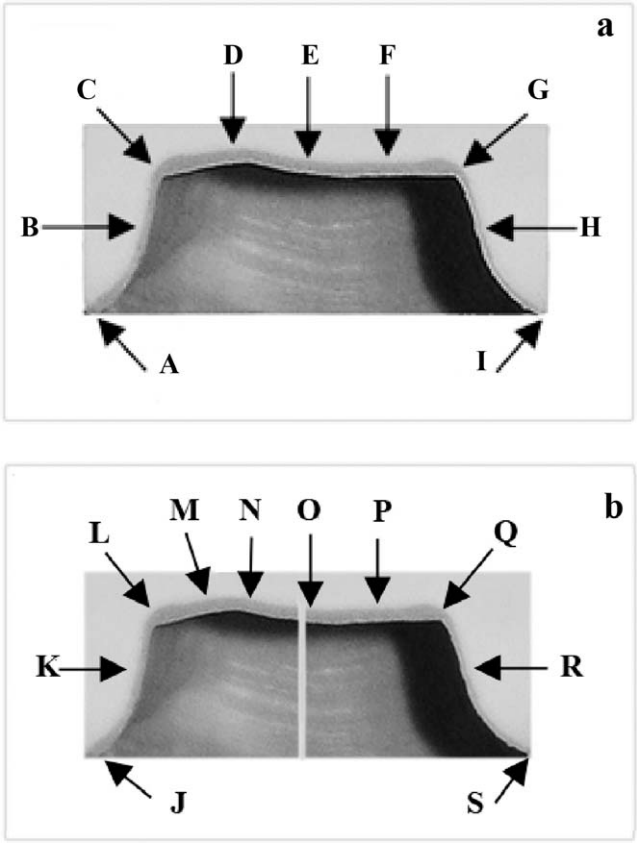

Figure 3. *Example of an addition silicone replica of the marginal width. The letters indicate the selected measuring points. Darker shade = light body; lighter shade = medium body. (a): Denotes the letters of the measuring points in the mesiodistal direction; (b): Denotes the letters of the measuring points in the buccolingual direction.*

taken with an addition-cured silicone (Provil Novo, Heraeus Kulzer GmbH). The crowns were filled with the addition-cured silicone light-body impression material (Provil Novo Light, batch number 310309, color green), placed on the prepared Frasaco tooth, and held in position using finger pressure for 4 minutes at room temperature. After the light-body impression material had polymerized, the crowns were removed and the thin film of the impression material that dressed the outside of the prepared Frasaco tooth or the inside of the crown was covered by a medium-body impression material (Provil Novo Medium, batch number 320079) that differed in color (yellow) from the light-body material. After polymerization, the light-body and the medium-body impression materials were removed en bloc. Three persons made the replicas: one person made 20 of the Lava Oral replicas, another person made 23, and a third person made two. Because the medium-body impression material joined the light-body replicas in one piece, they could be cut with a scalpel in two

directions: one corresponding to the mesiodistal (Figure 3A) and one to the buccolingual (Figure 3B). That is, the replicas were divided into four pieces, and because the light-body and the medium-body impression materials were different colors (green and yellow, respectively), the discrepancy between the master model and the frameworks representing the cement space could be distinguished (Figure 3a,b). The gap width was then measured using a Leitz UWM-DigS measuring microscope (Esselte Leitz GmbH & Co KG, Stuttgart, Germany) at 19 preselected locations (Figure 3a,b) on each half of the sectioned replicas at 20× magnification, giving a total of 38 measuring points for each crown. The gap width was measured as the thickness of the light-body impression material at the measuring points in Figure 3a,b. The same person carried out all the measurements without knowing the identity of the specimens.

Statistical Analysis

The values were statistically analyzed using an independent *t*-test at a significance level of $p<0.05$ after Bonferroni correction $p<0.01$. The data were analyzed using SPSS statistical software, version 20 (Statistical Package for Social Science, SPSS Inc, Chicago, IL, USA).

RESULTS

The results and statistical analyses are summarized in Tables 1 and 2. Statistical analysis (section A of Table 2) of all the measuring points, that is, measuring points A-S in Figure 3, revealed that the gap width was significantly wider ($p<0.001$) for the Empress crowns (control) than for the Lava Oral crowns, whereas there were no significant differences between Empress and Lava Die Stone, iTero Oral, and iTero Die Stone. The Lava Oral crowns showed a significantly ($p<0.01$) smaller gap width than the other groups.

Comparison of the marginal measuring points, that is, measuring points A, I, J, and S in Figure 3, showed that the marginal gap width was significantly ($p<0.01$) wider for Empress than for all the other groups (section B in Table 2).

For the proximal measuring points, that is, measuring points B, H, K, and R in Figure 3, there were no significant differences between the Empress and the other groups tested, whereas the gap width for the iTero Die Stone was significantly ($p<0.001$) wider than for the iTero Oral. For the Lava Die Stone, the proximal gap width was significantly wider ($p<0.001$) than for the iTero Oral crowns (section E in Table 2).

At the proximal-occlusal measuring points, that is, measuring points C, G, L, and Q in Figure 3, there were no significant differences between Empress and the other groups tested. The gap width for the iTero Oral crowns was significantly wider than for Lava Oral ($p<0.01$). No statistically significant differences were seen among the other groups of crowns studied for the proximal-occlusal measuring points (section D of Table 2).

The occlusal gap width, that is, measuring points D, E, F, M, N, O, and P in Figure 3, was significantly wider for the iTero Oral crowns than for the Empress, Lava Oral, and iTero Die Stone crowns ($p<0.001$). The Lava Die Stone crowns exhibited a significantly ($p<0.001$) wider occlusal gap width than the Empress, Lava Oral, and iTero Die Stone crowns (section E of Table 2).

DISCUSSION

The purpose of the present study was to evaluate the accuracy of ceramic crowns produced by means of in-office scanning of the preparation and subsequent computer-aided design/computer-aided manufacturing (CAD/CAM) production technologies. Today, dental CAD/CAM technology has expanded well beyond the initial version of the Cerec system introduced in the middle of the 1980s, and a number of CAD/CAM restorative technologies are currently on the market.[15] To evaluate the ability of the recently introduced in-office scanning and CAD/CAM restorative technologies to produce all-ceramic crowns with acceptable marginal and internal fit, two such systems, 3M ESPE Lava COS and Cadents iTero, were used. To copy the clinical situation as closely as possible, a digital impression of the scanned preparation was sent via the Internet to commercial dental laboratories trained in the technique of manufacturing this type of ceramic crown. In addition, the present study evaluated the marginal and internal fit of crowns made after the preparation was scanned from die stone models constructed from polyvinyl-siloxane impressions of the prepared phantom Frasaco model. Hot-pressed leucite-reinforced glass-ceramic crowns (IPS Empress) were used as a control. The IPS Empress crowns were selected for this purpose because this type of all-ceramic crown has been in clinical use for a long time.[16,17]

In the present work, replicas of the space between the inner surface of the crowns and the prepared

Table 1: *Summary of the Gap Widths at the Various Measuring Points*

	Empress	Lava Oral	Lava Die Stone	iTero Oral	iTero Die Stone
A. All measuring points (μm) (n=342 measuring points for each group)					
Mean±SD	183±90	162±65	177±60	181±72	174±60
Minimum	0	0	38	59	49
Maximum	597	331	327	346	420
Median	177	154	184	181	175
95% CI					
Lower	174	155	174	173	168
Upper	193	169	181	184	181
B. Marginal measuring points (μm) (n=72 measuring points for each group)					
Mean±SD	170±94	107±47	113±48	128±59	115±37
Minimum	0	32	38	59	49
Maximum	398	223	267	288	215
Median	145	97	47	59	37
95% CI					
Lower	148	96	102	114	106
Upper	192	118	124	141	123
C. Proximal measuring points (μm) (n=72 measuring points for each group)					
Mean±SD	141±75	130±55	149±44	115±40	160±52
Minimum	0	0	81	60	76
Maximum	367	265	286	243	334
Median	121	128	139	111	157
95% CI					
Lower	124	118	139	106	148
Upper	159	143	159	125	172
D. Proximal-occlusal measuring points (μm) (n=72 measuring points for each group)					
Mean±SD	210±116	176±54	189±48	201±46	195±57
Minimum	60	53	88	123	78
Maximum	597	293	327	310	393
Median	188	163	188	191	182
95% CI					
Lower	183	164	178	190	182
Upper	237	189	200	212	209
E. Occlusal measuring points (μm) (n=126 measuring points for each group)					
Mean±SD	200±65	203±54	224±32	237±50	204±47
Minimum	75	109	148	149	122
Maximum	359	331	294	346	420
Median	205	204	223	224	190
95% CI					
Lower	189	193	218	228	196
Upper	212	212	229	246	213

Abbreviations: SD, standard deviation; CI, confidence interval.

tooth were made, using a polyvinyl siloxane addition-cured silicone, to determine the gap widths. This technique was selected as it has been determined that using light-body addition-cured polyvinyl-siloxane impression materials for the replica technique is a reliable and commonly accepted method for evaluating clinical accuracy.[18-24] In addition, it has been shown that the gap-width values obtained using this technique are similar to those obtained after luting with glass-ionomer cement.[19] Other techniques for determining gap widths of dental restorations are using a light

Table 2: *Summary of the Statistical Analysis*[a]

	Empress	Lava Oral	Lava Die Stone	iTero Oral	iTero Die Stone
A. Statistical analysis of all measuring points (n=342)					
Empress	–				
Lava Oral	***	–			
Lava Die Stone	NS	***	–		
iTero Oral	NS	***	NS	–	
iTero Die Stone	NS	**	NS	NS	–
B. Statistical analysis of the marginal measuring points (n=72)					
Empress	–				
Lava Oral	***	–			
Lava Die Stone	***	NS	–		
iTero Oral	**	NS	NS	–	
iTero Die Stone	***	NS	NS	NS	–
C. Statistical analysis of the proximal measuring points (n=72)					
Empress	–				
Lava Oral	NS	–			
Lava Die Stone	NS	NS	–		
iTero Oral	NS	NS	***	–	
iTero Die Stone	NS	NS	NS	***	–
D. Statistical analysis of the proximal-occlusal measuring points (n=72)					
Empress	–				
Lava Oral	NS	–			
Lava Die Stone	NS	NS	–		
iTero Oral	NS	**	NS	–	
iTero Die Stone	NS	NS	NS	NS	–
E. Statistical analysis of the occlusal measuring points (n=126)					
Empress	–				
Lava Oral	NS	–			
Lava Die Stone	***	***	–		
iTero Oral	***	***	NS	–	
iTero Die Stone	NS	NS	***	***	–

[a] *NS (not significant):* $p>0.01$; ** $p<0.01$; *** $p<0.001$ *(independent t-test and Bonferroni corrections).*

microscope directly after the restoration is placed on the prepared tooth or die stone[8] or luting the restorations on extracted teeth and then sectioning the crowns and teeth to determine the gap width.[8] When using light microscopy directly, only the marginal gap width can be determined,[8] and when using a number of prepared teeth and then sectioning the crowns/teeth, the gap width could be influenced by such factors as the design of the prepared teeth.[25-28] It should be noted, however, that with the replica technique used in the present study the crowns were filled with light-body impression material and held in place with finger pressure. This could have resulted in a variable force applied on the crowns, but the experimental setup imitates the way crowns are usually placed in patients.

In the present study all the crowns produced using scanning and CAD/CAM production, irrespective of type or whether they were made via die stone models or directly from the prepared tooth, exhibited marginal fit that was smaller than for the control. At the proximal and proximal-occlusal measuring points, no significant differences could be seen compared with the control, whereas occlusally the control exhibited a significantly smaller gap width than the iTero Oral and Lava Die Stone crowns. It should be noted that the controls were manually produced using the lost wax technique and die spacer, whereas Lava and iTero were digitally produced crowns. Because it is difficult to exactly reproduce the die spacer thickness and manually produce the crowns, this can be one of the reasons for

the discrepancy in gap widths and the error distribution observed between the manually and digitally produced crowns (Table 1).

However, it is still not known what gap distance is clinically optimal or what effect the selection of the location of the measuring points has. McLean and Fraunhofer[29] conclude that 120 μm is the maximum tolerable marginal opening, but previous studies reported mean gap widths ranging from 9 to 215 μm in dental restorations and a gap width of 420 μm at the marginal measuring point and 850 μm at the occlusal measuring point.[30-34] In a previous study of heat-pressed Empress crowns cemented to dies with zinc phosphate cement the marginal gap widths reported range between 26 and 548 μm.[35] In a recently presented study using Lava COS and Cerec intraoral scanning systems (Sirona, Bensheim, Germany), the gap widths determined using silicone replicas ranged between 0 and 552 μm.[36] That is, in the previous studies[30-36] there was a huge spread in the gap widths, which supports the findings in the present study. Thus, the results obtained in the present study are within the ranges reported in previous studies, although the values reported for the Empress crowns were obtained for crowns luted to dies with zinc phosphate cement and sectioned before measuring the gap width.[35] It should be noted, however, that conditions in the studies varied and there is no standardized method for determining the accuracy of dental crowns, making comparison difficult. Most of the studies referred to earlier[30-36] include restorations produced by means of conventional impression techniques or scanning from die stone models, and in a survey of the literature, only one article was found that addressed the internal and marginal fit of ceramic crowns made using in-office scanning of the preparation and subsequent CAD/CAM production.[36] One interesting finding in the previous study of Lava crowns[36] was that the values were almost identical to those obtained in the present study, with the exception of those for the marginal fit. Possible explanations for the divergent gap width determined in the three studies could be that the there were slight differences in convergence angle and/or in the finishing margin design of the prepared teeth and/or differences in preparation depth.[25-28]

Regarding the internal fit, internal discrepancies may weaken the ceramic crowns, so the marginal and internal fits are among the important criteria for the long-term success of ceramic restorations. For example, because the space between the tooth and the restoration exposes the luting material to the oral environment, a wide gap discrepancy may cause cement solubility, plaque accumulation, marginal leakage, and crown failures. Inadequate marginal fit is also said to be the most significant factor in the development of caries.[37] Today the recommended method for many types of all-ceramic restorations, such as IPS Empress, is adhesive luting using resin composite, which may be less soluble in oral fluids than conventional zinc phosphate and glass-ionomer cements.[38,39] However, it should be emphasized that restorations made of oxide ceramics may be luted with conventional zinc oxide phosphate or glass-ionomer cements.[40-42] In this context it should be noted that glass-ionomer and zinc oxide phosphate cements exhibit higher solubility than resin composite.[38,39] In addition, the clinical experience of those types of cements is mainly based on metal or porcelain-fused-to-metal restorations, and mean values for the marginal fit of such crowns have been reported to range between 31 and 72 μm.[43-45] More long-term follow-up studies are, therefore, necessary to determine the optimal internal and marginal fit of all-ceramic dental restorations.

CONCLUSION

All the crowns in the present study made via scanning and subsequent CAD/CAM production showed marginal fit that was less than that of the control. At the proximal and proximal-occlusal measuring points, however, there were no significant differences between the studied crowns and the control. Occlusally, the gap width for iTero Oral and Lava Die Stone was wider than for the control. Thus, based on the findings of the present study, an in-office digital-impression technique can be used in vitro to fabricate CAD/CAM ceramic single crowns with a marginal and internal accuracy that is on the same level as that of a conventional hot-pressed glass-ceramic crown. In the present study, slight differences could be seen between the two types of ceramic crowns studied with respect to internal fit. However, it should be noted that the gaps determined in the present study are almost two times higher than those reported for cast metal or porcelain-fused-to-metal crowns and that the present study is an in vitro comparison study. More studies are, therefore, necessary to validate the ability of in-office digital-impressions in clinical situations.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature

or kind in any product, service, and/or company that is presented in this article.

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Evaluation of Color Changes in the Vitapan Classical Shade Guide After Disinfection

SE Alshethri

Clinical Relevance

The shade guide must remain consistent in the shade-matching process. If there is a perceptible color change in the shade guide, the result will be inconsistent shade determination and, thus, an unacceptable restoration.

SUMMARY

The purpose of this study was to evaluate how one, two, and three years of simulated treatments affect the colors of Vitapan Classical Shade Guide tabs after being chemically disinfected. Ten shade tabs (one, control; nine, disinfection) were evaluated visually and by chromameter for color changes after disinfection. Results showed that 0.62 ΔE was found after three years of disinfection. The color changes in the shade guide tabs were perceptible or noticeable to the human eye in eight out of 45 shade tabs (17.8%) after two years and in 13 out of 45 shade tabs (28.9%) after three years of treatment. It was concluded that one shade guide should be retained as a control and periodically compared with the shade guide in use to determine when the shade tabs in use should be replaced or discarded.

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INTRODUCTION

The demand for esthetic dental restorations has grown significantly in recent years because of the development of new esthetic materials and increased patient awareness of and interest in improving their dental esthetics; this has also lead to an increased need for accurate shade-matching of the tooth being restored to the adjacent teeth.[1-3] In addition, the effects of advertisements in the popular media promoting the demand for various esthetic dental treatments cannot be ignored.[4] In a survey conducted in New Zealand, most participants reported an increased demand for tooth-whitening (77.8%) and veneers (54.8%) subsequent to the broadcasting of television programs promoting dental esthetics.[4] It has been reported, in a study of patients' perceptions of dental attractiveness, that one of the most important considerations in judging the attractiveness of a finished dental restoration is its shade,[5] which constituted 77% of the smile attractiveness variables for men and 61% for women; other variables, such as natural teeth, display, symmetry, and lip line, constituted the remaining 23% for men and 39% for women.[5] It has also been reported that any difference in the shade of the restoration compared with that of the natural tooth must be perceptible to the patient to be important.[6]

Shade selection is a critical and sometimes demanding step in the shade-matching process for a completed dental restoration. Inconsistencies in shade determination are the result of multiple factors, including the physiological and psychological color vision status of the person selecting the shade.[7,8] Environmental factors such as light conditions also play an important role in shade selection.[8-11]

Although there have been improvements in esthetic dental materials, intraoral shade matching has not changed appreciably since its initial use.[12,13] Visual shade selection based on a shade guide is the most frequently used method of color determination in dental practice.[14] It has been reported that a shade guide can reduce the precision of shade selection to approximately 48%, producing inconsistent results.[9,15]

Shade guides are usually used in dentistry to determine shade and evaluate tooth color in restorative and bleaching treatments. A shade guide is made of a set of shade tabs intended to cover the range of colors present in human teeth.[16]

The Vitapan Classical Shade Guide (Vita Zahnfabrik H. Rauter GmbH & Co KG, Bad Säckingen, Germany) is one of the guides used for shade selection in dentistry. It is important to note, however, that each Vita shade tab has cervical, body, and incisal colors over an opaque backing and is identified and named by the body shade.[17] The Vita shade range is divided into four groups designated by the letters A, B, C, or D. According to the manufacturer, these shades are reddish-brownish, reddish-yellowish, grayish, and reddish-gray, respectively. Shade tabs of a specific letter group have similar hue, and each hue group includes several tabs of increasing chroma and decreasing value, designated in numeric order (eg, A_1, A_2, A_3, $A_{3.5}$, and A_4).[18-20]

The CIE color system (CIELAB) was determined by the Commission Internationale de l'Eclairage (International Commission on Illumination) in 1978.[21] This method of color evaluation is related to human color perception according to three attributes or variables. The CIE system is becoming more widely used in dental research.[19,22] The three attributes are L*, a*, and b*, where L* is the lightness variable (increased L* value means a lighter shade) and a* and b* are chromaticity coordinates, which designate positions on the red/green and yellow/blue axes, respectively (+a = red, –a = green, while +b = yellow, –b = blue).[20,22]

Regardless of the shade guide used, it must remain consistent throughout the shade-matching process. It should not change color with routine clinical use. If a perceptible color change occurs in the shade guide, it could lead to inconsistent shade determination and unacceptable restorations. However, the shade guide must be disinfected after each use, in compliance with Occupational Safety and Health Administration (OSHA) requirements. It has been suggested that this procedure can cause color changes in the shade tabs, but few studies have investigated this. Disinfectants have been shown to alter the colors and surface characteristics of denture resins[23,24] and cast restorations.[25] In 1999, it was reported that there were no perceptible color changes in pressable ceramic and ceramometal porcelain after immersion in various surface disinfectants.[25] In 2007, in a study of how disinfectant use affects color changes of Vitapan Classical Shade Guide tabs, color changes (ΔE) of 2.5 and 1.8 were found after two and three years of simulated treatment, respectively.[26] Those authors also reported that there was a statistically significant increase in the value (L*) and chroma (C*) after two and three years of simulated treatments. This increase was not visually apparent to the investigators after three years of simulated treatments.[26] In 2008, it was reported that all disinfectant solutions used in the study produced perceptible changes in the colors of shade guide tabs (1.0 to 1.6 ΔE) with the immersion technique.[27]

The purpose of this study was to evaluate how one, two, and three years of simulated treatments affect the colors of Vitapan Classical Shade Guide tabs after being chemically disinfected.

MATERIALS AND METHODS

The Vitapan Classical Shade Guide was selected for use in this study because it is among the most widely used shade guides in dentistry. Shade tabs, labeled A_2, $A_{3.5}$, B_1, C_2, and D_3, were selected from 10 shade guides in order to include all basic hues and a wide range of saturations and values. The 10 shade guides were first evaluated visually for any perceptible or noticeable color differences among tabs within the same shade group. Two dentists who were not involved in the research methodology carried out the visual evaluation after passing an Ishihara color-blindness test. The shade tab name was blocked for all tabs used during the visual evaluation. Shade tabs were held over a light-blue card background and under a light-correcting device (Demetron Shade Light, Kerr, Orange, CA, USA) at the same angle

and distance, and any differences were recorded. A time restriction of 5-7 seconds was imposed for each assessment to minimize eye fatigue. A shade guide tab was ranked as visually different from the untreated tab at each evaluation period if the two visual evaluators simultaneously reported differences.

One tab of each shade group was randomly selected not to be disinfected and was used later for visual comparison with the treated tabs at each evaluation period (years 1, 2, and 3) by the same aforementioned visual evaluation method. The remaining nine tabs of each shade group were used for disinfectant application.

A ShadeEye NCC dental chromameter (SHOFU In., Kyoto, Japan) was used for measurements. The contact tip of the measuring device was placed on the body (middle third) of each tab, as recommended by the manufacturer, for all measurements. The plastic contact tip diameter is 5 mm, and the flash portion (actual measurement area) of the measuring device is 2 mm. A repositioning device was used to ensure that the same area was selected for each measurement (Figure 1). A light-blue card was used as background during all measurements. The chromameter was calibrated before measurements for each year, by means of the calibration cap supplied by the manufacturer. The chromameter measures the colors of shade tabs based on the CIE L* a* b* color space system. Total color differences or distances between two colors (ΔE) were calculated according to the following formula[21]:

$$\Delta E_{Lab\ *} = \left[(\Delta L\ *)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right]^{1/2}$$

Three measurements for each shade tab were recorded and averaged to set the baseline data (year zero) before treatments.

MinutenSpray disinfectant (Arabian Products Factory for Medical Disinfectant, Riyadh, Saudi Arabia) was used in the study because it is the disinfectant used in the clinics of the College of Dentistry of King Saud University, and it complies with the recommendations of the shade guide manufacturer for the disinfection of shade guide tabs. According to the Material Safety Data Sheet, MinutenSpray is a colorless or transparent alcohol-based surface disinfectant with a 70% ethanol and isopropanol mixture. The remaining nine shade tabs of each shade group were sprayed until wet with MinutenSpray disinfectant and allowed to sit for one minute, as recommended by the manufacturer. The shade tabs were then rinsed with water and wiped dry with 2×2 gauze. It was assumed that each individual shade guide would be used twice per day for five days per week for 48 weeks per year. Thus, each shade guide is used and disinfected at least 480 times per year. The process of disinfection was repeated 480 times to simulate one year's usage. Color measurements for one year were recorded following the same procedure as for baseline measurements. The process was repeated to simulate two and three years of use, and color measurements were recorded for two and three years.

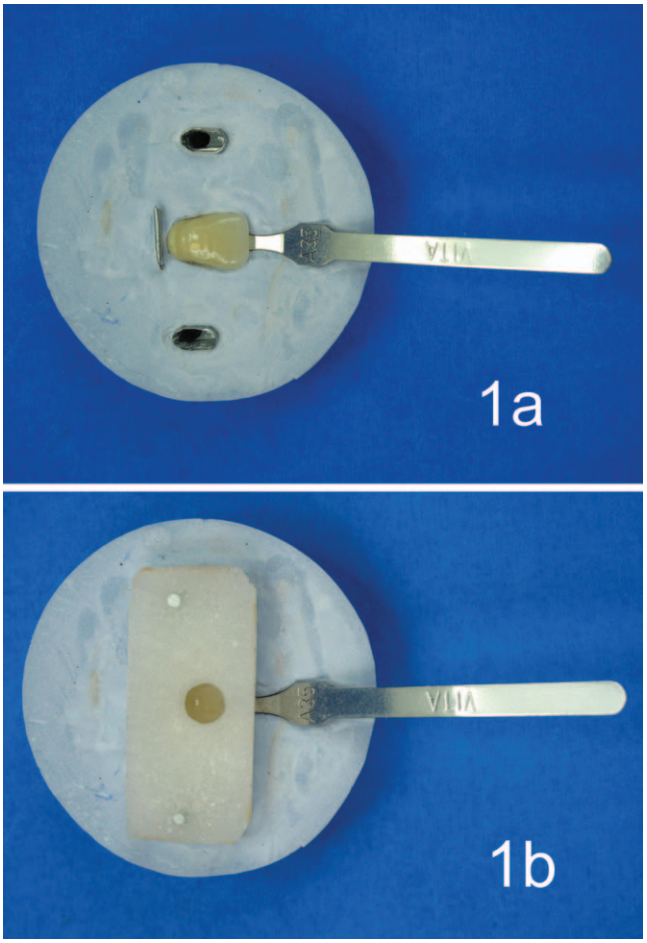

Figure 1. *Repositioning device used to ensure that the same area was selected for each measurement.*

Data were analyzed with SPSS Pc+ version 21.0 statistical software (IBM SPSS Statistics, Armonk, New York, USA). Descriptive statistics (mean, standard deviation, median, and interquartile range) were used to describe the symmetric and skewed variables. One-way analysis of variance (ANOVA) was used to compare the mean values of

Table 1: *Mean (SD) Values of Color Changes (ΔE) and Color Variable Changes (ΔL*, Δa*, and Δb*) of Shade Guides for Each Evaluation Period*

Evaluation Period	ΔL* Mean (SD)[a]	Δa* Mean (SD)[b]	Δb* Mean (SD)[c]	ΔE Mean (SD)[d]
Year 1	0.5215 (0.69)	0.0711 (0.09)	0.1585 (0.13)	0.6887 (0.57)
Year 2	0.4882 (0.67)	0.0859 (0.11)	0.1748 (0.16)	0.6129 (0.38)
Year 3	0.4778 (0.63)	–0.0348 (0.09)	0.1889 (0.13)	0.6171 (0.45)

[a] p=*0.94;*
[b] p<*0.0001;*
[c] p=*0.75 (Kruskal-Wallis test);*
[d] p>*0.05 (one-way ANOVA, F-test).*

symmetrical quantitative outcome variables in relation to the categorical study variables. The Kruskal-Wallis test was used to compare the mean ranks of skewed outcome variables in relation to the categorical study variables. Kappa statistics were computed to observe agreement between the categorical responses of the two examiners. A p-value of ≤ 0.05 was considered statistically significant.

RESULTS

The mean changes in color (ΔE) and color variables (ΔL*, Δa*, and Δb*) of shade guides for each evaluation period relative to baseline are shown in Table 1. Statistical analysis was done to test for significant association between changes in color and the evaluation period (one, two, and three years). Statistically significant differences were found only in the mean rank values of Δa*, in which the mean rank values of Δa* at year 3 were significantly lower than the values at year 1 and year 2, but no statistically significant differences were found between year 1 and year 2 values at $p<0.0001$ (Kruskal-Wallis test). Furthermore, there was no statistically significant difference in the mean rank values of other color variables (ΔL*and Δb*) at $p=0.94$ and $p=0.75$, respectively (Kruskal-Wallis test) and mean values of color change (ΔE) at $p>0.05$ (one-way ANOVA, F-test) among the three periods of evaluation.

It is important to examine the direction of the linear changes in the color variables (lightness and chromacity coordinates) for each evaluation period, because the overall changes in color (ΔE) are directionless. Table 2 shows the mean color components (L*, a*, and b*) for each evaluation period. Testing for significant association between changes in the color components (L*, a*, and b*) for each evaluation period in relation to baseline showed no statistically significant difference in the mean values of L* and b* at $p=0.81$ and $p=0.99$, respectively (one-way ANOVA, F-test) and in the mean rank values of a* at $p=0.76$ (Kruskal-Wallis test).

Table 3 shows the mean value of color changes (ΔE) in shade guide tab groups for each evaluation period. It shows that tab group B_1 had the lowest mean color changes at all evaluation periods, but the differences of ΔE_1, ΔE_2, and ΔE_3 were not statistically significant at $p=0.54$, $p=0.40$, and $p=0.86$, respectively (Kruskal-Wallis test).

When significant associations between changes in L*, a*, and b* and ΔL*, Δa*, and Δb* were examined for the three evaluation periods for each shade guide tab group (A_2, $A_{3.5}$, B_1, C_2, and D_3), only the mean rank values of Δa* of groups $A_{3.5}$, B_1, and D_3 were statistically significantly lower at year 3 compared with those of year 1 and year 2 at $p=0.47$, $p=0.2$, and $p=0.005$, respectively (Kruskal-Wallis test). There was no significant association between changes in other color variables at the three evaluation periods for each shade guide tab group.

It was decided in this study that a shade guide tab would be ranked as visually different from the untreated tab at each evaluation period if the two visual evaluators simultaneously reported a difference. Table 4 shows the examiners' visual evaluations for color differences when comparing treated shade guide tabs with untreated shade

Table 2: *Mean (SD) Values of Color Variables of Shade Guides (Lightness and Chromaticity Coordinates) for Each Evaluation Period*

Evaluation Period	L* Mean (SD)[a]	a* Mean (SD)[b]	b* Mean (SD)[c]
Baseline	69.1333 (3.0)	0.0511 (0.94)	13.1237 (3.12)
Year 1	69.6548 (2.9)	0.1222 (0.93)	13.2822 (3.10)
Year 2	69.6215 (2.9)	0.1370 (0.91)	13.2985 (3.10)
Year 3	69.6111 (2.9)	0.0163 (0.92)	13.3126 (3.10)

[a] p=*0.81 (one-way ANOVA, F-test);*
[b] p=*0.76 (Kruskal-Wallis test);*
[c] p=*0.99 (one-way ANOVA, F-test).*

Table 3: *Mean (SD) Values of Color Changes (ΔE) Among Shade Guide Tab Groups for Each Evaluation Period*

Shade Guide Tab Groups	ΔE_1 Mean (SD)[a]	ΔE_2 Mean (SD)[b]	ΔE_3 Mean (SD)[c]
A2	0.70 (0.43)	0.68 (0.42)	0.66 (0.37)
A3.5	0.86 (0.89)	0.72 (0.54)	0.65 (0.67)
B1	0.49 (0.33)	0.43 (0.26)	0.49 (0.32)
C2	0.71 (0.41)	0.66 (0.30)	0.62 (0.36)
D3	0.68 (0.57)	0.58 (0.31)	0.66 (0.50)

[a] p=*0.54;*
[b] p=*0.40;*
[c] p=*0.86 (Kruskal-Wallis test).*

guide tabs at all evaluation periods. It shows no visually perceptible color differences detected at baseline and year 1. The percentage of shade guide tabs ranked as different increased noticeably for years 2 and 3 (17.8% and 28.9%, respectively). The examiners' reliability for color difference comparison between treated and untreated shade guide tabs at all evaluation periods was analyzed (Table 5). It was found that the two examiners were consistent in their observations at the baseline and year 1 evaluation periods. There was a statistically significant agreement between the two examiners at years 2 and 3, when they compared treated shade guide tabs with untreated shade guide tabs for color differences. The overall agreement between the first examiner and second examiner for all four time periods was statistically significant (kappa= 0.45; $p<0.0001$). Table 6 shows the numbers and percentages of shade guide tabs visually evaluated by the two examiners as "different" among tab groups. This table shows that no visually perceptible color differences were detected in shade guide tab B_1 at all evaluation periods, while shade guide tab $A_{3.5}$ had higher

Table 4: *Examiners' Visual Evaluation of Color Differences, Comparing Treated Shade Guide Tabs With Control Shade Guide Tabs at All Evaluation Periods*

Time Period	No Difference Observed		Difference Observed[a]	
	Total	%	Total	%
Baseline	45	100.0	0	0
Year 1	45	100.0	0	0
Year 2	37	82.2	8	17.8
Year 3	32	71.1	13	28.9

[a] *The shade guide tab was ranked differently if both examiners detected differences simultaneously.*

Table 5: *Examiners' Reliability for Color Difference Comparisons Between Treated and Untreated Shade Guide Tabs at All Evaluation Periods*

Time Period	Examiner 1 vs Examiner2	
	Kappa Value	*p*-Value
Baseline[a]	–	–
Year 1[a]	–	–
Year 2	0.42	0.005
Year 3	0.54	< 0.0001

[a] *The two examiners were consistent in their observations.*

"different" scores (44.4% and 55.6% at years 2 and 3, respectively).

DISCUSSION

Surface disinfection is the most popular method used in dental clinics to disinfect and clean shade guides after use. This technique complies with regulations established by OSHA in 2005, which ranked shade guides as semi-critical items that can be disinfected with an intermediate-level disinfectant and approved by the Environmental Protection Agency. This technique is more convenient, and more aggressive methods can destroy some portions of shade guides.

Several studies have investigated how much of the color change detected by a chromameter or colorimeter is perceptible to the human eye. It was reported that one unit of ΔE was detectable by 50% of human observers in controlled conditions,[28] and color differences between 2.0 and 3.7 units were visually detectable under clinical conditions.[29] It was also reported that ΔE greater than 2.75 units is clinically unacceptable,[30,31] whereas other investigators have reported that ΔE greater than 3.0[32] or 3.3[33] is clinically unacceptable. All the afore-

Table 6: *Numbers and Percentages of Shade Guide Tabs Evaluated Visually by the Two Examiners as "Different" Among Shade Guide Tab Groups* [a]

Shade Guide Tabs	Baseline No (%) (n=9)	Year 1 No (%) (n=9)	Year 2 No (%) (n=9)	Year 3 No (%) (n=9)
A2	0	0	0	1 (11.1)
A3.5	0	0	4 (44.4)	5 (55.6)
B1	0	0	0	0
C2	0	0	2 (22.2)	3 (33.3)
D3	0	0	2 (22.2)	4 (44.4)

[a] *The shade guide tab was ranked differently if both examiners detected differences simultaneously.*

mentioned researchers were discussing two different points: visual perceptibility and the clinical acceptability of color difference. Douglas and Brewer[34] stated that the visual thresholds of perceptibility (mean 0.4 ΔE units) were significantly lower than the visual thresholds of acceptability (mean 1.7 ΔE units) for metal ceramic crowns differing in their chroma. They also reported that the visual thresholds of color acceptability were significantly lower for metal ceramic crowns differing in their red chroma or, in other words, differing in the a* coordinate (mean 1.1 ΔE units) compared with those of metal ceramic crowns differing in their yellow chroma, or b* coordinate (mean 2.1 ΔE units).[34] In this study and after 1440 cycles (1440 minutes) of disinfection, which simulated three years of clinical use, the mean ΔE for shade guide tabs was 0.62 units, which is almost one-third of that found by Pohjola and others,[26] who also used a spray technique to disinfect shade guide tabs. They found a statistically significant chromametric color change (1.8 ΔE units) after three years of simulated treatment, but they reported that this color change was not visually observable.[26] In the current study, the color change was observable in 17.8% of the sample (eight out of 45 shade tabs) after two years of treatment and in 28.9% of the sample (13 out of 45 shade tabs) after three years of treatment. The mean value of 0.62 ΔE units found in this study lies above the visual thresholds of perceptibility reported by Douglas and Brewer[34] (0.4 ΔE units). It is not known if this perceptible color difference in shade guide tabs will directly result in clinically acceptable restorations, as it is below the reported visual thresholds for acceptability (2.75 ΔE units), but it can be hypothesized that a perceptible color difference in shade guide can lead to incorrect shade selection, resulting in unacceptable restorations.

Another interesting point is that most of the chromametric color change took place after one year of treatment, after which no statistically significant difference occurred, but the color difference was visually perceptible after two and three years of treatment. It was found in this study that only the a* coordinate (red and green chroma) registered a significant change at year 3, while the L* and b* coordinates were consistent over years 1, 2, and 3 (Table 1). This concurs with the finding of Douglas and Brewer,[34] who reported that visual perception is more sensitive to objects differing in their red chroma. It is important to note that although the mean values are given in Tables 1, 2, and 3, the median values and the appropriate nonparametric statistical tests were used for statistical analysis; mean values are given for simplification of presentation.

When color change over time was examined relative to shade guide tab group, B_1 had the lowest mean chromametric color changes at all evaluation periods (Table 3), despite the fact that the differences in ΔE_1, ΔE_2, and ΔE_3 were not statistically significant, in agreement with the visual evaluation performed in this study. No visually perceptible color differences were detected in shade guide tab B_1 at all evaluation periods. It is also interesting to note that the visual detection of color change increased as chroma increased and value decreased, which can be explained by the shade tabs ranked as different (55.6% of $A_{3.5}$, 33.3% of C_2; and 44.4% of D_3). The $A_{3.5}$ tab has very high chroma and very low value, while C_2 is high in chroma and low in value. The D tab, as a group, has a red characteristic; meanwhile, the D_3 tab has very low value and high chroma. This could also be explained as was reported by Yab and others,[32] who reported that the difference in color-matching between visual evaluation and computerized colorimetry is shade dependent. They found that the differences occurred in very light or very dark shades. Generally, one shade guide should be retained as a control for comparison, and it should be periodically compared with the shade guide in use to determine when the guide in use should be replaced.

It is important to note that the overall agreement between the first and second examiners for all four time periods was nearly 50% for all observations, indicating that the two examiners did not agree in almost half of their observations, which supports the contention that color perception varies both between persons and within persons over time.[35-37]

Another concern about shade guide tabs is the fabrication of the tab itself. Some shade guide tabs are made of porcelain and prepared by a layering technique, as is the case of Vitapan Classical Shade Guide tabs; other guides use the technique of adding surface characteristics and staining, then glazing. Commercial shade guides are sometimes manufactured from plastics. The manufacturing technique used to fabricate shade guide tabs may affect any shade change caused by surface disinfection.[26] It is worth noting that most of the changes in overall color (ΔE) occurred after one year of treatment, whereas the changes after two and three years were

not statistically significant. Alamri[27] reported that immersion time has the most significant effect on color change and thus recommended a periodic check for color changes of shade guide tabs. In the present study, 1440 cycles (1440 minutes) of disinfection equaled a total of 24 hours, where there was direct contact between the disinfectant and the shade guide tabs, as they were sprayed wet. Recommendations from the American Dental Association Council on Scientific Affairs for the disinfection of prosthetic materials include spray or immersion with an appropriate material. The incorrect application of the disinfectant may affect the physical and/or mechanical properties of the material undergoing the disinfection process.[38,39] Agents containing organic solvent, such as alcohol, should be generally avoided, as they can cause degradation of some materials, such as plastics or resins.[24] The MinutenSpray disinfectant, as reported in its material safety and data sheet, is a highly alcohol-based disinfectant (a 70% ethanol and isopropanol mixture), which could account for the color changes after three years of simulated treatment. Pohjola and others[26] did not report whether the CaviCide disinfectant used in their study had low or high alcohol content.

Finally, any color change that occurred could be due to the effect of the chemical disinfectant or the wiping action during the disinfection process. In addition, surface residues from the disinfectant's components may cause some color change.

CONCLUSION

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

1. The mean chromametric color change (ΔE) found after three years of disinfection was 0.62 units. There was no statistically significant difference with evaluation periods.
2. The color changes in shade guide tabs were perceptible or noticeable to the human eye in eight out of 45 shade tabs (17.8%) after two years and in 13 out of 45 shade tabs (28.9%) after three years of treatment.
3. The color changes were perceptible or noticeable to the human eye in shade guide tabs with high chroma and low value ($A_{3.5}$, C_2, and D_3).
4. Generally, one shade guide should be retained as a control and periodically compared with the shade guide in use in order to determine when the shade tabs in use should be replaced or discarded.

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Conflict of Interest

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

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Polymerization Shrinkage of Different Types of Composite Resins and Microleakage With and Without Liner in Class II Cavities

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Clinical Relevance

The use of RMGIC liner with composite resin restorations reduces microleakage. The silorane-based composite showed lower volumetric polymerization shrinkage than methacrylate-based composites.

SUMMARY

Aim: To determine the volumetric polymerization shrinkage of four different types of composite resin and to evaluate microleakage of these materials in class II (MOD) cavities with and without a resin-modified glass ionomer cement (RMGIC) liner, in vitro.

Materials and Methods: One hundred twenty-eight extracted human upper premolar teeth were used. After the teeth were divided into eight groups (n=16), standardized MOD cavities were prepared. Then the teeth were restored with different resin composites (Filtek Supreme XT, Filtek P 60, Filtek Silorane, Filtek Z 250) with and without a RMGIC liner (Vitrebond). The restorations were finished and polished after 24 hours. Following thermocycling, the teeth were immersed in 0.5% basic fuchsin for 24 hours, then midsagitally sectioned in a mesiodistal plane and examined for microleakage using a stereomicroscope. The volumetric polymerization shrinkage of materials was measured using a video imaging device (Acuvol, Bisco, Inc). Data were statistically analyzed with Kruskal-Wallis and Mann-Whitney U-tests.

Results: All teeth showed microleakage, but placement of RMGIC liner reduced microleakage. No statistically significant differences were found in microleakage between the teeth restored without RMGIC liner ($p>0.05$). Filtek Silorane showed significantly less volumetric polymerization shrinkage than the methacrylate-based composite resins ($p<0.05$).

Conclusion: The use of RMGIC liner with both silorane- and methacrylate-based composite resin restorations resulted in reduced microleakage. The volumetric polymerization

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shrinkage was least with the silorane-based composite.

INTRODUCTION

Interest in esthetic dentistry has resulted in composite resin restorations being increasingly used not only as a replacement material for failed or unesthetic amalgams but also as the first choice to restore posterior teeth.[1] Mechanical performance, wear resistance, and esthetic potentials of composite resins have significantly improved over the past few years. On the other hand, polymerization shrinkage of composite resins remains a challenge and still imposes limitations in the application of direct techniques.[2]

Polymerization shrinkage causes detachment of the enamel margins and/or can form gaps that result in marginal microleakage that allows the passage of bacteria, fluids, molecules, or ions between the cavity surface and composite resin.[3] Microleakage of posterior composite restorations is a matter of concern to the clinician, especially at the margins of the proximal box of class II cavities, as it leads to staining at the margins of restorations, recurrent caries, hypersensitivity, and pulp pathology.[4]

Packable composites are claimed to eliminate some of these shortcomings. Increased filler loading of these materials gives them a different consistency compared with hybrid composites. They are recommended for use in stress-bearing posterior regions and offer improved handling properties, such as increased sculptability and handling characteristics similar to amalgam restorations, and produce acceptable interproximal contacts. These allow them to be safely and successfully used in class II restorations.[5,6]

Recently, because of an increasing demand for a universal restorative material indicated for all types of direct restorations, including posterior teeth, a new category of resin composite was developed named nanofilled composites. Short-term (one-year) clinical studies have revealed that nanocomposites show high translucency, high polish, and polish retention similar to those of microfilled composites while maintaining physical properties and wear resistance equivalent to those of several hybrid composites[7] and exhibit sufficient compressive strength and wear resistance to justify their use in high stress-bearing areas, such as the occlusal surfaces of posterior teeth.[8]

To overcome the problem of polymerization shrinkage, extensive efforts have been invested over the years to develop low-shrinkage composite resins. As a result, dental composite research has focused on the use of ring-opening systems like oxirane-based resins cured under visible light conditions. Weinmann and others[9] described the synthesis of a new monomer system named silorane obtained from the reaction of oxirane and siloxane molecules. The novel silorane-based resin is claimed to have combined the two key advantages of the individual components: low polymerization shrinkage due to the ring-opening oxirane monomer and increased hydrophobicity due to the presence of the siloxane species.

Reduction of polymerization shrinkage can be achieved by reducing the mass of the restorative material with the use of liners. Use of glass ionomer liners under composite resins has reduced the stresses generated at the cavity walls during polymerization.[10] Resin-modified glass ionomer cements (RMGICs) might be a better material of choice for the liner because of their higher mechanical strength compared to the conventional material and their ability to set on command. They are also known to be less technique sensitive. Furthermore, RMGICs have been recommended as liners under resin composites to reduce the amount of polymerization shrinkage, potential microleakage, and secondary caries.[11-13]

The current study aimed to assess the volumetric polymerization shrinkage of four different composite resins and their microleakage in MOD cavities, with and without an RMGIC liner, *in vitro*. The tested hypothesis of the study was that placement of RMGIC liner under the composite resin restorations results in reduced microleakage, and silorane-based composite resin shows lower volumetric polymerization shrinkage than methacrylate-based composite resins.

MATERIALS AND METHODS

Selection of Teeth

One hundred twenty-eight upper premolar teeth, extracted for orthodontic purposes, were selected. The teeth were free from caries, hypoplastic defects, and cracks on visual examination. The teeth had been stored in distilled water for a maximum of three months prior to use. Using a hand scaler, calculus deposits were carefully removed and the teeth were stored in water at room temperature (23 ± 1°C) except when aspects of the experimental procedure required isolation from moisture.

Table 1: *Composite Resins Used in the Current Study*

Product	Batch Number	Ingredient	Manufacturer
Filtek Supreme XT Nanofilled Composite Resin	20080117	Inorganic fillers (59.5%), bis-GMA, UDMA, bis-EMA, TEGDMA, silica nanofillers (5-7 nm), zirconia/silica nanoclusters (0.6-1.4 μm)	3M ESPE
Filtek P60 Packable Composite Resin	20081004	Inorganic fillers (61%), bis-GMA, UDMA, bis-EMA, zirconia/silica nanofillers (0.01-3.5 μm)	3M ESPE
Filtek Z250 Hybrid Composite Resin	20090406	Inorganic fillers (60%), bis-GMA, UDMA, bis-EMA, zirconia/silica nanofillers (0.01-3.5 μm)	3M ESPE
Filtek Silorane Low Shrink Composite Resin	N105399	Inorganic fillers (55%), hydrophobic resin matrix	3M ESPE

Cavity Preparation

The teeth were divided into eight groups of 16 teeth, and standardized large MOD cavities were prepared whereby the bucco-palatal width (BPW) of the proximal box of each cavity was prepared to two-thirds of the BPW of the tooth and the occlusal isthmus was prepared to half the BPW. The cavity depth at the occlusal isthmus was also standardized to 3.5 mm from the tip of palatal cusp and 1 mm above the cementoenamel junction at the cervical aspect of the proximal boxes. The cavosurface margins were prepared at 90 degrees, and all internal line angles were rounded. The facial and lingual walls of the cavity were also prepared parallel to each other in accordance with a previously reported procedure.[11,14] Diamond fissure burs (DIATECH, Heerbrugg, Switzerland) were used in a high-speed hand piece with water coolant and changed after every five cavity preparations.

Restorative Procedures

All the teeth were restored with the same manufacturer's composite resin and its associated bonding system in accordance with the manufacturer's instructions and light cured by light-emitting diode (Radi Plus, SDI, Victoria, Australia). The cavity preparations and restorative procedures were conducted by the same dentist. The composite resins used in this study are listed in Table 1.

Group 1: Etching of enamel and dentin was performed with 35% phosphoric acid (Scotchbond, 3M ESPE, St Paul, MN, USA) according to the manufacturer's instructions. Two consecutive coats of Adper Single Bond 2 (3M ESPE) were applied using a microbrush for 15 seconds, followed by gentle air drying and then light curing for 10 seconds. Filtek Supreme XT (Shade A3B) was placed and light cured for 20 seconds.
Group 2: Teeth were restored with Filtek P60 (Shade A3) as previously described.
Group 3: Teeth were restored with Filtek Z250 (Shade A3) as previously described.
Group 4: Silorane Adhesive System primer (3M ESPE) was applied using a microbrush for 15 seconds, followed by gentle air drying and then light curing for 10 seconds. After that the Silorane Adhesive system bond (3M ESPE) was applied, followed by a gentle stream of air, and light cured for 10 seconds. Filtek Silorane (Shade A3) was placed and light cured for 20 seconds.
Group 5: Teeth were lined with a thin layer of Vitrebond (3M ESPE) on the pulpal and axial walls with approximately 1-mm thickness and light cured for 30 seconds. Then the teeth were restored with Filtek Supreme XT (Shade A3B) using the same method as for group 1.
Group 6: Teeth were restored with Filtek P60 (Shade A3) using the same method as for group 5.
Group 7: Teeth were restored with Filtek Z250 (Shade A3) using the same method as for group 5.
Group 8: Vitrebond was applied as previously described and teeth were restored with Filtek Silorane (Shade A3) using the same method as for group 4.

Eight nominally triangular increments of approximately 2-mm thickness were used to restore the teeth, three for each proximal box and two for the occlusal surface.[11,14,15] Each increment was cured for 20 seconds as per the manufacturer's instructions. The occlusal aspect of the restorations was carved to approximate the normal occlusal anatomy of an upper premolar tooth. Each tooth was restored by placing a transparent matrix (Auto matrix II, combination matrix intro-kit, Dentsply, Petrópolis, Brazil). The matrix band was held by finger pressure against the gingival margin of the cavity so that the preparations could not be overfilled at the gingival margin.[16] This also allowed the light to be directed only in an apical direction when curing the composite resin. The matrix band was removed after the restorations were completed. The restored teeth were finished with Sof-Lex Finishing discs (3M

ESPE) in a slow hand piece and 15-μm-grit finishing diamond burs (DIATECH) used in an air turbine hand piece under water coolant.

Thermocycling and Gingival Marginal Microleakage Evaluation

Root apices were sealed with a composite resin and polymerized for 20 seconds. All tooth surfaces were sealed with nail varnish, with the exception of a 1-mm band around the margins of each restoration, and the teeth replaced in water when the varnish dried. The specimens were thermocycled between two water baths maintained at 55 ± 1°C and 5 ± 1°C so that the restored teeth were submerged for 60 seconds with a 30-second transfer from water bath to water bath for the time equivalent for 1000 cycles.[17] The teeth were then immersed in 0.5% basic fuchsin dye for 24 hours, and a vertical section was made through each restored tooth midsagitally in a mesiodistal plane using a low-speed diamond blade (IsoMet, Buehler Ltd, Lake Bluff, IL, USA). Sectioned restorations were examined under a stereomicroscope (Olympus SZ 61, Olympus Corporation, Japan) at 40× magnification, and the extent of the gingival marginal microleakage was recorded. Accordingly, the degree of gingival margin microleakage was scored[18] as follows (Figure 1): 0 = no evidence of dye penetration, 1 = superficial penetration not beyond the dentinoenamel junction (DEJ), 2 = penetration beyond the DEJ but limited to two-thirds of the gingival wall length, and 3 = penetration beyond two-thirds of the gingival wall length. Two examiners scored the restorations independently, any discrepancies between them were reevaluated by both, and a consensus was reached. For each restoration, one score, by convention the worst, was used for the analyses.

Volumetric Polymerization Shrinkage Determination

Volumetric polymerization shrinkage was measured using a video imaging device (AcuVol, BİSCO, Inc, Schaumburg, IL, USA). This device has been described by Sharp and others[19] and provides data comparable to those obtained using a mercury dilatometer. Small semispherical samples of composites were manually formed and placed on the rotating pedestal of the AcuVol, in equal amounts, and left undisturbed for 10 minutes to take their final shape (n=16). After 10 minutes, they were light cured following the manufacturer's instructions. Shrinkage values were recorded continuously for 10 minutes after curing, and the final shrinkage value was recorded as percent shrinkage. Five values were taken for each material, and the mean values were calculated and used for evaluation.

Statistical Analysis

The microleakage scores and volumetric polymerization shrinkage data were statistically analyzed using a nonparametric one-way analysis of variance (Kruskal-Wallis) test followed by paired group comparisons using a Mann-Whitney U-test. Statistical significance was set in advance at the 0.05 confidence level. All data were analyzed by means of SPSS 11.5 for Windows (SPSS Inc, Chicago, IL, USA).

RESULTS

The microleakage scores for the different composite resins with and without RMGIC liner are shown in Table 2. None of the groups showed complete prevention of dye penetration. Group 7 showed the best marginal sealing. Although groups 5, 6, and 8 showed similar results ($p>0.05$), they were superior to groups 1 to 4 ($p<0.05$). No statistically significant differences were found in microleakage between the teeth restored without RMGIC liner ($p>0.05$).

When comparing each group individually, microleakage was lower in the groups in which RMGIC liner had been used. Filtek Supreme XT and Filtek Z250 with RMGIC liners had significantly less microleakage than those without liners ($p<0.05$).

The mean volumetric polymerization shrinkage values for the composite resins used in this study are shown in Table 3. The rate of shrinkage was least with Filtek Silorane and highest with Filtek P60, and these values were significantly different than those of all the other materials ($p<0.05$).

DISCUSSION

Microleakage is one of the most common problems of composite resin restorations, especially at the margins of the proximal box of class II cavities. Microleakage may result from many factors, including adaptation of resin material to the tooth surface, the adhesive system used, and polymerization shrinkage of materials used.[20,21]

Dye penetration is one of the most frequently used methods to evaluate microleakage.[21,22] In the current study, a dye penetration test was used because it is simple and relatively cheap and provides quantitative and comparable results. This method does have some limitations, however, such as

Table 2: *Gingival Microleakage Scores of Groups Evaluated*

Groups	n	Microleakage Scores				Mean ± SD
		0 (%)	1 (%)	2 (%)	3 (%)	
Group 1 (Filtek Supreme XT)	16	0 (0)	11 (68.75)	3 (18.75)	2 (12.5)	1.44 ± 0.72
Group 2 (Filtek P60)	16	1 (6.25)	8 (50)	4 (25)	3 (18.75)	1.56 ± 0.89
Group 3 (Filtek Z250)	16	1 (6.25)	9 (56.25)	5 (31.25)	1 (6.25)	1.38 ± 0.71
Group 4 (Filtek Silorane)	16	2 (12.5)	9 (56.25)	3 (18.75)	2 (12.5)	1.31 ± 0.87
Group 5 (Vitrebond + Filtek Supreme XT)	16	6 (37.5)	8 (50)	2 (12.5)	0 (0)	0.75 ± 0.68
Group 6 (Vitrebond + Filtek P60)	16	3 (18.75)	11 (68.75)	2 (12.5)	0 (0)	0.94 ± 0.57
Group 7 (Vitrebond + Filtek Z250)	16	6 (37.5)	10 (62.5)	0 (0)	0 (0)	0.63 ± 0.50
Group 8 (Vitrebond + Filtek Silorane)	16	3 (18.75)	9 (56.25)	3 (18.75)	1 (6.25)	1.13 ± 0.80

subjectivity of reading and high diffusability of dyes due to their low molecular weight.[23]

In vitro evaluation of restorative materials fails to simulate the intraoral thermal changes during eating and drinking. Thermocycling is a widely acceptable method used in microleakage studies to simulate the effects that restorations are subjected to in the mouth.[20,24,25] Some researchers, however, consider it a questionable method since the temperatures used may not be the real temperatures of hot and cold beverage tolerated by patients.[26-28]

The results of the present study highlighted that microleakage was similar between the teeth restored without RMGIC liner. In agreement with this, Hardan and others[29] and Sadeghi[30] have reported that Filtek Supreme and Filtek Z250 showed similar microleakage in class II cavities. The results of our study also agree with those reported by Fleming and others[31] and Tredwin and others,[16] who found similar marginal adaptation and microleakage using Filtek P60 and Filtek Z250 in class II cavities.

In the current study, when compared with methacrylate-based composite resins, Filtek Silorane did not significantly reduce the amount of microleakage, contrary to other studies.[32-34] In accordance with our results, Ernst and others[35] also reported similar microleakage results with silorane- and methacrylate-based composite resins.

The methods utilized in the current study during composite resin placement replicated those commonly used in clinical practice. Different microleakage scores were obtained in the current study than other reported scores, probably because of differences in experimental design.

Several methods have been developed to improve marginal sealing and reduce microleakage. The use of RMGIC liners under composite resin restorations is one of these methods because of the stress-buffering capacity of these materials to resist the debonding stress during polymerization contraction. The use of liners may also reduce the effects of C-factor (the ratio of bonded to unbonded surfaces) and lower the internal stresses within the placed restoration. However, the benefit of using RMGIC liners under composite resin restorations for reducing polymerization shrinkage and microleakage is still controversial. While some researchers[36,37] have reported that using RMGIC liners failed to reduce gap formation and marginal sealing, some have reported significant effects of RMGIC liners in reducing microleakage.[38,39] In the current study, RMGIC liner usage resulted in less gingival microleakage regardless of the composite resin used.

The polymerization reaction was accompanied by a dimensional change that resulted in shrinkage for all composite resins used in the current study. As expected, Filtek Silorane shrank less than the methacrylate-based composite resins. Cationic ring opening polymerization of the cycloaliphatic oxirane moieties is the reason for silorane-based composites' low shrinkage and low polymerization stress. The cationic cure starts with the initiation process of an acidic cation, which opens the oxirane ring and generates a new acidic center, a carbocation. After the addition of an oxirane monomer, the epoxy ring is opened to form a chain, or, in the case of two- or multifunctional monomers, a network is formed.[9]

Table 3: *Mean Shrinkage and Standard Deviation (SD) of the Restorative Materials Evaluated*

Material	Mean Shrinkage ± SD
Filtek Supreme XT	1.75^{a} ± 0.06
Filtek P60	1.97^{b} ± 0.02
Filtek Z250	1.75^{a} ± 0.04
Filtek Silorane	0.88^{c} ± 0.04

Mean values exhibiting different superscript letters were significantly different.

Despite lower volumetric polymerization shrinkage values, Filtek Silorane did not yield the lowest scores for dye penetration. If only contraction stresses determined the extent of microleakage, Filtek Silorane was expected to show the best sealing ability. This result could be related to the adhesive systems used. There are conflicting results about the effectiveness of different adhesive systems on microleakage. While some authors have reported that etch-and-rinse and self-etch adhesives produce similar results in terms of marginal adaptation and microleakage,[40,41] some of them reported higher dye penetration scores with the use of self-etch adhesives.[42-44] In the current study, the same manufacturer's composite resin and its associated bonding system was used in accordance with the manufacturer's instructions. A self-etch adhesive system (Silorane System Adhesive) and an etch-and-rinse adhesive system (Adper Single Bond 2) was used with Filtek Silorane and the methacrylate-based composite resins, respectively.

CONCLUSION

Within the limitations of this *in vitro* study, the hypothesis was accepted in that the use of RMGIC liner as the first gingival increment of class II restorations with both silorane- and methacrylate-based composite resin restorations resulted in reduced microleakage. The volumetric polymerization shrinkage was least with the silorane-based composite. However, further clinical research is needed to support these findings, as the volumetric polymerization shrinkage of the restorative materials was evaluated without cavity factor and bonding influences.

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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Award

American Academy of Gold Foil Operators' 2013 Distinguished Member of the Year Award

It gives me great pleasure to present the distinguished member award to a most deserving member. This year's recipient is Dr. David William Thorburn. His Patients call him Dr. Thorburn, his friends call him Dave, his climbing buddies call him Hey wait up, and there is a rumor that the Vancouver music underground calls him Disco Dave. He is indeed a man of many talents. His artistic ability shows in the fabulous art drawings he has completed. In fact he was so prolific that at one point he had an art studio in Vancouver filled with his own art. Then again, he also built up custom race engines for Datson race cars, so prized, that he had one of them stolen right in front of his office. When it comes to mountain climbing, Dave is really out there. He has climbed in Canada, the United States, and Europe, from Mt. Assiniboine, and Mt. Rainier to the Matterhorn. Of course he is also an avid skier, traveling all the way to the mountains in South America to ski during our summertime. However, it is Dr. Thorburn's dental talents that we are honoring today.

Dave graduated from the University of British Columbia School of Dentistry in 1981. He immediately went to work treating patients in Fort Nelson, B.C., White Rock, B.C., Surrey B.C., Sardis B.C., and Vancouver B.C., before opening his own dental practice in Vancouver in 1989. He joined the Walter K. Sproule Gold Foil Study Club, and the Vancouver Crown and Bridge Study Club to hone his operating skills to the point where his name has become synonymous with exceptionally fine quality restorative dentistry. His patients are very fortunate to have him as their dentist.

Dave served on the executive council of this Gold Foil Academy from 2001 through 2003, served as vice president in 2004, president elect in 2005 and President of the American Academy of Gold Foil Operators in 2006.

Never selfish, but always trying to help other dentists at his own expense, David has made a special effort to pass on the skills he has perfected to dental students and the general dental community. He has been a clinical instructor for the Vancouver Community College Dental Hygiene Department, a clinical instructor for the 3rd year students at the University of British Columbia School of Dentistry, a clinical instructor for the 4th year students at the University of British Columbia School of Dentistry. Dave also presented a two-day Gold Foil course in Corona, Italy and taught the Vancouver Gold Foil Seminar, a one-week course limited to practicing dentists, for 15 years. In addition, he provided hundreds of patients for the course from his own private practice. He has given many lectures and presentations to various dental organizations, but it is his clinical demonstrations on live patients that are unsurpassed, providing treatment that will last a lifetime. It has been shear enjoyment to watch him create dental works of perfection for the patient, that, as the observer, I could take pleasure in, as you would enjoy any fine piece of art. Please rise and join me in congratulating Dr. David William Thorburn, our Distinguished Member.

Submitted by Dr Richard D Tucker
Presented at the 2013 AAGFO Meeting
Lincoln, NE 26 October 2013

The Award of Excellence

The Award of Excellence is given to an individual who has made great contributions and/or reached a high level of technical skill in the field of Operative Dentistry. The 2014 Award of Excellence goes to a very deserving recipient Dr. Richard D. Tucker. He has contributed immensely to furthering the cause of exquisite, refined operative procedures by simplifying many of the steps in fabricating direct and cast gold restorations. He has made cast and direct gold restorations practical and economical for patients to afford, desire and appreciate. He has also developed techniques to make gold restorations extremely esthetic which in todays world seems to be the driving force. Many of you who have seen Dick operate at various national and local meetings, can attest to the fine results that he is able to achieve in a timely manner.

He has furthermore developed practical methods to move teeth with a suckdown appliance, to practically make stents for simple implant placements and an extrusion technique for restoring fractured teeth.

Dick was born in 1949 to Dr Richard V. and Elaine Tucker in Bellingham, WA and spent his youth in Ferndale, WA graduating from Ferndale High School in 1967. He attended Western Washington University where he graduated with a degree in Chemistry in 1971 and an Education degree in 1972. He was accepted that same year to the University of Washington-School of Dentistry Class of 1976.

Upon graduating, passing the state boards and taking a two week foil course, he started his private practice in Bellingham, Washington that Fall. He also in the same year joined the George Ellsperman Gold Foil Study Club, which he has been a member of for many years before becoming the mentor. He is or has been a member of four other study groups during his time in private practice.

Dick is a member of the ADA and its constituent societies as well as the American Academy of Gold Foil Operators, Academy of Operative Dentistry, Restorative Academy, Associated Ferrier Study Clubs, and CAIC, as well as a Fellow in the ACD and ICD. He has also held offices in his local study clubs and dental society, the Academy of Gold Foil Operators and as a councilor for the AOD.

This year's recipient has given over fifty lectures on both direct and cast gold in the United States, Canada, Mexico and Germany. He has presented more than thirty-five clinical demonstrations and table clinics on gold foil and cast gold at meetings in the USA, Canada and Germany. He has received the Clinician of the Year Award from the American Academy of Gold Foil Operators and the Distinguished Member Award in 2011. Dick has done several research projects on the hardness and expansion of die stones and the wear resistance of gold foil versus platinized gold foil.

He is currently mentoring two operating cast gold study clubs, one in Seattle and the other in Vancouver, Canada as well as two operating gold foil in Vancouver, BC. He has conducted a yearly undergraduate elective cast gold course at the University of Washington - School of Dentistry for the past fifteen years. He is currently the primary instructor for the Tucker Institute Course held each year in June in Seattle at the University of Washington - School of Dentistry.

Dick's other passions are mountain climbing, skiing, fishing and sailing. He has summited five of the highest peaks on five continents. He is very active in the National Ski Patrol at Mt Baker Ski Area both in instructing classes in First Aid and Avalanche Training as well as on-the-hill patrolling. He has twice raced his dad's former sailboat from Victoria in British Columbia, Canada to the island of Maui in Hawaii.

He has also been involved with the Big Brothers and Sisters Organization and has actively been helping and mentoring a young man for the past

three years. He is mentoring him in areas that will be useful to him presently and also later in life.

Dick and his lovely wife Christina have two grown children Christopher and Laura. The family will be expanding soon as they will be gaining a son-in-law in August of this year. They also have a lovely home overlooking Chuckanut Bay just south of Bellingham where the evening sunsets and the crab feeds are spectacular.

It is my distinct honor at this time to present the 2014 Award of Excellence to my good friend and colleague Dr. Richard D. Tucker.

Submitted by Dr Warren K Johnson, DDS
Presented at the 2014 AOD Meeting
Chicago, Illinois February 21, 2014

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Faculty Positions

INDIANA UNIVERSITY SCHOOL OF DENTISTRY
Chair of the Newly Formed Department Of Cariology and Operative Dentistry

Indiana University School of Dentistry invites applications for the position of Chair of the newly established Department of Cariology and Operative Dentistry. The department includes the disciplines of Operative Dentistry, Preventive Dentistry and Cariology. The successful candidate will have the title of the Indiana Dental Association Endowed Chair (of Restorative Dentistry).

The administrative expectations of the Chair include leadership of the department of faculty, staff, and students, programmatic oversight in both pre-doctoral and post-doctoral education, strategic recruitment of outstanding faculty, fiscal responsibilities and planning. The successful candidate should have academic leadership experience, a national reputation in disciplinary excellence, documented success in leading strategic initiatives and demonstrate a strong record of scholarship and research.

The Endowed Chair responsibilities include collaboration with the Indiana Dental Association leadership, state-wide presentations in clinical dentistry and interact with and serve as a clinical resource for the Indiana dental community.

Qualified applicants should be eligible for tenure at the rank of full professor at Indiana University Purdue University Indianapolis. Minimum credentials include both a DDS or DMD from a CODA accredited program or equivalent with preferred credentials to include formal advanced education and credentials in preventive dentistry, operative dentistry, public health, or a related field. Candidates must be eligible for licensure in the State of Indiana. Rank and salary will be commensurate with the candidate's qualifications, experience and credentials.

Please send a complete electronic application with the following documents:

- Signed letter of intent
- Complete curriculum vitae
- Names of three professional references (letters will be required prior to an interview).
- For tenure, three additional professional references of persons who will be able to provide an objective assessment of the candidate's academic contributions and scholarship will be required (letters will be required prior to an interview).
- Documents should be sent to dsexecaf@iupui.edu with the subject line reference posting #IN-DENT 14002.

Review of applicants will begin immediately with an anticipated appointment start date of July 1, 2014. Indiana University is an equal employment opportunity/equal access/affirmative action employer and a provider of ADA services.

Indiana University School of Dentistry is located on the IUPUI campus near the heart of downtown Indianapolis. The School of Dentistry is the only dental school in the Hoosier state and educates 80% of the dentists practicing in Indiana and offers an extraordinary learning environment in which teaching, research and community service are uniquely combined to prepare tomorrow's dental professionals.

Indiana University School of Dentistry http://www.iusd.iupui.edu/

DEPARTMENT OF CARIOLOGY, RESTORATIVE SCIENCES AND ENDODONTICS
CRSE Restorative Faculty Search

The University of Michigan invites applications and nominations for a full-time clinical or tenure track faculty member at the level of Assistant or Associate Professor in the Division of Restorative Dentistry. The School and Department are fully engaged in refining a new model for dental education that includes evidence-based dentistry and inter-professional education. At the same time, the Department is involved in a broad range of areas including educational research, cariology, tissue engineering, cancer biology, materials science, clinical research, and public health and policy research. The successful candidate for the clinical track should demonstrate sound preparation for teaching, potential for clinical scholarly activity, and clinical experience. Candidates for clinical track should have a DDS/DMD degree and the ability to be licensed in Michigan. A MS degree in a field relevant to the position is

desirable, but not necessary. For the tenure track, candidates who demonstrate a record of ongoing scholarly activity and strong potential for obtaining extramural research funding are encouraged to apply. Candidates for tenure track should have a DDS/DMD degree and/or PhD degree (or equivalent). The department has an active mentorship program for both clinical and tenure-track faculty and will provide ample opportunities for clinic-based patient care and development of collaborative research programs. Opportunities are available for participation in the School of Dentistry Faculty Practice. Salary and level of academic appointment will be commensurate with qualifications and experience.

A State of Michigan Clinically Limited Academic License may be available for qualified candidates. Applicants should submit curriculum vitae, statement of interests and goals, and the names of three references via the secure website: (**http://facultyrecruiting.dent.umich.edu**). Applications will be accepted and evaluated on an ongoing basis until the position is filled. Further information may be obtained by consulting the departmental website at: http://dent.umich.edu/about-school/department/crse/cariology-restorative-sciences-and-endodontics-crse.

Questions: Dr. Tilly Peters, Search Committee Chair, c/o Jean Klark at jklark@umich.edu

The University of Michigan is an Affirmative Action\Non-Discriminatory employer.

SOUTHERN ILLINOIS UNIVERSITY SCHOOL OF DENTAL MEDICINE

Southern Illinois University, School of Dental Medicine is seeking applications for a full-time tenure track faculty position at the assistant/associate professor level in the Department of Restorative Dentistry, Section of Operative Dentistry. Responsibilities include didactic, pre-clinical, and clinical teaching in Operative Dentistry at the pre-doctoral level, with some teaching at advanced levels and service to the University. Independent research and scholarly activities are also expected. Excellence in teaching is integral to the mission of Southern Illinois University and the School of Dental Medicine. Applicants must have a DDS/DMD degree from an institution accredited by the Commission on Dental Accreditation or equivalent and be eligible for licensure in compliance with the Illinois Dental Practice Act. Candidates should have experience in teaching Pre-doctoral students in General Dentistry and expertise in Operative Dentistry or Advanced Education in General Dentistry training. Expertise in evidence-based teaching and dental materials sciences are highly desirable. Salary and rank are commensurate with qualifications and experience. Opportunity for extramural private practice is available. Review of applications will begin immediately and continue until the position is filled. Submit a letter of intent, curriculum vitae, and three letters of reference to Dr. Bruce Rotter, Dean, Southern Illinois University School of Dental Medicine, 2800 College Avenue, Alton, IL 62002. SIU-SDM is an EEO/AA employer. Women and minorities are encouraged to apply. SIUE is a state university - benefits under state sponsored plans will not be available to holders of F-1 or J-1 visas.

Erratum

Reference 13 in: S Ardu, O Duc, I Krejci, and R Perroud (*2013*) Amelogenesis Imperfecta: A Conservative and Progressive Adhesive Treatment Concept. Operative Dentistry: May/June 2013, Vol. 38, No. 3, pp. 235-241. is missing the title. The correct reference should read:

13. Silva NR, Thompson VP, Valverde GB, Coelho PG, Powers JM, Farah JW, & Esquivel-Upshaw J (2011) Comparative reliability analyses of zirconium oxides and lithium disilicate restorations in vitro and in vivo *Journal of the American Dental Association* **142(Supplement 2)**:4S-9S.

We apologize for this error.

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Retentiveness of Metal Coping Luted to Teeth of Uremic Patients Undergoing Hemodialysis Using Five Different Luting Cements

MH Ghazy • MM Aboumadina • SH Mahmoud

Clinical Relevance

Dental changes have been reported to the teeth of uremic patients that consequently affect bonding of that tissue to fixed restorations.

SUMMARY

Objectives: This study aimed to assess the retention of metal copings luted to uremic teeth with five different luting agents.

Methods: A total of 35 sound natural molars was collected from uremic patients and randomly assigned into five groups (n=7). The teeth were prepared for metal copings using diamond tips and water coolant. Metal copings with a loop on the occlusal surface were fabricated using base metal alloy (Rexillium III). The copings were luted using Fuji I, glass ionomer (GI); Fuji Plus, resin-modified glass ionomer (RMGI); Panavia F 2.0, resin cement; Rely X Unicem, self-adhesive cement (SA); and Adhesor, zinc phosphate cement (ZPh). All specimens were incubated at 37°C for 24 hours, conditioned in artificial saliva for 7 days, and then thermocycled for 5000 cycles (5°C-55°C). The dislodging force was measured using a universal testing machine at a crosshead speed of 2 mm/min. The mode of failure of the loaded adhesive copings was evaluated. Statistical analyses were performed using one-way analysis of variance and Tukey post hoc test.

Results: GI and SA cements had the highest and the lowest mean retentive strength, respectively (580.90±17.3, 406.6±12.7). There was no significant difference between ZPh, SA, and resin cements. These cements were inferior to GI and RMGI cements ($p<0.05$), which showed statistically similar retentive strengths.

Conclusions: The results of this study support the use of glass ionomer and resin-modified glass ionomer cements for luting of metal copings to uremic teeth with retentive preparations.

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INTRODUCTION

The retention of crown restorations has always concerned the dental professional, as it affects the longevity of the indirect restorations. Multiple factors affect the success of indirect restorations, such as preparation design, oral hygiene/microflora, mechanical forces, and restorative materials. However, a key factor to success is the choice of a proper luting agent and the cementation procedure. Loss of crown retention was found to be the second leading cause of failure of crowns and fixed partial dentures.[1-3]

Dental luting agents provide a link between the restoration and prepared tooth, bonding them together through some form of surface attachment, which may be mechanical, micromechanical, chemical, or a combination thereof. Zinc phosphate cement has been the most popular luting material for more than 90 years. Excellent clinical performance has been reported for indirect restorations cemented with zinc phosphate cement despite its high solubility and its lack of adhesion.[4,5] Nevertheless, to prevent pain during cementation and to achieve better retention of cast restorations, alternative cements, such as polycarboxylate and glass ionomer cements, were introduced.[6] The adhesion of glass ionomer has been suggested to occur as a result of chemical bonding between negative carboxylate groups in the water-suspended polymer and positive calcium ions in the dental hard tissue.[7] A considerable level of adhesion to both dentin and enamel has been measured *in vitro* for these cements,[8,9] which was also partially confirmed by other clinical studies.[10,11]

With the current advancements of adhesive dentistry, resin cements play an important role for restorative dentistry. These products have several advantages when compared to conventional powder/liquid cements: better retention, minimum solubility in the oral environment, less microleakage, and acceptable biocompatibility.[12,13] Additionally, these materials have the potential of bonding to both substrates (tooth and restoration), favor tooth structure reinforcement, and allow esthetic treatment success.[14,15]

The bond to dentin is obtained by surface pretreatment with acid, followed by application of an adhesive system containing hydrophilic and hydrophobic components.[16] These steps either remove or modify the smear layer and demineralize the dental surface to expose a collagen layer for resin monomer infiltration, consequently forming the hybrid layer.[17]

Self-adhesive resin cements were recently launched into the dental market aiming to simplify the clinical steps and diminish the sensitivity of the previous multiple step technique.[18] The material is directly applied onto tooth surface, without any pretreatment.

The smear layer is partially incorporated by the acid monomers that promote micromechanical retention to tooth structure; chemical retention may occur by the reaction between acid monomers and hydroxyapatite present in tooth hard tissues.[19] Several *in vitro* studies have assessed the impact of luting agents on casting retention.[18-23] These retention tests, which typically were performed by removing a standardized casting from a stylized crown preparation with direct tensile loading, focused on the effects of the type of cement,[23-25] type of metal,[24,25] preparation taper,[20,26] preparation height,[20] surface roughness,[21] and application of dentinal desensitizing agents.[23] The studies related to crown retention and luting cement type reported that adhesive resins had consistently greater retention than zinc phosphate.[18,23,24]

Chronic renal failure (CRF) is defined as a progressive decline in renal function associated with a reduced glomerular filtration rate as measured clinically by the creatinine clearance rate. The interaction between oral health, CRF, and renal replacement therapy has been the subject of many studies during the last 10 years. This scientific interest refers directly to the rising number of end-stage renal failure (ESRF) patients and renal transplanted patients worldwide.[27,28] In the last three to four decades, improvements in dialysis and transplantation have reduced morbidity and mortality among patients with ESRF. As survival improves, more attention must be focused on other areas, such as dental health, which appears to be yet another area where attention has been lacking.[29]

Dental changes including enamel hypoplasia of the primary and permanent teeth with or without brown discoloration and narrowing or calcification of the pulp chamber of teeth of adults with CRF[30] have been reported. In addition, characteristic changes analogous to those seen in bone were detected in dentin of erupted teeth in patients with CRF.[31] Mahmoud and others[32] investigated the influence of uremia on the shear bond strength of resin composite to enamel and dentin substrates with assessment of the micromorphological pattern of etched enamel and dentin surfaces using atomic force microscopy. They reported that uremia adversely affects bonding of resin composite to enamel and dentin and confers

an altered micromorphological etching pattern. A recently published *in vitro* study investigated the effect of phosphoric acid concentration and etching duration on surface roughness of enamel and dentin substrates of uremic patients receiving hemodialysis supported the use of 42% phosphoric acid for etching uremic hard tooth tissues for 60 seconds.[33]

Considering the preceding information and because the success of modern luting cements is greatly dependent on the quality and the performance of their bonds to dental substrates,[16,17] deteriorated or weak bonding of these materials to tooth tissues of uremic patients could be expected. This hypothesis has not been hitherto confirmed or even dismissed in spite of the recent improvements in the chemistry of adhesive systems that have succeeded to a great extent in offsetting the difficulties associated with bonding to different tooth tissues.[34] Accordingly, the aim of this laboratory study was to evaluate and compare the retentive strength of metal copings luted to teeth of uremic patients undergoing hemodialysis using a glass ionomer cement, resin-modified glass ionomer cement, resin cement, and a self-adhesive resin cement with that luted with a zinc phosphate cement.

METHODS AND MATERIALS

Thirty-five sound natural mandibular molars of nearly similar size and shape extracted for periodontal reasons were collected from uremic patients under maintenance hemodialysis. The buccolingual and mesiodistal widths of the selected molars were measured in millimeters allowing a maximum deviation of 10% from the determined mean. The teeth were obtained according to a protocol approved by our Institutional Committee for Ethics of Research. Uremic patients were seeking help for their dental pain at the Outpatient Dental Clinic, Faculty of Dentistry, Mansoura University. They had been referred from the Outpatient Clinic of Mansoura Urology and Nephrology Center. The patients were under regular maintenance hemodialysis treatment using a biocompatible membrane dialyzed with a volumetric machine using bicarbonate dialysate three times weekly for 4 hours each time (12 h/wk). The average length of time that the patients had been receiving hemodialysis was 5.6 years. No patient had decompansated organs other than the kidney. They had serum creatinine above 7 mg/dl and a creatinine clearance rate 10 ml/min; 13 patients were normotensive, and 22 had controlled hypertension. All collected teeth were subjected to thorough scaling (Varios 550, NSK Nakanishi, Kanuma, Japan) to get rid of both hard and soft deposits. All teeth were kept in 1% thymol solution at room temperature for 2 weeks. The teeth had their roots embedded in a cylindrical PVC ring (1.4×2.5 cm) using a self-cure acrylic resin (Duracrol, Sofa-Dental, Prague, Czech Republic) up to 1 mm below the cemento-enamel junction.

Teeth Preparation

All teeth were prepared in a standardized manner using number 837.012 diamond tips (Edenta GmbH, Lustenau, Austria) loaded in an industrial lathe cutting machine (BV series bench lathe, Bengbu, China) aiming to get tooth cylinders having their occlusal plane perpendicular to the long axis of the tooth, 10° axial taper, 7 mm in diameter, and 4 mm high. All of the preparations were made by one experienced operator throughout the study. A polyvinyl siloxane impression (Virtual, Ivoclar Vivadent, Schaan, Liechtenstein) was taken for each prepared tooth and poured with type 4 improved stone (GC, Fuji Rock, Leuven, Belgium) to obtain stone dies.

Construction of Metal Copings

The resultant dies were covered with two coats of die spacer (Yeti Dental, Engen, Germany) 1 mm above the cervical finish line to ensure good marginal adaptation. The dies were lubricated (Die lube, Dentaurum, Ispringen, Germany) and then used to fabricate indirect wax patterns (Plastodent G, DeguDent, GmbH, Hanau-Wolfgang, Germany) using a specially designed split stainless-steel counter die. A wax loop was fabricated and centrally attached to the occlusal surface of the wax pattern parallel to the long axis of the prepared teeth for performing the dislodgment test.

The wax patterns were invested in a phosphate-bonded investment (Ceravest Quick, GC, Tokyo, Japan) and cast in a base metal alloy (Rexillium III, Pentron, Wallingford, CT, USA). After divesting and cleaning with an ultrasonic cleaner and hydrofluoric acid, the inner surface of the castings were inspected under magnification (×4), and surface irregularities were removed with a small round carbide bur. The metal copings were checked for fit using a silicon disclosing medium (Fit Checker, GC Co, Tokyo, Japan), and further potential interferences of castings were evaluated and adjusted if necessary. The intaglio surfaces of all copings were sandblasted using 50-μm aluminum oxide.

Table 1: *Luting Agents Tested*

Product Name	Manufacturer	Lot Number	Type of Luting Agent	Mixing Method and Ratio
Fuji I	GC Co, Tokyo, Japan	0812051	Glass ionomer cement	Automix capsule, 10 s mixing at 4000 rpm
Fuji Plus	GC Co	0905261	Resin-modified glass ionomer cement	Automix capsule, 10 s mixing at 4000 rpm
Panavia F2.0	Kurary Medical Inc, Kurashiki, Japan	00162A, 0023B	Resin-based cement	Hand mix, equal length of base and catalyst
Rely X Unicem	3M ESPE AG, Seefeld, Germany	446227	Self-adhesive resin cement	Aplicap capsule (295 mg per capsule)
Adhesor	SpofaDental a.s., Prague, Czech Republic	2056105	Zinc phosphate cement	Hand mix, 8 g powder with 0.3 cc liquid

Five commercially available luting agents, (Fuji I, Fuji Plus, Panavia F2.0, Rely X Unicem, and Adhesor) cements were evaluated in this study (Table 1). Each cement was mixed according to the manufacturer's instruction and applied to intaglio surface of the copings (n=7). The copings were gently seated on the abutments and held in place under a 5-kg load for 10 minutes using a special device. After initial setting of the cement, the excess cement was removed with an explorer.

Testing

The specimens were stored in a 37°C incubator for 24 hours, immersed in artificial saliva for 7 days (Save-A-Tooth, Phoenix Lazerus, Inc, Pottstown, PA, USA) and thermocycled in distilled water for 5000 cycles between 5±2°C and 55±2°C with a dwell time of 30 seconds and a transfer time of 5 seconds. After the aging process, the dislodging force of the copings was measured using a universal testing machine (Type 500, Lloyd Instruments, London, UK) at a crosshead speed of 2 mm/min (Figure 1). The fitted surfaces of the separated castings were examined visually to determine the mode of cement failure: adhesive, cohesive or a combination. Adhesive failure meant luting cements were totally separated from the casting or tooth surface. Cohesive failure meant failure occurred within the luting agent or tooth structure. Mixed failure meant both cohesive and adhesive. All specimens were fabricated and measured by the same operator.

Figure 1. *Universal testing machine (Type 500, Lloyd Instruments, London, UK).*

Table 2: *Retentive Strength Means (n), Standard Deviation (SD), and Tukey Test (p<0.05)*

Groups	Means[a]	SD	Maximum	Minimum
1: Fuji I	580.90 A	17.35	607.70	562.40
2: Fuji Plus	557.66 A	18.81	576.20	544.10
3: Panavia F2.0	420.39 B	14.30	422.20	391.10
4: Rely X Unicem	410.61 B	12.74	431.20	388.70
5: Adhesor	395.65 B	18.25	420.76	382.13

[a] *Means followed by different letters are statistically different at the 5% significance level.*

Table 3: *Distribution of Mode of Cement Failure in Percentages*

Groups	Cement Totally on Tooth	Cement on Tooth and Casting	Cement Totally on Casting	Dentinal Fracture
1: Fuji I	—	71.4%	—	28.6%
2: Fuji Plus	—	85.7%	—	14.3%
3: Panavia F2.0	—	28.5%	71.5%	—
4: Rely X Unicem	—	14.2%	85.8%	—
5: Adhesor	—	85.7%	14.3%	—

Statistical Analysis

The statistical package for Social Science Version 19 (SPSS Inc., Chicago, IL, USA) was used for the statistical analysis. Retentive force data was analyzed by one-way analysis of variance (ANOVA) and Tukey test with the level of significance set at 5% ($p<0.05$).

RESULTS

ANOVA showed statistically significant differences among the experimental groups, and Tukey test identified the differences ($p<0.05$). Groups 1 and 2 exhibited the highest retentive strength means, respectively, while the lowest mean retentive strength was exhibited by group 4 (Table 2). Tukey test revealed no significant difference between groups 1 and 2. Also, no significant differences were detected among groups 3, 4, and 5 ($p>0.05$). The distribution of the cement mode failure is given in Table 3. Uncommon horizontal fracture of coronal dentin occurred in 28.6% specimens cemented with Fuji I and 14.3% of specimens cemented with Fuji Plus.

DISCUSSION

The permanent cementation of an indirect restoration is a critical step in the overall treatment procedure. If the cement does not live up to its promise, in the worst case a new restoration has to be made. This is time consuming and annoying for dentists as well as for patients. Dentin is a more heterogeneous and physiologically dynamic substrate than enamel. Among other variables, this explains why bonding to dentin is still a challenge despite the improvements in dental adhesive technology and advances in bonding knowledge.[35,36] The literature contains reports[31,37,38] that uremia produced micromorphological changes of dentin and altered etching pattern with reduced surface roughness, which negatively influenced the bonding of resin composite to dental tissues.[32]

Retention is considered an important requirement in the fixation of prosthetic crowns. Clinically, a crown would hardly undergo such great tensile efforts as those applied in this study, but the tested experimental conditions serve as parameters to evaluate behaviors of the luting materials used with uremic dentin substrate. The results of the present study may be explained by the bonding efficacy of the luting agents. Although other factors may influence crown retention, the preparations were standardized (cervical diameter, taper, roughness, and piece fit), thus eliminating or minimizing the interference of these variables on the results.

Concerning the luting agents, the results showed greater retention (Table 2) for the glass ionomer cement (Fuji I) when compared to the zinc phosphate cement (Adhesor), probably due to the chemical diffusion–based adhesion to dentin, improving the retention compared to conventional cements.[39] Moreover, better mechanical properties of glass ionomer cement in relation to zinc phosphate cement also influence their tensile, compressive, and shearing strengths. The lower tensile strength of the zinc phosphate cement may be related to its composition, which makes this material friable and less resistant to tensile forces.[40] Zinc phosphate cement does not have chemical adhesion to any dental substrate, acting only as a luting agent by mechanical or frictional retention. Thus, the height, taper, and area of the preparation are important aspects for its success as a luting material.[40,41] Therefore, in healthy individuals and situations where preparation retention is deficient, such as a short clinical crown and accentuated taper of the preparation, the choice for a luting agent lies with resin cement, leading to a more favorable clinical prognosis.[42]

The retention values obtained in this study by resin cement (Panavia F2.0) and self-adhesive resin (Rely X Unicem) luting agents were lower than those obtained by glass ionomer and resin-modified glass

ionomer luting agents. The inferior retention obtained by these luting agents in the current study could be explained by the impaired adhesion of these cements to uremic dentin substrate. Panavia F2.0 adhered to tooth structure chemically and mechanically. Bonding to dentin may be linked to a form of *in situ* tissue changes, producing a collagen scaffold by acid etching that is infiltrated and stabilized by resin.[43] Since etching appears to be essential for the dentin components in order to form a resin-reinforced hybrid structure, it is important to have sufficient demineralization to allow adhesive penetration. The formed hybrid layer bonds chemically with the resin cement.[44] On the other hand, the unique self-adhesive resin cement Rely X Unicem has made the use of strong resin cement very easy and predictable. This cement is essentially a filled, self-etching primer that provides the physical properties of resin cement.[45] An important factor that determines the technique sensitivity of adhesive systems is individual and locational variation in structural characteristics and mechanical properties of dentin with regard to their high impact on dentin bonding.[46] It was reported that uremia had a deleterious effect on the nature of dentin substrate and reflected negatively on the bonding mechanism of resin-based materials.[30,32] Dentin is a dynamic substrate subject to continuous physiologic and pathologic changes in composition and microstructure. A comparative ultrastructural (scanning electron microscopic) analysis of dentin in patients suffering from chronic renal failure and in patients undergoing chronic hemodialysis revealed a wide spectrum of changes, ranging from mild disturbance with increasing tubule irregularity and focal obliteration of tubule lumens to widespread formation of dysplastic dentin exhibiting numerous mineralized, largely atubular globules with only occasional large, irregular tubules.[38] Daley and others[47] suggested that characteristic changes analogous to those in bone occur in dentin of erupted teeth with ESRF. Wysocki and others[31] carried out morphometric studies on teeth extracted from normal human individuals and compared them with those extracted from patients suffering from CRF; their findings revealed that the predentin in patient suffering from CRF was significantly thicker than normal. Galili and others[48] found narrowing of dental pulp of patients with ESRF and transplanted patients compared to healthy individuals. All of these morphologic and structural transformations of dentin induced by ESRF resulted in a dentinal substrate that is less receptive to adhesive treatment than is normal dentin.

Recently, Mahmoud and others[33] supported the use of 42% phosphoric acid for the etching of uremic hard tooth tissues for 60 seconds. The lower performance of the resin cement and self-adhesive resin cement in comparison to both the glass ionomer and the resin-modified glass ionomer cement can be attributed to the lack of the effect of a surface-conditioning procedure before luting and hybrid layer formation. Thus, the authors recommended that resin cement or self-adhesive resin cements be used in combination with extensive surface-conditioning agents to obtain the best results. In the same context, it should be noted that the lowest retentive values recorded for resin cement and self-adhesive resin cements were still higher than the retentive values obtained with zinc phosphate cement.

The mode of cement failure distribution revealed that fracture occurred at both the cement-metal and the cement-tooth interfaces for copings luted with the zinc phosphate. In no situation was cement observed to completely remain on the prepared tooth. However, cement was completely retained on the casting for 71.5% and 85.8% of copings luted with adhesive resin cement and self-etch adhesive cement, respectively. The chemical pretreatment of polyacrylic acid to tooth structure appeared to enhance a superior bond of glass ionomer and resin-modified glass ionomer cement to tooth structure. On the other hand, in copings luted with the resin cement and self-adhesive resin cement, debonded cement was observed to be retained totally on the metal surface for the majority of the specimens. This shows the inadequate bond of resin cement and self-adhesive resin to the dentinal surface, most likely resulting from the dentinal micromorphological and ultrastructural changes of dentin due to uremia. These changes lead to lack of the effect of conditioner and primer, causing the weak link of the cemented coping assembly to occur at the cement-tooth interface during tensile debonding. Cohesive dentin fracture was observed for 28.6% of copings cemented with the glass ionomer and 14.3% of copings cemented with resin-modified glass ionomer cement. These results confirm the highest retentive values recorded for these cements. These findings were attributed to the fact that dentin of uremic teeth is a totally different substrate compared to normal or even sclerotic dentin. The literature reported that different ultramorphological and structural changes occurred in dentin as a result of uremic syndrome and secondary hyperparathyroidism, making this tissue different than normal.

In this study, the author deals with uremic dentin, a unique substructure, and evaluated the retentive strengths of five different luting cements on base metal alloy copings. Although glass ionomer cement and resin-modified glass ionomer cement showed higher retentive strengths, all tested cements provided retentive strengths exceeding clinically expected debonding forces.[22] Thus, it can be concluded that all five test cements can be used satisfactorily when they are prepared according to the manufacturers' recommendations. The use of glass ionomer and resin-modified glass ionomer cements seems to be advantageous with uremic teeth.

CONCLUSIONS

Under the conditions of this study, the following conclusions could be drawn:

1. All of the tested cements can be used to satisfactorily lute to uremic teeth prepared following conventional mechanical principles.
2. Glass ionomer and resin-modified glass ionomer cements seem to be better choices in luting uremic teeth of retentive preparations.
3. Cohesive dentin fracture occurred with glass ionomer and resin-modified glass ionomer cemented copings.

Conflict of Interest

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

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Influence of Chemical Degradation on the Surface Properties of Nano Restorative Materials

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Clinical Relevance

This study demonstrated that the incorporation of nanoparticles does not improve the surface properties of the ionomeric cements when exposed to simulated dietary solutions. However, the nanofilled composite resin was more resistant to chemical degradation.

SUMMARY

Objectives: The aim of this *in vitro* study was to investigate the effect of chemical degradation on the surface roughness (R_a) and hardness (Knoop hardness number [KHN]) of nano restorative materials.

Methods: Disc-shaped specimens (5-mm diameter; 2-mm thick) of Filtek Z350 and TPH Spectrum composites and the Vitremer and Ketac Nano light-curing glass ionomer cements were prepared according to the manufacturers' instructions. After 24 hours, polishing procedures were performed and initial measurements of R_a and KHN were taken. The specimens were divided into 12 groups (n=10) according to material and storage media: artificial saliva, orange juice, and Coca-Cola. After 30 days of storage, the specimens were reevaluated for R_a and KHN. The pH values of the storage media were measured weekly. Data were tested for significant differences by repeated-measures three-way analysis of variance and Tukey tests ($p<0.05$).

Results: Composites were found to present lower roughness values and higher hardness values than the ionomeric materials under all storage conditions. After degradation, the KHN of all experimental samples decreased significantly, while the R_a of the ionomeric materials increased, depending on the media, with a markedly negative impact of Coca-Cola and orange juice. There was no difference among the storage media for Filtek Z350 with

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Table 1: *Materials Used in This Study*

Material	Composition	Mean Filler Size, μm	Manufacturer/Batch No.
Ketac Nano (3M ESPE)	Paste A: silane-treated glass, silane-treated zirconia oxide silica, polyethylene glycol dimethacrylate (5%-15%), silane-treated silica, HEMA, Bis-GMA (<5%), TEGDMA (<5%), HEMA (1%-10%) Paste B: silane-treated ceramic, silane-treated silica, copolymer of acrylic and itaconic acids, HEMA (1%-10%)	1 μm (cluster) 5-25nm (nanofiller) <3.0 μm (glass)	3M/ESPE M3M3
Vitremer™ (3M ESPE)	Powder: fluoroaluminosilicate glass; redox system Liquid: aqueous solution of a modified polyalkenoic acid, HEMA (15%-20%)	3.0 μm	3M/ESPE P: 6LP L: 6FH
Filtek™ Z350 (3M ESPE)	58%-60 vol% (78.5 wt%) combination of aggregated zirconia/sílica cluster filler with primary particles size of 5-20 nm and nonagglomerated 20-nm silica filler, Bis-EMA, Bis-GMA; UDMA; TEGDMA	5-20 nm 0.6-1.4 μm (clusters)	3M/ESPE 8NU
TPH Spectrum™ (Dentsply)	Polymer matrix: Bis-GMA, Bis-EMA, and TEGDMA; filler: 57 vol% of Ba-Al-borosilicate glass and coloidal sílica with mean particle size of 0.8 μm	4.4 μm	Dentsply Ind. E Com Ltda L797977

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

regard to the KHN values. Nanofillers did not show any influence on the roughness and hardness of resin-modified glass ionomer cements and resin composites concerning their degradation resistance.

INTRODUCTION

The ability of restorative dental materials to withstand occlusal forces and exposure to various substances in the mouth is an important requirement for their clinical performance over a considerable period of time. The chemical factors known to cause deleterious effects include low pH due to cariogenic biofilm,[1] consumption of acidic drinks or food stuffs,[2,3] and the action of enzymes,[4] which can soften and roughen the outermost layers of restorative materials. Glass-ionomer cement degradation is a complex process of extracting metal cations from the cement matrix and incorporated glass particles,[5] resulting from fluid uptake by the matrix and its solubility. Still, the highly hydrophilic hydroxyethyl methacrylate (HEMA) present in resin-modified glass ionomer (RMGI) makes this material also susceptible to the disintegration of its matrix, in a variable performance heavily dependent on the resin matrix composition and polymerization reactions.[6]

The application of nanotechnology to composite resins was introduced by Filtek Supreme (3M-ESPE, St. Paul, MN, USA), which contains a unique combination of nanofillers (5-75 nm) and nanoclusters embedded in an organic polymer matrix. In addition to improved optical properties, nano materials present better mechanical behavior,[7] since the particle size increased the nanofiller load in the restorative materials.[8] Some studies have shown that nano composites presented higher surface hardness values and lower brushing abrasive wear than did microfilled and hybrid composites.[9,10] Recently, a new RMGI was introduced for operative dentistry. Ketac Nano, a light-curing nano-ionomer (3M ESPE), includes fluoroaluminosilicate glass, nanofillers, and nanofiller clusters combined with HEMA, bisphenol glycidyl methacrylate (Bis-GMA), and triethylene glycol dimethacrylate (TEGDMA) as resin monomers, (3M ESPE, internal data).[11]

According to the manufacturer, Ketac Nano presents mechanical properties such as compressive and flexural strength, diametral tensile, and wear resistance greater than most other glass ionomers tested.[11] Still, the new material showed high polish and surface roughness after tooth brushing, similar to composite.[11] Some studies *in vitro* have shown that the inclusion of nanotechnology in Ketac Nano really provided an ionomeric material with better properties under chemical (biofilm interaction and pH cycling) and mechanical challenge,[12,13] applications of acidulated phosphate fluoride,[14] and after polishing procedures.[15] Nevertheless, a randomized clinical trial with one-year results showed enamel marginal deficiencies and color mismatch more prevalent for Ketac Nano.[16]

Whereas the resin matrix of this material has also undergone modifications (additional monomers to HEMA) and this material is indicated for erosion lesions,[11] often caused by frequent ingestion of acidic foods and drinks, it would be important to evaluate the effects of different beverages on the surface roughness and hardness of nano restorative materi-

Table 2: *Main Ingredients in the Storage Solutions Studied*

Food/Drink	Main Ingredients	pH
Coca-Cola	Carbonated water, sugar, caramel color, phosphoric acid, natural flavors, caffeine	2.49
Orange juice (Minute Maid)	Water, orange juice, sugar, citric acid, natural flavor, and antioxidant ascorbic acid	3.23
Artificial saliva	Calcium (0.1169 g of calcium hydroxide/L of deionized water); 0.9 mM of phosphorus and potassium (0.1225 g potassium phosphate monobasic/L of deionized water); 20 mM TRIS buffer (2.4280 g TRIS buffer/L deionized water)	7

als (a nano-ionomer and a nanocomposite), when compared with another resin-modified glass ionomer cement and composite.

MATERIALS AND METHODS

Specimen Preparation and Initial Analysis

Four different types of tooth-colored restorative materials were used in this present study (Table 1): two resin-modified glass ionomers (Vitremer and Ketac Nano, 3M ESPE) and two composites (Filtek Z350 [3M ESPE] and TPH Spectrum [Dentsply Ind & Com Ltda, Petropolis, Brazil]). Thirty specimens of each material were prepared according to the manufacturers' instructions under aseptic conditions and inserted into plastic molds with internal dimensions of 5-mm diameter and 2-mm thick. The surface of each specimen was covered by a polyester strip and pressed flat by a glass slab. The top surface of all materials was cured according to manufacturers' cure times using an Elipar Trilight curing light unit (3M ESPE), with a mean intensity of approximately 800 mW/cm^2 as checked with a curing light meter (Hilux Dental Curing Light Meter, Benliglu Dental Inc, Turkey). The surface of Vitremer was protected with finishing gloss (3M ESPE). All specimens were maintained at 100% relative humidity and 37°C for 24 hours. The surfaces were then wet-ground with water-proofed silicon carbide discs of decreasing abrasiveness (600, 1200, and 2000) and ultrasonically cleaned (Ultrasonic Cleaner, model USC1400, Unique Co, São Paulo, Brazil) in distilled water for 10 minutes to remove polishing debris. Then, the specimens were randomly distributed into three groups (n=10) according to the test storage medium: artificial saliva (control), orange juice, and Coca-Cola (Table 2).

Before chemical degradation in acidic drinks, all specimens were analyzed for surface roughness and Knoop hardness. For surface roughness testing, the specimens were analyzed using a Surfcorder SE1700 instrument (Kosaka Corp, Tokyo, Japan), with cutoff length of 0.25 mm, at a tracing speed of 0.1 mm/s. The mean surface roughness values (R_a, mm) of each specimen were obtained from three successive measurements of the center of each disk in different directions (total length analyzed of 3.750 mm). Next, hardness tests were carried out with a hardness tester (Shimatzu, Tokyo, Japan) using a Knoop indenter and a load of 50 g, with a dwell time of 15 seconds. Three readings were taken for each specimen, and the mean KHN was calculated for each disk. Then, 10 specimens with surface roughness and hardness values previously determined were distributed for each group of different storage solutions (n=10).

Chemical Degradation: Storage in Acidic Drinks

All specimens were immersed individually in 4 mL of storage solutions, Coca-Cola (pH 2.49), orange juice (pH 3.23), and artificial saliva (pH 7), for 30 days.[17] The solutions were changed weekly and pH tested using a portable pH meter (Orion Model 420A, Analyzer, São Paulo, Brazil). In all cases, the pH electrodes were calibrated immediately prior to use with the aid of standard buffer solutions at pH 4.0 and 7.0.

At the end of the storage period, the specimens were ultrasonically washed for 10 min, dried, and revaluated for roughness and hardness. A representative specimen from each group was also observed under scanning electron microscopy (model Jeol JSM 5600 LV, Tokyo, Japan) to illustrate the effect of the erosive challenge on the materials. Additional specimens from each material were made to serve as a baseline to compare baseline surfaces to chemically degraded surfaces.

Statistical Analysis

Data were evaluated using the PROC LAB from SAS to check the equality of variances and confirm a normal distribution. Hardness and roughness data were submitted to repeated-measures three-way analysis of variance (ANOVA) and Tukey test with a significance limit of 5%. Hardness data were transformed using root square to allow the use of ANOVA.

Table 3: *Mean (SD) Surface Roughness Values (μm) of Restorative Materials Submitted to Different Storage Solutions*[a]

Erosion	Material	Storage Solution		
		Artificial Saliva	Coca-Cola	Orange Juice
Before	Filtek Z350	0.14 (0.07)Ab	0.13 (0.03)Ab	0.13 (0.03)Ab
	TPH Spectrum	0.18 (0.04)Ab	0.18 (0.03)Ab	0.20 (0.04)Ab
	Ketac Nano	0.33 (0.12)Aa	0.37 (0.13)*Aa	0.31 (0.11)*Aa
	Vitremer	0.47 (0.19)Aa	0.39 (0.17)*Aa	0.34 (0.09)*Aa
After	Filtek Z350	0.11 (0.01)Ab	0.11 (0.01)Ab	0.11 (0.01)Ab
	TPH Spectrum	0.17 (0.02)Ab	0.17 (0.05)Ab	0.19 (0.05)Ab
	Ketac Nano	0.32 (0.14)Aa	0.48 (0.16)Aa	0.40 (0.11)Aa
	Vitremer	0.40 (0.09)Aa	0.48 (0.16)Aa	0.49 (0.15)Aa

[a] *Capital letters indicate comparison among storage solutions (horizontal). Lowercase letters demonstrate comparison among materials (vertical) within each storage solution and each erosion condition (before or after). Asterisks represent statistically significant difference between erosion effect (before × after). Groups denoted by the same letter/symbol represent no significant difference* ($p > 0.05$).

RESULTS

There was a significant interaction between the factors "materials" and "degradation effect" (before × after; $p=0.0439$) and between "storage solution" and "degradation effect" ($p=0.0074$). A significant interaction was not observed between "materials" and "storage solution" ($p=0.4733$) and among the three factors ($p=0.0699$). The means and standard deviations of surface roughness of each material under different storage conditions are displayed in Table 3. There was no significant difference among storage solutions (saliva/orange juice/Coca-Cola; $p=0.2010$) and between degradation effect (before/after; $p=0.2251$); however, there were statistical differences among the materials studied ($p<0.0001$).

Regardless of the storage solution, both composites (Filtek Z350 and TPH Spectrum) presented similar roughness values ($p>0.05$) and significantly lower roughness values than the ionomers, both before and after the chemical challenges. There was no statistical difference in roughness values between Ketac Nano and Vitremer in all storage conditions. In addition, when different storage solutions were compared for each material after chemical challenge, no statistically significant difference among them was observed. With regard to the degradation effects on the surface of each material, the exposure to acidic drinks (orange juice and Coca-Cola) resulted in chemical degradation (ie, significantly higher roughness values for both of the ionomeric materials tested). Artificial saliva did not produce any difference in the roughness results for all materials. Composite surfaces were not rough when kept in any storage solution evaluated.

Table 4 presents the mean and standard deviations of the Knoop hardness values of each material under the evaluated storage conditions. There was a significant interaction between the factors "materials" and "storage solution" ($p=0.0009$), "materials" and "degradation effect" ($p<0.0001$), and "storage solution" and "degradation effect" ($p<0.0001$), as well as among the three factors ($p=0.0022$). In addition, there was a significant difference among the materials ($p<0.0001$), storage solutions (saliva/juice/Coca-Cola; $p<0.0001$), and degradation effect (before/after; $p<0.0001$).

Before degradation in acidic beverages, both of the composites (Filtek Z350 and TPH Spectrum) presented similar and significantly higher hardness values than the ionomers evaluated, which presented similar values between them. Regarding the degradation effects, the exposure to any storage solution produced statistically lower hardness values for all materials tested. There was also an influence of the storage solution found for each material: the acidic beverages (Coca-Cola and orange juice) were more aggressive than artificial saliva for Vitremer and Keta Nano. Orange juice was also very detrimental to TPH Spectrum, while no difference was observed among the solutions for Filtek Z350. In addition, the composites presented significantly higher hardness values than did the ionomeric materials after chemical degradation by artificial saliva and Coca-Cola. However, after orange juice storage, Filtek Z350 showed the highest hardness values, followed by TPH Spectrum and, finally, by both ionomeric materials.

DISCUSSION

This current study evaluated the effects of acidic beverages on the surface roughness and hardness of nano and conventional restorative materials. Three storage media were selected: orange juice and Coca-Cola, due to their potential to cause chemical

Table 4: *Mean (SD) Knoop Hardness Number (KHN) of Restorative Materials Submitted to Different Storage Solutions*[a]

Erosion	Materials	Storage Solutions		
		Artificial Saliva	Coca-Cola	Orange Juice
Before	Filtek Z350	78.11 (8.55)*Aa	84.17 (10.79)*Aa	82.06 (12.31)*Aa
	TPH Spectrum	81.84 (11.15)*Aa	79.93 (9.11)*Aa	79.43 (10.97)*Aa
	Ketac Nano	41.16 (5.29)*Ab	39.65 (5.79)*Ab	39.64 (6.83)*Ab
	Vitremer	39.12 (4.53)*Ab	40.31 (7.83)*Ab	39.41 (8.43)*Ab
After	Filtek Z350	65.33 (5.80)Aa	57.97 (6.60)Aa	65.13 (7.46)Aa
	TPH Spectrum	64.30 (5.22)Aa	52.35 (5.76)ABa	42.85 (4.96)Bb
	Ketac Nano	27.38 (4.18)Ab	18.92 (2.18)Bb	15.53 (2.69)Bc
	Vitremer	28.91 (2.76)Ab	16.29 (4.19)Bb	19.45 (4.27)Bc

[a] *Capital letters indicate comparison among storage solutions (horizontal). Lowercase letters demonstrate comparison among materials (vertical) within each storage solution and each erosion condition (before or after). Asterisks represent statistically significant difference between erosion effect (before × after). Groups denoted by the same letter/symbol represent no significant difference* ($p > 0.05$).

degradation, and artificial saliva, as a positive control. Coca-Cola contains phosphoric acid and has low titratability. Orange juice contains citric acid, which has a high titratability and buffering capacity.[18] Overall, both storage solutions caused a significant increase in the surface roughness values for resin-modified glass ionomers cements (RMGIC) studied and a significant decrease in hardness for all materials, confirming their potential to degrade resin-based restorative materials.

Before the degradation process, higher roughness values were observed for RMGIC when compared with composite resins. The differences observed at the baseline conditions among materials regarding their means of surface roughness values are mainly related to differences in their filler particle size, shape, volume, and distribution and their interaction with the organic matrix, allowing better polishing characteristics for the composites.[19] Still, the handling of two pastes or powder/liquid can trap air in the ionomeric material structure, resulting in surface bubbles and exposure of porosities after finishing/polishing procedures.

Prior to acidic challenge, similar roughness values between the nanofilled and conventional materials were observed, for both the composite and ionomer groups. However, Mitra and Holmes[7] and Cavalcante and others[20] demonstrated that nanofilled composites presented lower roughness values and better polishing characteristics than did hybrid composites because of the presence of nanofillers. The same behavior was observed for Ketac N100 compared with other ionomeric materials.[15] Nevertheless, the resinous matrix of the materials used in this study was not probably totally removed by the initial finishing/polishing procedures, leaving a thin matrix layer over the fillers (as observed in micrographs), which interfered in the superficial nanofillers' presence and exposition.

Composite resins and RMGICs performed differently with regard to their surface roughness when evaluated for the 30-day chemical degradation period. The chemical challenge caused no effects on the roughness values for both composites. The ethoxylated version of the Bis-GMA (Bis-EMA) existing in the composition of Filtek Z350 and TPH Spectrum matrixes probably contributed to their hydrolytic and biochemical stability, due to the hydrophobicity of this monomer. Yap and others[21] also showed that the surface roughness of a Bis-EMA–based composite was not affected by acidic beverages. Bis-EMA shows a decreased flexibility and increased hydrophobicity due to the elimination of the hydroxyl groups when compared with composites formulated with Bis-GMA.[22] Hence, the reduction in water uptake may be partially responsible for the chemical stability of composites that contain Bis-EMA.

Regarding RMGIC, an acid environment has been shown to have a severe effect on surface degradation.[18] The degradation by orange juice or Coca-Cola caused a significant increase in roughness surface for Ketac N100 and Vitremer (Figure 1; Table 3). The presence of HEMA, a highly hydrophilic monomer in the organic matrix from RMGICs, can increase the solubility of RMGICs.[23] Rogalewicz and others[24] observed HEMA, TEGDMA, and additive decomposition products eluted from a RMGIC after seven days of immersion in acidic media. It is possible that the loss of components from the two Vitremer and Ketac N100 matrixes (polyacrylate-inorganic and polymer-organic) leads to changes in surface roughness and hardness. Therefore, it could be speculated that the acidic environment corroded the RMGIC

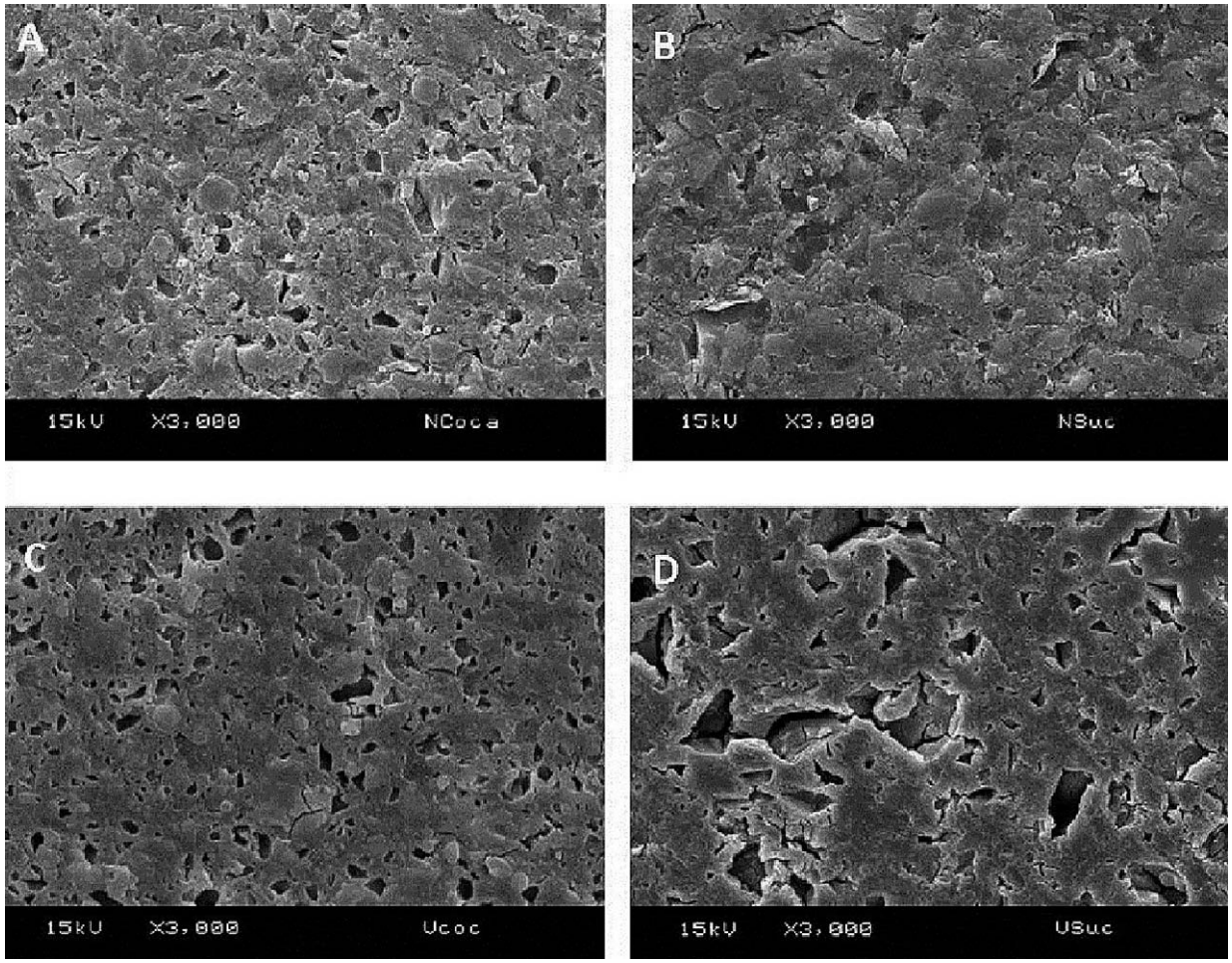

Figure 1. *Representative scanning electron micrographs of glass ionomer cement after degradation. (A): Ketac Nano/Coca-Cola. (B): Ketac Nano/orange juice. (C): Vitremer/Coca-cola. (D): Vitremer/orange juice. Note corroded resin matrix provided by chemical erosion; extrusion fillers. Original magnification 3000×.*

matrix, promoting an increase in roughness (Figure 1; Table 3), also seen in micrographs. Thus, regarding roughness results, it is evident that the composition of the matrix influenced the surface roughness of materials subjected to a chemical challenge[25] and that the incorporation of nanoparticles in the composite and glass ionomer cement did not interfere with their chemical degradation resistance.

Hardness is a property that is used to predict the wear resistance of a material and was the parameter most affected by the chemical challenge in the current study. According to the present results, both composites (Filtek Z350 and TPH Spectrum) presented higher hardness values than the RMGICs. The different contents of organic matrixes and higher filler loading, as well as the higher degree of conversion for the resin composites, could explain the behavior of these materials compared with ionomers. However, their high filler loading (79% by weight, following manufacturer data) contributed to the similar initial hardness values between Filtek Z350 and TPH Spectrum, regardless of inclusion of nanofillers in only one.

After degradation in acidic beverages, all materials showed a significant reduction of hardness, and RMGICs showed a greater loss of hardness than the resin composites studied. The decreased hardness observed for all storage solutions seems to have originated from hydrolysis, since the more hydrophilic organic matrixes experienced greater hydrolysis.[26] According to Sakar,[27] corrosive wear begins with water absorption that diffuses internally through the resin matrix, filler interfaces, pores, and others defects, accelerated by the low pH of the solution. Thus, the chemical degradation rates of different materials depend on their hydrolytic stability, which is mainly related to the resin matrix.[28] As the resin matrix of composites is known

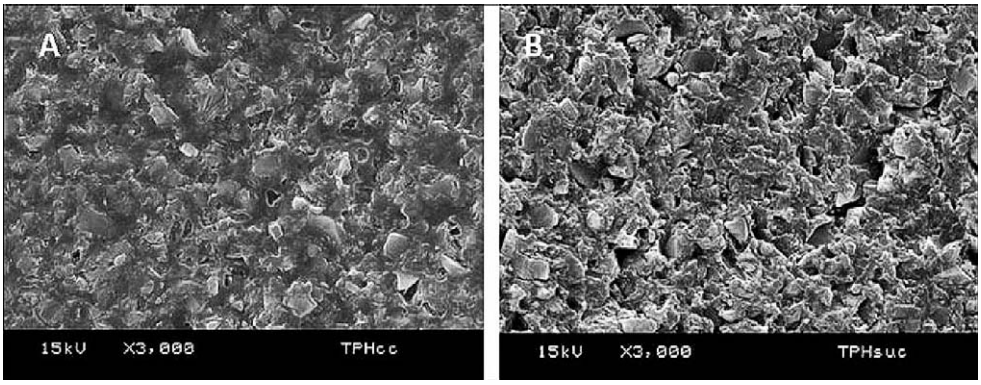

Figure 2. *Representative scanning electron micrographs of TPH Spectrum after storage in (A) Coca-Cola and (B) orange juice. Severe corroded aspect of the resin matrix pointed out by marking it after storage in orange juice (B). Original magnification 3000×.*

to absorb a small percentage of water,[29] composites were more resistant to degradation than were hydrophilic materials, such as RMGICs.[30] In addition, the storage solutions promoted dissolution near the glass particles, as seen in micrographs, which could be the result of dissolution of the siliceous hydrogel layer of RMGICs.[31]

It was observed that Filtek Z350 was the unique material that was not influenced by storage medium. This result can be supported by the hypothesis that the prime deleterious action resulted from the water and not from the acidic environment.[32] Despite a minor difference in the percentage load of the composites tested, the higher filler loading with smaller particle size provides a reduction in the interstitial spacing (less matrix exposition) and enhances the overall resistance of Filtek Z350 to chemical degradation[33] when compared with TPH Spectrum. Moreover, the greater part of TEGDMA from that resin composite was replaced with a blend of urethane dimethacrylate (UDMA) and Bis-EMA (ethoxylated bisphenol-A dimethacrylate). Pearson and Longman[34] determined that UDMA has lower water sorption than Bis-GMA, due to a higher conversion and cross-linking, evidencing the importance of the type of resin matrix in chemical degradation resistance.

Concerning the resin composite TPH Spectrum, a significant loss of surface hardness was observed after storage in orange juice. This could be related to its inorganic fillers, as suggested by Soderholm and others.[35] It was shown that materials containing barium glass fillers are more susceptible to acid attack. Moreover, the corrosive effect of the storage solutions did not depend only on their intrinsic pH value but also on their buffering effect, type, and chelating properties of the acid, among other acid characteristics.[36] According to Owens[37] and Cheng and others,[38] orange juice has a greater buffering capacity and corrosive effect than does Coca-Cola, explaining the lower hardness values of TPH Spectrum composite when stored in orange juice (Figure 2; Table 4). Still, the size and dimension of the citrate molecule induces the formation of stable complexes with metallic ions present at fillers.

Although all of the materials degraded with storage in all solutions, Coca-Cola and orange juice produced greater reductions in the hardness values for TPH Spectrum and RMGICs, with or without nanofiller inclusion.

CONCLUSION

It was concluded that different beverages (Coca-Cola and orange juice) provided great changes in surface roughness for RMGIC, regardless of the presence of nanofillers. Overall, Coca-Cola and orange juice provided a decrease in the hardness for all materials studied. The most intense decrease on hardness was observed for RMGICs immersed in both acidic solutions and TPH Spectrum in orange juice. Nanofillers did not influence the roughness and hardness of RMGIC or resin composites studied.

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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Effects of Different Surface Treatments on Bond Strength Between Resin Cements and Zirconia Ceramics

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Clinical Relevance

The use of different surface treatments (airborne particle abradion or tribochemical silica coating/silane coupling system) on zirconia ceramic for different adhesive resin cements (dual or self-cure) may improve bond strength between resin cements and zirconia ceramic.

SUMMARY

This study compares the bond strength of resin cement and yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramic with different surface conditioning methods. Two hundred presintered Y-TZP ceramic specimens were prepared, sintered (4 × 4 × 4 mm), and randomly assigned to four equal groups as control (C, no conditioning); airborne particle abraded (APA, air abrasion with 11 μm Al_2O_3); tribochemical silica coating/silane coupling system (TSC, Rocatec, air abrasion with 110 μm Al_2O_3, 30 μm silica-coated Al_2O_3 and silane); and laser (L, Er:YAG laser irradiation treated at a power setting of 200 mJ). After specimen preparation, composite resin cylinders were prepared and cemented with resin cements (Clearfil Esthetic, Panavia F 2.0, Rely X-U100, Super Bond C&B, and Multilink Automix) on the ceramic surfaces and kept in an incubator at 37°C for 60 days. All specimens were tested for shear bond strength with a universal testing machine, and fractured surfaces were evaluated by environmental scanning electron microscopy. Statistical analysis was performed using Kruskal-Wallis and Mann-Whitney U-tests (α=0.05). The bond strengths for C and L groups were not significantly different according to adhesive resin cement. APA and TSC resulted in increased bond strength for Panavia F 2.0 and Rely X-U100 resin cements. Additionally, TSC presented higher bond

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strength with Multilink Automix. Adhesive fracture between the ceramic and resin cement was the most common failure. Complete cohesive fracture at the ceramic or composite cylinders was not observed. Regardless of the adhesive resin cement used, laser treatment did not improve resin bond strength.

INTRODUCTION

Zirconia-based restorative materials have been used as core materials for single crowns and fixed dental prostheses due to their superior esthetics, biocompatibility, and high mechanical strength.[1] Early recommendations for luting zirconia restorations included the use of conventional cements, such as zinc phosphate or resin-modified glass ionomer cements, although adhesive cementation provides high retention, improves marginal adaptation, prevents microleakage, provides fracture resistance of the restored tooth and the restoration, and improves longevity of ceramic restorations.[1,2] In addition, resin cements offer the advantage of sealing minor internal surface flaws created by acid etching or airborne particle abrasion, significantly strengthening ceramic materials.[3] To achieve exceptional bonding durability between luting agents and a sintered zirconia surface, the surface area of the bonding surface must be increased and an active surface produced.[4-6] Airborne particle abrasion (APA), tribochemical silica coating (TSC), acid etching, plasma spraying, low-fusing porcelain layers, application of a glaze layer, silane application, heat-induced maturation-selective etching techniques, fusion sputtering, and lasers have been evaluated on yttrium partially stabilized tetragonal zirconia polycrystalline (Y-TZP) surface-conditioning methods.[7-14]

Unlike glass ceramics, etching is not possible for Y-TZP due to the high crystalline content and glass-free structure.[5,15-17] The zirconia surface has been shown to be minimally affected by conventional roughening techniques, although the strength may be enhanced.[18-21] APA is a suitable method to increase surface area to produce micromechanical interlocks of the bonding interface.[4,22] Tribochemical silicoating by Rocatec™ has been introduced as an alternative to APA.[23] In this technique, the ceramic surface is first air abraded with Al_2O_3 particles to remove contaminants and provide microroughness. Then the surfaces are airborne-particle abraded with aluminum trioxide particles modified with silica. The blasting pressure results in the embedding of the silica-coated alumina particles on the ceramic surface, rendering the silica-modified surface chemically reactive to the resin through silane coupling agents.[24] Bonding between a ceramic surface and resin luting agent may be facilitated by a silane coupling agent. The alkoxy groups (RO_3Si-) of the silane molecule react with water to form silanol groups (SiOH). The silanol groups further react with hydroxyl (OH) groups on a ceramic surface with available Si and O to form siloxane (-Si-O-Si-O-) covalent bonds. The monomeric end of the silane molecules reacts with the methacrylate groups of the resin luting agent. Thus, a strong network of siloxane covalent bonds is formed between the ceramic surface and methacrylate resin.[16] This chemical reaction is not applicable to zirconia-based ceramics because it lacks a silica phase.[4] A high initial tensile bond strength of resin to zirconia-based ceramics was achieved with silicoating followed by silanization.[4,7,20,24-26] Different laser systems (Er:YAG, CO_2, and Nd:YAG) have been used to alter and improve bond strength of resin cements to a zirconia surface.[12,13,27-29] Most reports, however, have demonstrated a significant reduction in bond strength after simulated artificial aging.[4,7,8,24]

The aim of this study was to compare bond strengths after three different surface treatments to zirconia and the use of five different adhesive resin cements. The null hypotheses of this study were that different types of surface treatment would not change the bond strength between the zirconia and adhesive resin cement and that the different types of adhesive resin cement would not affect the bond strength of zirconia.

MATERIALS AND METHODS

Preparation of Core Ceramics

Two hundred ($5 \times 5 \times 5$ mm) cubes were milled from presintered 3% Y-TZP (Zircon Ice Zirconia®, Zirkon-Zahn, Bruneck, Italy [ZrO_3; specifications, Y_2O_3 % 4-6, Al_2O_3 % <1, SiO_2 % max. 0.02, Fe_2O_3 % max. 0.01, Na_2O % max. 0.04]) using an electrical high-precision saw (IsoMet 1000 Precision Saw, Buehler Ltd, Lake Bluff, IL, USA) under water irrigation with a diamond wafering blade (6-inch Diamond Waferin Blade, Series 15 LC Diamond No 11-4776, Buehler). The cubes were sintered at 1500°C for 7 hours in a high-temperature sintering furnace for zirconia (Keramikofen 1500, ZirkonZahn). The dimensions of the cubes were $4 \times 4 \times 4$ mm following 20% volumetric shrinkage[30] associated with the sintering.

All specimens were ultrasonically cleaned (Professional Ultrasonic Cleaner CD-4800, Codyson, ShenZ-

hen, China) successively in 75% ethanol, 25% deionized water, 50% ethanol, 50% deionized water, 25% ethanol, and 75% deionized water for 15 minutes to remove factors that inhibit adhesion. The specimens were dried in the natural atmosphere and placed in a sterile cell culture dish (Costar 96 Well Cell Culture Cluster, Corning Inc, Corning, NY, USA).

Ceramic Surface Treatments

The cubes were assigned to one of the following four groups (n=50):

1) Control group (C): Underwent no mechanical surface treatment following sintering.
2) APA: The cubes were abraded using 110-μm alumina (Al_2O_3) particles (Sheraaluminiumoxid 110 MY, Shera Werkstoff Technologie, Hanover, Germany) at 2.8-bar pressure for 13 seconds from a distance of 10 mm perpendicular to the treatment surface of the cubes.
3) TSC: The bonding surface of each specimen was air abraded with 110-μm Al_2O_3 particles (Rocatec Pre, 3M ESPE, St Paul, MN, USA) at 2.8-bar pressure from a distance of 10 mm perpendicular to the treatment surface of the cubes for 10 seconds. Then 30-μm silicatized Al_2O_3 particles (Rocatec Plus) were applied at 2.8-bar pressure for 10 seconds at a distance of 10 mm in accordance with the manufacturer's recommendation. Silane coupling agent was applied to the silica-modified surface with a disposable brush, which was then dried with air.
4) Laser (L): Because ceramic surfaces reflect the laser beam, surfaces of the specimens were coated with graphite (pencil) to increase the laser energy absorption. The surfaces were then treated with erbium:yttrium-aluminum-garnet (ER:YAG) laser (AT Fidelis Plus III, Fotona, Ljubljana, Slovenia) (2 W, 10-Hz frequency, and 200-mJ energy, 2940 nm) from a distance of 10 mm for 10 seconds.

Environmental Scanning Electron Microscopy

After surface treatments, zirconia surfaces were examined at 500×, 1000×, 2000×, and 5000× magnification under an environmental scanning electron microscope (ESEM) (Quanta Feg 250, FEI, Oregon, USA).

Preparation of Composites Discs

Composite resin (Filtek™ P60, 3M ESPE) was prepared by injecting the resin composite into a glass pipette (Ø 3 mm) and was light polymerized using an LED polymerization light (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) at 1350 mW/cm^2 for 40 seconds from two different directions for a total of 80 seconds

Two hundred (Ø 3 × 3 mm) composite cylinders were prepared and ultrasonically cleaned (Professional Ultrasonic Cleaner CD-4800, Codyson) by the same method and stored in a closed box to prevent contamination.

Cementation

Five different adhesive resin cement systems—Panavia F 2.0 (Kuraray Co. Ltd, Tokyo, Japan), RelyX U 100 (3M ESPE), Clearfil Esthetic (Kuraray Medical Co, Osaka, Japan), Super Bond C&B (Sun Medical, Shiga, Japan), and Multilink Automix (Ivoclar-Vivadent)—were used to lute the composite resin discs to the zirconia discs according to the manufacturers' instructions. Properties of all used materials are listed in Table 1. Each cement was mixed and applied on the surface of the composite resin disc and seated on the zirconia disc. The polymerization was completed under 700 g of constant load for 60 seconds using a custom-made jig modified from a parallelometer. Simultaneously, the excess cement was wiped off, and the specimen was light polymerized (except Superbond C&B) with the same unit at four different locations for 60 seconds each. Oxygen-blocking gel (Oxyguard II, Kuraray) was applied for 3 minutes, then washed with air/water spray for Panavia 2.0 cement. The polymerization unit was tested for power output after every 10 specimens.

Wet Storage

All bonded specimens were placed inside the compartments of special cell boxes in an incubator (Incubator IF110 Plus, Memmert, ,Büchenbach, Germany) at 100% humidity and 37°C (±2°C) for 60 days before being stressed with the notched shear bond test method.

Shear Bond Strength Test

The specimens were mounted in the jig for shear bond strength testing described in ISO/TR 11405 of a computer-controlled universal testing machine (Autograph AG-50kNG, Shimadzu, Kyoto, Japan) and were loaded in shear at a constant crosshead speed of 0.5 mm/min until failure. The maximum load to failure was recorded by the corresponding software in newtons (N), and the shear bond strength was calculated in MPa by dividing the failure load (N) by

Table 1: *List of Brand Names, Curing Types, Main Compositions, Batch Numbers, and Manufacturers of Resin Cements Investigated*

Material	Type	Characteristics	Batch No.	Zirconia Primer	Batch No.	Manufacturer
Multilink Automix	Dual curing/ self-etch	Liquid: DMA, HEMA, Ba-glass fillers, ytterbium fluoride, spheroid mixed oxide A primer: aqueous solution of initiator B primer: HEMA and phosphoric acid and acrylic acid monomers Metal/zirconia primer: phosphoric acid acrylate and methacrylate cross-linking agents in an organic solution	L12252	Monobond S + Heliobond	K41829 K30706	Ivoclar Vivadent
Panavia F 2.0	Dual curing	10-methacryloxydecyldihydrogen-phosphate (MDP) Paste A: BPEDMA, MDP, DMA Paste B: Al-Ba-B-Si glass/silica-containing composite	00303B 00052A	Porcelain Bond Activator + SE Bond Primer	00004C 00799A	Kuraray Medical Co.
Clearfil Esthetic	Dual curing	Paste A: Bis-GMA, TEG-DMA, methacrylate monomers, silanated glass filler, colloidal silica Paste B: Bis-GMA, TEGDMA, methacrylate monomers, glass filler, colloidal silica, benzoyl peroxide, pigments	0005BC	Ceramic Primer	00004E	Kuraray Medical Co.
Rely X U 100	Dual curing/ self-etch	Glass powder, dimethacrylate, silanated silica, Sodium p-toluene sulfinate, calcium hydroxide	327580	ESPE Sil 3-		
		methacryloxypropyltrimethoxysilane (MPS) in ethanol	192348	3M ESPE		
Super Bond C&B	Self-curing	Powder: PMMA, Liquid: MMA, 4-META Catalyst: TBB + Liquid: MMA, 4-META	ME12F MS1 (monomer)	Porcelain Liner M	TE1	Sun Medical

Abbreviations: DMA, dimethacrylate; HEMA, 2-hydroxyethylmethacrylate; BPEDMA, bisphenol-A polyethoxydimethacrylate; DMA, aliphatic dimethacrlyate; TEGDMA, triethylene glycol dimethacrylate; BisGMA, bisphenylglycidyl dimethacrylate; 4-META, 4-methacryloxyethyl trimellitate anhydride; MMA, methyl methacrylate; PMMA, poly(methyl methacrylate); TBB, tri-n-butyl borane catalyst.

the bonding area (mm^2). The same operator prepared and tested all specimens to eliminate interoperator variability.

Failure Mode

The mode of failure of each specimen was determined by inspecting the bonding surfaces of each specimen under optical magnification (10× handheld loupe, Carl Zeiss AG, Oberkochen, Germany). Failure mode was classified into three types: (1) adhesive failure (failure between the resin luting agent and the resin composite or between the resin luting agent and the ceramic), (2) cohesive failure within the resin luting agent, and (3) mixed mode of failure, including the first and second types ESEM images of representative specimens were captured.

Statistical Analysis

Statistical analyses were performed by using SPSS 18.0 System for Windows (SPSS, Chicago, IL, USA). As a result of the Shapiro-Wilk normality test for the nonparametric methods applied to data that do not meet the normal distribution, p-values less than 0.05 were considered to be statistically significant in all tests. Kruskal-Wallis followed by a Mann-Whitney U-tests were used to comparatively assess the data.

RESULTS

The data of shear bond strength in all subgroups are listed in Table 2. For Panavia F 2.0, Clearfil Esthetic, and Rely X U100 adhesive resin cements, shear bond strength values of control and laser groups were statistically different from TSC ($p<0.000$) and APA ($p<0.000$; C: $p<0.019$; L: $p<0.002$ for Clearfil Esthetic resin cement) groups. In addition, there was a significant difference between the two resin cement groups (Clearfil Esthetic and Rely X U100) and the two surface treatment methods (APA and TSC) ($p<0.000$). For adhesive resin cement Multilink Automix, TSC and

Table 2: *The Median and Min-Max MPD Values (MPa) of Surface Finishing Groups and Adhesive Cements (n=10)*[a]

Cements Surface Treatment	Panavia F 2.0		Clearfil Esthetic		RelyX U100		Multilink Automix		Super Bond C&B	
	Median	Min–Max	Median	Min–Max	Median	Min–Max	Median	Min–Max	Median	Min–Max
Control (C)	0.2^{A}	0.0–0.3	0.0^{A}	0.0–0.2	0.3^{A}	0.0–0.4	0.0^{A}	0.0–0.5	0.2^{A}	0.0–3.0
Airborne-particle-abraded (APA)	$12.2^{B,1}$	9.9–14.8	$1.8^{B,2}$	0.0–5.7	$12.7^{B,1}$	10.9–20.1	$0.3^{A,2}$	0.0–0.8	$0.4^{A,2}$	0.3–1.1
Tribochemical silica coating/silane coupling system (TSC)	$13.3^{B,1}$	7.1–19.8	$8.8^{C,2}$	5.1–13.0	$10.5^{C,2}$	6.8–13.2	$10.1^{B,1,2}$	7.5–13.0	$0.9^{A,3}$	0.2–1.5
Laser (L)	0.4^{A}	0.0–5.0	0.1^{A}	0.0–0.4	0.1^{A}	0.0–2.6	0.0^{A}	0.0–0.4	0.4^{A}	0.0–1.6

[a] *Different uppercase letters represent statistically significant differences within each row. Different numbers represent statistically significant differences within each column.*

other surface treatment groups were statistically different (C: $p<0.000$; APA: $p<0.000$; L: $p<0.000$). There was no difference in shear bond strength between Superbond CB resin cement and surface treatment methods ($p>0.05$). The bond strength for control and laser groups were not significantly different according to adhesive resin cement ($p>0.05$).

The ESEM images (1000× and 5000×) showed morphological differences among Y-TZP specimens after surface treatment. Zirconia specimens with no treatment showed microstructures of grained Y-TZP ceramics (Figure 1A,B). The surface roughness of APA specimens was remarkably increased compared with the control surface (Figure 1C,D). ESEM images of TCS showed the smooth surface of zirconia ceramic caused by silanization (Figure 1E,F). Er:-YAG laser irradiation created a smooth surface, with cracks and loss of material, although the surface was not altered (Figure 1G,H).

Failure modes of the groups are presented in Figure 2 All shear bond strength specimens demonstrated adhesive failures at the resin/zirconia interface. Adhesive failure was mostly observed with an average of 83.5% between the ceramic and resin-luting agent. Fourteen percent of the specimens were associated with mixed failures (Figure 3). Resin cement residue was noted on the ceramic surface. Fractures always occurred at the ceramic/cement interface. Cohesive failure of the ceramic and composite was not observed.

DISCUSSION

Mechanical and chemical modifications of the prepared surface of fixed restorations are well-documented methods of establishing a reliable bond between the restoration and the adhesive cement. Adhesive resin cements are used not only to prevent the dislodgement of ceramic restorations but also to obtain a strong and long-lasting cementation between tooth and ceramic restoration.[18] Thus, it is very important to select the best combination of resin cement and surface treatment for durability. However, hydrofluoric acid etching is not sufficient to improve the bond strength between zirconia ceramics and resin cements, as zirconia lacks silica and is resistant to acid etching. With these considerations in mind, hydrofluoric acid etching was excluded from this study.

To promote micromechanical retention in zirconia ceramics, the APA method can be used instead of acid etching.[31] APA might create subcritical microcracks and phase transformation within the zirconia surface and thereby deteriorate the mechanical properties of zirconia.[32] Quaas and others[33] found that contaminating zirconia surfaces by air abrasion led to higher resin-ceramic bond strength than cleaning the contaminated ceramic surfaces with phosphoric acid or alcohol. The results of a study by Blatz and others[34] also confirm that air-particle abrasion of zirconia surfaces with Al_2O_3 increases bond strength values of self-adhesive luting cements to ceramic surfaces. Derand and Derand[35] reported that acid etching and airborne-particle abrasion had only minor influence on bond strengths. In the present study, the outcome of APA treatment was found to be comparable to that of the TSC treatment, as bond strength values were higher than the laser and control groups. Lüthy and others[36] found that the bond strength of the phosphate monomer (MDP)-containing composite resin P21 to the sandblasted zirconia surface was more than the bond strength of other cements tested (Ketac-Cem, Nexus, Rely X Unicem, Superbond C&B, and Panavia F). This might depend on the fact that the phosphate ester group of the MDP can directly bond to metal oxides.[37] Similarly, APA treatment results were similar with TSC in MDP-containing adhesive resin cements, and high bond strength values were obtained in the present study.

Theoretically, air-particle abrasion could improve bond strength by enhancing surface microroughness

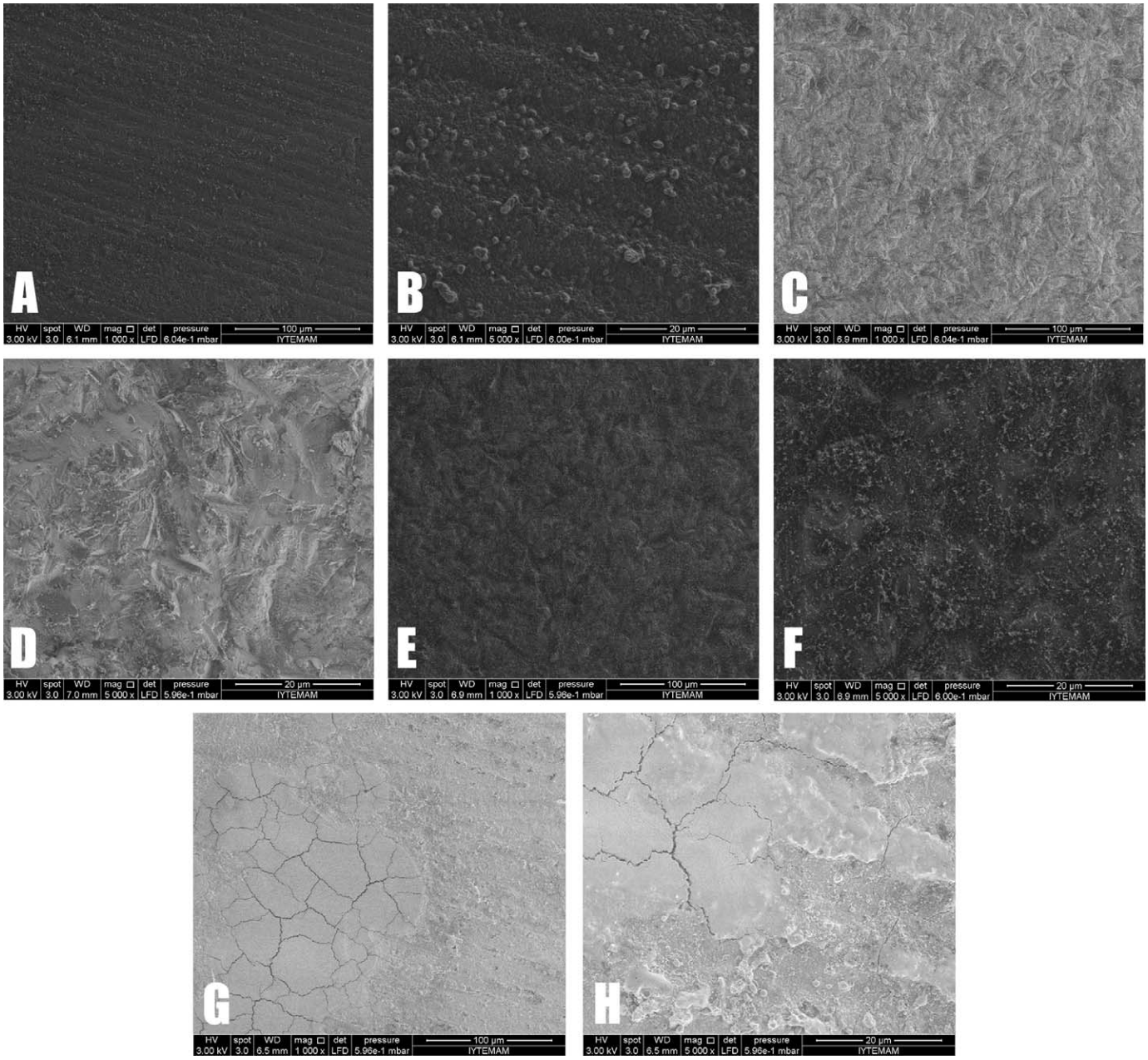

Figure 1. *Environmental scanning electron micrographs (1000× and 5000× original magnification) of zirconia ceramic specimen surfaces. (A and B): Control. (C and D): Airborne particle abraded. (E and F): Tribochemical silica coated. (G and H): Laser applied.*

and generating more hydroxyl groups on the ceramic surface to react with the silanol groups of the silane. The silane molecules alternatively chemically bond to the methacrylate groups of the luting resin.[16] It has also been suggested that the silane agent promotes resin bonding by increasing surface energy and thus wettability of the bonding substrate.[17] Xible and others[20] reported a strengthening effect of tribochemical airborne-particle abrasion using a larger particle size (Rocatec, 110 μm; 3M ESPE) at 2.8-bar pressure. Kern and Wegner[4] reported good initial bond strength of a dual-polymerizing bis-GMA resin cement to APA zirconia. Adding a silane, however, did not improve the bond strength. Blatz and others[8] reported that a coupling agent containing an adhesive phosphate monomer can achieve superior long-term shear bond strength to APA zirconia restorations when using a resin cement. The silicoated/silanated group achieved the highest bond strength among the groups.[15,38] This outcome

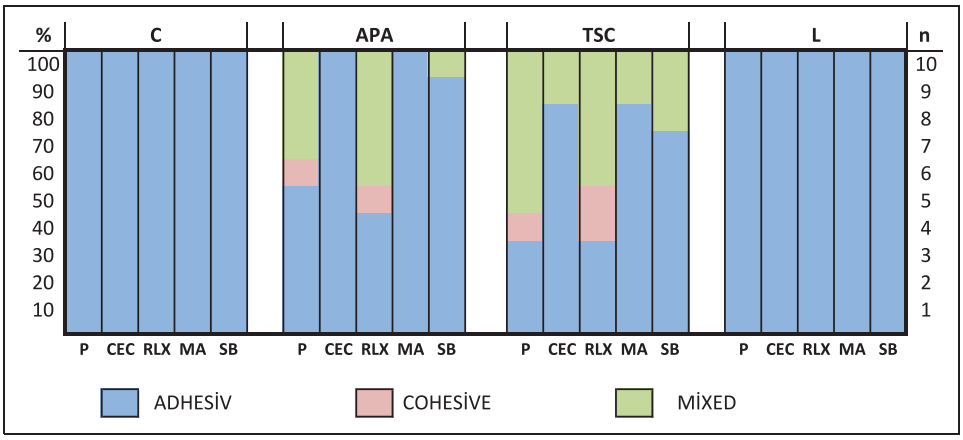

Figure 2. *Distribution of failure modes of experimental groups (C: Control; APA: Airborne particle abraded; TSC: Tribochemical silica coating/silane coupling system, L: Laser groups and P: Panavia F 2.0; CEC: Clearfil Esthetic; RLX: Rely X-U100; MA: Multilink Automix; SB: Super Bond C&B cement).*

is in line with the findings of Kim and others,[25] who demonstrated a high tensile bond strength of resin to zirconia after silicoating and silanization. In the present study, high bond strength values were obtained with the TSC method, and this finding is also in line with previous reports.

Akyil and others[13] reported that air abrasion with 110 μm Al_2O_3 and silica coating with CoJet-Sand were the most effective surface treatment methods with Clearfil Esthetic resin cement. The shear bond strength between a zirconium oxide ceramic and composite resin using an MDP-containing resin luting agent (Panavia F) was increased with chair-side TSC.[17] Passos and others[39] reported that groups conditioned using silica coating and silanization showed higher bond strengths in both dry and aged conditions. Two microtensile bond studies[40,41] on zirconium oxide ceramics using composite resin block as substrate showed that tribochemical silica coating with 50-μm Al_2O_3 particles modified by silica was ineffective in improving the bond strength of three luting agents, including an MDP-containing resin luting agent (Clearfil Esthetic Cement, Kuraray). In the present study, TSC treatment increased bond strength values of MDP-free resin adhesive cements more than MDP-containing resin.

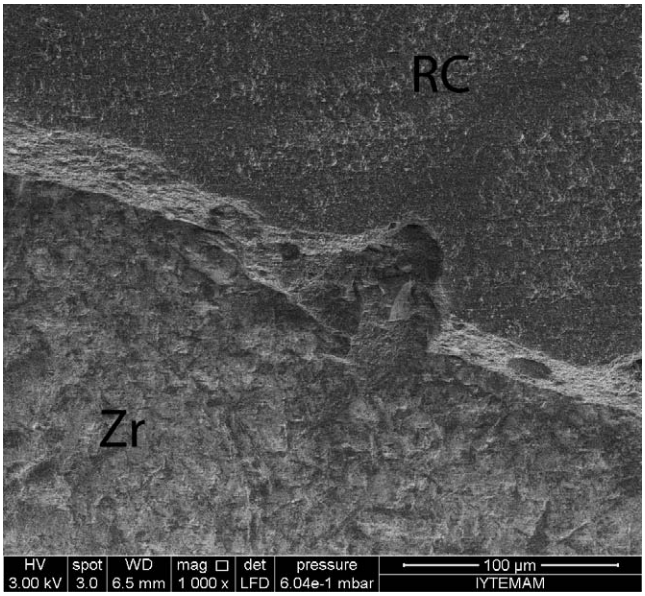

Figure 3. *SEM photograph (1000× original magnification) of APA treated/Rely X U100 adhesive cement failed surface (RC, resin cement; Zr, zirconia).*

In this study, a lower power setting of 200 mJ was selected for the Er:YAG laser with meticulous water cooling to prevent internal tensions related to heating/cooling-dependent local temperature changes. Bond strength results indicated that adhesion was not improved by laser treatment. Er:YAG laser–treated and –untreated surfaces presented similar results. These results agree with those of Cavalcanti and others,[27] who evaluated the influence of surface treatments and metal primers on the bond strength of resin cements to a Y-TZP ceramic with microshear bond test. In addition, our findings are in line with those of Foxton and others,[12] who aimed to evaluate the durability of the bond of conventional dual-cured resin cements to Procera Al_2O_3 and zirconium oxide ceramics after surface treatment with air abrasion and erbium laser. Akyıl and others[13] found that the Er:YAG laser irradiation (200 mJ/pulse, 10 Hz, 10 seconds) increased the bond strength compared to that of untreated surface and created a rough surface similar to that of air abrasion with SEM evaluations. In this study, ESEM evaluations showed that the irradiated surface exhibited a

bubbled blister-like appearance and unusual microcracks. This appearance may have been due to the development of a heat-damaged layer caused by the application of Er:YAG laser on the Y-TZP surface. This heat-damaged layer, containing bubbles, may be poorly attached to the infralayer of the substrate and may account for specimen rupture when a low force is applied during the shear bond test.

Long-term water storage and thermal cycling are commonly used to simulate aging of resin bond interfaces.[8] In the literature, there is no consensus on a relevant regimen for artificial aging.[42] Kern and Wegner[4] evaluated resin bond strength to zirconia after water storage for 150 days and 2 years, combined with thermal cycling. They indicated that artificial aging with thermal cycling decreased the tensile bond strength of resin to silicoated (Rocatec, 3M ESPE) zirconia by almost one-third of the initial bond. Strong degradation at the Y-TZP ceramic/resin cement interface has been noted when the specimens are submitted to aging with stored in distilled water at 37°C for 7 days.[43] Ozcan and others[44] found no adhesion (0 MPa, debonding during aging) between Y-TZP ceramic air abraded with Al_2O_3 and different resin cements (Panavia F2.0, Multilink, Superbond C&B, and Quadrant Posterior Dense) after thermocycling (6000×, 5-55°C). Ozcan and others,[45] after subjecting the specimens to water storage at 37°C for 3 months in the dark, compared bond strength of two resin cements to Y-TZP ceramic using 3-methacryloxypropyltrimethoxysilane (MPS) or MPS/4-methacryloyloxyethy trimellitate anhydride (4-META) silanes. They did not conduct thermocycling, although the effect of thermocycling is controversial.[4,6] Blatz and others[8] noted a decrease in the tensile bond strength values between Y-TZP and two resin cements (adhesive application + RelyX ARC and adhesive application + Panavia F) after 24 hours of storage in water. Passos and others[39] compared dry conditions and 90 days of water storage at 37°C and thermocycling (12,000×, 5-55°C) bonding to Y-TZP with resin cements and showed that bond strength was reduced dramatically after aging. Oyagüe and others[40] evaluated the hydrolytic stability (6 months of water storage at 37°C) of different dual-cure resin cements when luted to zirconia ceramic. They found that the bond strength of Clearfil Esthetic Cement significantly decreased after water storage. The studies showed similar results, as thermocycling and wet storage decreased the bond strength between zirconia and resin cements regardless of the aging process. In this study, wet storage was done by holding the incubator at 37°C for 60 days, and some of the cement bond strength values obtained were zero in the laser and control groups.

CONCLUSIONS

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

1. Bond strengths of resin cements in untreated control and laser-applied specimens were low. APA and TSC methods can improve the bond to the resin cement.
2. Phosphate monomer–containing adhesive resin cements (Panavia 2.0, Clearfil Esthetic, and Rely X U100) in combination with air-particle abrasion or tribochemical silane application of ceramic surface produced a higher bond strength.
3. The tribochemical silica coating/silane coupling system increased bond strength values of phosphate monomer–free adhesive resin cement (Multilink Automix).
4. Regardless of the dual and self-cure resin cement used, laser treatment did not improve resin bond strength.

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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Reinforcement of Teeth With Simulated Coronal Fracture and Immature Weakened Roots Using Resin Composite Cured by a Modified Layering Technique

RS Seyam • EH Mobarak

Clinical Relevance

Reinforcement of teeth with coronal fractures and immature weakened roots with resin composite that was cured by the modified layering technique could be a promising approach for practitioners.

SUMMARY

Objective: The purpose of this study was to evaluate the strengthening effect of resin composite, cured by a modified layering protocol, for teeth with simulated coronal fracture and weakened immature roots.

Methods: Fifty maxillary teeth were decoronated and their apices sectioned to standardize the length to 12 mm. Prepared teeth were equally distributed into five groups. Group 1VF root apices were flared with Pesso drills up to size 6. The roots were flared until a dentin thickness of only 1 ± 0.2 mm remained. Root ends were filled with mineral trioxide aggregate. The canals were backfilled with Vertise Flow following a modified layering protocol using two light-transmitting posts size 6 and 3. Next, a DT light post size 2 was cemented using the same material. Groups 2TS/MF and 3ED/PF were prepared and cured in the same way as group 1VF but filled with Clearfil Tri-S Bond/Majesty Flow and ED Primer II/Panavia F2.0 respectively. Group 4UF was similarly prepared but left unfilled (control). In group 5NW, roots were unflared but similarly filled as in group 3ED/PF. After 24 hours of storage, the fracture load was measured. The degree of cure for each tested material was indirectly measured using microhardness at

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different root levels (cervical, middle, and apical). Data were analyzed using one-way analysis of variance followed by Newman-Keuls post hoc test.

Results: Fracture load results revealed that groups 1VF and 2TS/MF had no statistically significant difference from group 5NW ($p>0.05$). For each tested material, no significant difference was found among microhardness values at different root levels.

Conclusion: It may be possible to reinforce the teeth with coronal fracture and immature weakened roots to be comparable with unweakened ones when composite is applied and cured by the modified layering technique.

INTRODUCTION

Traumatic dental injuries with coronal fracture can lead to pulpal necrosis with cessation of root formation, resulting in an immature root.[1] The immature root can have an open apex coupled with thin dentinal walls, which create endodontic and restorative challenges.[1,2] For management of open apices, mineral trioxide aggregate (MTA) apical barrier has been recommended.[3-5]

Even when treatment of the open apex has been successful, the thin dentinal walls of immature teeth remain a restorative problem because of the high susceptibility to fracture from normal forces of mastication.[1,6] Therefore, when restoring such teeth, it would be advantageous to reinforce the roots in hopes of increasing their resistance to fracture. Researchers have tested the reinforcing effect of different materials including glass ionomer cements, hybrids of glass ionomer cements, and resin composites with different post systems including metal or fiber posts.[1,7,8] Fiber posts have further extended the application of adhesive dentistry in endodontics and have been advocated because of their advantages of corrosion resistance, esthetic appeal, single-visit office placement, and easier removal for endodontic retreatment.[9] In addition, posts with a resin composite that bonds to dentin have surpassed other approaches in increasing the resistance to fracture.[1] Nevertheless, multistep adhesives present with technique sensitivity, difficulty in material application as well as curing effectiveness, and a high C-factor (ratio of bonded to unbonded surfaces) in the root canal. This makes bonding to root dentin a real challenge.

In coronal restorations, to overcome the problems of multistep adhesives and their technique sensitivity, adhesive application steps were simplified, resulting in the most recent single-step self-adhering resin composite.[10] Also, the use of a layering technique was found to increase curing effectiveness,[11] to help in volumetric shrinkage compensation,[12] and to decrease the C-factor.[13]

In root canals, plastic light-transmitting posts and, recently, the newer versions of fiber posts were developed to help in light transmission to increase the depth of resin cure.[14] However, it was reported that light curing from the top of post spaces is insufficient to optimally polymerize light-curing adhesives and resin cements in the apical part of the canal.[15] The use of prolonged curing time was reported to aid in the effective curing of the apical part and thus in obtaining homogenous bonding throughout the canal length.[16] Also, the additional use of the light-transmitting posts in successive sizes to apply resin material in layers to control the C-factor while achieving effective curing presents an attractive solution to bonding to weakened root canals. Previous work highlighted the success of this approach to achieve proper bonding to different root levels.[17] The effect of this approach to reinforce weekend roots has not yet been investigated.

The degree of cure has been tested using different approaches, among them the surface microhardness testing that was used in numerous previous studies,[18-20] since it was found to be a good indicator for the degree of conversion.[21-23]

Thus, the purposes of this study were 1) to compare the fracture load of immature roots restored using different resin materials when applied using a modified layering technique with the aid of successive light-transmitting posts and 2) to indirectly test the efficacy of using the modified layering technique in curing the tested materials by measuring their microhardness at different root levels.

MATERIALS AND METHODS

Tooth Selection and Standardization

Intact human maxillary incisors were collected, cleaned, and disinfected and then stored in saline until use.[24] Root length, mesiodistal, and buccolingual diameters at the cemento-enamel junction (CEJ) of the collected teeth were measured with a digital caliper (Mitutoyo digital caliber, Mitutoyo Corp, Kawasaki, Japan).[8] Teeth (n=50) with similar root sizes and lengths were selected (Figure 1A). The overall ranges of root dimensions of all selected teeth measured at the CEJ were 5 (±1) mm mesiodistally

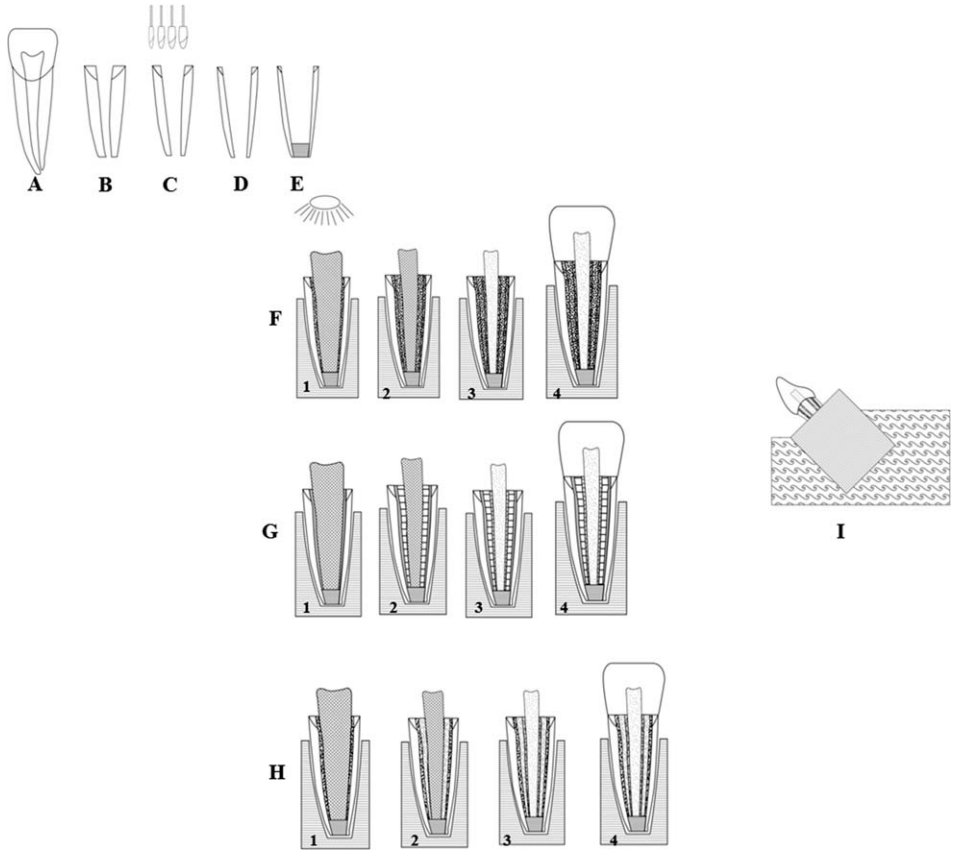

Figure 1. *Schematic drawing illustrating tooth preparation with simulated coronal fracture and weakened immature root (A, B, C, and D). Root end was filled with 3 mm mineral trioxide aggregate apical plug (E). Group 1VF specimens were reinforced by Vertise Flow (VF) in three layering applications (F1, 2, 3, and 4). Group 2TS/MF specimens were reinforced by Clearfil Tri-S Bond (TS) and flowable resin composite Clearfil Majesty Flow (MF) in three layering applications (G1, 2, 3, and 4). Group 3ED/PF specimens were reinforced by dual-cure self-etch primer adhesive/cement ED Primer II(ED)/Panavia F2.0 (PF) in three layering applications (H1, 2, 3, and 4). For each specimen, core buildup was constructed with Filtek Supreme using a former (Figures 1F4, 1G4, 1H4). Each specimen was placed in a specially fabricated jig, subjected to 45° load using a universal testing machine at a crosshead speed of 0.5 mm/min to the palatal surface of cores of all specimens (I).*

and 6 (±1) mm buccolingually. The root length was approximately 15 (±1) mm.

Tooth Preparation and Immature Weakened Canal Simulation With Coronal Fracture

To simulate the worst case scenario of an immature tooth with weakened canal walls and coronal fracture, the anatomical crowns of the teeth were sectioned perpendicular to the long axis of the roots, leaving 2 mm coronal to the CEJ of the buccal surfaces using a saw (Isomet, low-speed saw, Lake Bluff, IL, USA) under water coolant. The root length was standardized to 12 mm by sectioning the root apices (Figure 1B). Forty teeth were flared to simulate the immature root canal walls. Apically, the canals were enlarged to a depth of 12 mm using Peeso reamers (Dentsply Maillefer, Ballaigues, Switzerland) of ascending sizes from size 3 (1.1 mm diameter) to size 6 (1.7 mm diameter; Figure 1C). The canal spaces were flared throughout the canal length using high-speed tapered diamond burs (Dentsply Maillefer), leaving approximately 1 ± 0.2 mm of dentin thickness between the internal prepared root canal wall and the external root surface at the cervical margin (Figure 1D).[25] The remaining thickness was confirmed with a digital caliper (Mitutoyo digital caliber, Mitutoyo Corp).[25] After flaring, buccolingual and mesiodistal radiographs were taken to ensure the homogeneity of root flaring. The other 10 teeth were prepared in the same way apically but without the coronal flaring.[6]

After preparation, each tooth was irrigated using 2 mL NaOCl 5.25% (Clorox, Alexandria Detergent of Chemicals Company, Alexandria, Egypt). Pro-Root MTA (Dentsply Dental, Tulsa, OK, USA) was mixed according to the manufacturer's instructions and used to form a 3-mm apical plug (Figure 1E). A Messing gun (EndoGun, Medidenta, Woodside, NY, USA) was used to place the material as close to the

Table 1: *Materials Descriptions, Manufacturers, Compositions, and Batch Numbers*

Material (Manufacturer)	Description	Composition and batch number
Clearfil Tri-S Bond: Kuraray Medical Inc, Tokyo, Japan	Light-cure single-step self-etch adhesive system	MDP, Bis-GMA, HEMA, photoinitiators, ethanol, water, silanated colloidal silica (061232)
Clearfil Majesty Flow: Kuraray Medical Inc, Tokyo, Japan	Light-cure flowable resin composite	TEGDMA, silanated barium glass filler, silanated colloidal silica, hydrophobic aromatic dimethacrylate, di-camphorquinone (00308A)
Vertise Flow: Kerr Corp, Orange, CA, USA	Light-cure self-adhering flowable resin composite	Matrix: GPDM and methacrylate co-monomers fillers (70wt%): prepolymerized filler, barium glass, nano-sized colloidal silica, nano-sized ytterbium fluoride (3358782)
Panavia F2.0: Kuraray Medical Inc, Osaka, Japan	Dual-cure single-step self-etch resin cement	ED Primer II; Liquid A: HEMA (30%-50%), MDP, N-methacryloyl-5-aminosalicylic acid, water, accelerator (61185); ED primer II liquid B: N-methacryloyl-5-aminosalicylic acid, accelerator, water, sodium benzene sulfinate (61185); Paste A: hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, sodium aromatic sulfinate (TPBSS), N,N-diethanol-p-toluidine, surface-treated (functionalized) sodium fluoride <10%, silanated barium glass (61185); Paste B: MDP, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica, photo initiator, dibenzoylperoxide (61185)

Abbreviations: Bis-EMA: ethoxylated bisphenol A glycol dimethacrylate; Bis-GMA, bisphenol-A glycol dimethacrylate; GPDM: glycerol phosphate dimethacrylate; HEMA, 2-hydroxy ethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate.

apex as possible. A hand plugger and the thick end of moistened paper points (Dentsply Maillefer) were used to condense the material to the apex. Radiographs were taken to ensure proper placement and increment thickness. Teeth were then stored at 37°C and 100% humidity for at least 24 hours. Aluminum foil was wrapped around the roots of each specimen up to 2 mm below the CEJ to act as a spacer.[26] The roots were then centrally embedded in polyester (polyester #2121, Hsien, Taiwan) along their long axis following the methodology described by Mobarak and others.[27] After the setting of the resin, the foil was replaced by polyether light impression material (Examix NDS, GC Corporation, Tokyo, Japan) to simulate the periodontal ligament. In each canal, the smear layer was removed using 5 mL of 17% EDTA followed by 5 mL of 5.25% NaOCl as an irrigant. Final irrigation was accomplished with 10 mL of distilled water,[28] then air dried with high-pressure airflow for five seconds.

Specimen Grouping

The embedded specimens (n=50) were equally divided into five groups according to the preparation and reinforcing material used. Materials used in the present study are presented in Table 1. The tested groups were as follows.

Group 1VF—Ten flared specimens received Vertise Flow (VF) material (Kerr Corp, Orange, CA, USA), a light-cure self-adhering flowable resin composite, in three layering applications. A light coat of VF was first agitated onto the canal walls using a disposable microbrush applicator (Microbrush International Co., Grafton, WI, USA). A light-transmitting plastic post size 6 (Luminex, Dentatus USA Ltd, New York, NY, USA) was then inserted centrally in the root canal at the level of the apical MTA, leaving approximately 0.5 mm thickness of VF resin composite material circumferentially (Figure 1F1). Curing was done on top of the light-transmitting post for 80 seconds using Elipar S10 (3M ESPE Dental Products, St Paul, MN, USA). The light-curing unit had an intensity >800 mW/cm^2 that was checked using a radiometer (Demetron LED Radiometer, Kerr Corp). After curing, the light-transmitting plastic post was removed. A second layer of VF was then applied and cured with the aid of a size 3 light-transmitting plastic post (Luminex, Dentatus USA Ltd) to obtain a circumferential resin composite with thickness of less than 2 mm (Figure 1F2). This layer was also cured for 80 seconds in the same way as the first layer. After curing, the light-transmitting post was removed. The remaining space was then filled with VF, then a DT light post (size 2, Bisco Inc, Schaumburg, IL, USA) was placed slowly and cured for an additional 80 seconds (Figure 1F3). All posts were mounted so the long axis of the post coincided with the long axis of the root. The posts were standardized to a length of 11 mm, and the tip of the curing unit was positioned on top of the post.

Group 2TS/MF—Specimens (n=10) were filled similar to group 1VF, but the light-cure single-step self-etch adhesive system Clearfil Tri-S Bond and flowable resin composite Clearfil Majesty Flow (Kuraray Medical Inc, Tokyo, Japan) were used instead. Clearfil Tri-S Bond was applied according to the manufacturer's instructions (Figure 1G1). Light curing through a size 6 light-transmitting plastic post for 80 seconds was done. The canals were then filled with Clearfil Majesty Flow using the other successive light-transmitting posts (Figure 1G1 and 1G2) and finally the DT Light Post (size 2) following the same protocol applied to the VF group (Figure 1G3).

Group 3ED/PF—Specimens (n=10) were filled and cured following the previously mentioned protocol for the VF and TS/MF groups (Figure 1H1, 1H2, 1H3) but using dual-cure primer adhesive/cement ED Primer II/Panavia F2.0 (Kuraray Medical Inc) that was applied according to the manufacturer's instructions.

Group 4UF—In this group, the flared teeth were left unfilled, except at the apical 3-mm MTA barrier (control). A cotton pellet was placed at a level just below the facial CEJ before core buildup using resin composite but without using intraradicular posts.[6]

Group 5NW—Nonflared teeth were used in this group. The post space was prepared with a specific size 2 drill (Bisco Inc) of 1 mm diameter that corresponds to the DT light post size 2. The canal space was then filled with Panavia F2.0 as in group 3ED/PF following the manufacturer's instructions. However, the material was applied in one increment using only size 2 DT light post and cured for 80 seconds.

For each specimen, the core buildup was constructed with Filtek Supreme (3M ESPE Dental Products) using a former (Figures 1F4, 1G4, 1H4)[29] after Clearfil Tri-S Bond adhesive system application. All specimens were stored in distilled water at 37°C for 24 hours before mechanical testing.

Fracture Load Measurement

Each specimen was placed in a specially fabricated jig. Specimens were subjected to a compressive load on the palatal surface of the cores at an angle of 45° to the long axis of the tooth [5] (Figure 1I) using a universal testing machine (Model LRX-Plus, Lloyd Instruments Ltd, Fareham, UK) at a crosshead speed of 0.5 mm/min (Figure 1I). The maximum load required to cause fracture was recorded for each specimen. Failed teeth were examined for failure mode determination and presented as in Chuang and others.[30]

Table 2: *Fracture Resistance (N) Results of Experimental Groups (n=10)*[a]

Experimental Group	Fracture Resistance, Mean (SD)
Group 1VF	313.43 (103.7)[a]
Group 2TS/MF	324.52 (114.8)[a]
Group 3ED/PF	243.47 (81.4)[b]
Group 4UF	169.23 (61.6)[c]
Group 5NW	370.10 (86.6)[a]

[a] *Groups with the same lowercase subscript letter are not statistically significant (Newman-Keuls test, p>0.05).*

Microhardness Evaluation

Three representative teeth from groups 1VF, 2TS/MF, and 3ED/PF were prepared and restored in the same way. After 24 hours, using the Isomet saw, one section (2- ± 0.05-mm thickness) was obtained from each of the cervical, middle, and apical levels. Surface hardness was determined using the Vickers Micro-hardness Tester (Model HVS-50, Laizhou Huayin Testing Instrument Co, Ltd, Laizhou, Shandong, China). A load of 200 g was applied to the surface of the specimens for 10 seconds. Three indentations were done 100 μm apart from each other for each of the different root levels (cervical, middle, and apical) of each specimen, from which a mean Vickers hardness number (VHN) value was calculated.[31]

One-way analysis of variance (ANOVA) was performed to detect any intergroup differences. The Newman-Keuls test was used for post hoc pairwise multiple comparisons. The level of significance was set at $p>0.05$. All statistical calculations were done using computer programs Microsoft Excel 2007 (Microsoft Corporation, New York, NY, USA) and SPSS (Statistical Package for the Social Science; SPSS Inc, Chicago, IL, USA) version 15 for Microsoft Windows.

RESULTS

The results of the mean failure values (N) and standard deviation (SD) for all tested groups are shown in Table 2. The results of the one-way ANOVA test indicated a significant difference among tested groups ($p<0.001$). Group 1VF and group 2TS/MF were not statistically different from group 5FW (the nonflared group), while they were statistically significantly higher than group 3ED/PF and group 4UF (Newman-Keuls test, $p<0.01$). Modes of failure

Table 3: *Modes of Failure Among the Tested Groups*

	Fracture Plane Location			Fracture Plane Direction		
	C	M	A	Oblique	Horizontal	Cracked Vertically
Group 1VF	8	—	2	10	—	—
Group 2TS/MF	10	—	—	10	—	—
Group 3ED/PF	10	—	—	10	—	1
Group 4UF	10	—	—	10	—	—
Group 5NW	9	—	1	9	1	2

Abbreviations: ED/PF, ED primer II/Panavia F2.0; NW, unweakened roots; TS/MF, Clearfil Tri-S Bond/Clearfil Majesty Flow; UF, unfilled flared canals; VF, Vertise Flow.

of each group are presented in Table 3. The predominant mode of failure was cervical.

The descriptive data and test of significance of microhardness results are shown in Table 4. The one-way ANOVA test revealed no significant difference among the cervical, middle, and apical microhardness values of each tested group.

DISCUSSION

Endodontic treatment of fractured immature anterior teeth with thin dentinal walls is one of the worst-case scenarios that can face practitioners. The quantity of residual coronal structure and the residual root dentin thickness is a crucial factor that designates the type of definitive restoration.[32] When coronal damage is minimal, additional retention effect gained from post placement is not that critical. However, it is widely agreed that in a severely damaged coronal structure, placement of a post provides sufficient retention to the core.[32,33] Tooth reinforcement was also suggested to decrease the incidence of root fracture with thin dentinal walls.[1,6,8]

Maxillary human anterior teeth were used for this study as they are more susceptible to traumatic injuries. In the present study, extreme care was taken in the selection of the tooth dimensions, their distribution within the experimental groups, and their standardized preparation, thus minimizing the effect of human tooth variation, as was recently discussed by Tanalp and others.[34] Artificial crowns could alter the distribution and transmission of stresses into a post-root complex,[35] masking the reinforcing effect of tested approaches. Therefore, in this study as well as in other studies,[25,29] no crown restorations were made for teeth to allow evaluation of these approaches. Although the test standards and conditions are not identical to the clinical situation, they allow the comparison of different materials within given standards, as explained by Kivanc and others.[25] Further laboratory studies should consider the impact of completed crowns.

The results of the present study revealed that there was a significant increase in fracture load when reinforcing materials and the new fiber post were used compared with unfilled flared canals. Three-dimensional finite element analysis simulations have pointed out the relevance of using restorative materials with elastic properties similar to dentin, such as resin composite, for more favorable performance of the restored teeth under stress.[9] Previous studies on fracture resistance, although different in methodology, confirmed the reinforcing effect of resin composite materials used with the fiber post.[7,8] In these studies, either weakened or immature roots were simulated. Yet, in the current study, immature weakened roots (thin-walled tapered canals with immature apices) were simulated. To simulate immature tooth preparation, tubular canals were prepared apically. In addition, coronally, teeth were decoronated and further flared out, simulating thin-walled canals. Nevertheless, these previous studies[7,8] and ours were common in that the root canals were enlarged more than 1.5 mm in diameter and fracture load was tested using static loading. Although dynamic loading would have replicated the clinical situation, static loading was used to minimize the experimental variables.[7]

Table 4: *Vickers Microhardness Results*

Material	Coronal	Middle	Apical	*p* Value
Vertise Flow (Group 1VF)	39.81 (2.9)	38.94 (5.5)	38.43 (3.9)	0.77
ClearFil Majesty Flow (Group 2TS/MF)	42.86 (5.9)	39.50 (4.4)	41.20 (5.9)	0.45
Panavia F2.0 (Group 3ED/PF)	37.83 (5.0)	37.31 (6.1)	36.19 (2.7)	0.76

The achieved reinforcement in the current study may be due to characteristics of the materials used. For Majesty Flow, the high filler content up to 62% by volume and polymerization shrinkage of 1.88%, as reported,[36] might render it to be comparable to the conventional micro-hybrid resin composite while maintaining the recommended flow behavior. For the VF, it belongs to the new category of restorative materials defined as "self-adhering resin composite." These materials are claimed to eliminate the need for a separate bonding application step, thus simplifying the restorative procedure. For this reason, VF may be considered to start the eighth generation of dental adhesive systems or to represent a cross-link between all-in-one adhesive systems and flowable resin composite. VF was suggested to bond to tooth structure in two ways: primarily through the chemical bond between the phosphate functional groups of a glycerol phosphate dimethacrylate monomer and calcium ions of the tooth and, secondarily, through a micro-mechanical bond as a result of an interpenetrating network formed between the polymerized monomers of VF and collagen fibers of dentin.[10] Recent studies have raised a concern about the high water sorption properties of the VF that might affect its mechanical properties and sealing ability.[10,37] However, VF in this study was enclosed in the root canal by MTA apically and core material coronally, which might have had a positive effect on isolating this material from the direct deteriorating hydrolytic effect of surrounding fluids. Although the self-adhering material represents an attractive approach to clinicians, long-term success is not yet validated, as these materials have only been recently introduced into the market.

Contraction stresses are affected by volume and flow of the used resin as well as the cavity configuration (C-factor). Within a root canal, the C-factor might exceed 200.[32] In the present study, minimizing the bonded to the unbounded ratio was done through the modified layering technique, in which the volume of each resin layer was decreased; thus, the total shrinkage stress is expected to decrease accordingly.[32] The bonding to root dentin results obtained in previous research,[17] when the modified layering technique was used, may support this suggestion. Also, through the use of the light-transmitting posts, the layers toward the post were considered to be unbonded. The easy removal of the light-transmitting post, Luminex, in the pilot study after curing confirmed the lack of bonding between the post and the resin composite.

The predominant mode of failure in the present study was the cervical obliquely directed mode. Most of the obliquely oriented fracture lines extended from the palatal to the buccal side. This oblique orientation was explained by Fukui and others[29] to be due to specimens being loaded at 45° to their long axis. The reported mode of failure corroborates other studies explaining the distribution of stresses along the fiber post due to its modulus of elasticity being similar to dentin.[25,29,30,35]

Microhardness results revealed no significant difference between the different root levels (cervical, middle, and apical) in each tested group. The light-transmitting post, Luminex, was used with the aim of allowing a better polymerization in the deepest canal regions.[38] For such purpose, the Elipar S10 curing unit was also used. This unit has been reported to have the capacity to maintain, according to the manufacturer, an intensity $\geq$800 mW/cm^2 at a depth of 7 mm. Having layers of resin composite of less than 2-mm thickness,[36] with the aid of different sizes of light-transmitting posts, might have also helped the light-transmitting post to activate the thin resin layer surrounding the post surface, enabling the resin to cure promptly along the post.

Based on this study, reinforcement was achieved with the light-cured materials that were applied and cured using the modified layering approach presented in the current study. Nevertheless, further long term in vitro and in vivo studies are still required. Also, the impact of fatigue loading and thermal cycling on fracture should be considered.

CONCLUSION

It may be possible to reinforce teeth with coronal fractures and immature weakened roots to be comparable with unweakened teeth when composite is applied and cured by the modified layering technique.

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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Degree of Conversion and Hardness of a Silorane-Based Composite Resin: Effect of Light-Curing Unit and Depth

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Clinical Relevance

Forty seconds of light exposure in increments up to 3 mm of a novel silorane-based composite resin produced higher degree of conversion and hardness than thicker increments, using halogen or LED units.

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SUMMARY

Purpose: **To investigate the effect of different light-curing units and depths on the degree of conversion (DC) through Fourier transform infrared spectroscopy (FTIR) and Knoop Hardness Number (KHN) of a silorane-based composite resin (Filtek LS, 3M ESPE, St Paul, MN, USA) (LS).**

Materials and Methods: **LS specimens mounted in a particular designed matrix were photoactivated by three light-cure units (LCUs) at depths of 2, 3, 4, and 5 mm. The DC was determined in a FTIR spectrometer with an attenuated total reflectance accessory. The KHN was measured in an automatic microhardness tester. The results were analyzed using the Friedman and Spearman statistical tests (α=0.05).**

Results: **There was no effect of LCUs on the DC (p=0.472) or KHN (p=0.174) for all of the studied depths. The highest DC and KHN means were found at 2-mm depth, which were**

not statistically different from 3-mm depth, but were higher than 4-mm and 5-mm depths (p=0.007). Spearman analysis found a positive linear correlation between the variables KHN and DC (r=0.858, p<0.000).

Conclusions: The LCUs' effect was not verified. Values of DC and KHN for LS decreased with increasing depth. The highest values for both DC and KHN were obtained at depths of 2-3 mm.

INTRODUCTION

Direct composite resin (CR) restorations have been widely used in restorative dentistry procedures, in both anterior and posterior regions.[1-4] However, despite the recent innovative improvements in techniques and materials, clinical problems are still observed in CR restorations.[5]

Regarding the materials' properties, polymerization shrinkage stress is still considered to be one of the main drawbacks of CRs.[6] It is dependent on the composite constitution, cavity configuration (C-factor), elastic modulus, and degree of conversion (DC). To reduce the phenomena of volumetric contraction and shrinkage stress during polymerization, studies have sought to change the proportion, size, quantity, and shape of inorganic fillers present in CRs.[7] Other studies analyzed the proportion of organic matrix/inorganic particles and its relationship with the polymerization shrinkage, concluding that an excessive amount of fillers (above 80 wt%) could modify the rheology of the CR, reducing its ability to flow during the initial curing stage (pre-gel state) and increasing the polymerization shrinkage stress and the contraction of the remaining tooth structure.[8-10] Those factors could lead to debonding, microleakage, postoperative sensitivity, marginal discoloration, secondary caries, and/or eventual restorative failures. Another method to minimize the disadvantages of CRs is to modify the chemical structure of the organic matrix used. During attempts to improve the Bis-GMA molecule, different formulations were developed, such as urethane matrix,[1,2,4,11] the espiro-orthocarbonates with their variations,[12-14] and Ormocer (organic modified ceramic), in which silica particles were inserted into the organic matrix, reducing polymerization shrinkage and increasing the conversion sites.[15,16] More recently, a silorane-based resin composite was established that claimed to present a lower shrinkage rate compared to conventional methacrylate CRs. This material is a combination of a siloxane backbone and oxirane rings. The siloxane block shows high hydrophobicity, and the oxirane rings are responsible for the decrease in volumetric contraction during curing, based on a distinct process: cationic ring-opening polymerization.[17] Studies of silorane-based composites report polymerization shrinkage values less than 1% in volume,[17,18] excellent resistance to bending,[18,19] favorable physicochemical and mechanical properties relative to methacrylate-based composites,[17-20] adequate biocompatibility,[21-25] and satisfactory short-term clinical performance.[26,27]

Adequate polymerization is a crucial factor for the optimization of the physical and mechanical properties of CRs, such as hardness and color stability. For a light-cured resin, such as the silorane CR, the polymerization is influenced, among other factors, by irradiation time, intensity of power, and the type of unit activation (eg, halogen, light-emitting diode [LED]). Factors such as the capacity for conversion of the monomers and its relation with different types of light-curing units (LCUs), depth of cure, and mechanical properties have been discussed for methacrylate CRs.[1,17,28-32] However, silorane CR presents a particular polymerization initiator system based on the cationic opening of the oxirane rings. As the initiation process differs from the conventional and well-established photoinitiation of the methacrylate CR based on camphorquinone, it is important to test the silorane CR under different light sources.

Few studies have determined the degree of conversion for silorane-based composites,[20,33,34] its relation with hardness at different depths,[20,35] or the effect of the light source. This study aimed to evaluate the effect of different light-curing units

Table 1: *Emission Spectrum Measures*

Light-Curing Units	Radii-cal	Bluephase G2	Optilux 501
Spectral range, nm	420-520 (S)	390-520 (S)	385-515 (S)
	440-480 (M)	380-515 (M)	400-505 (M)
Maximum peak of emission, nm	Peak: 460 (S)	Peak 1: 412 (S), 410 (M)	Peak: 494 (S)
	Peak: 460 (M)	Peak 2: 455 (S), 460 (M)	Peak: (M)[a]

Abbreviations: M, data provided by the manufacturer; S, data found in the present study measured by spectrometer (USB 4000, Ocean Optics, Dunedin, FL, USA).
[a] *Data not provided by manufacturer.*

Table 2: *Power Density and Energy Density for Specific Regions of Spectrum of Each Light-Curing Unit*[a]

Light-Curing Units Spectrum Characteristics	Power Density, mW/cm^2			Energy Density, J/cm^2[b]		
	Radii-cal	Bluephase G2	Optilux 501	Radii-cal	Bluephase G2	Optilux 501
Manufacturers' total spectrum	1200	1200	800-900	48	56	32-36
Study total spectrum	894	1430	832	35.76	57.20	33.28
Spectral range (400-515 nm)	836	1316	805	33.44	52.64	32.20
Spectral range (190-400 nm)	30	66	24	1.2	2.64	0.96
Spectral range (>500 nm)	53	85	43	2.2	3.4	1.72

[a] *Power measured by potentiometer (Ophir 10A-V2-SH, Ophir Optronics LTD, Jerusalem, Israel). Power density (mW/cm^2) = Power (mW) × Area (cm^2). Calculation of power density in each spectral range provided by data analysis software (OriginPro 8.0, OriginLab Corp, Northampton, MA, USA).*
[b] *Energy density for a time of 40 seconds.*

and depths on the degree of conversion by Fourier transform infrared spectroscopy (FTIR) and Knoop Hardness Number (KHN) of a silorane-based composite (Filtek LS, 3M ESPE, St Paul, MN, USA) (LS).

METHODS AND MATERIALS

Design of Experiment and Materials Employed

This *in vitro* experimental study was outlined in four randomized complete blocks. This design allowed for control of unknown sources of variation, with each block containing one combination of each factor being studied. The study factors were the depth of cure at four levels (2, 3, 4, and 5 mm) and the light-curing units RC (LED, Radii-cal, SDI Limited, Bayswater, Australia), BP (LED, Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein), and OP (Halogen light, Optilux 501, Kerr Corp, Orange, CA, USA). The LCUs were previously tested for spectral distribution, irradiance (power density), and energy dose (Tables 1 and 2). The experimental units were specimens (n=48) made of silorane-based composite resin Filtek LS, color A3 (Lot: 9NER; 3M ESPE) (Table 3). The response variables were the degree of conversion and Knoop microhardness.

Characterization of Filtek LS

Differential thermal analysis (DTA) and thermogravimetric analysis (TG) were carried out using the same equipment (SDT 2960, TA Instruments, New Castle, DE, USA). Micro-Raman spectroscopy was performed using a spectrometer (IHR550, Horiba Jobin Yvon, Kyoto, Japan). The energy dispersive x-ray spectroscopy (EDX) measurements were performed in a fully automatic sequential fluorescence x-ray spectrometer (ZSX Primus II, Rigaku Corp, Tokyo, Japan).

Specimen Preparation

A matrix consisting of a nylon cylinder with a depth of 3 mm with a central perforation 5 mm in diameter in the bottom was designed. Three interchangeable rings with a thickness of 1 mm with a central perforation 5 mm in diameter were constructed to be placed into the master cylinder to provide different depths. Specific disks were constructed to guide the tip of each LCU to a distance of 2 mm from the bottom of the first specimen. This matrix was developed in order to separately evaluate the depth of cure of the polymerized resin at each depth, without needing to section the specimens (Figure 1).

For the 5-mm deep specimen, the matrix was placed over a 0.1-mm thick glass plate. Its central perforation was then filled with LS in a single 1-mm thick, 5-mm diameter increment. A 0.1-mm thick cover glass was placed on top, and the piece was subjected to a weight of 1200 g for 20 seconds. For the 4-mm depth, the first interchangeable ring was placed over a glass plate. Its central perforation was filled with LS in a single 1-mm thick, 5-mm diameter increment. A 0.1-mm thick cover glass was placed on top, and the piece was subjected to a weight of 1200 g for 20 seconds. Then, the ring containing the LS was placed into the matrix, from bottom to top. The same

Table 3: *Material Safety Data Sheet : Filtek LS (3M ESPE)*

Composition	No. CAS	%/Wt
Silane treated quartz	100402-89-9	60-76
Yttrium trifluoride	13709-49-4	5-15
Bis-3,4-epoxycyclohexylethyl-phenyl-methylsilane	154265-59-5	5-15
3,4-Epoxycyclohexylcyclopolymethylsiloxane	—	5-15
Mixture of other by-products	Mixture	<5
Mixture of epoxy-mono-silanole by-products	Mixture	<5
Mixture of epoxyfunctional di- and oligo-siloxane by-products	Mixture	<5
Mixture of alpha-substituted by-products	Mixture	<5

Abbreviation: CAS, Chemical Abstracts Service

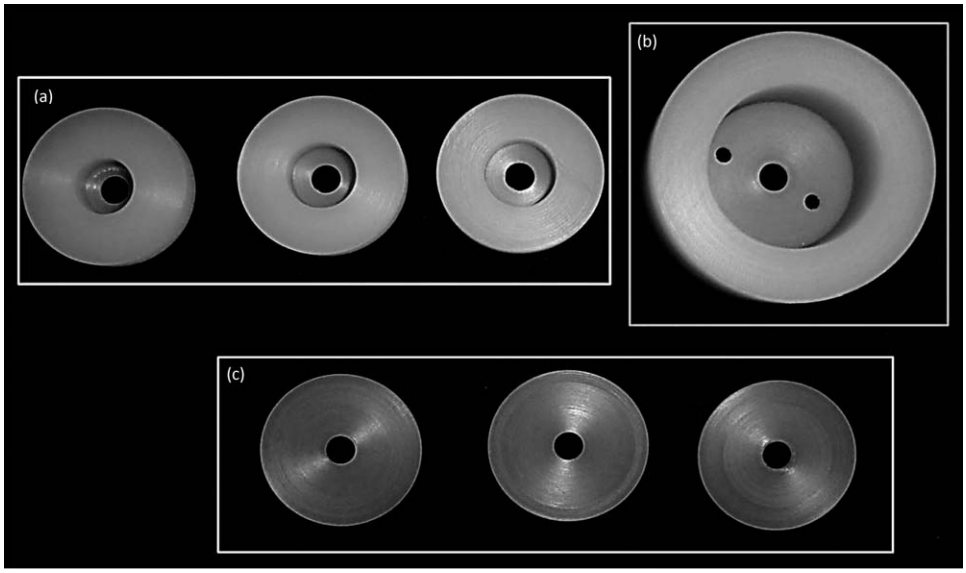

Figure 1. *Matrix used in the study, showing all of its components. (a): Special rings for each light-curing unit. (b): Central cylinder. (c): Interchangeable rings.*

procedure was performed for the 3- and 2-mm depths. The special rings were placed for each LCU, and the set was polymerized for 40 seconds (Figure 2). After photoactivation, the specimens were removed from the nylon matrix, identified (bottom and top), and grouped according to the previous randomization. Then, they were stored at 37°C in the absence of moisture and light for a period of seven days.

Determination of Degree of Conversion

A FTIR spectrometer (Nicolet 6700, Thermo Fisher Scientific Inc, Fitchburg, WI, USA) coupled to a microscope (Centaurus, Thermo Fisher Scientific Inc) and an attenuated total reflectance accessory (Micro-ATR- Ge, Thermo Fisher Scientific Inc) was used. Micro-FTIR spectra in the region of 650-4000 cm^{-1} were collected in absorption mode with a spectral resolution better than 4 cm^{-1} and acquired with 256 accumulations under a dry nitrogen purge. The spectrometer was configured as follows: everglow source, KBr beam splitter, Ge-coated, and HgCdTe detector. Three distinct spectra were measured on the bottom of all 48 specimens. The first was in the center of the specimen, the second was 1.25 mm to the left of the first, and the third was 1.25 mm to the right of the first.

The spectrum of the uncured resin was determined using the previous characterization as a basis for calculating the DC. The bands used as internal standards in this study were a "C-O-C" (883 cm^{-1})

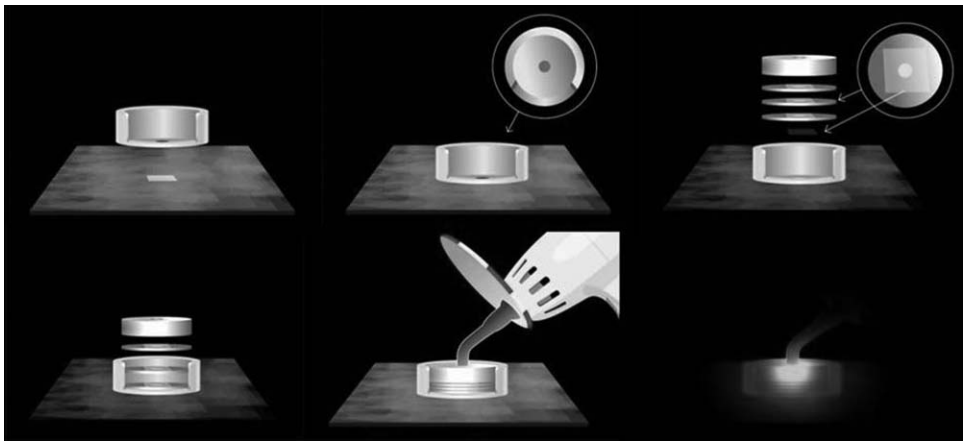

Figure 2. *Representation of the experimental sequence.*

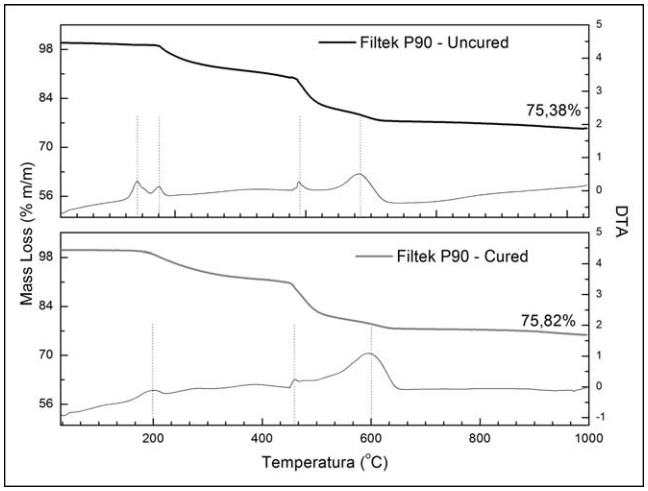

Figure 3. *Measures of TG/DTA carried out for cured and uncured LS.*

stretching band and a "C-H" (2919 cm^{-1}) stretching band. The following calculation was performed in order to reach the DC:

$$DC = \left\{ \frac{\left[\left(\frac{AC-O-C}{AC-H} \right)_{UP} - \left(\frac{AC-O-C}{AC-H} \right)_{P} \right]}{\left(\frac{AC-O-C}{AC-H} \right)_{UP}} \right\} \times 100(\%)$$

DC indicates degree of conversion; A, height of the bands; UP, unpolymerized monomer; and P, polymerized monomer.

Determination of Knoop Microhardness

The KHN was determined in an automatic microhardness tester (FM-ARS-9000, Future Tech Corp, Kawasaki, Japan). Five indentations were made on the bottom of the 48 specimens under a force of 50 gf for 50 seconds. The first indentation was located in the center of the sample. The others were located 1.25 mm from the center. The KHN was calculated using the following formula: $KHN = 14229 \times P / d^2$, where L, P= force (gf) and d= length of long diagonal (μm).

Statistical Analysis

The Kolmogorov-Smirnov test and the Levene test were used to check the normality and the variances equality of data obtained for KHN and DC. As the data did not present a normal distribution, the nonparametric Friedman test was used to evaluate the effect of the individual factors. Spearman correlation test was used to investigate the relationship between the hardness and degree of conversion. A significance level of 5% was utilized for all tests.

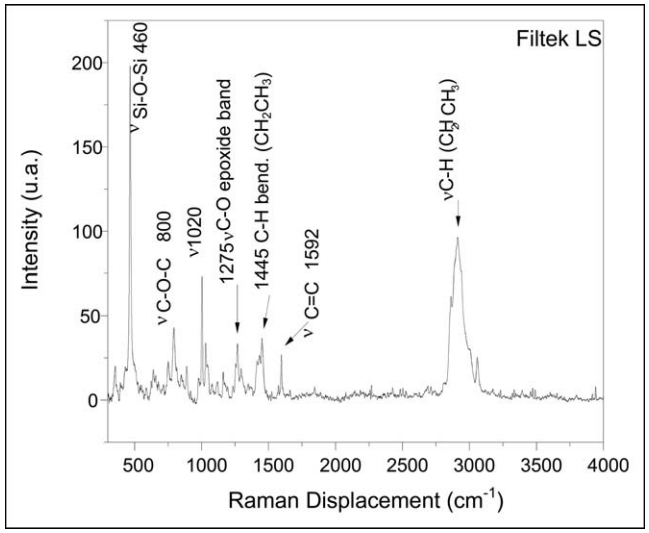

Figure 4. *LS spectrum obtained by Raman spectroscopy.*

RESULTS

Characterization of LS

Thermal analysis revealed a filler/weight ratio of 75.82% (Figure 3). Energy-dispersal x-ray spectroscopy demonstrated the presence of yttrium and iodine as components of the initiation system. Raman spectroscopy qualitatively verified the bands related to the vibrational modes of the epoxy groups and the presence of C=C and C-H bonds (Figure 4).

Determination of Degree of Conversion

DC results for each study factor (depth of cure and light-curing units) in each block were obtained. The Friedman test showed no significant effect (p=0.472) of LCUs on the DC (Table 4). However, a significant effect (p=0.007) was observed when the depths were varied (Table 5). The highest DC mean was found at 2-mm depth, which was not statistically different from 3-mm depth, but was higher than 4-mm and 5-mm depths (p=0.007). The absorbance spectra obtained during the FTIR showed that while the band of the C-H bond (2919 cm^{-1}) remained unchanged (internal standard) after the polymeriza-

Table 4: *Friedman Test to Assess the Effect of Light-Curing Units (LCUs) on the Degree of Conversion of the Filtek LS**

LCUs	Blocks	Median (Min-Max)	Sum of Orders
Bluephase G2	4	56.94 (32.15-62.12)	11.0
Optilux 501	4	50.79 (33.14-60.08)	7.0
Radii-cal	4	47.29 (37.96-54.40)	6.0

* p=*0.472.*

Table 5: *Friedman Test to Assess the Effect of Depths on Degree of Conversion*

Depth, mm	Blocks	Median (Min-Max)	Sum of Orders*
2	4	72.85 (65.71-78.66)	16.0[a]
3	4	65.05 (54.61-70.29)	12.0[a,b]
4	4	40.78 (10.27-56.86)	8.0[b,c]
5	4	21.15 (7.08-31.34)	4.0[c]

** Values followed by different letters indicate significant difference (p=0.007; least significant difference=7.96).*

tion (Figure 5), a variation in the bands related to the oxirane rings was found (883 cm^{-1}) (Figure 6), suggesting the opening of the oxirane rings.

Determination of Knoop Microhardness

The Friedman test showed no significant effect (p=0.174) of the LCUs on the KHN (Table 6). The same test showed a significant effect (p=0.007) of depth on the KHN (Table 7). The highest KHN mean was found at 2-mm depth, which was not statistically different from 3-mm depth, but was higher than 4-mm and 5-mm depths (p=0.007).

Correlation Between KHN and the Degree of Conversion

Spearman analysis revealed a positive linear correlation between the KHN and DC for LS (r=0.858, p<0.000) (Figure 7).

DISCUSSION

The physical and mechanical properties of CRs are known to be directly influenced by the degree of conversion obtained during the polymerization. For methacrylate-based composites, the DC can vary from 43% to 75%, depending on their composition (photoinitiator system, form and amount of particles, and type of organic matrix), the thickness of resin layers, the intensity and spectrum of light radiation, the exposure time, and the distance from the light-curing unit.[31,36,37] More recently, a silorane-based composite resin (LS) was developed and became available on the market. Therefore, it is essential to study the depth of cure of this particular CR.

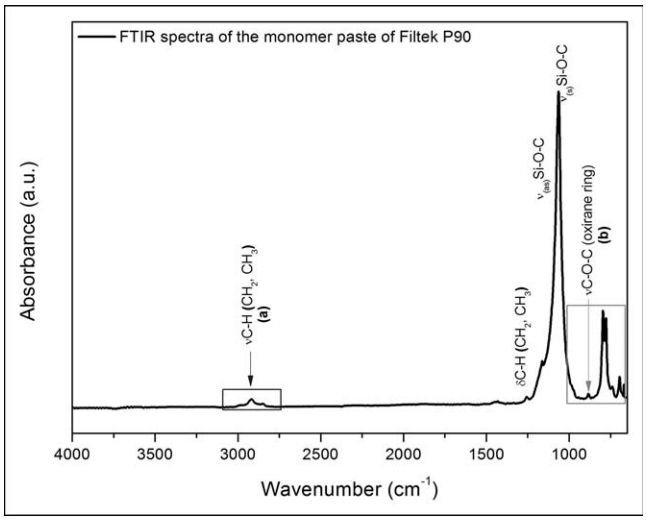

Figure 5. *Identification of bands used to measure the degree of conversion. (a:) Bonding "C-H" (2919 cm^{-1})/internal standard. (b): Band related to the oxirane ring (883 cm^{-1}).*

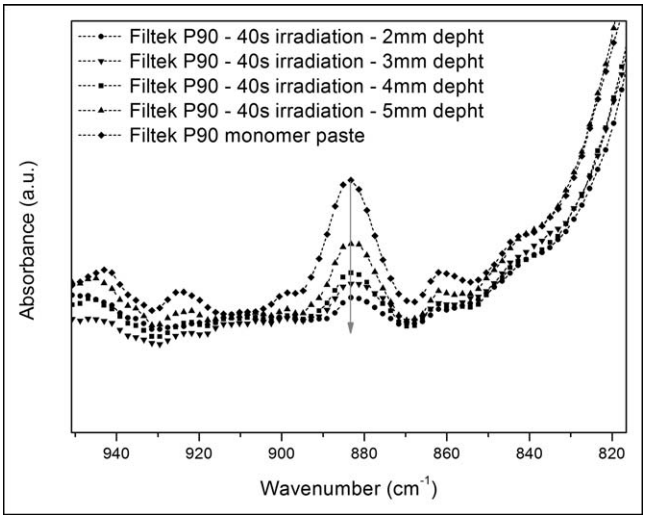

Figure 6. *View the reduction of the band "C-O-C" (883 cm^{-1}) suggesting the opening of the rings in different depths.*

In the present study, the emission spectrum, power density, and energy emitted by the three LCUs were initially determined in accordance with ISO/TS 10650. The lack of uniformity in the emission spectrum of LCUs reported by manufacturers justifies the need for verification.[20] For the spectral range between 190 to 400 nm and recommended power densities up to 100 mW/cm^2, none of the devices tested exceeded this value. Between 400 and 515 nm, it is recommended that the power density should not be less than 300 mW/cm^2 or more than 1000 mW/cm^2. It was found that only the BP exceeded the recommended values, featuring a power density of

Table 6: *Friedman Test to Assess the Effect of Light-Curing Units (LCUs) on the Knoop Hardness Number (KHN)**

LCUs	Blocks	Median (Min-Max)	Sum of Orders
Bluephase G2	4	33.10 (27.33-36.88)	11.0
Radii-cal	4	34.66 (28.10-33.71)	7.0
Optilux 501	4	32.69 (27.21-34.28)	6.0

** p=0.174.*

Table 7: *Friedman Test to Assess the Effect of Depth on the Knoop Hardness Number**

Depth, mm	Blocks	Median (Min-Max)	Sum of Orders*
2	4	41.76 (39.98-41.82)	16.0[a]
3	4	38.67 (37.38-38.71)	12.0[a,b]
4	4	33.09 (20.11-34.74)	8.0[b,c]
5	4	20.23 (10.22-24.81)	4.0[c]

** Values followed by different letters indicate significant difference (p= 0.007; least significant difference=7.96).*

1316 mW/cm^2. High levels of photons can generate faster polymerization, impairing CR to flow during the pre-gel state and increasing the stress in the tooth-restoration interface. Above 515 nm, only OP followed the recommendation of up to 50 mW/cm^2, showing a 43-mW/cm^2 power density. None of the light emission spectra of each LCU supplied by the manufacturers was similar to those found by spectroscopic tests in the present study, which may have impacted the power density values. However, there was no influence of LCUs in the two response variables, perhaps because of the 40-second light-curing time, which provided energy density higher than the minimal dose recommended by the manufacturer (20 J/cm^2). Furthermore, DC and KHN are not only dependent on power density but also on the composition of the resin (photoinitiator, type, and size of particles), time of curing, and distance of composite to light source.[7,20,31]

The LS characterization was performed by DTA and revealed a 76% load (weight/weight), which was in accordance with the information provided by the manufacturer. The EDX qualitatively demonstrated the presence of yttrium in the inorganic particles and iodine in the photoinitiator system. These findings reinforce the mechanisms of LS polymerization by opening the oxirane rings with acid cations.[17] Raman spectroscopy was performed to qualitatively verify the bands related to vibrational modes of the epoxy groups and the presence of C=C and C-H bonds. This test would also be used to verify the DC; however, the composite exhibited a luminescence incompatible with the accomplishment.

Some studies have determined the depth of cure of composites by determining DC and hardness.[20,30,35] The DC can be measured by FTIR,[20,30,32-34,38] Raman spectroscopy,[37,39,40] nuclear magnetic resonance,[38] differential scanning calorimetry,[41] and differential thermal analysis.[42] An adequate polymerization of composites is directly related to the DC, providing optimal physical-chemical and mechanical properties as well as satisfactory clinical performance. In the present study, in order to determine the DC, a nylon cylindrical matrix with a central 5-mm diameter hole, sectioned at four depths separated by 1 mm, was used for specimen preparation. Nylon was chosen to facilitate specimen removal and, because of its similarity to the optical characteristics, reflection and refraction of the dental structures. A specific ring for each LCU was developed to perfectly guide the LCU tip, ensuring the exact depth. The construction of this matrix sectioned at depths of 1 mm aimed to prevent the DC from the heat caused by cutting the specimens.[20] The absorption of water from the cooling process could also change the visualization of the bands during the FTIR analysis.[31]

With respect to the DC of silorane composite, studies have been conducted with distinct objectives using different methodologies, making it difficult to compare the results.[33,34,38,43,44] Only one study assessed the DC, hardness, and the correlation between them at different depths.[20] The present study showed a decrease in the DC as a function of the increased depth, which is in accordance with other studies.[30,34] The mean DC found for the depth of 2 mm in this study was 72.85%, which is higher than the maximum results obtained for silorane composite found in the literature, which ranged from 50% to 64.9%.[33,34,38,43] This difference may be explained by the distinct power densities and curing times selected. As in this present study, the DC did not differ for the 2- and 3-mm depths, and it would be acceptable to use increments of 2 to 2.5 mm as recommended by the manufacturer.

Hardness is an indicator of ductility, smoothness, and abrasion capacity, and may predict the surface wear resistance of a CR.[18] Previous studies that used

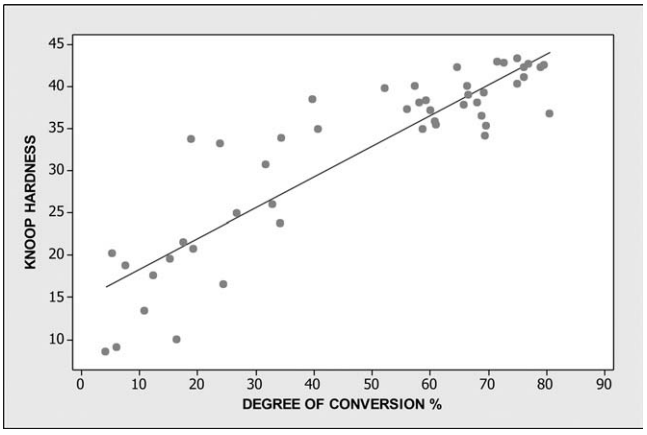

Figure 7. *Scatter diagram of the DC results (%) in the x-axis, plotted as a function of KHN in the y-axis.*

methacrylate composites found that the increase of the amount of inorganic particles by volume produced a high surface hardness and improved the DC.[45] As observed for the DC tests, silorane hardness results are difficult to compare due to methodologic differences.[7,19,20,32,33,35,46,47] The Knoop microhardness mechanical test was used to indirectly determine the depth of cure. This test can be used to measure the hardness of thin regions because the indentation is narrower than that of the Vickers hardness test. Additionally, it is not influenced by the phenomenon of elastic recovery that is common to polymeric materials. A mean KHN of 41.76 was found at the depth of 2 mm. This value is relatively smaller than those found in the literature for methacrylate CRs.[18,47] This can be explained by the low proportion of filler by volume (55%).[18] The KHN results for the silorane composites found in the literature ranged between 43 and 60.[18,47] Similar to the DC, the KHN decreased with depth, and it was significantly higher at a depth of 2 mm compared to 4- and 5-mm depths, but did not differ from 3 mm. A previous study agreed with these results showing that the silorane-based composite was capable of acceptable (80%) bottom-to-maximum microhardness ratios at increments up to 3 mm, at both low and high irradiances.[35] These findings differ from other results[20] that have found no statistically significant difference between the 2- and 6-mm depths using the Vickers microhardness test. This variance in the results may be due to the different hardness tests employed and to the fact that the last study light cured each increment separately.

Similar to the results of another study, a strong correlation was observed between the DC and KHN, which indicated that hardness is a suitable mechanical indicator of DC.[48] Even though there was a positive linear correlation between hardness and DC, it is necessary to further investigate the polymerization process of silorane composites because this material exhibited lower hardness compared to methacrylate composites, even though the DC was acceptable.

CONCLUSION

After 40 seconds of light exposure, different LCUs (halogen or LED) did not affect the DC and KHN of novel silorane-based composite resin at the depths of 2, 3, 4, and 5 mm. Values of DC and KHN decreased with increasing depth. The highest values for both DC and KHN were obtained at depths of 2-3 mm. Although the LS presented a satisfactory degree of conversion, the low Knoop hardness values suggest the need for further studies that address the wear resistance of these materials.

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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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