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## **OPERATIVE DENTISTRY**

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

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# Clinical Evaluation of Stress-reducing Direct Composite Restorations in Structurally Compromised Molars: A 2-year Report

S Deliperi • DN Bardwell • D Alleman

### Clinical Relevance

If a bonding preservation technique and a stress-reducing protocol are adopted, direct large-size composite restorations performed equally well regardless of the adhesive system used after two years of clinical service.

### **SUMMARY**

Objective: To evaluate the clinical performance of class II large-size direct composite restorations.

Materials and Methods: Fifty (50) patients 18 years or older were included in this clinical trial restoring 75 vital molar teeth with large-size cavities.

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Inclusion Criteria: Occlusal extension greater than two-thirds the intercuspal distance and proximal extension greater than half the distance between line angles. Teeth with residual cavity walls less than 1 mm and with one or more cusps involved were excluded. Teeth were randomly divided in three groups. Group 1: Opti-Bond FL; group 2: Scotchbond 1 XT; group 3: PQ1. Total-etching was performed using 35% phosphoric acid followed by the application of 2% chlorhexidine gluconate in the three groups. All teeth were restored using Vit-l-escence microhybrid composite resin. The proximal surface was built up first, followed by dentin and enamel occlusal surface stratification; wedge-shaped increments of composite resin were placed and cured using the Ultra-Lume V curing light through a combination of pulse and progressive curing techniques.

Results: Restorations were evaluated at sixmonth intervals during the two-year period

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using a modified US Public Health Service criteria by two independent evaluators precalibrated at 85% reliability. No failures were reported and  $\alpha$  scores were recorded for all parameters. Statistical analysis was performed using a  $\chi^2$  test and the Fisher exact test ( $\chi^2$ =10.6; p=0.001). No teeth exhibited sensitivity in the three groups both at the two-week recall and two-year follow-up.

### INTRODUCTION

Amalgam has been used in the restoration of structurally compromised posterior teeth for many years. The evolution of both composite materials and adhesive systems has advanced rapidly over the past 20 years. Modern etch-and-rinse adhesive systems produce bond strengths that allow clinicians to bond to tooth structure without the use of aggressive retentive cavity preparations. 1 Etch-and-rinse adhesives include either three-step or one-bottle two-step systems, which both adopt a separate phosphoric acid etch-and-rinse phase. In the three-step approach, hydrophilic primers are used before applying a uniform layer of hydrophobic resin to complete hybridization.<sup>2</sup> The simplified one-bottle two-step systems combine hydrophilic primers and hydrophobic resin into one application. Three-step etch-andrinse adhesives are considered the gold standard for bonding resin-bonded composite (RBC) to tooth structure. Conversely, the dentin bond of one-bottle two-step etch-and-rinse adhesives was reported to be less effective than the three-step systems in the long term.4 Acetone-based etch-and-rinse adhesives demonstrated a tendency for lower retention rates than ethanol-based etch-and-rinse adhesives due to their higher technique sensitivity.<sup>5</sup> Semidirect and indirect inlay/onlay composite restorations have progressively replaced amalgam restorations over the last two decades.<sup>6,7</sup> Lately, single-visit direct RBC restorations also have been used as a viable alternative to conventional indirect restorations.8 However, the most recent American Dental Association (ADA) statement on resin-bonded composites (RBCs) endorses the use of posterior composites in 1) smalland moderately sized restorations, 2) conservative tooth preparations, and 3) areas where esthetics are important. These include classes I and II, replacement of failed restorations, and primary caries. Despite the ADA recommendation, dentists are stretching the clinical indications for direct RBC restorations.8,10

The drawbacks of direct RBC are well known. Beyond composite wear<sup>11</sup> and less than ideal

bonding to dentin, 12 stress from polymerization shrinkage still remains one of the main concerns. 13,14 Stress developed at the tooth restoration interface may result in postoperative sensitivity, marginal enamel fractures, premature marginal breakdown, and staining. Three different strategies to reduce polymerization stress have been identified: <sup>13,15</sup> 1) modification to the placement technique; 2) altered curing schemes; and 3) use of a resilient liner on dentin. Combining composite stratification with wedge-shaped increments and polymerization with a low-intensity approach is mandatory to reduce stress in the restoration. The composite mass (per increment) is reduced and the class II high Cfactor configuration is transformed into multiple low C-factor configurations (maximizing the unbonded free surface to enhance stress relief); the soft-start curing protocol allows more time for composite flow into the direction of the cavity walls, resulting in stress release during polymerization shrinkage and increased cross-linking. 16,17 The application of a thin layer (0.5 to 1 mm) of flowable composite limited to the dentin floor has been also suggested as an adjunctive strategy to counteract stress from polymerization shrinkage. 18,19 This layer may act as a stress-absorber because flowable composite may deform to absorb some of the overlaying composite shrinkage strain. Restorations performed according to this protocol are termed stress-reducing direct composite (SRDC).  $^{20}$ 

The goal of this paper is to evaluate the clinical performance of direct RBC restorations placed on structurally compromised posterior teeth using both a bonding preservation and stress-reducing protocol. There were two null hypotheses: 1) both three- and two-step etch-and-rinse adhesives would eliminate postoperative sensitivity; 2) no difference would be detected in the clinical performance of RBC in the three groups after two years of clinical service.

### **MATERIALS AND METHODS**

Fifty patients 18 years or older were included in this clinical trial restoring 75 vital molar teeth with large-size cavities.

A rubber dam was placed and any existing restoration was removed using no. 2 and no. 4 round burs (Brasseler, Savannah, GA). The cavity was prepared in a very conservative manner, just removing either the existing restoration or the decayed dental tissue and trying to preserve the remaining sound tooth structure according to the basic guidelines for direct adhesive preparations. A caries indicator (Sable Seek, Ultradent Products,

South Jordan, UT, USA) was applied to the dentin; stained, nonmineralized, and denatured dental tissues were removed with a spoon excavator. Residual enamel sharp angles and unsupported prisms were smoothed using the SD and SB partially diamond-tipped ultrasonic tips (EMS, Nyon, Switzerland); the SB instrument was also used to smooth sharp angles located on dentin. No bevels were placed either in the occlusal or gingival margins.

Once the preparation was completed, an assessment was made as to whether the facial-lingual occlusal extension (isthmus) was greater than twothirds the intercuspal distance, the proximal extension was greater than half the distance between line angles, and the mesiodistal extension was greater than half the distance between marginal ridges. Cavosurface margins were located on enamel. Teeth not matching these criteria as well as teeth with residual cavity walls less than 1 mm and teeth with one or more cusps involved were excluded from entering the study. Teeth were randomly divided into three groups. Three different etch-and-rinse filled ethanol-based adhesive systems were selected for this study: group 1: Opti-Bond FL (Kerr, Orange, CA, USA); group 2: Scotchbond 1 XT (3M ESPE, Seefeld, Germany); group 3: PQ1 (Ultradent Products).

Sectional matrices (OmniMatrix, Ultradent Products) were placed and interproximal matrix adaptation secured by using pink and blue dental wedges (Hawe Sycamore Interdental Wedges, Kerr). The teeth were etched for 15 seconds using a 35% phosphoric acid (UltraEtch, Ultradent Products). The etchant was removed and the cavity rinsed with water spray for 30 seconds, being careful to maintain a moist surface. The cavity was disinfected with a 2% chlorhexidine gluconate antibacterial solution (Consepsis, Ultradent Products). In group 1, the Opti-Bond FL (Kerr) three-step adhesive system was applied using a hydrophilic primer before applying a uniform layer of hydrophobic resin. In the remaining two groups, the one-bottle two-step adhesive systems Scotchbond 1 XT (3M ESPE) and PQ1 (Ultradent Products) were applied using a mix of hydrophilic primers and hydrophobic resin. In the three groups, the adhesive systems were gently air-thinned and light-cured for 20 seconds using a LED curing light (UltraLume V, Ultradent Products).

Vit-l-escence microhybrid composite resin (Ultradent Products) was used to restore the teeth. Stratification was initiated using multiple 1- to 1.5-mm triangularly shaped (wedge-shaped) increments. The proximal surface buildup was completed using

the Pearl Smoke (PS) enamel shade. Stratification of dentin was started placing a 1- to 1.5-mm even layer of A-3.5 flowable composite (PermaFlo, Ultradent Products) on deeper dentin, which was followed by the application of dentin wedge-shaped increments strategically placed on only two bonded surfaces, decreasing the cavity configuration or C-factor ratio. The C-factor is defined as the ratio between bonded and unbonded cavity surfaces; increasing this ratio also increases the stress from polymerization shrinkage.<sup>21</sup> For the same reason, single increments of PS enamel shade were applied to one cusp at a time; each cusp was cured separately, achieving the final primary and secondary occlusal morphology. In order to reduce stress from polymerization shrinkage, the authors used a previously described polymerization technique, based on a combination of pulse (enamel) and progressive (dentin) curing techniques through the tooth. The pulse curing protocol was adopted for the proximal and occlusal enamel buildup polymerization; it was accomplished by using a very short curing time (1 or 2 seconds) per each increment. The progressive curing technique was used for the polymerization of the dentin increments; it was performed by placing the light tip in contact with the external cavity walls to start the polymerization through the wall (indirect polymerization) at a lower intensity. Final polymerization was then provided at a higher intensity and extended curing time (Table 1). Initial occlusal and proximal adjustment of the restoration was performed using no. 7404 and no. 7902 carbide burs (Brasseler, Savannah, GA). Patients were recalled after 48 hours to complete the occlusal adjustment and perform the final polishing. Figures 1 to 10 show the step-by-step procedure used to restore a large class II restoration according to a SRDC protocol and the result at the follow-ups.

Table 1: Recommended Photo-curing Times and Intensities for Proximal and Occlusal Enamel and Dentin

Buildup Location	Polymerization Technique	Intensity, mW/cm <sup>2</sup>	Time, s
Proximal enamel	Pulse	800	2 (20)
Dentin	Progressive	800	20 <sup>a</sup> + 20
Occlusal enamel	Pulse	800	1 + 20 <sup>b</sup>

 <sup>&</sup>lt;sup>a</sup> 20 seconds of indirect polymerization through the facial and palatal walls
 <sup>b</sup> 20 seconds per each surface (palatal, facial, and occlusal surfaces).



Figure 1. Preoperative view of teeth no. 36 and no. 37 showing incongruous tooth-colored restorations.



Figure 2. Once cavity preparation was completed, sectional matrices were placed and etching was performed using 35% phosphoric acid.

### **RESULTS**

Restorations were evaluated at six-month intervals during the two-year period using a modified US Public Health Service criteria (Table 2) by two independent evaluators precalibrated at 85% reliability. The evaluators were double-blinded. No failures were reported and  $\alpha$  scores were recorded for all parameters. Statistical analysis was performed using a  $\chi^2$  test and the Fisher exact test ( $\chi^2$ =10.6; p=0.001) using the Statistical Package for the Social Sciences version 11.0 (SPSS Inc, Chicago, IL, USA).

In group 1, there were 15 out of 25 teeth that exhibited pre-op tooth sensitivity; no post-op sensitivity was recorded either at the two-week recall or two-year follow up. Patient drop out was as follows: one patient at the 18-month follow-up and one patient at the two-year follow-up. In group 2, there



Figure 3. After applying a 2% chlorhexidine gluconate solution on dentin, a two-step etch-and-rinse ethanol-based adhesive system was applied on both enamel and dentin.



Figure 4. Build up of proximal surfaces was completed first followed by the application of a thin layer of flowable composite on deep dentin.



Figure 5. Dentin stratification was performed by using wedge-shaped increments of composite dentin shades.



Figure 6. Restoration was completed with the application of PS shade to each cusp in order to develop cusp ridges and supplemental morphology.



Figure 8. Result at the 12-month recall.



Figure 7. Initial polishing was performed under rubber dam isolation.

Figure 9. Result at the 24-month recall.

were 12 out of 25 teeth that exhibited pre-op tooth sensitivity; no post-op sensitivity was recorded either at the two-week recall or two-year follow up. Patient drop out was as follows: one patient at the 12-month follow-up and one patient at the two-year follow-up. In group 3, there were 18 out of 25 teeth that exhibited pre-op tooth sensitivity; no post-op sensitivity was recorded either at the two-week recall or two-year follow up. Patient drop out was as follows: one patient at the six-month follow-up and one patient at the two-year follow-up (Table 3). In summary, no teeth exhibited sensitivity in the three groups both at the two-week recall and two-year follow-up (group 1:  $\chi^2$ =20.1; p<0.0001; group 2:  $\chi^2$ =16.2; p<0.0001;  $\chi^2$ =29.6; p<0.0001). Post-op sensitivity was evaluated through air-syringe blow at a distance of 1-2 cm; patients having unrestored class V defects were excluded from the study to avoid bias.

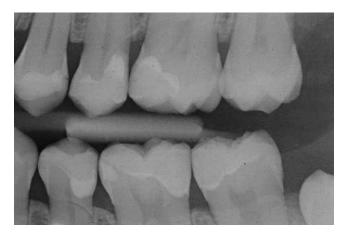


Figure 10. Postoperative x-rays at the 24-month follow-up.

Table 2: Evaluation of Restorations Using Modified US Public Health Service Criteria							
Score	Score Alpha		Charlie	Delta			
Surface texture	Sound	Rough	_	_			
Anatomical form	Sound	Slight loss of material (chipping, clefts), superficial	Strong loss of material (chipping, clefts), profound	Total or partial loss of the bulk			
Marginal integrity (enamel)	Sound	Positive step, removable by finishing	Slight negative step not removable, localized	Strong negative step in major parts of the margin, not removable			
Marginal discoloration (enamel)	None	Slight discoloration, removable by finishing	Discoloration, localized not removable	Strong discoloration in major parts of the margin not removable			
Secondary caries	None	Caries present	_	_			
Gingival inflammation	None	Slight	Moderate	Severe			
Restoration color stability	No change	Change of color comparing to baseline condition	_	_			
Pre-op sensitivity (air)	None	Yes	_	_			
Post-op sensitivity (air)	None	Moderate	Severe	_			

### **DISCUSSION**

It is well known that human dentin contains endopeptidases called matrix metalloproteinases-2 (MMP-2). MMP-2 are enzymes that may be involved in the degradation of the polymer matrix of the hybrid layer as well as the collagen fibrils network. Deterioration of the dentin-composite bond may compromise the longevity of RBC restorations.

Number of Teeth With Sensitivity - Without Table 3: Sensitivity at each Time Point Sensitivity Group 1 Group 2 Group 3 Pre-op 15-10 12-13 18-7 2 wk 0-25 0-25 0-25 1 y 0 - 250 - 240 - 242 y 0-23 0-23 0-23 Patient drop out 2 2 2

Chlorhexidine was demonstrated to be effective in the inhibition of MMP-2s. The application of a similar inhibitory agent in the clinical bonding procedure may result in a more satisfactory performance of bonding interfaces over time. 12,22 It cannot be extrapolated from this study whether the use of chlorhexidine may have been responsible for the lack of difference in tooth sensitivity for the three adhesive systems over the two-year evaluation period; separate clinical studies specifically designed to test the role of chlorhexidine on post-op sensitivity should be conducted. Unfortunately, only two clinical studies from the same research group tested the long-term clinical performance of etch-and-rinse adhesive systems. Wilder and colleagues<sup>3</sup> tested a previous version of the Opti-Bond FL three-step etch-and-rinse adhesive system over a 12-year period. They reported an overall retention rate of 89%; surprisingly, the retention rate was higher in the selective etching group (etching of enamel only) than in the total-etch group (etching of both enamel and dentin). Swift and coworkers<sup>23</sup> reported a 93.3% retention rate for the ethanol-based one-bottle twostep etch-and-rinse adhesive system OptiBond Solo after three years of clinical service; however, the

retention rate dropped to 65.6% at the eight-year recall.<sup>4</sup> A similar trend was reported for the acetone-based one-bottle two-step etch-and-rinse adhesive system Prime & Bond 2.1. Neither of these two studies were with class II restorations using the chlorhexidene preservation and the SRDC protocols. Other studies reported increased durability of the two-step etch-and-rinse adhesive system when the preparation margins were completely in enamel.<sup>24,25</sup>

A meta-analysis of studies conducted in the 1990s reported an annual failure rate of 2.2% for direct posterior composite restorations, 2.9% for resin composite inlays, and 1.9% for ceramic restorations.<sup>26</sup> However, this meta-analysis did not refer to large-size restorations. Brunthaler and colleagues<sup>27</sup> completed a review of prospective studies on the clinical performance of direct RBC restorations published between 1996 and 2002. They found a linear correlation between the size of the restoration and the observation period and the failure rate. The observation period (from one to 17 years) and the related failure rate range (between 0% and 45%) suggest caution when considering these data. Nevertheless, their review also included clinical studies of restorative materials removed from the market due to the too-high failure rate. Few studies tested the clinical performance of direct large-size RBCs. Brackett and others<sup>28</sup> reported no difference in the clinical performance of small- vs medium- vs largesize direct RBCs. They included class I, class II, and cusp-replacing restorations, and the observation period was limited to 18 months. Deliperi and Bardwell<sup>8</sup> reported no failure for class II direct cusp-replacing RBC restorations after two years of clinical service using both a bonding preservation and stress-reducing protocol. A two-step etch-andrinse adhesive system (PQ1) was used in this study. Preoperative tooth sensitivity was solved two weeks after completing the restoration and was not detected at the next follow-ups. This positive trend has been confirmed over a six-year evaluation period (unpublished data). The pilot study did not include a control group and the number of restorations was limited to 25. The same research group reported similar results for direct large-size RBCs placed on endodontically treated teeth in anterior teeth over a five-year period.<sup>29</sup> The results of the current study are also very encouraging and seem to match the findings of the former study on direct cusp-replacing RBC restorations. Although both three- and two-step etch-and rinse-adhesive systems were used in this study, no difference was found among the three groups in this study. It is interesting that neither

marginal deterioration nor marginal discoloration were detected after two years; postoperative sensitivity was also eliminated after completing the restoration. Hayashi and Wilson<sup>30</sup> tried to predict factors responsible for future failure of RBC restorations. They reported that restorations with marginal discoloration at three years were 3.8 times more likely to have failed by five years than restorations with no marginal discoloration; by the same token, restorations with marginal deterioration at five years were 5.3 times more likely to have failed by five years than restorations with no marginal deterioration. The combination of both marginal discoloration and deterioration prelude an even higher failure rate of RBC restorations. According to this prediction, we should expect a very low failure rate for the large-size restorations included in this study over the next five years.

### CONCLUSION

Both of the null hypotheses were accepted. Direct class II large-size composite restorations performed equally well regardless of the adhesive system used after two years of clinical service. The selection of specific layering and curing schemes may protect the RBC restoration from polymerization shrinkage stress; strategies used to prevent the degradation of the hybrid layer may help to avoid deterioration of the dentin-composite bond. Further clinical studies with a long-term observation period are required to better support the findings of this report.

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# Clinical Evaluation of a Low-shrinkage Composite in Posterior Restorations: One-Year Results

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### Clinical Relevance

Filtek Silorane showed acceptable clinical performance after one year. However, the low-shrinkage resin combined with the self-etch adhesive did not provide any advantage over the methacrylate-based composite combined with the total-etch adhesive.

### **SUMMARY**

Objectives: The aim of this study was to compare the one-year clinical performance of three restorative systems, which included a novel low-shrinkage composite and two bonding strategies.

Materials and Methods: Twenty-five patients received three Class I (occlusal) or Class II restorations performed with one of three re-

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storative systems: Filtek Silorane Restorative System (FS); Adper Scotchbond 1 XT, a two-step etch-and-rinse adhesive, with Filtek Z250 (XT); and Adper Scotchbond SE, a two-step self-etch adhesive, with Filtek Z250 (SE). All materials were applied following the manufacturer's instructions. Two independent observers evaluated the restorations at baseline, after six months, and after one year, according to the United States Public Health System modified criteria. The Kruskal-Wallis test and the Mann-Whitney U-test were computed to compare the behavior of the restorative systems; Friedman and Wilcoxon tests were used to analyze the intrasystem data ( $\alpha$ =0.05).

Results: All restorations were evaluated at one year. FS and XT performed statistically similarly at one year, but marginal staining for SE was statistically worse. Intrasystem comparisons between baseline and one year also showed deterioration of marginal staining for SE, while a deterioration of the marginal adaptation was recorded for both SE and FS. XT was the only system for which there was no

statistical change of the parameters measured in this study.

Conclusions: Both restorative systems using self-etch adhesives showed a tendency to degradation of marginal adaptation after one year of clinical use, compared to baseline values. Although the clinical performance of FS was deemed acceptable after one year, this study did not find any advantage of the silorane-based composite over the methacrylate-based composite. The low-shrinkage associated with FS may not be a determinant factor for clinical success.

### INTRODUCTION

The improvements in dental adhesives and resin composites, along with a minimally invasive approach to caries treatment, have made these restorative materials very popular for direct posterior restorations. Nevertheless, the longevity of these restorations is still affected by the consequences of their polymerization shrinkage. The volumetric reduction due to polymerization generates stress within the material, at the adhesive interface, and in the tooth structure. The physical mismatch between the shrinkage-prone restorative material and the stiffer tooth structure may result in microleakage, marginal staining, gap formation, postoperative sensitivity, and enamel microcracks or cusp deflection. 2,3

Filtek Silorane, introduced in 2007, is the first commercially available resin composite not based on bisphenol A diglycidyl methacrylate or urethane dimethacrylate, the dimethacrylate monomers most commonly used.4 This novel silorane-based resin takes its name from the combination of its chemical blocks, siloxanes and oxiranes. The silorane molecule has a siloxane core with four attached oxirane rings that open upon polymerization to bond to other monomers. 5,6 This mechanism implies a slight reduction of the initial distance between monomers, which results in a volumetric shrinkage of less than 1%, which might generate less stress on the adhesive interface.<sup>5,7,8</sup> This characteristic has been validated by other *in vitro* studies, <sup>9,10</sup> in which the siloranebased resin resulted in a significantly lower cusp deflection when applied to MOD preparations, in comparison to methacrylate-based resins. Moreover, the silorane-based resin has been shown to have adequate physical and mechanical properties, which make it suitable for clinical application.<sup>6,11</sup>

The specific chemistry and curing mechanism of the silorane-based resin composite required the

development of a dedicated adhesive by the respective manufacturer. In the case of Filtek Silorane, a two-step self-etch adhesive was developed. This adhesive is composed of a self-etch primer and a hydrophobic bonding resin. <sup>12,13</sup> Self-etch adhesives have become increasingly popular as they are more user-friendly, less technique-sensitive, and may reduce postoperative sensitivity 14,15 compared to etch-and-rinse adhesives. However, the adhesion to enamel achieved by etch-and-rinse adhesives is still considered the "gold standard" 14,16,17 as a result of the deep etching pattern created by the low pH of phosphoric acid. Therefore, the performance of selfetch adhesives on enamel may depend on their aggressiveness. "Strong" self-etch adhesives result in a more stable and satisfactory enamel bond than do "mild" self-etch adhesives, especially on ground, aprismatic enamel. 18-20 In fact, selective etching of enamel margins with phosphoric acid has been  $recommended^{21-23}$  in clinical situations prior to applying "mild" self-etch adhesives.

Clinical trials are the ultimate test with which to measure the clinical effectiveness and durability of adhesives and resin composites.24 This is of paramount relevance, as there is no clinical evidence to back the deleterious effect of polymerization stress on restoration longevity.25 Accordingly, the aim of this study was to compare the one-year clinical performance of three restorative systems in posterior restorations: the low-shrinkage silorane-based resin composite with its proprietary self-etch adhesive and a widely studied methacrylate-based resin composite, Filtek Z250, used with either a two-step etchand-rinse adhesive or with a two-step self-etch adhesive. The null hypothesis was that there would be no differences in clinical performance for the three restorative systems after one year.

### **MATERIALS AND METHODS**

Before participating in the study, subjects signed a written informed consent. Both the consent and this research protocol had previously been reviewed and approved by the Ethics Committee of the Rey Juan Carlos University.

All patients, with ages ranging from 18 to 60 years (average 29.8 years), required at least three Class I (occlusal) and/or Class II restorations (Table 1). The dental health status of patients was normal in all other respects. Specific exclusion criteria were as follows:

- Fewer than 20 teeth;
- History of existing tooth sensitivity;

Restorative System	Number of Restorations	Toot	h		Class	3	
		Premolars	Molars	olars I	II		
					OM or OD	MOD	Total
Filtek Silorane Restorative System (FS)	25	12	13	12	10	3	13
Adper Scotchbond 1 XT + Filtek Z250 (XT)	25	8	17	14	10	1	11
Adper Scotchbond SE + Filtek Z250 (SE)	25	13	12	12	12	1	13
Total (%)	75 (100)	33 (44)	42 (56)	38 (50.6)	32 (42.6)	5 (6.6)	37 (49.3

- Periodontal disease:
- Extremely poor oral hygiene;
- Bruxism;
- Known allergy to resin-based materials or other materials used in this study;
- Pregnancy or breast-feeding; or
- Chronic use of anti-inflammatory, analgesic, and psychotropic drugs.

Further, excluding criteria for the teeth to be restored were as follows:

- Nonvital teeth:
- Abutment teeth for fixed or removable prostheses; and
- Teeth without a normal occlusal relationship with natural dentition or without at least one adjacent tooth contact.

Bitewing radiographs of the teeth to be restored were taken preoperatively, unless the patient had radiographs taken within the previous year. There was an even distribution of the restorations that replaced existing restorations with clinical or radiographic signs of recurrent caries or esthetic failures and restorations that were performed to treat primary caries lesions.

All operative procedures were performed by the same operator (B.B.). Restorations were placed under local anesthesia with rubber dam isolation. The cavity design was restricted to eliminate carious tissues from primary caries lesions or to remove the restorative material and carious tissues when existing restorations were replaced. Cavities were prepared using diamond burs (Komet-Brasseler, Lemgo, Germany) with no intentional bevels on enamel cavosurface margins. In deep cavities, dentin was covered

with a resin-modified glass ionomer cement (Vitrebond, 3M ESPE, St Paul, MN, USA). An appropriate matrix system (Palodent, Dentsply, Konstanz, Germany) and wooden wedges were applied to the cervical margins of proximal preparations.

The restorative systems evaluated in this study were the Filtek Silorane Restorative System, Adper Scotchbond 1 XT + Filtek Z250, and Adper Scotchbond SE + Filtek Z250 (Table 2).

Initially, the three restorative systems were randomly assigned to each of the three teeth for which restorative treatment was needed, regardless of the characteristics of the tooth and restoration class. However, interference in the randomization procedure within patients was eventually performed in order to equally distribute materials into some important variables, such as tooth type and position, restoration class, and restoration size, in such a way that the influence of those factors was minimized.<sup>26</sup> All adhesive systems were applied according to the manufacturer's instructions (Table 2). Resin composites were placed in 2-mm increments. Each increment was light-cured for 20 seconds using a LED Demetron I polymerization unit (Kerr, Orange, CA, USA) with a minimum light output of 550 mW/cm<sup>2</sup>.

After polymerization, coarse finishing was accomplished with carbide burs under water cooling and, if needed, with a #12 blade and aluminum-oxide disks (Sof-Lex, 3M ESPE). Final finishing of the occlusal surface was accomplished with polishing points (Enhance and PoGo, Dentsply).

### **Clinical Evaluation**

All restorations were evaluated after one week (baseline), six months, and one year for the following

Adhesives (Batch No.)	Composition	Instructions for Use	Туре
Silorane System Adhesive (also known as LS System Adhesive or P90 System Adhesive) (Primer: 8AP; Adhesive: 8AK)	Primer: phosphorylated methacrylates, Bis-GMA, HEMA, water, ethanol, silane-treated silica filler, Vitrebond™ copolymer, initiators, stabilizers	Primer: application for 15 sec with black microbrush, followed by gentle air dispersion and 10 sec of light-curing	Two-step self-etch
	Adhesive: hydrophobic DMA, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers	Adhesive: application with green microbrush, followed by gentle air dispersion and 10 sec of light-curing	
Adper Scotchbond 1 XT (also known as Adper Single Bond Plus or Adper Single Bond 2) (318655)	HEMA, Bis-GMA, GDMA, water, ethanol, silane-treated silica nanofiller, photoinitiator	Acid etch: phosphoric acid (Scotchbond™ Etchant, 3M ESPE): 35% (15 sec). Rinse (10 sec). Blot excess water using a cotton pellet or minisponge. Do not air-dry	Etch-and-rinse
		Adhesive: apply two to three consecutive coats of adhesive for 15 sec with gentle agitation using a fully saturated applicator. Gently air thin for 5 min to evaporate solvent. Lightcure for 10 sec	-
Adper Scotchbond SE (also known as Adper SE Plus) (Liquid A: 7AF; Liquid B: 8AL)	Liquid A (colored wetting solution): water, HEMA, surfactant, rose bengal dye	Liquid A: apply to the cavity so that a continuous red-colored layer is obtained on the surface	Two-step self-etch
	Liquid B (adhesive): UDMA, TEGDMA, TMPTMA, HEMA phosphate and MHP, bonded zirconia nanofiller, initiator system based on camphorquinone	Liquid B: scrub into the entire wetted surface of the bonding area for 20 sec. Red color will disappear quickly, indicating that the etching components have been activated. Air-dry thoroughly for 10 sec. Apply second coat to the entire bonding surface. Light air application. Light-cure for 10 sec	-
Resin Composites	Organic Matrix	Inorganic Filler	
Filtek Silorane (8BH)	3,4-Epoxycyclohexylethylcyclopoly- methylsiloxane, bis-3,4-	Silanized quartz particles: 50% volume, 7	'0% weight
	epoxycyclohexylethylphenylmethylsilane, yttrium fluoride (15%), camphorquinone, iodonium salt, stabilizers, pigments	Size: 0.1–2 μm	
Filtek Z250 (7LY)	Silane-treated ceramic, bisphenol A polyethylene glycol diether dimethacrylate,	Quartz and zirconia particles: 60% volum	e, 78% weight
	UDMA, Bis-GMA, TEGDMA, water (<2%)	Size: 0.01–3.5μm (0.6 μm, on average)	

parameters: color match, retention, marginal adaptation, anatomic form, surface roughness, marginal staining, sensitivity, and secondary caries (Table 3). Pre- and postoperative sensitivity was determined

with a dental syringe placed 2 cm from the tooth surface. Two clinicians (L.C. and E.C.) evaluated the restorations blindly at each recall using the modified United States Public Health Service (USPHS)

Table 3: Modified	USPHS Criteria Used	
Criteria	Code	Definition
Color match	Alfa Beta Charlie	Restoration matches adjacent tooth structure in color and translucency.
		Mismatch is within an acceptable range of tooth color and translucency.
		Mismatch is outside the acceptable range.
Retention	Alfa Beta Charlie	Full retention.
		Partial retention.
		Restoration is lost.
Marginal adaptation	Alfa Beta Charlie	Restoration closely adapted to the tooth. No crevice visible. No explorer catch at the margins, or there was a catch in one direction.
		Explorer catches. No visible evidence of a crevice into which the explorer could penetrate. No dentin or base visible.
		Explorer penetrates into a crevice that is of a depth that exposes dentin or base.
Anatomical form	Alfa Beta Charlie	Restorations continuous with existing anatomic form.
		Restorations discontinuous with existing anatomic form but missing material not sufficient to expose dentin base.
	•	Sufficient material lost to expose dentin or base.
Surface roughness	Alfa Beta Charlie Delta	Surface of restoration is smooth.
	•	Surface of restoration is slightly rough or pitted, but can be refinished.
	•	Surface deeply pitted, irregular grooves, and cannot be refinished.
		Surface is fractured or flaking.
Marginal staining	Alfa Beta Charlie	No staining along cavo-surface margin.
		<50% of cavo-surface affected by stain (removable, usually localized).
		>50% of cavo-surface affected by stain.
Sensitivity <sup>a</sup>	Alfa Beta Charlie Delta	None.
	•	Mild but bearable.
	•	Uncomfortable, but no replacement is necessary.
		Painful. Replacement of restoration is necessary.

Table 3: Modified USPHS Criteria Used (cont.)						
Criteria	Code	Definition				
Secondary caries	Alfa Beta	Absent.				
		Present.				
<sup>a</sup> Postoperative sensitivity at	baseline was registered o	one week after the restoration insertion.				

criteria as adapted by Wilson and others<sup>27</sup> (Table 3). When disagreements arose during evaluations, the examiners had to reach a consensus. To help with the evaluation of marginal discoloration, intraoral color photographs were collected at baseline and at the recall appointments. Clinical photographs consisted of digital images at 1.3× magnification taken with a Nikon D80 camera with a 105-mm Micro-Nikkor lens (Nikon USA, Melville, NY, USA).

The statistical analyses were carried out with the SPSS 16.0 for Windows software (SPSS Inc, Chicago, IL, USA) using the nonparametric Kruskal-Wallis test and Mann-Whitney U-test to compare the behavior of the three restorative systems at baseline, six months, and one year. Friedman and Wilcoxon nonparametric tests were used to compare the data obtained for each restorative system at each evaluation period. The level of confidence was set at  $\alpha = 0.05$ .

### **RESULTS**

A total of 75 restorations were placed in 25 patients. The distribution of the restorations was similar between Class I (38) and Class II (37) cavities (Table 1). All patients attended the six-month and one-year recalls (100% recall rate). The results are summarized in Table 4.

### Comparison of the Performance of the Three Restorative Systems at One Year

Adper Scotchbond SE + Filtek Z250 resulted in significantly worse marginal staining than did the other two restorative systems at one year (p=0.028). This deterioration had already been detected at the six-month recall appointment (p=0.013) (Figure 1).

All restorative systems resulted in a percentage of Alfa ratings above 90% at one year for the categories of retention and anatomical form. However, Alfa ratings for surface roughness, and in particular, marginal adaptation, decreased for all of the restorative systems, although this reduction did not result

in statistical differences among them. Secondary caries was only detected in one tooth restored with Filtek Silorane, which had previously shown a fracture of the material at the six-month evaluation. Both findings had no statistical repercussions.

### Baseline vs One-year Evaluation for Each Restorative System

Filtek Silorane Restorative System—Marginal adaptation was significantly worse at one year compared to baseline (p=0.005), as seven of 25 restorations were rated Bravo and one was rated Charlie. Additionally, surface roughness was statistically similar at baseline and after one year, but it was different at six months (p=0.02), as 28% of the restorations were rated Bravo at this recall. Filtek Silorane was the only system that rated Bravo in secondary caries and retention, Charlie in adaptation and anatomical form, and Delta in surface roughness. However, all of these ratings came from a single restoration and did not lead to any statistical significance. Only one restoration showed a true color modification over time, and two did not match adjacent tooth structure because of the yellowish and very opaque aspect of the Filtek Silorane resin composite.

Adper Scotchbond 1 XT + Filtek Z250—Marginal staining, surface roughness, and in particular marginal adaptation parameters resulted in worse rankings at one year (four restorations were rated Bravo), although there were no statistical differences (p>0.05). No Charlie ratings were assigned to this restorative system for any of the criteria. Postoperative sensitivity (slight discomfort associated with cold beverages) was found in one patient during the first week after the restoration was placed.

Adper Scotchbond SE + Filtek Z250—Marginal adaptation and marginal staining were significantly worse at one year compared to baseline. Adaptation deficiencies increased (p=0.002) in the last six months, as seven restorations rated Bravo and one was rated Charlie at one year. Marginal staining

Criteria	Code	Baseline			6 months			1 year		
$\textbf{Materials} \rightarrow$		FS	ХТ	SE	FS	XT	SE	FS	XT	SE
Color match	Α	23	25	23	22	24	23	22	24	22
	В	2	_	2	3	1	1	3	1	1
	С		_	_	_	_	1	_	_	2
Retention	Α	25	25	25	24	25	25	24	25	25
	В	_	_	_	1	_	_	1	_	_
	С	_	_	_	_	_	_	_	_	_
Marginal adaptation	Α	24	25	25	20	23	24	17	21	18
	В	1	_	_	4	2	1	7	4	7
	С	_	_	_	1	_	_	1	_	_
Anatomic form	Α	25	25	25	24	25	25	24	25	25
	В	_	_	_	_	_	_	_	_	_
	С	_	_	_	1	_	_	1	_	_
Surface roughness	Α	23	24	25	17	21	22	22	22	21
	В	2	1	_	7	4	3	2	3	4
	С	_	_	_	_	_	_	_	_	_
	D	_	_	_	1	_	_	1	_	_
Marginal staining	Α	25	25	23	24	24	18	23	22	16
	В	_	_	2	_	1	6	1	3	8
	С	_	_	_	1	_	1	1	_	1
	D	_	_	_	_	_	_	_	_	_
Sensitivity	Α	25	24	24	25	25	25	25	25	25
	В	_	1	1		_				
	С		_		_		_			
	D	_	_	_	_	_	_	_	_	_

Table 4: Number of Evaluated Restorations in Each Criterion for Each Experimental Group (cont.)												
Criteria	Code		Baseline 6 months		Baseline		6 months			1 year		
Materials-	÷	FS	ХТ	SE	FS	ХТ	SE	FS	ХТ	SE		
Secondary caries	А	25	25	25	25	25	25	24	25	25		
	В	_	_	_	_	_	_	1	_	_		

Abbreviations: FS, Filtek Silorane Restorative System; SE, Adper Scotchbond SE, a two-step self-etch adhesive, with Filtek Z250; XT, Adper Scotchbond 1 XT, a two-step etch-and-rinse adhesive, with Filtek Z250.

appeared during the first six months (p=0.014) and remained stable at the one-year recall, when eight restorations rated Bravo and one was rated Charlie. This restorative system was the only one that resulted in one Charlie rating for color match. Additionally, one patient experienced postoperative sensitivity after restoration placement, which disappeared gradually after a few days.

### **DISCUSSION**

In this study, Filtek Silorane and the etch-and-rinse adhesive Adper Scotchbond 1 XT + Filtek Z250 resulted in statistically similar clinical parameters at one year. The other system formed by the self-etch adhesive Adper Scotchbond SE and Filtek Z250 resulted in increased marginal staining at one year. Thus, the first null hypothesis must be partially rejected.

Early marginal staining is usually a clinical sign that a restoration is prone to failure or that the adhesive interface undergoes degradation with time.<sup>28</sup> Marginal discoloration may be caused by several factors, including the presence of excess filling materials, a deficient restoration around the margin, and the formation of gaps.<sup>29</sup> However, the nature of the adhesive system is a determinant factor. The marginal staining associated with Adper Scotchbond SE + Filtek Z250 restorations must have been caused by the adhesive itself, since the other system using the same resin composite showed no alteration in this parameter. Adper Scotchbond SE is a strong self-etch system (pH=1).30 Although marginal discoloration has been associated with a poor etching ability of self-etch adhesives at the enamel margins, 21,23,28 significant marginal staining and color changes have been reported for self-etch adhesives with a pH similar to that of Adper Scotchbond SE.<sup>28</sup> Adper Scotchbond SE is a two-

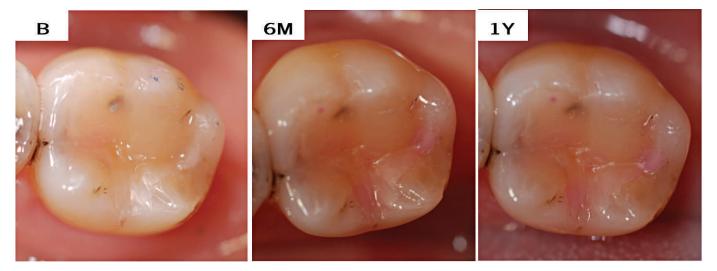


Figure 1. First molar. Occlusal restoration with Adper Scotchbond SE and Filtek Z250. Staining around this Class I restoration was observed at sixmonth and one-year evaluations, being rated Charlie (>50% of cavo-surface is affected). Furthermore, the one-year photograph shows that staining has progressed in depth across the adhesive interface. This stain also caused color changes in the resin composite close to the bonded walls. B, baseline; 6M, six-month recall: 1Y, one-year recall.



Figure 2. First molar. Occlusal restoration with Adper Scotchbond 1 XT and Filtek Z250. This restoration preserved its original aspect after six months and one year. No signs of adhesive deterioration were found. B, baseline; 6M, six-month recall; 1Y, one-year recall.

step self-etch adhesive in which water is separate from the adhesive solution to increase stability of the material.<sup>31</sup> In fact, conventional methacrylate monomers undergo rapid hydrolysis under acidic aqueous conditions.<sup>31</sup> Liquid A is a 2-hydroxyethyl methacrylate (HEMA)-water solution without etching capacity, and Liquid B is the solution containing the acidic monomers. The combination of acidic hydrophilic and hydrophobic monomers in the same solution (in this case, Liquid B) may cause a low degree of carbon double bond (C=C) conversion and increased permeability of adhesive interfaces. Other factors may be responsible for the marginal staining observed in the current study with SE. The change of color from pink to yellow that results from the adequate mix of Liquid A and Liquid B confirms that the acidic monomers have been activated (ionized). However, the activation of Liquid B into an etching agent, based on the superficial moisture provided by the 80% water in Liquid A, may result in an incomplete conversion of the acidic monomers. Their inclusion in a HEMA-rich and still-colored adhesive interface would enhance their susceptibility to hydrolytic degradation and, consequently, marginal staining. 30,32 This mechanism was corroborated by the presence of the characteristic pink color of Liquid A in most of the stained margins around Adper Scotchbond SE + Filtek Z250 restorations (Figure 1).

A recent study<sup>32</sup> has revealed high color instability after water immersion of a self-etch adhesive (One-Up Bond F, Tokuyama Dental Corporation, Tokyo, Japan), with a very similar color-change mechanism to that of Adper Scotchbond SE. Water sorption and

discoloration of the adhesive interface may affect the color appearance of the restoration. This phenomenon is highly consistent with what evaluators found in the present study, as all the restorations with Adper Scotchbond SE + Filtek Z250 that rated Bravo or Charlie for color match presented a variable saturation of pink. Some of these restorations already had slight pink marginal staining at the baseline evaluation (Figure 1).

Regarding the comparisons between the baseline and one year for each restorative system, only Adper Scotchbond 1 XT + Filtek Z250 was statistically invariable for all of the parameters tested (Figure 2). Therefore, the second null hypothesis must also be partially rejected. The only system including an etch-and-rinse adhesive obtained the best clinical outcome, which is consistent with the results of previous clinical research. <sup>21,28</sup> Moreover, in a literature review <sup>33</sup> focused on marginal integrity, significantly better *in vitro* and *in vivo* enamel marginal adaptation were found with etch-and rinse adhesives compared to self-etch systems.

Restorations performed with Adper Scotchbond SE + Filtek Z250 exhibited a statistically lower number of Alfa ratings for marginal adaptation and marginal staining parameters at one year. Filtek Silorane also resulted in significantly worse marginal adaptation after one year. Marginal adaptation is influenced by many factors, such as the polymerization shrinkage of the composite resin or the adhesive system used. Both factors could influence the clinical results of this study, since restorative systems were made of different resin composites and adhesives.

Polymerization shrinkage of resin composites may be a potentially harmful factor for the clinical survival of direct restorations as a result of the transfer of stresses to the adhesive interface. Ideally, marginal adaptation, which depends on polymerization shrinkage and resulting stress, should be assessed at baseline because both shrinkage and resulting stress take place during the placement of the restoration. Other clinical factors, such as wear and the integrity of the adhesive interface, may have induced changes in marginal adaptation over the one year of clinical use.

As Filtek Silorane Restorative System has been conceived by its manufacturer for posterior restorations only, Class I and II lesions were selected for this study. The resulting cavity designs produce high C-factor values, which contribute to higher shrinkage stress. The application technique of the resin composite may also influence the bonding effectiveness. In this study, the incremental technique was used in all restorations, as it has been demonstrated to benefit the bond strength of both methacrylate-based 37,38 and silorane composites. 36

The higher polymerization shrinkage of Filtek Z250 combined with high C-factor may create a more unfavorable environment than is associated with Filtek Silorane, as the silorane composite has been reported to undergo up to 1% volumetric shrinkage,<sup>5</sup> according to the manufacturer's information. However, recent research has found Filtek Silorane's volumetric shrinkage to be slightly higher (1.4%)<sup>6,10</sup> and close to the 1.7% total volumetric shrinkage determined for Filtek Z250.<sup>6</sup> Moreover, the elastic

modulus of Filtek Silorane is also higher than that of Filtek Z250, which might be attributed to a more significant influence of the organic matrix on composite stiffness.<sup>6</sup> Although the relationship between elastic modulus and polymerization stress is still not well defined, the in vitro study by Boaro and others<sup>6</sup> measured a greater polymerization stress for Filtek Silorane than for Filtek Z250, which contradicts the belief that lower polymerization shrinkage is related to lower polymerization stress values, as was originally expected.<sup>39</sup> These studies confirm that reduced shrinkage per se does neither guarantee attenuation of stress in restored teeth, <sup>10</sup> nor does it improve the interfacial integrity of the restoration,6 which is in line with the findings of previous clinical studies. 25,40 The authors of another clinical report<sup>41</sup> related to Filtek Silorane analyzed exclusively the marginal adaptation and reported better marginal adaptation for the methacrylate-based resin composite (Ceram.X, Dentsply) compared to that of Filtek Silorane.

Many of the marginal defects detected in the present study appeared to result from the fracture of thin areas of resin composite flash that extended to non-instrumented enamel surfaces adjacent to the cavity margins. Better contouring at polishing should eliminate these areas of marginal flash. Mild self-etch systems have a less stable bonding capacity to enamel, probably because of a shallower etching pattern. The use of adhesives with a more efficient etching capacity may have reduced the occurrence of such defects, especially in high-stress-

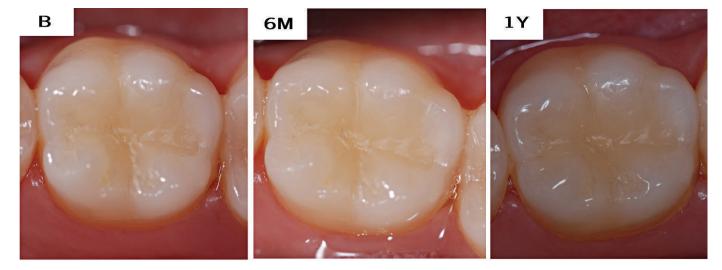


Figure 3. First molar. Occlusal-palatine restoration with the Filtek Silorane Restorative System. Although Filtek Silorane composite displayed a yellowish and opaque appearance in some restorations, the restorations shown were rated Alfa for color match. No staining was detected. B, baseline; 6M, six-month recall; 1Y, one-year recall.

bearing areas, because of the improvement of enamel etching.  $^{44}$ 

The adhesive system that accompanies Filtek Silorane requires separate light-curing of the primer and the bonding, thereby establishing the bonding mechanism to dentin in the first application step, resembling one-step self-etch adhesives. This bonding mechanism uses a form of nano-interaction, typical of ultra-mild self-etch adhesives, which is related to the relatively high pH (2.7) of the respective primer. Mine and others<sup>13</sup> observed a tight superficial interaction and very slight inter- and intracrystallite demineralization with subsequent resin infiltration, when bonded to enamel. This nano-interaction bonding mechanism is clinically relevant for a methacryloyloxydecyl dihydrogen phosphate (MDP)-based twostep self-etch adhesive 45 and may also occur with the polyalkenoic acid copolymer<sup>46</sup> (or Vitrebond<sup>™</sup> copolymer) incorporated in the two-step self-etch Silorane System Adhesive used in the present study. Recent in vitro research46,47 found evidence of the Vitrebond carboxylic acid reacting with calcium ions. It has been demonstrated 13,47 that the Silorane System Adhesive provides a tight, stable, and water-resistant adhesion to dentin. However, information about its performance when it is bonded to enamel is still scarce.

As mentioned above, the Filtek Silorane Restorative System has been specially designed for posterior restorations, for which the esthetic requisites are not so relevant. Accordingly, the manufacturer only provides four shades. At the six-month and oneyear assessments three restorations were rated as Bravo. It is noteworthy that the two Bravo ratings with Filtek Silorane at baseline were caused by the poor esthetic characteristics of the silorane-based resin composite (Figure 3). In fact, evaluators deemed these restorations too yellow and very opaque; thus, their translucency differed from that of tooth structure (Figure 3). Both restorations were also rated Bravo in the subsequent follow-up assessments; therefore, only one restoration showed a real color modification over time. These observations derived from the *in vivo* analysis are consistent with recent in vitro research demonstrating low translucency and high color stability of siloranebased resin composite compared to those of methacrylate-based resin composites. 48,49

### **CONCLUSIONS**

The clinical performance of the Filtek Silorane Restorative System was found acceptable after one year. Additionally, stable adhesion to enamel with self-etch adhesives is still a challenge, as both restorative systems including these products showed a deterioration of their marginal adaptation after one year.

Despite the limitations of this study, the clinical outcomes led to the perception that the Filtek Silorane Restorative System did not provide any remarkable advantage for the evaluated criteria when compared to the other systems, and they reinforced the findings that etch-and-rinse adhesives are still the benchmark when it comes to clinical performance. Further recalls are planned to follow up with regard to the clinical performance of these restorations, as wider differences between the restorative materials might surface at later stages.

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## Comparison of Fracture Strength of Endocrowns and Glass Fiber Post-Retained Conventional Crowns

GR Biacchi • RT Basting

### Clinical Relevance

Restorations of the endocrown type are options for restoring endodontically treated molar teeth with extensive loss of coronal structure. The endocrown has advantages over the conventional crown because its mechanical performance is better, it costs less, and it takes less clinical time to complete.

### **SUMMARY**

The aim of this in vitro study was to compare the fracture strength of full ceramic crowns using two techniques—indirect conventional crowns retained by glass fiber posts, and endocrowns with an "anchorage" in the pulp chamber—and analyze the failure mode. For this purpose, 20 healthy mandibular molars were divided into two groups (n=10): Group GC contained teeth with indirect conventional crowns, filling cores, and glass fiber posts;

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Group GE contained teeth with restorations of the endocrown type. Teeth were endodontically treated and prepared for ceramic restorations fabricated by the injection technique (IPS e.max Press, Ivoclar-Vivadent), forming the GC and GE groups. Specimens were mounted in a universal test machine (EMIC) and were submitted to an oblique compression load, at an angle of 135 degrees to the long axis of the tooth, until failure. Statistical evaluation performed by the Mann-Whitney nonparametric test showed significant differences between the two groups (p=0.002), with Group GE shown to be more resistant to compressive forces than Group GC. The predominant failure pattern in both groups was fracture of the tooth on the side of force application and/or consequent displacement of the restoration on the opposite side.

### **INTRODUCTION**

Restoration of endodontically treated teeth with extensive coronal loss has always followed a strict

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protocol, with the fabrication of total crowns supported on metal cores and/or glass fiber posts. <sup>1-4</sup> Initially, it was believed that this procedure would provide better reinforcement of the remaining dental structure. <sup>5,6</sup> However, it has been observed that the use of intracanal retainers only promoted retention of the prosthetic crown. As a result of removing a healthy dental structure to enable the placement of rigid elements devoid of mechanical behaviors similar to those of the tooth, <sup>7-10</sup> the remaining tooth could be weakened.

With the advent of adhesive dentistry, the need for using posts and filling cores has become less evident. Moreover, the appearance of ceramics that had high mechanical strength and were capable of being acid etched (such as those reinforced with leucite or lithium disilicate), allied with the adhesive capacity of adhesive systems and resinous cements, made it possible to restore posterior teeth, especially molars, without cores and intraradicular posts. <sup>11</sup> Thus, it became feasible to restore posterior teeth with extensive coronal destruction by means of onlay and/or overlay restorations and, more recently, with endocrowns, without the use of radicular posts and while using the entire extension of the pulp chamber as a retentive resource. <sup>12-14</sup>

Pissis<sup>12</sup> was the forerunner of the endocrown technique, describing it as the "mono-block porcelain technique." The nomenclature endocrown was described for the first time by Bindl and Mörmann<sup>13</sup> in 1999 as adhesive endodontic crowns, and was characterized as total porcelain crowns fixed to depulped posterior teeth. These crowns would be anchored to the internal portion of the pulp chamber and on the cavity margins, thus obtaining macromechanical retention provided by the pulpal walls, and microretention would be attained with the use of adhesive cementation. It is a method particularly indicated in cases in which there is excessive loss of hard tissues of the crown, interproximal space is limited, and traditional rehabilitation with post and crown is not possible because of inadequate ceramic thickness. 15 This technique is easily performed, demands less clinical time when compared with conventional crowns, costs less because of the fewer number of steps involved, overcomes the patient's lack of available time, and has good esthetic acceptance because it is made of ceramic.<sup>2</sup>

In a clinical study, Bindl and Mörmann<sup>16</sup> evaluated the performance of 208 endocrowns cemented to premolars and molars and observed that the premolars presented more failures than the molars. It is suggested that this occurs because premolars have a

smaller adhesion surface when compared with molars. Additionally, premolars have greater crown height, which, consequently, compromises the mechanical properties of the endocrown. It is also suggested that endocrowns should be made only with reinforced ceramics. This has been shown to be an advantageous technique because the procedure is easy. <sup>11</sup>

Nevertheless, because of the absence of information about the biomechanical behavior of endocrowns and the expectation that this type of restoration would behave similarly or superiorly to conventional crowns (because of the potential to be retained in the pulp chamber by micromechanical retention given by the adhesive system and resin cement), the present study has endeavored to evaluate the fracture strength of endodontically treated molars with extensive coronal loss, restored by the conventional technique (glass fiber post and ceramic crown) and by endocrowns, in addition to observing the failure mode when they are submitted to an oblique compressive force.

### **METHODS AND MATERIALS**

This study was approved by the Human Research Ethics Committee of the School of Dentistry and Dental Research, São Leopoldo Mandic (Process Number 2010/0240).

### **Specimen Preparation**

Thirty mandibular molars with complete root formation were collected from the Tooth Bank of the School of Dentistry of the Health Science Center, Federal University of Santa Maria, and were cleaned and stored in 1.0% thymol. They were sectioned in the enamel 1 mm above the cementoenamel junction (CEJ). Twenty teeth were selected according to the following inclusion criteria: presence of enamel on the crown margins, wide pulp chamber, and similar mesiodistal and vestibulolingual diameters.

The teeth were individually fixed with acrylic resin (VIPI Flash, Pirassununga, SP, Brazil) in polyvinyl chloride (PVC) rings (Tigre SA, Joinville, SC, Brazil), leaving the CEJ 1 mm above and parallel to the acrylic resin. Then, teeth were randomly distributed into two groups (n=10): the endocrown group (GE) and the conventional crown and intraradicular post group (GC).

### **Endocrown Preparation**

Preparations for endocrowns and for conventional crowns were made by using a preparation standard-

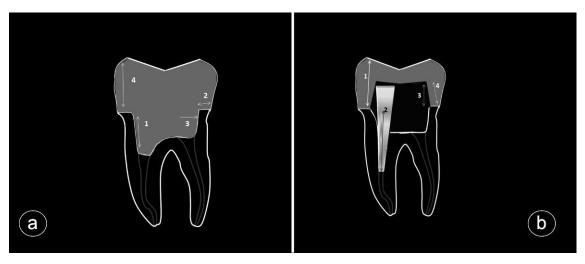


Figure 1. Schematic representation of the preparation. a) Endocrown: 1) height between 5 and 3.70 mm from the cervical margin up to the distal canal entrance; 2) preparation margins between 2.2 and 2.7 mm; 3) expulsive degree of approximately 8 to 10 degrees; and 4) crown height of 7 mm from the gingival margin. b) Conventional crown: 1) crown height of 6 mm; 2) post height of 9 mm to the cervical margin; 3) core height of 3 mm; and 4) expulsive degree of approximately 6 to 8 degrees.

ization device adapted from an optical microscope. Preparations were limited to removal of the pulp chamber roof, excessively retentive areas, and alignment of the pulpal walls, which was done up to the limit of the anatomic configuration of the chamber itself with an internal taper of 8 to 10 degrees. Entrances and undercuts of mesial and distal canals (1 and 3 mm depth, respectively) were protected using an adhesive system (Adper Single, Bond 2, 3M ESPE, St Paul, MN, USA) and a flowable resin (Natural Flow, DFL, Rio de Janeiro, RJ, Brazil). Preparations were finalized, allowing a path of draw without interferences. The distal canal was deepened between 3.7 and 5 mm from the cervical margin, limited by the canal anatomy. The procedure of smoothing and rounding the internal angles of the margins began with the use of the same diamond tip and ended with polishing of the margins and internal angles with an abrasive rubber tip.

### **Conventional Crown Preparation**

Roots of Group GC received glass fiber posts #1 (White Post, FGM, Joinvile, SC, Brazil) and a resin composite filling core (Tetric N-Ceram, Ivoclar-Vivadent AG, Schaan, Liechtenstein), in which the gutta percha was removed from the distal canal up to the limit of 9 mm and was measured from the preparation margins. After this, the root canal was widened and enlarged with the bur included in the post system with the cursors duly positioned. The post was cut 3 mm above the gingival margin and was cemented using an adhesive system (Adper

Single Bond, 3M ESPE) and dual resin cement (RelyX ARC, 3M ESPE).

After post cementation, the filling core was made with increments of resin composite (Tetric N-Ceram, Ivoclar-Vivadent AG). A transparent addition silicone mold (Transil, Ivoclar-Vivadent AG), which allowed for standardizing the core height, was filled and with the mold sitting on the tooth was light activated for 60 seconds.

Each tooth with a prefabricated core was taken to the standardizing device for adjustment of the preparation margins to a final width of 1.7 mm. Small adjustments were also made in cases in which the core exceeded the height of 3 mm. Final characteristics of endocrown and conventional crown preparations and their schematic representations may be seen in Figure 1.

### **Laboratory Phase**

Endocrowns and conventional crowns were shaped with the use of light and heavy polyvinyl siloxane impression (Hidroxtreme, Coltène/Whaledent, Altstätten, Switzerland). The process of die-casting with special type IV stone plaster (Durone, Dentsply, Petrópolis, RJ, Brazil) began, and metal cylinders were used to make the dies and to facilitate manipulation afterward.

Laboratory procedures began by making the crowns in wax (Pro-mod VKS, Horgensell, Germany) on the dies of conventional crowns and endocrowns, maintaining the same proportion in height. Crowns

Table 1: Classification of the Pattern and Failure Mode						
Failure Pattern	Description					
1. Fracture of the <i>endocrown</i> or conventional crown	Fracture of the ceramic restoration					
2. Fracture of the tooth	Fracture of the tooth or root					
3. Fracture with displacement	Fracture of the tooth or restoration with displacement (loss of adhesion) of the endocrown or conventional crown					
4. Displacement without fracture	Displacement (loss of adhesion) of the endocrown or conventional crown without fracture of the restoration or tooth					

were made of IPS e.max Press (Ivoclar-Vivadent AG) by the injection technique as per the manufacturer's materials and instructions.

After polishing, the crowns were prepared with 10% hydrofluoric acid (Condac Porcelana, FGM), silane agent (Prosil, FGM), and adhesive agent (Adper Scotchbond Multi-Purpose, 3M ESPE). This was followed by a light jet of air and then light activation for 20 seconds. They were cemented after the teeth adhesive system (Adper Scotchbond Multi-Purpose, 3M ESPE) and dual resin cement (RelyX ARC, 3M ESPE) were applied. Specimens were kept in a humid environment for 72 hours before they were submitted to the compressive strength test.

### **Compressive Strength Test**

To perform the compression test, each specimen was put into a fixation device and placed obliquely on the base of a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil). A compressive load was applied at a 135-degree angle to the long axis of the tooth, on the internal and central face of the vestibular cuspid of all ceramic restorations. This was done by means of a metal rod 6 mm in diameter at a speed of 1 mm/min until failure occurred, represented by fracturing and/or debonding of the tooth and/or crown. All values involving displacement of the metal rod and the load exerted on the tooth restoration set were recorded by a software program. The value of the force required to cause failure was recorded for each specimen in N.

Failure pattern characteristics of each specimen were defined by observation under a stereoscopic loupe at 40× magnification (EK3ST, Eiconal, São Paulo, SP, Brazil) and were classified according to the four failure modes shown in Table 1.

Values obtained in the compressive strength test and data on the fracture pattern of each group were submitted for statistical evaluation by Mann-Whitney tests to detect significant variations between groups.

### **RESULTS**

Mean fracture strength values for the different groups, standard deviations, and results of the Mann-Whitney test are presented in Table 2. The results of the Mann-Whitney test showed higher fracture strength values for Group GE than for Group GC (p=0.002). Results of the fracture pattern are presented in Table 3.

A high prevalence of fracture of the tooth or restoration with displacement (loss of adhesion) was noted for both groups (Figure 2), as was a low prevalence of three types of fracture: 1) fracture of one conventional crown (10%), which occurred in Group GC; 2) fracture of the tooth (10%), which occurred in Group GE; and 3) displacement without fracture (10%), which occurred in Group GC. It was observed that fractures in the teeth occurred on the

Table 2: Median Fracture Strength Values*†						
Group	Median (±SD)	Minimum Value- Maximum Value				
Endocrown	674.75 <sup>A</sup> (158.85)	543.00-1095.64				
Conventional crown	469.90 <sup>B</sup> (129.83)	316.26–787.62				

<sup>\*</sup> Values expressed in Newtons (N), standard deviations (SD), minimum and maximum values, and results of the Mann-Whitney test.

<sup>†</sup> Different superscript capital letters indicate significant differences by the Mann-Whitney test (p=0.002).

Table 3: Failure Mode for the Groups Under Study in Percentages, %							
Group	Fracture of the Endocrown or Conventional Crown	Fracture of the Tooth	Fracture With Displacement	Displacement Without Fracture			
Endocrown	0	10	90	0			
Conventional	10	0	80	10			

side on which the test force was applied, and displacement occurred on the opposite side. Only one specimen, which belonged to Group GE, presented root fracture (apical third), and only one, which belonged to Group GC, presented fracture of the cusp of the porcelain crown.

### DISCUSSION

The restorative procedure performed with the conventional crown, the resin composite filling core, and the glass fiber post attempts to reproduce the biomechanical behavior and the esthetic of the enamel and the resilience of the dentin. <sup>17,18</sup>

An Endocrown preserves root tissue and limits internal preparation of the pulp chamber to its anatomic shape. It uses ceramic throughout the entire extension of the cavity<sup>13,14</sup> and, because of its rigidity, does not mimic dentinal tissue mechanically. Nevertheless, under oblique compression forces, higher strength values were detected in Group GE. The thickness and quantity of ceramic used in the restoration of Group GE were significantly greater than those used in Group GC. The high bonding

capacity of lithium disilicate ceramics to the dental structure and the smaller number of bond interfaces probably make the dentin/enamel/ceramic group more resistant when compared with the dentin/enamel/post/resin/ceramic group.

For the endocrowns, 90% of failures were of the tooth fracture type associated with displacement of the restoration on the opposite side of the incidence of force. Only 10% presented fracture of the tooth (in the apical third of the root portion). On the other hand, in the group of ceramic crowns, 80% of test specimens fractured and displaced the restoration on the opposite side of the incidence of force. Nevertheless, with the exception of one specimen from Group GC, no "debonding" of restorations from the teeth occurred, even after fracture. This adequate resistance to displacement is due to the adhesive property of lithium disilicate-based ceramics, which can be acid etched, and promotes micromechanical interlocking with the resinous cement and with the adhesion between resin cement and tooth surface. 19 In cases of endocrowns, lithium disilicate can be considered one of the best restorative materials.

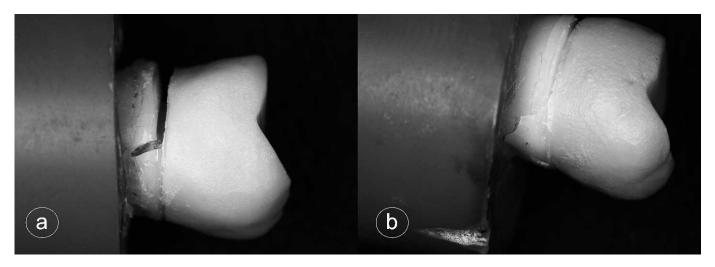


Figure 2. Type of fracture. a) Fracture of the tooth with displacement (loss of adhesion) of the endocrown; and b) fracture of the tooth with displacement (loss of adhesion) of the conventional crown.

Results showed significantly higher fracture strength for endocrowns when compared with conventional crowns; this is consistent with the findings of other *in vitro* studies. <sup>15</sup> *In vivo* studies have also demonstrated the favorable performance of endocrown restorations. <sup>11,14,16</sup> Moreover, it should be taken into account that these restorations are easy to perform but must be made only with reinforced ceramics. <sup>11,14,16</sup>

When the finite element method was used, the favorable performance of endocrown restorations was observed, <sup>20</sup> even in premolars. <sup>21</sup> However, *in vivo* and *in vitro* studies contradict each other concerning the possibility of using restorations of the endocrown type for premolar teeth. <sup>16,22</sup> This is a result of the smaller bond surface and greater crown height of premolars when compared with molars.

Thus, it should be considered that restorations of the endocrown type present advantages for depulped molar teeth, in that they promote adequate function and esthetics, in addition to the biomechanical integrity of the compromised structure of nonvital posterior teeth.<sup>23</sup> Moreover, they appear to be a solution for teeth with a short clinical crown and atresic, calcified, curved, or short root canals that make it impossible to use posts. They are made easily by the dentist, demand less clinical time when compared with conventional crowns, and have good esthetic acceptance because they are made of ceramics. Through elimination of the post and filling core, the number of bond interfaces is reduced, thus making the restoration less susceptible to the adverse effects of degradation of the hybrid layer.<sup>24</sup>

It is worth remembering that the restorative approaches studied simulate extreme situations with extensive loss of dental tissue, which do not allow the use of a ferrule. Knowing that the ferrule increases fracture strength and loss of bond of prosthetic restorations,<sup>3</sup> one understands that with ferulization, in both Group GE and Group GC, responses of greater resistance to oblique compression forces might have been observed.

Results obtained by the present study reinforce the advantages that have been presented in the clinical experiences of various authors. Given the two parameters evaluated—strength and failure mode—the mechanical superiority of restorations of the endocrown type was observed. It is known that *in vitro* tests have limitations in attempts to produce the mechanisms responsible for the occurrence of clinical failure. Therefore, although the method used endeavored to simulate the clinical situation in all

stages, difficulties are inherent to the *in vitro* nature of the study. The results of the present study do not necessarily reflect the clinical performance of the restorative approaches tested. Therefore, from the results obtained, it may be concluded that restorations of the endocrown type are restorative options for endodontically treated molar teeth with extensive loss of coronal structure. They are able to replace conventional crowns supported on posts and filling cores and provide advantages in terms of mechanical performance, cost, and clinical time.

### **CONCLUSION**

Endocrown restorations presented greater fracture strength than indirect conventional crowns associated with glass fiber posts and resin composite filling cores. For both groups, the failure pattern was characterized by fracture of the tooth associated with displacement of the restoration on the opposite side.

### **Conflict of Interest Declaration**

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

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# Effect of Tooth Surface Preparation on the Bonding of Self-Etching Primer Adhesives

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### Clinical Relevance

The effectiveness of some self-etching primer adhesive systems is not significantly affected by the mode of rotary instrumentation used in dentin preparation.

### SUMMARY

The aim of this study was to determine the bonding effectiveness of four self-etching primer adhesives after various tooth preparation protocols. Enamel/dentin specimens were prepared from 84 permanent molars, divided into three enamel preparation groups (silicon carbide paper [SiC]; erbium, chromium:yttrium, scandium, gallium, garnet [Er,Cr:YSGG]

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laser [EL] and diamond bur [DB]) and five dentin preparation groups (SiC, EL, DB, steel [SB], and ceramic burs [CBs]). In each group, specimens were equally divided into four subgroups and were bonded using Clearfil SE Bond (CSE, Kuraray), Xeno IV (XE, Dentsply), Tokuyama Bond Force (TK, Tokuyama) and Filtek Silorane System Adhesive (FS, 3M ESPE), as well as a hybrid resin composite (Clearfil Majesty Esthetic, Kuraray) for CSE, XE, and TK, and Filtek Posterior Restorative (3M ESPE) for FS). After 24 hours of water storage at 37°C, microshear bond strength (µSBS) testing was carried out. Data were analyzed using analysis of variance (ANOVA)-Tukey test at  $\alpha$ =0.05 and bond failure modes assessed. Representative debonded specimens were prepared and examined under the scanning electron microscope (SEM). All adhesives exhibited no significant differences in µSBS on enamel and dentin under the clinical cavity preparation protocols, except for TK on dentin. SEM revealed areas of altered subsurface enamel/dentin following EL ablation.

### INTRODUCTION

The bond strengths of enamel-dentin adhesives to enamel and dentin may be affected by various factors. 1-3 Surface characteristics of tooth structure resulting from different preparations of protocols may affect bonding effectiveness, but this may depend on the type of adhesive system used. Generally, total-etch adhesives are less affected by surface characteristics than are self-etching primer adhesives. 4-13 On enamel, surface roughness 11 while on dentin, surface roughness, <sup>6,11,14</sup> and smear layer quality, 5,8,15 may influence bond strengths for selfetching primer adhesives. Variability in bonding effectiveness of this group of adhesives may be attributed in part to their pH and etching aggressiveness on the enamel and dentin substrate, 16,17 and to the particular enamel-dentin adhesive used.<sup>17-20</sup>

Self-etching primer adhesives simultaneously demineralize and infiltrate dentin; therefore theoretically, more complete resin infiltration may be accomplished than with total-etch adhesives. Because functional and cross-linking monomers are present in a single mixture, resin infiltration to the same depth as dentin demineralization may be more likely in "all-in-one" adhesives than in their two-step counterparts. Results of a previous study by the present authors, in which four recent "all-in-one" adhesives exhibited no significant differences in microshear bond strength, irrespective of dentin tubule orientation and depth, <sup>21</sup> in contrast to two-step self-etching primer adhesives, appear to support this.

Tooth preparation is accomplished with the aid of hand and/or powered cutting instruments, including rotary and laser instruments. Conventional cavity preparation using rotary instruments usually involves the use of more than one type of such instruments. It is important to ascertain the effects on enamel and dentin bond strength of various surface characteristics that may result from such cavity preparation.

With the advent of "minimal intervention" dentistry, more conservative modes of tooth preparation have been introduced. The use of erbium lasers for enamel and dentin preparation has been proposed. However, conflicting reports have described the effectiveness of resin bonding following laser tooth preparation. Increased resistance to acid etching of Er:YAG-irradiated enamel has been reported. Other authors have reported a significant increase in calcium and phosphate concentrations in irradiated dentin at the cavity floor following

erbium, chromium:yttrium, scandium, gallium, garnet (Er,Cr:YSSG) irradiation.<sup>29</sup> Another study reported a significant increase in quantities of calcium in Er,Cr:YSSG-irradiated canine mandibular bone, although the calcium:phosphate ratio was not significantly affected.<sup>30</sup> A more mineralized, acidresistant enamel surface may resist etching by the weak acid of a self-etching primer adhesive.<sup>17</sup> A more mineralized enamel surface, such as could arise following laser irradiation, may result in poorer etching and lower bond strengths.

More recently, a novel innovation for the excavation of soft, carious dentin, <sup>31</sup> a ceramic bur (KISM-Cera Bur, Komet, Lemgo, Germany) was developed. There are as yet no reports on the effect on cavity preparation of using the ceramic bur on resin bonding.

A resin composite based on a new technology using silorane resin was recently introduced. The name "silorane" is derived from its chemical building blocks of siloxanes and oxiranes.<sup>31</sup> The combination of the properties of siloxanes and oxiranes results in a resin composite that the manufacturer claims is a biocompatible, hydrophobic, low-shrinkage product.<sup>31</sup> The novel resin matrix requires a specific two-part self-etching primer adhesive. Although the primer component of the adhesive is made up of hydrophilic methacrylate-based resins similar to those of other adhesive systems,<sup>31</sup> the hydrophobic adhesive bond, developed to be compatible with the new silorane restorative resin,<sup>31</sup> has been reported to exhibit lower polymerization stress and shrinkage. The microshear bond strengths exhibited by this adhesive to enamel and dentin after different tooth preparation conditions are not known.

The aim of this study was to determine the microshear bond strengths of one two-step self-etching primer adhesive, two "all-in-one" adhesive systems, and the silorane-based adhesive to enamel and dentin, prepared using a high-speed flat-fissure medium-grit diamond bur, a slow-speed cross-cut flat-fissure steel bur, a round ceramic bur, and an Er,Cr:YSGG laser. The null hypothesis tested was that there is no difference in microshear bond strengths of one two-step self-etching primer adhesive, two "all-in-one" adhesives, and the silorane-based adhesive to enamel and dentin prepared using various tooth preparation methods.

### **MATERIALS AND METHODS**

Ethics approval was obtained from the University of Melbourne Human Research Ethics Committee for the collection and use of 84 whole human permanent molar teeth from the Royal Dental Hospital in Melbourne. The teeth were stored in 1% chloramine T (pH = 9.1) solution for two weeks, transferred into phosphate-buffered saline solution (pH = 7.2) at  $4^{\circ}$ C, and used within six months of extraction. Twentyfour teeth were used for enamel specimen preparation and were sectioned at the cementoenamel junction and perpendicular to the occlusal surfaces mesiodistally and buccolingually to obtain 96 enamel specimens. Sixty-four enamel specimens from 16 teeth were mounted in dental stone in plastics molds with the enamel surface exposed, labeled according to tooth number and ground with 600-grit silicon carbide paper (SiC). These specimens were divided into two groups for tooth preparation using 600-grit SiC paper as control and the erbium laser (EL, Waterlase; Biolase Technology Inc, San Clemente, CA, USA); each group comprised 32 specimens from eight teeth. The 32 enamel specimens from the remaining eight teeth were mounted as above but were not ground with SiC paper. These constituted the specimens that were prepared with a high-speed medium-grit flat-fissure diamond bur (DB, average particle size 100 µm, DB 835 314 012). Surface enamel was abraded by the diamond bur in a handheld high-speed handpiece (Trend, TC 95BC, W & H, Bürmoose, Austria) with two straight strokes to obtain a flat surface. The bur was changed after every four preparations.

The erbium laser was used with the following characteristics: wavelength 2780 nm, power output range of 0-6 W, pulse duration 140 μs, repetition rate 20 Hz, and pulse energy of 0-300 mJ. A G6 fiberoptic sapphire tip, 6 mm in length and with a spot size of 600 µm diameter, was used in a noncontact, focused mode held perpendicular and 1-2 mm away from the surface being ablated. The enamel was irradiated at a power setting of 5.5 W (energy density 171.9 J/cm<sup>2</sup>) with air pressure 90% and water pressure 80%, and was moved back and forth until the whole surface was ablated. An average enamel surface area of 4 mm × 4 mm was irradiated for five seconds. The 32 enamel specimens from each tooth preparation group were further divided into four subgroups comprising the eight specimens from two teeth for bonding with one of the two-step self-etching primer adhesives, Clearfil SE Bond (CSE, Kuraray Medical, Okayama, Japan); two "all-in-one" adhesives-Xeno IV (XE, Denstply Caulk, Milford, DE, USA) and Tokuyama Bond Force (TK, Tokuyama Dental Corp, Tokyo, Japan); and the Filtek Silorane Adhesive System (FS, 3M

ESPE, St Paul, MN, USA). Details of the materials are provided in Table 1. After tooth preparation, the enamel was dried, enamel/dentin adhesives were applied according to manufacturers' instructions (Table 2), and three to four 0.75-mm diameter and 1.5-mm high translucent polyvinylchloride microtubes were placed on the adhesive surface before curing and the adhesive light-cured using a lightemitting diode light unit with an output intensity of 800 mW/cm<sup>2</sup> (Bluephase C8, Ivoclar Vivadent, AG, Schaan, Liechtenstein). The intensity of the curing light was checked before use. A hybrid resin composite (Clearfil Majesty Esthetic, Kuraray Medical) was loaded into the tubes and cured for 20 seconds. For specimens bonded with FS, the Filtek Silorane Posterior Restorative (3M ESPE) was loaded into the microtubes and cured for 40 seconds.

The remaining 60 molar teeth were used in dentin specimen preparation. The occlusal thirds of the crowns were removed by sectioning perpendicular to the tooth long axes, the exposed dentin surfaces checked to confirm complete removal of the enamel, and the crowns sectioned at the cementoenamel junctions. The dentin discs were mounted in dental stone and wet-ground with 600-grit SiC paper. Dentin specimens were divided into five groups of 12 specimens each for tooth preparation using the DB, a slow-speed cross-cut flat-fissure steel bur (SB, S36204 014, Komet), a slow-speed round ceramic bur (CB, K4547 014, Komet), EL, and SiC paper as controls. The high-speed handpiece carrying the DB was run across the dentin surface in four straight strokes until the whole surface was cut. The SB and CB were used in a slow-speed handpiece (WD-75, W & H) and moved back and forth on the surface until the whole dentin surface was cut. Dentin was lased using the erbium laser at a power setting of 3.5 W (energy density 109.4 J/cm<sup>2</sup>), an air pressure setting of 65%, and a water pressure of 60%. An average dentin surface area of  $8 \text{ mm} \times 6 \text{ mm}$  was irradiated for 15 seconds. On completion of the tooth preparations, the 12 dentin specimens in each preparation group were further divided into four groups of three for bonding with the four dentin adhesives (Table 2). Resin bonding was carried out as described for the enamel specimens; however, six to eight microtubes were bonded per dentin disc.

The specimens were placed in distilled water in an incubator at 37°C for 24 hours. Plastics molds with the embedded specimens were mounted in a jig with the enamel/dentin surfaces flush with the external surface of the jig. A wire loop (0.35-mm diameter) was wound around the bonded cylinder with the

Adhesive	Code	рН	Contents	Manufacturer	Batch No.
Clearfil SE Bond	CSE	2.0	Primer: 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), 2-hydroxyethyl methacrylate (HEMA), hydrophilic dimethacrylate, dicamphoroquinone, <i>N,N</i> -diethanol- <i>p</i> -toluidine, water	Kuraray Medical, Okayama, Japan	51766
			Bond: 10-MDP, Bisphenol A diglycidylmethacrylate (Bis-GMA), HEMA, hydrophobic dimethacrylate, dicamphoroquinone, <i>N,N</i> -diethanol- <i>p</i> -toluidine, silanated colloidal silica		
Xeno IV	XE	~2.1	Mono-, di-, and trimethacrylate resins, dipentaerythritol penta acrylate monophosphate, cetylamine hydrofluoride, acetone, water	Dentsply-Caulk, Milford, DE, USA	080411
Tokuyama Bond Force	TK	2.3	Methacryloyloxyalkyl acid phosphate, C2-4 alkyl, HEMA, Bis-GMA, triethylene glycol dimethacrylate (TEGDMA), camphoroquinone, purified water, alcohol	Tokuyama Dental, Tokyo, Japan	YT11407
Clearfil	_	_	Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated Okayama, Jaj		00003C
Majesty Esthetic	_		barium glass filler, prepolymerized organic filler, di- camphoroquinone	Okayama, Japan	00004D
Filtek Silorane System Adhesive	FS	2.7	Primer: HEMA, Bis-GMA, phosphoric acid methacryloxy-hexylesters, 1,6-hexanediol dimethacrylate, copolymer of acrylic and itaconic acids, (dimethylamino)ethyl methacrylate, dicamphoroquinone, phosphine oxide, silane-treated silica, water, ethanol	3M ESPE, St Paul, MN, USA	20080311
			Bond: substituted dimethacrylate, TEGDMA, phosphoric acid methacryloxy-hexylesters, 1,6-hexanediol dimethacrylate, di-camphoroquinone, silane-treated silica		
Filtek Silorane Posterior Restorative	_	_	3,4-epoxycyclohexylcyclopolymethylsiloxane, bis-3,4-epoxycyclohexylethyl-phenyl-methylsilane, mixtures of resin and siloxane by-products, silane-treated quartz, yttrium trifluoride	3M ESPE, St Paul, MN, USA	20080311

composite-dentin interface at one end and was attached to a load cell connected to a computer at the other end. Microshear bond strength testing was carried out on a universal testing machine (Imperial 1000, Mecmesin, West Sussex, UK) using the corresponding computer software (Emperor, version 1, Mecmesin) at a cross-head speed of 1 mm/min until failure occurred. Maximum loads at failure were recorded and converted to MPa by dividing the failure load by the bonded specimen surface area. Logarithmic transformation of the data was done to satisfy the assumptions of the model for statistical analyses. Means and standard deviations were

obtained for each adhesive for the tooth preparation methods. Random effects mixed model analysis of variance (ANOVA) was used with the compound symmetry covariance matrix option for analyses of data obtained from samples from the same tooth. Bonferroni correction was used to compare preparation methods. Statistical analyses were carried out using the Statistical Package for the Social Sciences (SPSS), version 17 software for Windows (SPSS Inc, Chicago, IL, USA), at a significance level of p < 0.05. Bond failure modes were assessed using a light microscope at  $100 \times$  magnification and were classified as follows:

Adhesive	Priming	Bonding	Light Cure	Resin Composite/ Light Cure
Clearfil SE Bond	Primer applied, left undisturbed for 20 s, air-thinned for 5 s	Bond applied and gently air-blown for 3 s	10 s	Clearfil Majesty Esthetic/ 20 s
Xeno IV	Adhesive applied with a scrubbing action for 15 s, reapplied in same manner for 15 s, gently air-thinned for 5 s		10 s	Clearfil Majesty Esthetic/ 20 s
Tokuyama Bond Force	Adhesive applied, left in place for 20 s, air-thinned for 3 s		10 s	Clearfil Majesty Esthetic/ 20 s
Filtek Silorane System Adhesive	Primer applied, rubbed in for 15 s, air-thinned for 3 s, light-cured for 10 s	Bond applied, gently air-thinned for 5 s	10 s	Filtek Silorane Posterior Restorative/40 s

- A = Adhesive bond failure, involving more than 50% of the bonded surface
- $\bullet$  C = Cohesive failure in resin composite, involving more than 50% of the bonded surface
- M = Mixed bond failure involving up to 50% each of adhesive and cohesive failures

Debonded specimens representative of the adhesives and tooth preparation methods were retrieved from their molds, cleaned ultrasonically, embedded in epoxy resin (Epofix, Struers, Copenhagen, Denmark) for 24 hours, and sectioned perpendicular to the bonded surfaces. Exposed cross-sectional surfaces were polished with 600, 1200, 2000, and 4000-grit SiC papers, then 3 µm, 1 µm, and 0.25 µm diamond pastes; they were ultrasonically cleaned for 30 minutes, etched with 10% orthophosphoric acid for three to five seconds, rinsed for five seconds, placed in 5% NaOCl solution for five minutes, and rinsed under running water for five minutes. Specimens were dehydrated in an ascending ethanol:water series (10%, 30%, 50%, 70%, 90%, 100%) for one hour in each with at least three changes in 100% ethanol and critical point-dried (ethanol/CO<sub>a</sub>) (CPD 030, Bal-Tec AG, Balzers, Liechtenstein). Immediately after drying, the specimens were mounted on aluminum stubs, gold sputter-coated, and examined under the field emission scanning electron microscope (FE-SEM, Quanta 200F, FEI, Hillsboro, OR, USA). Two enamel and dentin specimens were prepared using each of the tooth preparation methods.

### **RESULTS**

Statistical analyses using random effects mixed model ANOVA showed that microshear bond strengths varied according to tooth preparation on enamel (p=0.002) and dentin (p=0.002), and also varied according to enamel-dentin adhesive on enamel (p=0.001). The results of mixed model ANOVA and Bonferroni correction for microshear bond strengths are shown in Tables 3 and 4. Results of analyses showed that bond strengths to enamel (Table 3) were significantly different between SiC and EL only for CSE (p=0.29) and XE (p=0.36). For TK, bond strengths to enamel were not significantly different between groups.

For dentin (Table 3), the microshear bond strengths of CSE and XE were not significantly affected by tooth preparation methods. Bond strengths for TK were significantly lower (p<0.05) following EL in comparison with other preparation methods.

At least 62% of bond failures in each enamel adhesive/tooth preparation group were mixed failures, followed by adhesive failures and a few cohesive failures (Figure 1). On dentin, bond failures in each adhesive/tooth preparation were also mainly mixed in nature (70%), except after bonding with XE on dentin prepared using the cross-cut flat-fissure steel bur, where bond failure was completely adhesive (Figure 2). Adhesive failures were also observed in the other adhesive/tooth preparation groups, but no cohesive failures were observed in dentin.

Table 3: Microshear Bond Strength Test Results*†					
Tooth Preparation Protocol	Clearfil SE Bond	Xeno IV	Tokuyama Bond Force		
Enamel (n=24)					
600-grit SiC paper	22.0 (4.48) <sup>A</sup>	14.6 (3.43) <sup>A</sup>	12.1 (3.40) <sup>A</sup>		
Medium (100-μm)-grit diamond bur	18.9 (5.12) <sup>A,B</sup>	11.6 (2.93) <sup>A,B</sup>	10.2 (4.44) <sup>A</sup>		
Er,Cr:YSGG laser	12.5 (2.80) <sup>B</sup>	10.1 (3.16) <sup>B</sup>	14.3 (4.19) <sup>A</sup>		
dentin (n=20)					
600-grit SiC paper	14.7 (4.85) <sup>A</sup>	13.0 (4.79) <sup>A</sup>	11.8 (3.21) <sup>A</sup>		
Medium (100-μm)-grit diamond bur	13.0 (5.05) <sup>A</sup>	12.2 (3.95) <sup>A</sup>	12.6 (2.96) <sup>A</sup>		
Steel bur	10.3 (4.58) <sup>A</sup>	12.6 (5.73) <sup>A</sup>	11.6 (4.47) <sup>A</sup>		
Ceramic bur	11.8 (5.37) <sup>A</sup>	11.3 (4.82) <sup>A</sup>	12.1 (3.41) <sup>A</sup>		
Er,Cr:YSGG laser	9.00 (3.35) <sup>A</sup>	10.4 (2.85) <sup>A</sup>	6.78 (2.12) <sup>B</sup>		

<sup>\*</sup> Means (SD) in MPa.

For FS (Table 4), microshear bond strengths to enamel varied according to enamel preparation (p<0.001). Bond strength of FS was significantly different between SiC and EL (p=0.005). On dentin, bond strengths between tooth preparation groups were not significantly different. Similar to observations made with the other three self-etching primer adhesives, bond failure modes for FS (Figure 3) on enamel were mainly mixed (85%), followed by a few adhesive and cohesive failures. On dentin, mixed failures constituted 70%, followed by adhesive failures. No cohesive failures were observed.

FE-SEM images of the cross-sectional surfaces of debonded specimens showed a remarkable difference between enamel and dentin prepared with SiC paper and the erbium laser. The typical keyhole appearance of enamel prisms was not observed in the enamel immediately underlying the lased surface (Figure 4a,b); instead there appeared to be collapse of the enamel prismatic structure. Subsurface crack formation with infiltration of adhesive resin into the cracks was observed within and underlying the lased enamel. This zone of altered enamel appeared distinctively different from the sound enamel below it. On dentin, cross-sectional views of the debonded

surface revealed that the dentin immediately underlying the lased surface appeared structurally different from the sound dentin below it and from dentin prepared with SiC paper (Figure 5a,b). An area of altered dentin was observed that exhibited fewer resin tags and appeared denser in consistency, with collagen fibrils less readily visible. A layer appeared to be demarcating altered dentin from the sound dentin below it.

### DISCUSSION

Reports have described the variability of bond strengths exhibited by self-etching primer adhesives under different tooth preparation protocols. 4-8,11,13,14,23,32 On enamel, conflicting reports have been put forth on the efficacy of laser preparations and the effectiveness of subsequent resin bonding. Some reports have found no significant difference in microtensile bond strengths of a two-step self-etching primer adhesive vs Er:YAG-lased enamel. 11,23 Other reports found significant lowering of enamel bond strengths of the same two-step adhesive, a one-step self-etching primer adhesive, and a total-etch adhesive following Er,Cr:YSGG<sup>33</sup> and Er:YAG laser ablation, 11 in comparison with a

<sup>†</sup> Statistical analyses were carried out using random effects mixed model ANOVA and Bonferroni test at  $\alpha$ =0.05. Logarithmic transformation of microshear bond strength data was used in the analyses to satisfy the assumption of the model. Within the same columns, values with different superscript letters are significantly different for either substrate.

Table 4: Microshear Bond Strength Results for Filtek Posterior Restorative System*†				
Tooth Preparation Protocol	Filtek Posterior Restorative System			
Enamel (n=20)				
600-grit SiC paper	17.1 (3.21) <sup>a</sup>			
Medium (100μm)-grit diamond bur	10.2 (2.16) <sup>a,b</sup>			
Er,Cr:YSGG laser	8.94 (1.75) <sup>b</sup>			
Dentin (n=20)				
600-grit SiC paper	6.37 (1.52) <sup>a</sup>			
Medium (100μm)-grit diamond bur	7.90 (2.14) <sup>a</sup>			
Steel bur	8.23 (2.59) <sup>a</sup>			
Ceramic bur	7.17 (2.09) <sup>a</sup>			
Er,Cr:YSGG laser	8.02 (1.53) <sup>a</sup>			

<sup>\*</sup> Means (SD) in MPa.

medium-grit diamond bur. In yet another study, the bond strength of one "all-in-one" adhesive was not different between Er,Cr:YSGG-lased and medium-grit diamond bur–cut enamel.<sup>33</sup>

Our study revealed no significant differences in bond strength on diamond bur-cut enamel and lased enamel for the two-step self-etching primer adhesive and two "all-in-one" adhesives. This result is similar in part to those of Cardoso and others, 33 who reported no change in microshear bond strength for the "all-in-one" adhesive used in their study but in part contrasts with reported significant lowering of bond strength of the two-step adhesive to enamel following Er, Cr: YSGG laser ablation compared with diamond bur-cut enamel. Kim and others<sup>28</sup> reported increased resistance to acid etching of Er:YAGirradiated enamel. Hossain and others<sup>29</sup> reported a significant increase in calcium and phosphate concentrations in irradiated dentin at the cavity floor following Er, Cr:YSSG irradiation. Furthermore, Kimura and others<sup>30</sup> reported a significant increase in

quantities of calcium in Er,Cr:YSSG-irradiated canine mandibular bone, although the calcium:phosphate ratio was not significantly affected. A more mineralized, acid-resistant enamel surface may resist etching by the weak acid of a self-etching primer adhesive. A more mineralized enamel surface, such as could arise following laser irradiation, may thus result in poorer etching and could explain in part the lower bond strengths observed for the two-step adhesive following laser irradiation. A recent finding by the same authors of a weak negative correlation between enamel microhardness and the bond strength of the two-step self-etching primer adhesive used in this study appears to support this argument.

Silicon carbide paper, usually a 600-grit surface, is used in the laboratory to prepare specimens, simulating clinical dentin/enamel preparation using a medium-grit diamond bur. Our study found no significant difference in bond strengths between the two methods on enamel and dentin for all adhesives; however, higher bond strengths were recorded with SiC. This suggests that laboratory microshear bond strengths using SiC may be higher than what may be obtainable under clinical tooth preparation conditions if a medium-grit diamond bur was used. However, significantly lower bond strengths were noted on lased enamel compared with SiC for three of the adhesives. This outcome may be explained by SEM findings of microcracks and structural alterations in enamel, which could have compromised resin bonding. SEMs of the subsurfaces of debonded lased enamel (Figure 4) revealed areas of altered enamel in which the typical keyhole appearance of enamel prisms was not present; instead, there appeared to have been collapse, cracking, and shattering of the enamel prismatic structure. The Er, Cr: YSGG laser is a hydrokinetic system; during irradiation, water ejected from an air-water spray onto the tooth surface is absorbed by incident radiation, causing heating and water evaporation, which results in high-stream pressure, which in turn induces microexpansion and explosion of dental hard tissues.<sup>33</sup> The Er,Cr:YSGG laser has been reported to result in enamel melting and recrystallization<sup>33</sup> and subsurface grooving.<sup>35</sup> Such a surface may compromise resin penetration and/or introduce weaknesses in the bond, which may lead to premature bond failure and low bond strengths.

On dentin, the adhesives exhibited no statistically significant difference in microshear bond strength after various tooth preparation protocols, except for

<sup>†</sup> Statistical analyses were carried out using random effects mixed model ANOVA and Bonferroni test at  $\alpha$ =0.05. Logarithmic transformation of microshear bond strength data was used in the analyses to satisfy the assumption of the model. Within the same columns, values with different superscript letters are significantly different for either substrate.

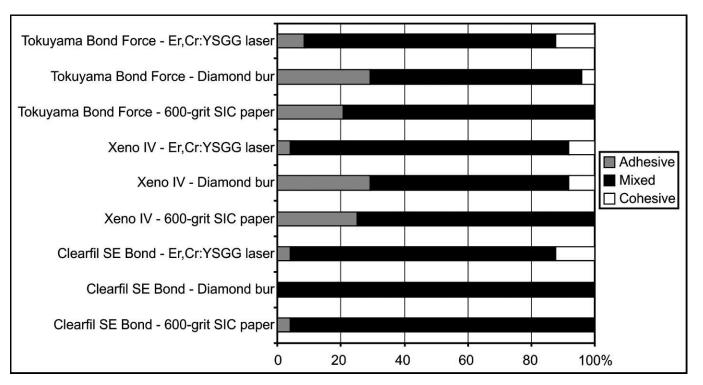


Figure 1. Bond failure modes observed on enamel. Adhesive bond failure—involving more than 50% of the bonded surface; cohesive failure in resin composite—involving more than 50% of the bonded surface; and mixed bond failure—involving up to 50% each of adhesive and cohesive failures.

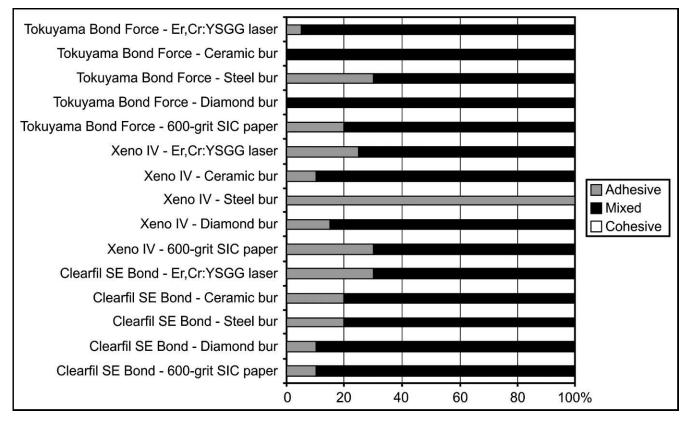


Figure 2. Bond failure modes observed on dentine. Adhesive bond failure—involving more than 50% of the bonded surface; cohesive failure in resin composite—involving more than 50% of the bonded surface; and mixed bond failure—involving up to 50% each of adhesive and cohesive failures.

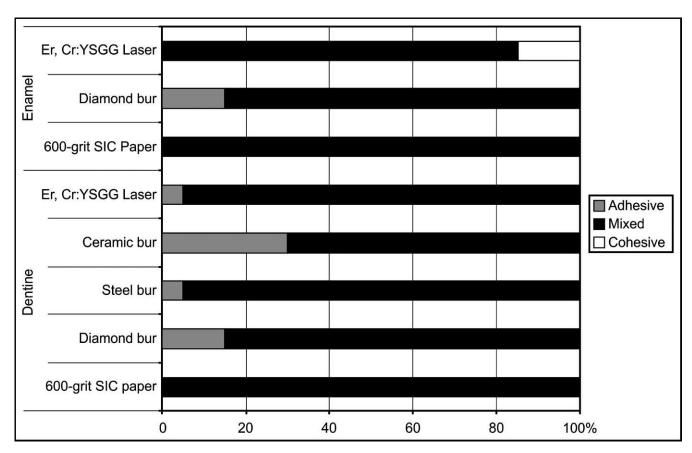


Figure 3. Bond failure modes observed for the Filtek Silorane Posterior Restorative. Adhesive bond failure—involving more than 50% of the bonded surface; cohesive failure in resin composite—involving more than 50% of the bonded surface; and mixed bond failure—involving up to 50% each of adhesive and cohesive failures.

one "all-in-one" adhesive, which reported lower bond strengths following Er,Cr:YSGG laser ablation.

Results suggest that bond strengths of the selfetching primer adhesives used in this study to dentin may not be affected by the type of rotary instrument used. However, variability in bond strengths of twostep self-etching primer adhesives in relation to "allin-one" adhesives to dentin surface preparation has been reported in previous studies. 10,14,36 Semeraro and others<sup>10</sup> reported that two "all-in-one" adhesive systems exhibited no significant differences in microtensile bond strength to dentin prepared with regular and superfine-grit diamond burs. In the same study, 10 another "all-in-one" adhesive and a two-step self-etching primer adhesive exhibited significantly higher bond strengths with a finer surface finish. In another study, however, Ermis and others<sup>37</sup> reported no significant difference in microtensile bond strength of the two-step selfetching primer adhesive and one "all-in-one" adhesive bonded to dentin prepared with medium-, fine-, and extra-fine-grit diamond burs. However, lower bond strengths were observed for another "all-inone" adhesive used.<sup>37</sup> In yet another study, Inoue
and others<sup>14</sup> reported significantly higher bond
strength of a one-step (two-bottle) self-etching
primer adhesive to dentin finished with a superfine-grit diamond bur compared with a regular-grit
diamond bur. The variable bond strength results
obtained may be explained by factors such as the
thickness of the dentin smear layer after preparation, <sup>8,15</sup> the aggressiveness of the adhesive primers, <sup>20</sup> and the nature of the dentin surface, whether
sound or sclerotic.<sup>38</sup>

In contrast to the results obtained in our study, Ogata and others <sup>13</sup> reported significantly lower bond strength of three two-step self-etching primer adhesives to dentin prepared with a cross-cut flat-fissure steel bur compared with a medium-grit diamond bur. The difference in results may lie in the quality of the smear layer produced following dentin preparation. In the above study, the authors reported that steel burs at a speed of 2000 rpm were used; those used in our study had a speed of up to 40,000 rpm. This

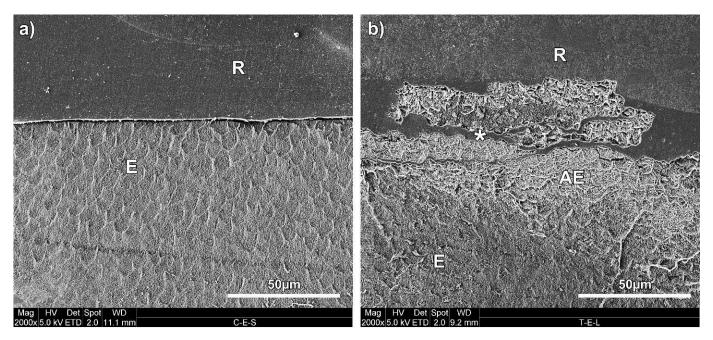


Figure 4. Representative FE-SEM images of cross-section of a) enamel surface prepared with 600-grit silicon carbide paper; and b) enamel prepared with the Er,Cr:YSGG laser. The typical keyhole appearance of enamel prisms (as seen in part a) is distorted in the enamel immediately beneath the lased surface. Collapse of the enamel prismatic structure and subsurface crack formation within the altered enamel are evident. Resin infiltration into the cracks has occurred in the underlying enamel as the result of crack formation (asterix). AE, altered enamel; E, sound enamel; R, resin

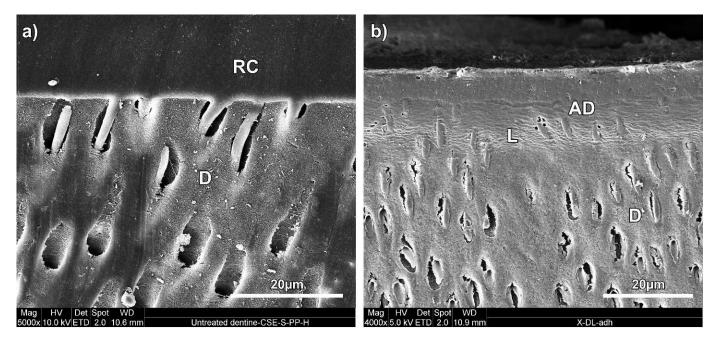


Figure 5. Representative FE-SEM of a) bonded interface of dentin prepared with 600-grit silicon carbide paper; and b) cross-section of debonded dentin surface prepared with Er,Cr:YSGG laser. The dentin immediately beneath the debonded surface (AD) appears to be different in structure to the sound dentin further below (D). This altered dentin appears to be denser in consistency and exhibits fewer resin tags, and a layer (L) appears to be demarcating the altered dentin from the sound dentin. AD, altered dentine; D, sound dentine; RC, resin composite.

difference in speed of cut may produce different stresses on dentin and different smear layer characteristics, which may in turn influence bond strengths. The novel ceramic bur resulted in comparable dentin microshear bond strengths to other tooth preparation protocols and therefore appears promising. Failure modes were mainly mixed in nature and consistent with other studies. <sup>21, 37</sup>

The lowest bond strengths were reported following dentin laser ablation for the two-step and both "allin-one" adhesives, but this was significant only for TK. Other changes brought about by laser tooth preparation that could weaken adhesion include formation of microcracks beneath the hybrid layer, collagen denaturation due to selective ablation of organic tissue resulting in less collagen being exposed, 23, 29 and deficient dentin hybridization. 36 Martinez-Insua and others<sup>35</sup> also reported the presence of widespread subsurface grooving in Er:YAG-lased enamel and dentin. Crack formation and other alterations observed in subsurface enamel and dentin in this study could be attributed to thermal changes caused by laser irradiation. SEM evaluation of the subsurface of the debonded lased dentin (Figure 5) revealed dentin that appeared more dense in consistency, exhibited fewer resin tags, and appeared demarcated from the sound dentin below it by a distinctive layer. These changes observed in the lased enamel could have weakened the dental tissue and impeded resin infiltration and dentin hybridization, predisposing to premature bond failure and thus significantly lower bond strength observed for the "all-in-one" adhesive. With the altered state of the dentin, the viscosity of the adhesive and its mode of application (i.e., with or without scrubbing) may play a more important role in assisting adhesive penetration into dentin, and this may have had an effect on the bond strength outcome on dentin.

The silorane-based adhesive exhibited no significant difference in microshear bond strength to enamel and dentin under the clinical tooth preparation protocols. Low microshear bond strengths were, however, observed with the adhesive on dentin. This newly developed resin composite is hydrophobic, and its low polymerization shrinkage is attributed to the cationic ring-opening reaction, which results in a gain in chain length and subsequent lower polymerization contraction compared with the radical addition polymerization of methacrylate resins.  $^{\rm 39}$  The pH of the silorane-based self-etching primer<sup>39</sup> is the least acidic of all the adhesives used in this study. The demineralizing aggressiveness on dentin of a self-etching primer adhesive has also been reported to be related to its pH.20 The adhesive primer is cured before application of the bond; dentin hybridization may thus be entirely dependent on the degree of demineralization, penetration, and cross-linking produced by the primer. The pH and the resultant hydrophilicity of the silorane adhesive primer may greatly determine the extent of resin permeation into dentin. Additional studies are required.

Findings in this study show that the microshear bond strengths of the two-step self-etching primer adhesive, one "all-in-one" adhesive, and the Filtek Silorane Posterior Restorative System were not affected by tooth preparation methods. However, the bond strength of one "all-in-one" adhesive was significantly lower after laser ablation. Therefore, the null hypothesis cannot be accepted.

#### **CONCLUSIONS**

Although the microshear bond strength of one "all-in-one" adhesive may be significantly affected by dentin laser ablation, the bond strengths of all other self-etching primer adhesives used in this study, including the silorane adhesive, were not significantly affected by tooth preparation methods. Alterations in subsurface enamel and dentin, which may compromise resin bonding, were observed following Er,Cr:YSGG laser ablation.

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## The Effect of Distance and Tooth Structure on Laser Fluorescence Caries Detection

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#### Clinical Relevance

Devices used to aid occlusal caries diagnosis are supposed to detect small lesions deep in the pit and fissure system. In detecting small occlusal caries, distance and tooth structure may separate the instrument and the carious lesion. In this study distance and tooth structure were found to reduce the ability of the DIAGNOdent to detect caries.

#### SUMMARY

The DIAGNOdent, a device used in caries detection, uses a laser to excite fluorescence from pigments in carious tooth structure. In clinical use assessing occlusal surfaces, distance and tooth structure may separate the instrument's tip from the fluorescent source. The aim of this *in vitro* study was to examine

the effect of distance and tooth structure on laser fluorescence (LF) readings.

In one set of experiments, a porphyrin pigment in oil suspension was used as a LF signal source. Thin slices of enamel and dentin were obtained from extracted molars. Pigment-induced LF readings were obtained when these slices were placed between the porphyrin pigment and the LF instrument's tip. The effect of either demineralized or intact tooth tissue on pigment-induced LF readings was assessed. In other experiments on extracted molars with small occlusal caries, LF readings were taken from pit/fissure sites before and after removal of the occlusal surface.

LF readings are proportional to pigment concentration and inversely proportional to the distance between the suspension and the instrument's tip. Enamel, demineralized enamel, dentin, and demineralized dentin all caused significant reductions in LF signal, all readings being taken with the same tip-pigment distance. Demineralized enamel (white with

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intact surface) caused the most reduction. After sectioning of carious teeth, there was a significant increase in LF readings.

The results of this study indicate that distance and the presence of tooth structure between the carious lesion and the instrument's tip reduce LF readings. These results indicate that anatomic factors interfere with the LF device's ability to assess occlusal caries. DIAGNOdent readings should not be relied on when making diagnostic decisions.

#### INTRODUCTION

Carious lesions affecting the occlusal surfaces of posterior teeth can be difficult to detect using conventional clinical examination techniques. 1,2 These lesions begin as areas of demineralization in the enamel lining the walls of pits and fissures that extend below the actual surface of the tooth.<sup>3</sup> In order to assist dentists in diagnosing small occlusal lesions, researchers have examined ways that the caries process can alter the optics and other physical properties of tooth structure. 4-6 This research has resulted in the development of diagnostic devices that are designed to detect inconspicuous lesions. One goal in developing caries diagnostic devices is to have the capability of detecting lesions at a stage before cavitation begins. Areas of subsurface enamel demineralization appear to the unaided eve as white spots. The altered porosity of enamel in these lesions change the tissue's light-scattering characteristics, a property that is used as a basis for a type of cariesdetection device.8 Certain caries-related bacteria produce metabolic products that have characteristic colors and fluorescent properties.<sup>9</sup> These bacterial pigments are responsible for the dark gray or brown color commonly observed in decayed dentin. Porphyrin compounds can be extracted from carious dentin. 10 These pigments fluoresce when excited by red light. The DIAGNOdent (KaVo, Biberach, Germany) is a laser fluorescence (LF) device that transmits 655 nm of laser energy to tooth structure via a specially designed handpiece. The instrument detects fluorescent radiation with wavelengths >680 nm. These wavelengths originate in porphyrincontaining carious tooth structure and penetrate enamel to a degree, allowing the device to detect lesions within the occlusal anatomy.11 This LF device has a digital display with a maximum reading of 99. Numerous studies have sought to determine the diagnostic sensitivity and specificity of LF, to determine the correlation of this instrument's readings with other methods of caries detection, and to

examine the relationship of LF readings with the histological extent of lesions. <sup>12–16</sup> These studies demonstrate a moderate correlation between LF and other methods of caries detection as well as a limited ability to determine the depth and extent of lesions.

When examining lesions set deep in the occlusal anatomy, the pigmented lesion and the LF instrument's tip will be separated by a distance that depends on the depth of the fissures. This separation is due to the fact that the DIAGNOdent's lightemitting and receiving tip is wider than the occlusal fissures and is restricted from entering the fissure. In addition to the physical separation that may exist between the DIAGNOdent tip and the pigmented portion of a carious lesion, intact and demineralized tooth structure lining the superficial portion of the fissures may stand in the light path between the deep areas of bacterial pigment and the LF tip. In small occlusal lesions partially demineralized enamel may cover stained dentin. The effect of this white, highly light-scattering type of tooth structure on the LF readings originating in deeper tissue has not been determined.

The ability of the LF device to detect caries through intact and demineralized tooth structure has not been extensively studied. Iwami and others <sup>17</sup> examined the effect of dentin slices ranging in thickness from 0.2-1.4 mm on the LF readings obtained from occlusal caries in extracted teeth. Dentin slices were prepared by either cutting parallel or perpendicular to the direction of the dentinal tubules. Both orientations of dentin were found to attenuate the LF signal strength in a thickness-related fashion, with slices cut parallel to the direction of the tubules attenuating more than disks cut perpendicular; this was attributed to the light-scattering effect of the tubules.

The purpose of this study was to examine the effect of signal source-LF tip distance and interposed tooth structure on the instrument's readings. By examining the effects of both distance and tooth structure, we simulated the anatomic factors separating the DIAGNOdent tip from the pigmented portion of the carious lesion. The null hypothesis tested was that distance and intervening tooth structure would have no effect on LF readings. In one set of experiments suspensions of porphyrin pigment acted as the LF signal source. Using this suspension, the relationship between the LF tip-signal source distance and the instrument's readings was examined. The impact of tooth structure on LF readings was examined by placing thin slices of

enamel or dentin between the signal source and the LF tip. These measurements were made through the enamel slices both before and after acid demineralization. This allowed us to assess the effect of demineralization on the tendency of enamel and dentin to attenuate the LF signal. The effect of distance and tooth structure on the ability of the LF to detect natural occlusal caries was examined in experiments where readings were obtained from extracted teeth having small occlusal caries before and after the occlusal tooth structure was removed to expose the lesion. It is hoped that the results of this study will not only lead to a better understanding of the LF's limitations, but also assist in developing protocols for evaluating other cariesdetecting devices.

#### **MATERIAL AND METHODS**

#### LF Signal Source

Experiments were performed to assess the effect of tooth structure on LF signal intensity. These experiments used a suspension of porphyrin pigment as a laser fluorescent source. Protoporphyrin IX (Aldrich, St Louis, MO, USA) was dispersed in United States Pharmacopeia mineral oil by grinding and mixing to form a homogeneous suspension that did not settle for at least 24 hours. The pigment in oil suspensions were placed in a 1-mL plastic well and covered with a glass cover slip. The LF handpiece was attached to a modified microscope (Wild, Heerbrugg, Switzerland) in such a way that the LF tip could be placed perpendicular to the cover slip covering the pigment suspension. A digital caliper (model CO 030150, Marathon Watch Company Ltd, Richmond Hill, ON, Canada) was also attached to the microscope, allowing the distance between the LF tip and the cover slip to be measured. LF readings obtained from mineral oil covered with a cover slip were negligible. Using this apparatus, the effect of pigment concentration and tip distance from pigment on LF reading could be determined. Also, small slices of tooth structure could be positioned between the LF tip and the fluorescent pigment without changing the distance between the instrument tip and the fluorescent source.

#### **Tooth Tissue Slice Preparation**

Sections of enamel and dentin (15 each) were obtained from 30 extracted human third molar teeth that were free of obvious caries or restorations. Due to the specific nature of the tooth sections required, one enamel or dentin slice was obtained from each tooth used. Patients from 18–30 years of age in the

New Jersey Dental School Oral Surgery clinic consented to donate their teeth for research purposes. The University's institutional review board approved the tooth collection procedure. Following extraction, teeth were stored in 1% phenol and debrided of soft and hard tissues. Any residual debris found to be adhering to the occlusal surfaces of the teeth was removed gently with a dental probe. The teeth were then examined for the presence of small carious lesions. Molars having small occlusal caries were separated from caries-free teeth. These two sets of teeth were used in separate experiments. All teeth were used within 30 days of collection.

The enamel slices were derived from the lingual surfaces of caries-free molars. First the crowns of the teeth were obtained by fracturing off the roots. Then the crowns were mounted in the sample holding chuck of a low-speed saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) with a diamond blade, so that the lingual surface of the tooth was parallel to the blade. Under water lubrication, the surface enamel was removed. Then an enamel ribbon, measuring approximately  $5 \times 3$  mm and 0.6-mm thick, was obtained by cutting the tooth superficially to the dentinoenamel junction. These ribbons were polished with 600-grit silicon-carbide paper (Buehler) with water lubrication. Following the first set of LF measurements, the enamel specimen was demineralized by immersing the ribbons for five days into a gel containing 0.1 M lactic acid, adjusted to pH 4.5 with sodium hydroxide and thickened with ethyl cellulose (Natrosol, Ashland Aqualon Inc, Parlin, NJ, USA). Following the demineralization treatment the enamel ribbons were rinsed with deionized water. These enamel specimens were then examined under 8-power illumination to ensure that a white lesion was seen when the tissue was wet and that the enamel surface was intact.

The coronal dentin disks (approximately 0.6-mm thick) free of enamel, pulp horns, and any discoloration suggestive of caries, were cut perpendicular to the long axis of caries-free teeth. These disks contained dentin from under the occlusal pits and fissures, allowing us to examine the effect of this tissue on pigment-evoked LF readings. The dentin disks were polished and subjected to demineralizing treatment, using the same procedure as was used for the enamel specimens. The success of the lactic acid treatment in producing dentin demineralization was assessed by determining whether the acid-treated dentin was softer to tactile examination than the intact dentin.

Both the enamel and dentin sections were prepared in such a way so that the enamel rods and dentinal tubules would be perpendicular to the specimen surface because this resembles the orientation of tissues on the occlusal aspect of molars. The lingual surface of molars is fairly flat, allowing us to create an enamel slice where the LF's light path runs approximately parallel to the enamel rods. In the coronal dentin disks, the dentinal tubules are roughly perpendicular to the dentin surface. The LF's light path would then be approximately parallel to the direction of the dentinal tubules.

## **Experiments to Determine the Effect of Enamel and Dentin on LF Readings**

A pigment suspension was placed into a 1-mL well and covered with a cover slip. The LF tip was then raised above the cover slip to a distance that allowed enamel or dentin slices to be positioned into the laser light path (Figure 1). The LF reading at this pigment-tip distance was then measured. Next, the enamel or dentin slices were gently slid on the cover slip until the center of the specimen was in the LF light path and readings recorded. Following demineralizing treatment, LF readings were taken through the same areas of the dentin and enamel specimens. During the positioning of the tooth tissue slices, the distance between the LF tip and the pigment remained unchanged. Tooth tissue slices were kept moist during these experiments. The slices were placed on the cover slip, covering the pigment suspension in such a way as to avoid trapping air between the tooth slice and the cover slip.

#### Experiments to Determine the Effect of Occlusal Anatomy on the LF Readings Obtained From Natural Caries

Third molar teeth having small, visually apparent, occlusal caries were used in these experiments. These teeth had dark areas at the base of enlarged occlusal pits and fissures. The occlusal surfaces were photographed at approximately 8-power magnification (DP12 Microscope Digital Camera System, Olympus, Tokyo, Japan). Then with the occlusal surface moist, LF readings were taken from multiple sites on the occlusal surface. These sites were selected to include all pit and fissure sites thought to represent areas of possible decay, as well as sites in the fissure system and areas on the cusp inclines observed to be caries free. Using Microsoft PowerPoint, the LF readings obtained from various occlusal sites were superimposed onto the photograph of the tooth, creating a LF map of the surface. The occlusal surface was then

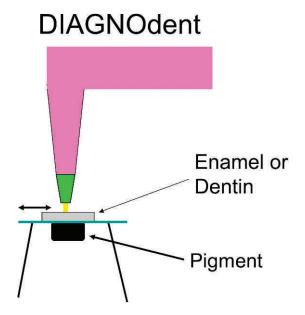


Figure 1. Experimental apparatus used to examine the effect of enamel and dentin slices on LF readings. A porphyrin pigment in oil suspension covered with a microscope-slide cover slip acts as the LF signal source. The DIAGNOdent handpiece is held in such a way that the tip is positioned at a fixed distance from the cover slip. Enamel or dentin slices can be slid between the instrument tip and the pigment and the effect of tooth tissue on the pigment-induced reading assessed. LF readings were taken through enamel and dentin slices before and after demineralization with lactic acid. LF readings were taken without the tooth slice, through the intact slices, and through demineralized tissues.

removed from the tooth by cutting perpendicular to the long axis of the tooth with the Isomet saw (Buehler) and water lubrication. This cut was made to a depth that exposed the enamel at the base of the occlusal fissures and differed from one tooth to another. The resulting new tooth surface was photographed as before and sites examined with the LF device, care taken to record measurements from the same sites as were examined on the original occlusal surface. In this way LF maps were created for the cut surfaces. In some teeth, a second cut was then made 0.5 mm below and parallel to the first and a third LF map created for that tooth. The teeth were not allowed to dry out during this process because desiccation has been observed to result in dentin crack formation and unstable LF readings. In total, 31 sites on 12 teeth were evaluated by this procedure. LF readings from the deepest cut were used in the data analysis, comparing the magnitude of the readings taken from the occlusal surface with those obtained from the cut surface.

#### LF Device Use

In all experiments the DIAGNOdent model 2095 was used with the A-tip because this tip is recommended

for occlusal examinations. The DIAGNOdent unit was calibrated prior to each use according to the manufacturer's instructions, using the instrument's ceramic standard. When measurements were being made on teeth, the instrument was zeroed with the tip in contact with healthy-colored enamel near a cusp tip. Readings from teeth were obtained with the tooth surface in a moist state with low ambient light. The A-tip was placed in gentle contact with the surface then rocked slightly; the peak reading obtained from each site was recorded for later analysis. Calibration exercises were held for the three examiners, using both extracted teeth and various concentrations of porphyrin in suspension. During these calibration exercises the LF readings obtained by the three examiners differed by fewer than ±5 units.

#### **Data Analysis**

LF readings are reported as mean ± standard deviation. A one-way analysis of variance with a pairwise Tukey-Kramer test was performed using the JMP statistical program (SAS Institute Inc, Cary, NC, USA) in order to determine whether significantly different LF readings were obtained from a protoporphyrin in oil suspension under the following conditions:

- 1) A small separation (0.75 mm) between the pigment and the LF tip.
- 2) Intact enamel slices (≈0.6 mm thickness) placed between the pigment and the LF tip.
- 3) Demineralized enamel slices placed between the pigment and the instrument's tip.
- 4) Intact dentin slices ( $\approx$ 0.6 mm thickness) placed between the pigment and the LF tip.
- 5) Demineralized dentin slices placed between the pigment and the instrument's tip.

When enamel or dentin was placed between the LF tip and the pigment, the tip-pigment distance was maintained at 0.75 mm. LF measurements were taken at this distance prior to taking readings through each enamel and dentin slice. Significance was set at p < 0.5. A sample-size calculation for this experiment was performed based on pilot study data. Twelve samples in each group would be the minimum number required in order to achieve a statistical power of 0.8 ( $\alpha$  level of 0.05).

In order to determine whether significant differences existed between LF readings obtained from the occlusal surfaces of molars with those obtained following removal of the occlusal surface, a two-

tailed *t*-test was performed. Significance was set at p<0.5.

#### **RESULTS**

#### Relationship Between Porphyrin Pigment Concentration and LF Readings

The relationship between the LF readings and the concentration of pigment dispersed into mineral oil is shown in Figure 2a. Six replicate readings were taken for each pigment concentration. These readings were taken when the distance between the pigment and the LF tip equaled 0. As pigment concentration in the suspension was raised, the LF reading increased. At a pigment concentration of 12.5 mg/mL, a mean LF reading of 95.4 was recorded. When a suspension containing 25 mg/mL was examined, a reading of 99 was recorded; this is the instrument's maximum reading. Further increases in pigment concentration did not cause the LF readings to increase further.

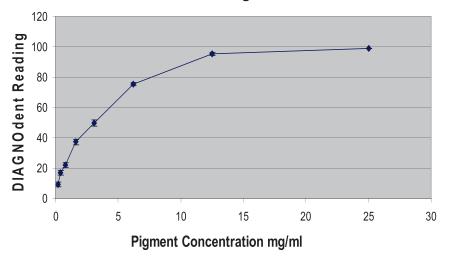
## **Effect of Fluorescent Signal Source Distance** on LF Readings

As shown in Figure 2b, LF readings dropped as the distance between the probe tip and the pigment increased. The relationship between distance and LF reading is shown for two pigment concentrations. One suspension contained a pigment concentration of 12.5 mg/mL. This concentration induced a mean reading of 95.4 when the distance between the instrument tip and the pigment equaled zero (three measurements per data point). With this pigment concentration, each 0.1-mm increase in distance resulted in a reduction in LF reading. The other suspension examined contained a higher (50 mg/mL) concentration of pigment. With this high pigment concentration (each data point represents one reading), increasing the distance between the tip and the pigment failed to reduce the LF reading from the maximum value displayed by the LF device (99) until the separation exceeded 0.4 mm. Beyond this separation, increasing the tip-pigment distance resulted in a drop in LF readings.

#### Effect of Enamel and Dentin on Pigmentinduced LF Readings

At a distance of 0.75 mm a suspension containing 25 mg/mL porphyrin induced a mean LF reading of 51.0  $\pm$  1.79 (n=30). At this LF tip-cover slip distance, enamel and dentin slices can be slid into the laser light path and the effect of tooth structure on LF

## a Porphyrin Concentration verse DIAGNOdent Reading



#### b DIAGNOdent readings verse distance

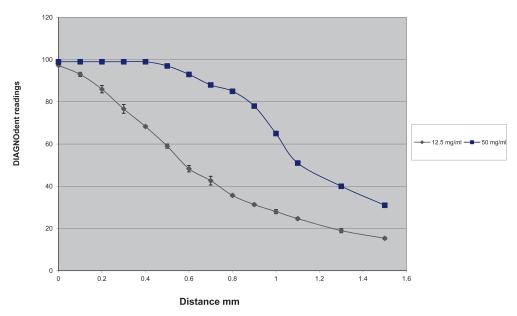


Figure 2. (A): Effect of protoporphyrin pigment concentration on LF readings, mean  $\pm$  standard deviation (n=6). (B): Effect of LF tip-pigment distance on readings measured from protoporphyrin pigment suspensions. Squares represent mean LF reading  $\pm$  standard deviation (n=3) using a pigment concentration just sufficient to elicit a mean LF reading of 95.4 (maximum LF reading, 99) when the distance between the instrument's tip and the pigment equals zero. The diamonds represent a single set of LF readings taken using a suspension containing a higher pigment concentration.

readings assessed. LF measurements were taken through both the intact and demineralized areas of the tooth slices (Figure 1). The mean thickness of the 15 enamel and 15 dentin slices used in this study was  $0.61 \pm 0.04$  mm and  $0.6 \pm 0.03$  mm, respectively. A two-tailed t-test indicated that the enamel and dentin slices did not differ significantly in terms of thickness (p < 0.05).

Placement of either dentin or enamel slices between the LF tip and the porphyrin pigment significantly reduced the amplitude of the instrument's reading compared with readings obtained without intervening tooth tissue (Figure 3). The mean reading through intact enamel was  $28.4 \pm 2.8$ , a significant 45.1% decrease compared with the readings obtained without enamel. The mean LF

## Effect of enamel and dentin slices on DIAGNOdent readings

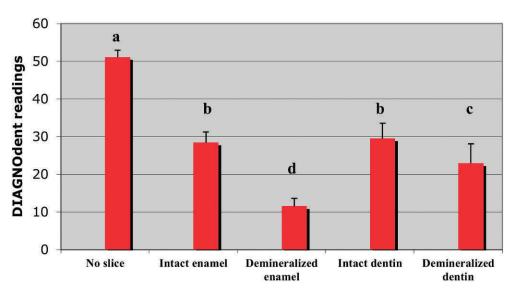


Figure 3. Effect of tooth structure on LF readings (mean  $\pm$  standard deviation) evoked by a porphyrin pigment suspension. Column on the left represents readings obtained with a 0.75 mm gap between the LF tip and the pigment (n=30). The remaining measurements were obtained when  $\approx$ 0.6 mm-thick slice of tooth tissue was placed into this gap and the readings repeated. LF readings, from left to right (starting with the second bar), taken through intact enamel (n=15), demineralized enamel (n=15), intact dentin (n=15), and demineralized dentin (n=15). Groups that had the same letter above the bar had LF readings that were not significantly different. Both enamel and dentin significantly attenuated the LF signal strength; demineralized enamel caused the most attenuation.

reading through demineralized enamel was 11.5 ± 2.1, corresponding to a 77.5% decrease. Demineralized enamel induced a significantly greater reduction in LF signal strength than did intact enamel. The mean LF reading obtained through intact dentin was  $29.5 \pm 4.1$ , a significant 42.2% reduction compared with the value for no tooth structure. The readings obtained through intact dentin were not significantly different from the readings obtained through intact enamel but were significantly higher than the reading measured when the laser passed through demineralized enamel. The mean LF reading through demineralized dentin was 22.9 ± 5.2, a significant 55.1% reduction compared with the measurements made with no tooth structure between the instrument's tip and the pigment. Though demineralized dentin attenuated the LF signal more than intact dentin or intact enamel, the readings obtained through demineralized dentin were significantly higher than those measured through demineralized enamel, indicating that demineralized dentin attenuated the LF signal less than did demineralized enamel.

The enamel and dentin specimens used in this study did not generate significant LF readings. Measurements of LF readings were taken from both

the intact and demineralized sides of these enamel and dentin slices. Intact and demineralized enamel induced a mean LF reading of 1 and 1.3, respectively (N=3). In the case of dentin, both intact dentin and demineralized areas induced a mean reading of 3.3 (N=3).

#### Comparison Between LF Readings Obtained From the Occlusal Surface With Those Taken Below the Surface

Photos of the occlusal surfaces of two extracted molars with dark areas on the occlusal fissures indicative of caries are shown in Figures 4a,d along with LF readings obtained from the areas over which the numbers are superimposed. Figures 4b,e present photographs and LF readings of the same teeth taken after the removal of the occlusal surface. White discoloration of the enamel lining the occlusal fissures is clearly visible, as are dark areas in the dentin adjacent to or at the base of the fissures. LF readings taken from these cut surfaces were higher than those measured at corresponding sites on the tooth surfaces. The surfaces shown in Figure 4c,f are from deeper sections. These reveal dark dentin having high LF readings. Twelve teeth with discolored occlusal fissures were evaluated by this proce-

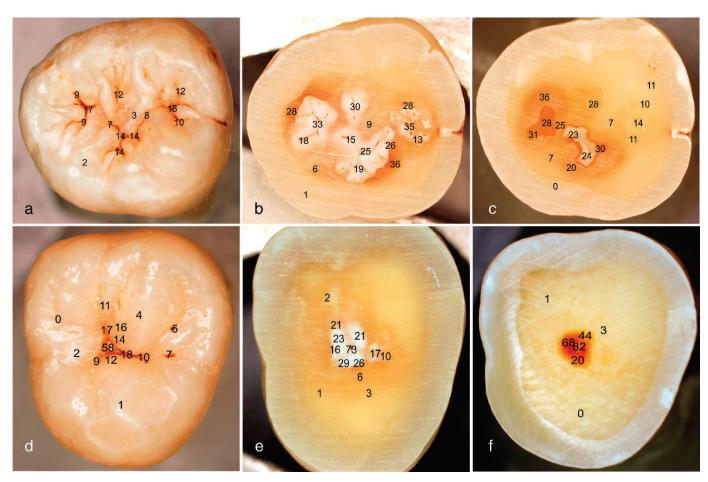


Figure 4. Photographs and LF readings of two molars having small occlusal caries. (A) and (D): Surface views of these teeth and LF readings obtained from occlusal sites. (B) and (E): photo and LF readings taken from these teeth following removal of the occlusal surface. Notice the areas of white-demineralized enamel lining the fissures and adjacent areas of stained dentin. LF readings from the cut surface are generally higher than those obtained from the original surface. (C) and (F): photograph and LF readings obtained following a second cut performed on the same two teeth. Notice the dark dentin area below the fissures with high LF readings.

dure. A total of 31 occlusal sites were measured with the LF before and after removal of the occlusal surface. The mean LF reading obtained from the surface was  $26.3 \pm 12.8$ . Following the removal of the occlusal surface the mean LF reading was  $58.9 \pm 26.5$ ; this value is significantly higher than the readings obtained from the intact surfaces (Table 1).

#### **DISCUSSION**

The basic premise behind the use of caries detectors is to enable clinicians to diagnose lesions that are not apparent with conventional examination methods. It is also desirable to quantitatively assess and record the severity of lesions so that changes over time can be monitored. The field of caries management is evolving rapidly. Nonsurgical treatment of early lesions along with caries management by risk reduction is considered appropriate care. <sup>18</sup> These trends do not mitigate the need for the accurate

diagnosis of occlusal caries. Detection of early lesions can help identify individuals and teeth at risk and in need of preventive intervention. Since its introduction, the DIAGNOdent has been subjected to

Table 1: Mean and Standard Deviation of LF Readings
Obtained From 31 Occlusal Sites on 12
Extracted Molars Obtained Before and After
Removal of the Superficial Portion of the
Occlusal Surface. All 12 Teeth Had Visual Signs
of Occlusal Caries.

Surface	Mean ± Standard Deviation
Occlusal surface	26.3 ± 12.8
Cut surface	58.9 ± 26.5*
*Difference significant, p<0.05.	

several investigations aimed at assessing its utility as a means of improving the diagnosis of occlusal caries. These studies examined the reproducibility of LF readings as well as diagnostic agreement with other methods. In general these studies support use of the LF as an adjunctive diagnostic method. <sup>14,20</sup> The purpose of this study was to examine the effect of anatomic factors on LF readings.

The LF device does not detect hard tissue demineralization directly but rather senses the presence of bacterially generated fluorescent pigments. <sup>10,11</sup> Noncarious stains, plaque, and pigmented material from prophylaxis pastes are sources of false positive readings influencing this instrument. <sup>20</sup> In contrast, clinically relevant factors such as the position of the pigmented part of the lesion with respect to the location of the LF-probe tip, factors that may reduce the LF signal strength, have not been investigated extensively.

In some of these experiments we used a porphyrin in oil suspension as a LF signal source in order to reproduce the LF signal generated by carious dentin. As has been observed previously, 10 we observed that the magnitude of the LF readings are related to the porphyrin pigment concentration. The maximum reading that is obtainable with the LF device is 99. At high pigment concentrations the instrument's response saturates and can increase no further. In clinical use, very dark lesions may possess quantities of pigment that saturate the instrument's response. We observed that the LF reading declines as the distance between the instrument's tip and the fluorescent pigment increased. We assessed this using a pigment concentration (12.5 mg/mL), below that which saturated the instrument's response. With this pigment concentration, a distance of 0.6 mm resulted in a 50% reduction in the LF signal strength compared with the signal measured with a distance of zero. Using pigment concentrations above those that saturate the instruments response, the effect of small separations between the LF tip and pigment are not seen. In experiments examining the effect of tooth slices on LF readings a pigment concentration of 25 mg/mL was used. This concentration was sufficient to induce a high enough LF reading to allow the attenuating effects of enamel and dentin to be assessed.

Due to the attenuating effect of distance, the LF device may underestimate lesions that exist deep in the pit and fissure system. These results underscore the need to correctly place the instrument tip during clinical use.

Tooth structure was also found to have an attenuating effect on the LF readings measured from porphyrin pigment dispersions. These experiments used slices of enamel and dentin placed between the porphyrin pigment and LF tip. Measurements were made before and after the enamel and dentin slices were demineralized using a pH 4.5 lactic acid gel. When performing this procedure, each slice was exposed to the acid on both its inner and outer flat surfaces. Care was taken to ensure that measurements were taken from the same area in both instances. Demineralized enamel attenuated the LF signal more than intact enamel, intact dentin, or demineralized dentin. This tissue has a white opaque appearance indicating a tendency to scatter light. This light scattering is the likely explanation for the reduced LF signal. The ability of dentin slices to partially block LF signal has been reported previously. 17 Next to demineralized enamel, demineralized dentin was the second most LF attenuating. The dentin specimens were cut in such a way that the LF's light path would be parallel to the direction of the dentinal tubules in an area corresponding to the dentin located below the occlusal fissures. The dentinal tubules scatter light.<sup>21</sup> This light scattering is probably responsible for the reduced LF signal strength. In dentin, lactic acid treatment results in the removal of the smear layer, demineralization of the intertubular dentin, and widening of the tubules. When exposed to caries acids, intratubular mineral precipitation occurs below the surface. 22 Although the appearance of dentin was not markedly changed by acid treatment, these results indicate that demineralization alters the optical properties of dentin, resulting in a greater attenuation of the LF signal. In contrast to demineralized tooth tissue, intact enamel and dentin attenuated the LF signal strength the least.

As seen in Figure 4, both intact and demineralized enamel line the fissures and may stand between the LF tip and pigmented dentin. Based on these features of occlusal anatomy, the observations concerning the LF signal—attenuating properties of intact and demineralized enamel are judged to be clinically relevant. The path taken by laser entering or fluorescent emissions exiting the carious lesions may also pass through dentin, depending on the instrument's position and the anatomy of the pit and fissure system. <sup>23</sup> In our observations of sectioned teeth with small occlusal lesions, it was noted that affected dentin is generally pigmented. Thus, the acid-demineralized dentin examined in this study may not represent a clinically relevant tooth tissue

type as far as its effect on caries detection is concerned.

The effect of distance and intervening tooth tissue on the LF device's performance was further evaluated in experiments where readings were recorded from small lesions before and after removal of the tooth's occlusal surface. Cutting revealed sites of enamel demineralization and dentin staining in teeth that appeared grossly intact. These subsurface areas were observed to be soft to tactile examination (performed after LF examination). Removal of the occlusal surface resulted in a significant increase in the LF readings obtained from 31 sites containing discolorations suggestive of early caries. Compared with readings obtained from the surface, measurements made after removal of the occlusal surface were higher even when the dentin was still covered by white-demineralized enamel, indicating that the attenuating properties of both distance and tooth structure are important in influencing the LF's readings.

The challenge for any caries detection device is to aid clinicians in the detection of lesions that are not apparent with conventional examination alone. The effect of lesion-detector distance and intervening tooth structure should be investigated when assessing the performance of technologies intended for use in occlusal caries detection. It has been noted that teeth stored in chemical preservatives lose LF signal strength with time.<sup>24</sup> Although the effect of phenol, the preservative used in this study was not assessed, the use of the nonoxidizing preservatives examined caused an approximately 30% reduction in LF signal strength over short time spans.<sup>24</sup> In this study the subsurface LF readings were 55.3% higher than the surface readings. The effect of the tooth storage conditions may have influenced the results but cannot account for the differences between the surface and subsurface readings.

The presence of factors that cause false positive LF readings as well as the factors described here that lead to underrating lesions indicate that LF readings must be interpreted with caution, and these readings should not be the exclusive basis for diagnosis. A further limitation is the inability of the LF device to distinguish between active and arrested caries. Although this study indicates that the occlusal anatomy hinders the ability of the LF to detect caries, we are not suggesting that clinicians use a low reading threshold when using LF in restorative treatment planning and decision making. When used in clinical practice, correlation between LF

readings and other diagnostic findings are critical to therapeutic decision making.

This instrument may be a useful tool in the longitudinal monitoring of occlusal lesions. Increases over time in a site's LF reading may be indicative of lesion progression because the distance and amount of intact tooth structure standing between the carious dentin and the instrument's tip would decline as the lesion expands and as the enamel walls of the fissure fail.

Many contemporary systems of caries detection seek to identify signs of caries that exist before cavity formation takes place, the goal being the institution of preventive measures aimed at preventing destruction of tooth structure. On occlusal surfaces, LF and/or other technologies can be used to identify suspicious areas in need of sealants or monitoring with other preventive measures. It is hoped that this and other caries detection devices will be used in diagnostic protocols aimed at identifying patients and teeth at risk<sup>25</sup> and not just as a means of justifying operative intervention.

#### **CONCLUSIONS**

The DIAGNOdent and other caries detector devices are supposed to aid in the identification of occlusal lesions that are difficult to diagnose by visual inspection. LF detects bacterial pigments that are deposited within carious tooth structure. This study examined the effect of distance and interposed tooth tissue on the ability of the LF to detect caries. Distance and interposed tooth structure interfered with the detecting ability of the LF device. These results indicate that when lesions occur deep inside the pits and fissures, the distance between the lesion and the LF tip as well as intervening tooth structure hinders the ability of this instrument to detect caries. In clinical use, low LF readings do not allow the presence of caries deep in the fissures to be ruled out.

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# Efficacy of Two Different CHX-Containing Desensitizers: A Controlled Double-Blind Study

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#### Clinical Relevance

CHX-containing desensitizers are used for treatment of hypersensitive teeth. This positive effect shows a durability of 3-month.

#### **SUMMARY**

The aim of this study was to compare the effectiveness and duration of action of the

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tooth desensitization agent Cervitec (C) vs that of the new Cervitec Plus (C+). In this monocentric, single-center, three-armed, controlled, double-blind study, 120 subjects were randomly assigned to one of three groups: group I received Cervitec Plus (C+), group II received Cervitec (C), and group III received placebo (P). Varnishes were applied after baseline determination of cervical dentin hypersensitivity using a pain score of one or higher. Re-evaluation was performed 1, 7, 30, and 90 days after application. Statistical evaluation was carried out using nonparametric statistics for relative effects and analysis of variance (ANOVA). Thirty days after application of C and C+, all hypersensitivity decreased significantly in relation to baseline measurements (p<0.001), with no changes taking place in the placebo group. Significant differences were observed between C and C+ vs placebo (p<0.001), whereas no significant difference between C and C+ was seen after 30 days (p=0.840). After 90 days, the reduction in

hypersensitivity with C+ was still significant compared with baseline measurements (p=0.001). However, C was not significantly different compared with baseline measurements (p=0.05). Analysis of all hypersensitive posterior teeth examined showed no significant difference between C and C+ after 90 days (p=0.362). For anterior teeth, the difference between C and C+ was significant (p=0.012). Both C and C+ reduce cervical tooth hypersensitivity, whereas C+ reduces hypersensitivity for a longer period of time.

#### INTRODUCTION

Dentin hypersensitivity affects up to 98% of the population. <sup>1–13</sup> Wide variation in hypersensitivity is due to the use of different diagnostic methods, different study setups in clinical studies, and different clinical situations.

Hypersensitivity may occur at exposed root surfaces or under restorations, and is characterized by transient pain in response to evaporative, tactile, thermal, or osmotic stimulation of exposed dentin.

The most common theory for the origin of dentin hypersensitivity is the Brannstrom hydrodynamic theory of dental pain. <sup>14</sup> It proposes that any stimulant that can cause fluid movement within the dentinal tubules can also stimulate nerve fibers and elicit a painful response. Hot or cold stimulation can cause expansion or contraction of the dentinal fluid, thereby initiating pain. An air stimulus applied to the dentin surface will desiccate or evaporate the dentin fluids with an immediate outward shift of these, which also causes pain.

Dentin hypersensitivity can result from enamel removal caused by attrition, parafunctional habits, tooth brushing, abrasion, erosion by acids, coronal fracture, defective restorations, gingival recession, or periodontal disease. A natural mechanism for reducing hypersensitivity is the adhesion of salivary proteins to the outer dentin surface, and of plasma proteins to the inner dentin surface, thereby blocking the dentin tubules. <sup>15</sup> Another form of natural protection given to the hypersensitive tooth is the production of tertiary dentin. <sup>16</sup> It is possible for the smear layer on the tooth to penetrate into the dentinal tubules and so block them, preventing the occurrence of hypersensitivity. <sup>17</sup>

In accordance with the Brannstrom hydrodynamic theory, one way of treating hypersensitivity is to seal the dentin tubules or to reduce or eliminate bacterial infiltration, which, in turn, will reduce dentin permeability and fluid flow. The natural way to reduce hypersensitivity is sclerosis. Over a period of time, minerals are deposited, resulting in a thicker layer of peritubular dentin and eventually bringing about closing up of the dentin tubules, thereby reducing sensitivity. If natural sclerosis does not occur, various other treatment methods are available. Two treatment modalities are used in the treatment of hypersensitivity: producing an alteration in fluid flow in the tubules, and blocking the pulp nerve response. To occlude the tubules and stop fluid flow, barriers can be erected by the application of toothpaste constituents, varnishes, dentin-bonding agents, composite resins, glass ionomer cements, and compomers that contain fluoride, strontium chloride, or oxalates. 18 These items often are used as components of various toothpastes<sup>19</sup> or are applied locally.20

However, a limited amount of data regarding the efficacy of desensitizers is available in the literature. Therefore, this study examined for the first time the effect of the chlorhexidine (CHX)-containing varnish Cervitec Plus on dentin hypersensitivity.

In the present study, the varnishes Cervitec and Cervitec Plus were used. The aim was to evaluate and compare the effectiveness of the two desensitizing agents Cervitec (C) and Cervitec Plus (C+). Long-term stability over a period of three months was also investigated, to assess whether one of the agents would show better long-term stability. The two desensitizing varnishes were compared with a placebo (P).

#### **MATERIALS AND METHODS**

#### **Products Used in the Study**

This monocentric, single-center, randomized, threearmed, parallel clinical study evaluated the efficacy of two different desensitizing varnishes.

The study was reviewed and approved by the Ethics Committee of the University of Goettingen (No. 5/9/06 from 19.09.2006).

Two desensitizing agents were used for the treatment of hypersensitivity: Cervitec Plus and Cervitec (both from Ivoclar Vivadent, Schaan, Liechtenstein). The third agent used was a placebo supplement that contained only water and ethanol (Table 1).

## Desensitizing Agents (Cervitec, Cervitec Plus, Placebo)

Both Cervitec and Cervitec Plus are protective agents designed to treat exposed root surfaces. They

Table 1: Composition of M.	able 1: Composition of Materials Used in the Study				
Function	Ingredients	Composition			
		Cervitec Plus	Cervitec	Placebo	
Solvent	Ethanol, water, ethyl acetate ethanol, water	-91%	88%-	-100%	
Varnish-building ingredients	(Poly)vinylbutyral (poly)vinylacetate copolymer	-7%	10%-	_	
Antimicrobial Thymol chlorhexidine diacetate-hydrate		1%/1%	1%/1%	_	

have an antimicrobial effect, which reduces bacterial plaque activity. Constituents of Cervitec and Cervitec Plus are shown in Table 1. Cervitec Plus does not contain the solvent ethyl acetate, which is replaced by an ethanol-water mixture. The concentration and origin of thymol and chlorhexidine are the same as in the Cervitec varnish. Therefore, indications and contraindications do not differ from those for Cervitec.

The placebo did not differ in smell or color from the desensitizing agents. The purpose of the placebo was conventional; it served only to "blind" the treating dentist and patients taking part in the study.

#### **Subjects**

One hundred twenty healthy volunteers with good oral hygiene (Quigley-Hein-Index <1) were included in this study. Only patients with restored and/or caries-free teeth showing cervical hypersensitivity were accepted. Patients with infectious disease, a high risk of endocarditis, or allergic reactions against components of the varnishes, as well as addicted patients, patients with epilepsy, and renal failure or immune-suppressed subjects, were excluded, according to the regulations of the Ethics Committee. Genders, ages, and smoking habits of the subject population are documented in Table 2.

All 120 subjects were allocated randomly to one of three groups (group I: Cervitec Plus; group II: Cervitec; and group III: placebo) of 40 subjects. All hypersensitive teeth from each patient were included in the study and were treated with one of the three varnishes.

Subjects were requested not to use any other desensitizing agents throughout the period of the study. Each subject was supplied with a toothbrush (Hager & Werken GmbH, Duisburg, Germany) and toothpaste (Elmex, Gaba GmbH, Lörrach, Germany)

to ensure standardized oral hygiene procedures for the period of the study.

#### **Evaluation of Tooth Hypersensitivity**

Investigation of hypersensitivity was performed at baseline, to determine the initial state of hypersensitivity, then at 1 day, 7 days, 30 days, and 90 days after application of the varnish. To define hypersensitivity, a gentle stream of air was applied to hypersensitive teeth with an air-blower (1 second) with the nozzle at a distance of 2 mm from the tooth. Hypersensitivity was graded on a scale from 0 to 4 (0 = no sensitivity, 4 = high sensitivity). 12

- Level 0: no sensitivity.
- Level 1: low sensitivity.

Table 2:

- Level 2: tolerable discomfort and/or pain after stimulation.
- Level 3: high sensitivity and/or pain during and up until 5 seconds after stimulation.
- Level 4: very high sensitivity and/or pain for 5 seconds and longer after stimulation.

Age, Gender, and Smoking Characteristics of

Subjects

Group I Group II Group III Cervitec Plus Cervitec Placebo

Number of subjects (n=120)

Render Female n=16 n=18 n=17

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Gender	Female	n=16	n=18	n=17
	Male	n=24	n=22	n=23
Age		35.4 ± 8.8	34.9 ± 8.1	36.0 ± 6.0
Smoker		n=17	n=16	n=20

#### Study Design

Before patients were recruited to the study, a preliminary oral examination was carried out and the medical history was taken, to assess the patient's general health condition and to exclude the presence or influence of other diseases. Oral examination consisted of inspection of the oral cavity and gingiva, as well as a dental examination (number of decayed, missing, or filled teeth [DMF-T]). Also, baseline hypersensitivity of all of the patients' teeth was evaluated.

Patients were randomly allocated to one of three groups (group I: Cervitec Plus; group II: Cervitec; group III: placebo).

Two observers were appointed for the study. All subjects were examined under standardized conditions by two calibrated dentists (kappa value > 0.8). Observer I performed the preliminary oral examination. Seven days after this, desensitizing varnish or placebo was applied to the buccal surface of each tooth showing hypersensitivity with a value of 1 or more by observer II (wisdom teeth were excluded). Neither observer I nor the patient knew which desensitizing varnish had been applied to the teeth. The teeth were dried off with a cotton ball and air, and varnish was applied with a dental brush (GlaxoSmithKline, Buehl, Germany) for 30 seconds, to allow penetration of varnish into the dentinal tubules. Patients were advised not to eat anything for three hours and not to brush their teeth on the day of application.

Evaluation of hypersensitivity and examination of the oral cavity were performed by observer I, as described previously, on day 1, day 7, day 30, and 90 days after application of the desensitizer. Group III was not re-examined 90 days after application. In group III, Cervitec was applied without re-evaluation after 30 days, for ethical reasons.

The study design is outlined in Table 3.

#### **Statistical Analysis**

Relative effects were used to compare the results of different treatments. The relative effect is a non-parametric comparative measure based on ranked data. It ranges between 0 and 1; the higher the value, the better is the effect. For computation of relative effects and confidence intervals, we used the SAS macro F1\_LD\_F1 (SAS Institute Inc., Cary, NC, USA). For testing of significance, ANOVA was used. For pair-wise comparisons of the treatment groups, no adjustment for multiple comparisons was necessary because of the closed testing procedure. To

adjust for post hoc comparisons of anterior and posterior teeth, the Bonferroni method was used.

#### **RESULTS**

In the course of the study, eight participants dropped out (group I: 4; group II: 1; group III: 3). These patients failed to turn up for their appointments.

Evaluation of group results revealed that for group I (C+), a significant reduction in hypersensitivity was noted after seven days compared with baseline (p=0.001). One day after application, no significant change was observed (p=0.05). At day 30 and day 90 after application, significant changes in hypersensitivity were still evident compared with baseline (p=0.001). In group II (C), one day after application of the varnish, no significant reduction in hypersensitivity was observed (p=0.05), whereas 7 and 30 days after application, hypersensitivity was reduced significantly (p=0.001). On day 90, hypersensitivity was not reduced compared with baseline (p=0.05). Group III (P) at no time showed any significant change compared with baseline: no reduction in hypersensitivity was noted.

Comparison of Cervitec Plus and Cervitec revealed no significant difference on day 1, at day 7, and at day 30 (day 1: p=0.8784; day 7: p=0.2724; day 30: p=0.8630). Ninety days after application of the varnishes, a significant difference in the reduction in hypersensitivity was observed (p=0.0001). When Cervitec Plus was compared with placebo, no significant difference was established after one day (p=0.2177). Seven and 30 days after application, we observed a significant difference between Cervitec Plus and placebo (p=0.0001). Cervitec and placebo showed no significance on day 1 (p=0.2309), but a significant difference in the reduction in hypersensitivity could be seen on day 30 (p=0.0001). Changes in intensity within the three groups after 30 days and 90 days are illustrated in Figures 1 and 2.

No significant difference after 30 days was observed between Cervitec Plus and Cervitec when anterior and posterior teeth were compared (p=1.0). When Cervitec Plus was compared with placebo, and Cervitec with placebo, significant differences between anterior teeth (groups I and III: p=0.001; groups II and III: p=0.001) and posterior teeth were found (groups I and III: p=0.006; groups II and III: p=0.002). Comparisons of effectiveness between anterior and posterior teeth at baseline and 90 days post application revealed a significant difference between Cervitec Plus and Cervitec for anterior teeth (p=0.012) (Figure 3). However, no significant

#### Table 3: Flow chart of the study

- Screening of subjects, including oral examination and medical history (observer 1)
- Evaluation of hypersensitivities (observer 1) as described before
- Stratified randomisation of subjects into 3 groups (each 40 subjects) by lot (observer 2):

Group I: Cervitec Plus

Group II: Cervitec

Group III: Placebo

• Application of desensitizing varnishes (observer 2)

• Re-evaluation (observer 1): - of hypersensitivities

- oral examination

1 day 7 days 30 days 90 days after application after application after application

• statistical analysis

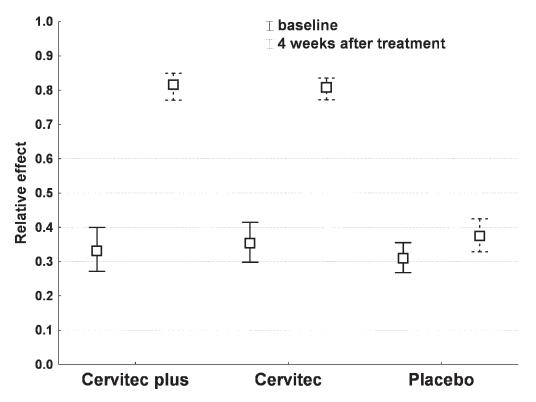


Figure 1. Changes of hypersensitivity 30 days after application of the varnish.

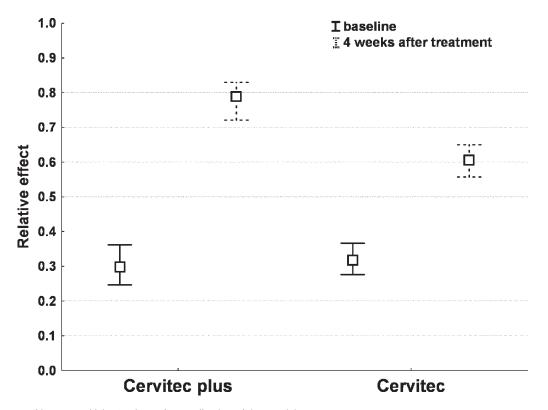


Figure 2. Changes of hypersensitivity 90 days after application of the varnish.

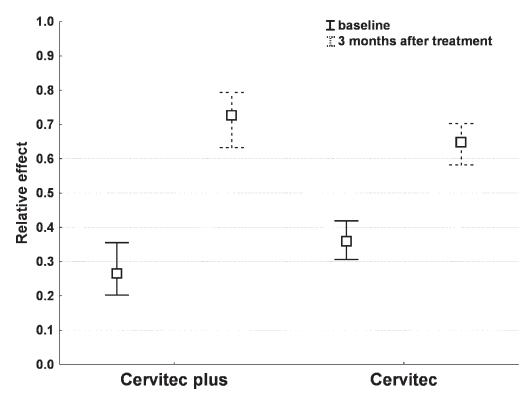


Figure 3. Effectiveness on anterior teeth at baseline and 90 days post application.

difference between Cervitec Plus and Cervitec was detected for posterior teeth (p=0.362) (Figure 4).

#### **DISCUSSION**

The aim of this study was to compare and evaluate the effectiveness and long-term stability of the two desensitizing agents Cervitec and Cervitec Plus against a placebo control.

Participants were requested not to use any additional medication for dentin hypersensitivity, such as fluoride rinsing solutions or special fluoride-containing toothpastes, as these could influence the results. Hypersensitivity was assessed using a gentle stream of air, by applying a method of assessment and grading that was used in a previous study.<sup>22</sup>

It was shown that Cervitec releases both CHX and thymol; at first the release is more rapid, later it slows down. <sup>23</sup> Combining both agents, CHX and thymol, showed a positive synergistic effect. Cervitec Plus contains the same amounts of CHX and thymol. Cervitec and Cervitec Plus reduce the hydraulic permeability of dentin, and this could explain the desensitizing effect. Furthermore, the adhesion of Cervitec Plus varnish is superior to that of Cervitec. This may explain the longer duration of the reduction in hypersensitivity of Cervitec Plus. From

a chemical point of view, the varnish polymer of Cervitec Plus is less hydrophobic than that of Cervitec. This allowed the omission of ethyl acetate from the formulation. Both the more hydrophilic solvent mixture and the more hydrophilic varnish polymer of Cevitec Plus increased moisture tolerance during application. This probably produced improved adhesion to the tooth structure with Cervitec Plus as compared with Cervitec.

Cervitec and Cervitec Plus reduce hypersensitivity equally for a certain period of time. The efficacy of Cervitec Plus could still be observed 90 days after application because of better adhesion of the varnish. Ignoring the dentist's instructions (no food for one hour after application, not brushing the teeth on the day of application) might be another reason why, in some cases, application of Cervitec and Cervitec Plus did not reduce hypersensitivity—a fact that can be ignored because it could be found in each group.

Hypersensitivity requires therapy that provides desensitization of hypersensitive dentin, resulting in a reduction in clinical symptoms. The success rate for a material or technique depends on the period of efficacy of the material or method used. Tooth hypersensitivity can be approached by decreasing the hydrodynamics of dentinal fluid or by decreasing the sensibility of tooth nerves. There is also the

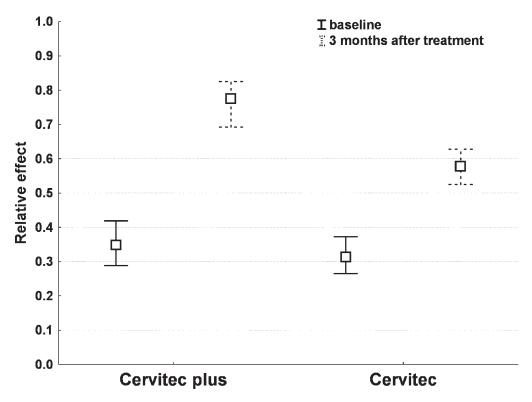


Figure 4. Effectiveness on posterior teeth at baseline and 90 days post application.

possibility of spontaneous desensitization; while odontoblasts create a barrier of reparative dentin, spontaneous remission of hypersensitivities in as many as 95% of all patients has been shown.<sup>24</sup>

To the best of our knowledge, up until now, no published studies have analyzed the effectiveness of Cervitec Plus, as we have done. However, several investigations of Cervitec have been published. A caries-protective effect was found in several clinical studies. <sup>25–28</sup> Compared with other CHX varnishes (EC40, Chlorzoin), <sup>29–32</sup> Cervitec produced better protection against caries. Caries protection has been demonstrated for fissures of the tooth <sup>33</sup> and the approximal region of teeth. <sup>34</sup> In addition to the property of providing protection against caries, Cervitec has antibacterial action and brings about a reduction in the quantity of streptococcus mutans. <sup>35–37</sup>

Many treatment options are available for managing dentin hypersensitivity. The nerve can be desensitized, or exposed dentin tubules can be covered, most frequently by using the topical application of an agent that does not irritate the pulp, is painless, and is easy to apply. It should act rapidly, should be permanently effective, and should not discolor the teeth. Nerve desensitization techniques most often make use of potassium. Tarbet

and others  $^{39,40}$  demonstrated that 5% potassium nitrate in a toothpaste was able to desensitize dentin for up to four weeks. Potassium is also available as a bioadhesive gel (5% and 10%); this has been shown to be effective.  $^{41}$  Potassium nitrate does not induce any changes in the pulp.  $^{42}$ 

One treatment option is the application of varnish to the dentin surface to seal the dentin tubules. In the study presented here, Cervitec, Cervitec Plus, and a placebo were used.

Cervitec Plus represents a newly developed modification of Cervitec. It does not contain ethyl acetate; this has been replaced by ethanol and water. This modification in the composition of the varnish provides better adhesion and desensitization. Concentrations of thymol and CHX have not been changed. The third agent, a placebo compound, has been used in several other studies. 43-45 In these studies, the placebo effect was found to be stronger than in the present study, in which only a mild effect was found. One possible explanation is that only one desensitizing agent was used for each patient. If no placebo effect was detectable with one tooth, it was possible that there was no effect on any other teeth in a particular patient. Because no effect on hypersensitivity occurred when placebo was used, no further effect was expected. Accordingly, we decided to apply Cervitec to the teeth after 30 days.

Apart from Cervitec and Cervitec Plus, several other varnishes have been studied. Panduric investigated the effectiveness of adhesives in reducing hypersensitivity. He compared the effectiveness of All Bond 2, Syntac Single Component, and One Step. Cervitec was used as a control. This study demonstrated that dentin adhesives can be used in the symptomatic therapy of dentin hypersensitivity. Syntac Single Component and fifth-generation One Step have much higher efficacy rates than fourthgeneration dentin adhesives and Cervitec. When dentin adhesives are used, efficacy decreases with time. 46 Another study investigated the one-bottle bonding agent One Step and glutaraldehyde-based HEMA over a period of nine months. Both produced a reduction in hypersensitivity for up to nine months. No significant differences were found between One Step and the Gluma Desensitizer.<sup>47</sup> Another study showed that strontium acetate and fluoride are significantly more effective than products containing strontium chloride or KCl. 48 Possible effects of the constituents of toothpaste in reducing hypersensitivity have also been investigated. An in vitro study measured the effects of toothpastes. The granular deposits are composed of abrasive components in the toothpastes and so have the potential to affect hydrodynamic mechanisms through partial or complete obturation of dentin tubules. 49

In the study presented here, agents were applied following a dental examination and determination of dental hypersensitivity. Only patients with caries-free teeth were included in this study, to exclude hypersensitivity arising from caries lesions. The diagnosis of hypersensitivity requires an appropriate differential diagnosis, because caries and dentin hypersensitivity can produce similar symptoms. <sup>50</sup>

The study presented here investigated the effects of Cervitec and Cervitec Plus on hypersensitivity. Previous studies demonstrated a caries-preventing effect and a reduction in plaque adsorption. We were able to show a reduction in hypersensitivity following the application of Cervitec and Cervitec Plus. The newly developed varnish Cervitec Plus even appears to produce higher and more sustained reduction in hypersensitivity than is produced by Cervitec. In addition to investigation of its protective properties in relation to caries, more research is needed on the treatment of dentin hypersensitivity using Cervitec Plus.

#### CONCLUSION

Both Cervitec and Cervitec Plus can substantially reduce tooth hypersensitivity. When Cervitec Plus is used, this effect is sustained for a substantially longer period. The placebo group showed no desensitizing effects.

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## SEM Analysis of Hybrid Layer and Bonding Interface After Chlorhexidine Use

D Lafuente

#### **Clinical Relevance**

More studies suggest that the bond between composite and dentin degrades over time because of the action of matrix metalloproteinase on collagen fibrils left unprotected by acid etching. Clinical actions should be taken to prevent this from happening and thus the failure of what should be a very long-term restoration.

#### SUMMARY

Objective: To evaluate the appearance of the hybrid layer of one total-etch and one self-etch bonding agent on human teeth with and without the application of 2% chlorhexidine after water storage.

Materials and Methods: Twelve human maxillary teeth (molars and premolars) had two Class II cavities (MO-OD) prepared. Teeth were separated in two groups (n=6) to receive either Adper Single Bond 2 or Adper SE Plus (3M ESPE). In one cavity, the dentin donding agent was applied following manufacturer's instructions. On the adjacent cavity, 2% chlorhexidine

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was applied for 30 seconds before the application of the bonding agent. Teeth were sectioned mesiodistally with a slow-speed diamond disk and stored in water at 37°C for four months. The teeth were prepared for scanning electron microscope observation. The appearance of the hybrid layer was observed and measured by two variables: clear image of hybrid layer and presence of resin tags in tubules. Data were analyzed with a Kruskal-Wallis test calculated at a 0.05 significance level.

Results: All groups treated with chlorhexidine had the clear presence of a hybrid layer, whereas only half the specimens without chlorhexidine had a clear hybrid layer. Chlorhexidine did not affect the presence of resin tags.

Conclusions: The use of 2% chlorhexidine before the application of a dentin-bonding agent

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## reduced the deterioration of the hybrid layer when exposed to water.

#### INTRODUCTION

When microtensile testing was introduced, a better study of the failure interface, especially under scanning electron microscopy (SEM) and transmission electron microscopy, 1,2 lead to the discovery that long-term storage in water changed the durability of the bonding interface.<sup>3–6</sup> The resin-dentin interface is a mixture of the adhesive resin with the exposed collagen fibers after the dentin has been demineralized with the use of an etchant. This interface was named the hybrid layer. 7,8 Many factors can affect the clinical longevity of this layer: occlusal forces, thermal changes produced by the different temperatures of foods, chemical agents in beverages, dentinal fluids, bacteria products, elution of resin monomers, and degradation of resin components.  $^{9-20}$ The use of 0.2% chlorhexidine gluconate for 60 seconds was found to inhibit collagenolytic activity,<sup>21</sup> thus maintaining the resin-dentin interface. Pashley and others<sup>21</sup> recommended the use of chlorhexidine on acid-etched dentin before using total-etch adhesives. It did not affect the in vitro bond strength of aged specimens tested in microtensile testing, and there were less cohesive failures in dentin or in the hybrid layer when dentin was treated with chlorhexidine than without such application.<sup>22</sup> The purpose of this study is to evaluate the appearance of the hybrid layer of teeth treated with and without 2% chlorhexidine after aging for four months (125 days) in water.

#### **MATERIALS AND METHODS**

MO-OD Class II cavities were prepared on 12 maxillary teeth, six molars, and six premolars, using a 558 carbide bur, leaving a 1 mm-thick enamel wall to separate the mesial cavity from the distal cavity. Cavities were prepared to a depth of 1 mm below the dentin-enamel junction with no axial wall but elimination of the proximal enamel ridge on both sides (Figure 1). Teeth were then separated into two groups, one to receive Adper Single Bond 2 (3M ESPE, St. Paul, MN) and the other to receive Adper SE Plus (3M ESPE).

#### **Adper Single Bond 2**

The mesial cavity of each tooth was etched with 37% phosphoric acid for 15 seconds and rinsed thoroughly for 30 seconds. Excess water was eliminated. Two consecutive coats of Adper Single Bond 2 (SB2) were rubbed over the dentin surface for 10 seconds, air

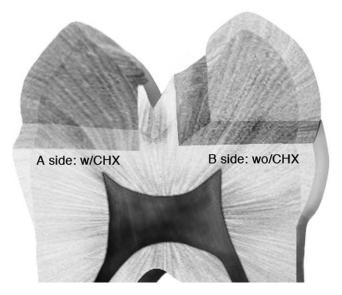


Figure 1. Diagram for the cavity preparation. The distal cavity of all specimens was treated with chlorhexidine before the bonding agent was applied.

thinned, and light cured for 20 seconds (Elipar 2500, 3M ESPE). A third coat of adhesive was applied over the dentin surface, air thinned, and light cured for another 20 seconds. A resin composite (Filtek Supreme XT, shade A2, 3M ESPE) was applied in three increments. The first increment was applied in less than 0.5 mm in thickness to minimize the negative effect of polymerization shrinkage. All increments were light cured for 20 seconds.

The distal cavity of each tooth was etched with 37% phosphoric acid for 15 seconds and rinsed thoroughly for 30 seconds. Excess water was eliminated. Two percent chlorhexidine (Consepsis, Ultradent Prods. South Jordan, UT) was applied to the cavity for 30 seconds and the excess eliminated before SB2 and composite were applied as before.

#### **Adper SE Plus**

After finishing the preparation, the mesial cavity was rinsed and excess water eliminated. Adper SE Plus (SEP) Liquid A (3M ESPE) was applied on the dentin surface for 10 seconds until the dentin was completely covered. Liquid B was rubbed over the dentin surface for 10 seconds, air thinned, and light cured for 20 seconds. A second coat of Liquid B was applied on the dentin surface, air thinned, and light cured for another 20 seconds. Resin composite (Filtek Supreme XT, shade A2) was applied in three increments. The first increment was applied in less than 0.5 mm thickness. All increments were light cured for 20 seconds.

Table 1:

SEP

H (corrected)

Ν

p-level

Chlorhexidine

Chlorhexidine

Without chlorhexidine

The distal cavity of each tooth was rinsed and excess water eliminated. Two percent chlorhexidine was applied to the cavity for 30 seconds. Excess chlorhexidine was eliminated before Adper SE Plus and composite were applied as before.

#### Specimen Preparation

All specimens were sectioned mesiodistally into two halves using a low-speed diamond saw. Specimens were placed under water at 37°C for 125 days. Then specimens were prepared for SEM (S-570, Hitachi, Tokyo, Japan) evaluation. All specimens were polished with silicon carbide paper under water, using sequentially 400, 600, 1000, and 2000 grit. The specimens were cleaned with 37% phosphoric acid for five seconds, rinsed in water for 30 seconds, and submerged in 3% NaOCl for five minutes. Then the specimens were placed in 70%, 80%, 90%, and 99% alcohol to eliminate all water present before being desiccated and prepared for SEM observation.

#### SEM Observation

All specimens were observed under 800× magnification, and an assessment of the dentin-bonding agent interface was made by two variables: clear image of hybrid layer on at least 75% of the length of the interface (yes=1, no=0) and presence of resin tags in tubules at least 75% of the length of the interface (present=1, not present=0). Scores were given for each parameter. Pictures of the most representative part of the interface image based on its score were taken for illustration purposes under 1000× magnification.

Nonparametric data were analyzed using a Kruskal-Wallis test calculated at a 0.05 significance level for each variable: quality of the hybrid layer and presence of resin tags.

#### **RESULTS**

The composite-dentin interface was given a score of 0 or 1 by two parameters, as shown in Table 1. Specimens treated with chlorhexidine before the application of the dentin-bonding agent (DBA) showed a higher presence of a hybrid layer in the interface when compared with the same bonding agent for the same tooth that was not treated with chlorhexidine, although no statistical difference was found in any of the variables in this study. Table 2 shows statistical analysis for variable: presence of hybrid layer.

SB2 produced the most uniform hybrid layer of the two bonding agents tested. The hybrid layer was

Variables

Treatment Hybrid layer (yes answer) Resin tags (yes answer)

SB2 Without chlorhexidine 3 3

Specimen Scores on the Evaluation for the Two

6

3

6

6

3

4

Table 2: Kruskal-Wallis Statistical Analysis for the Variable "Hybrid Layer Presence" With Single Bond 2			
		Sample size	Sum of ranks
SB2/- chlorhex	kidine	6	48
SB2/+ chlorhex	kidine	6	30
Н		2.07692	
Degrees of free	edom	1	

3.6

0.14954

12

present in the entire interface but varied in thickness in some areas of the specimen. SEP had two specimens in which, although the hybrid layer was identifiable, it was not as clear as other specimens treated with the same bonding agent or was not as clear as the one produced by SB2. For teeth not treated with chlorhexidine, both bonding agents produced a hybrid layer that was difficult to identify along most of the length of the interface for half the specimens evaluated (Figures 2–5).

Three specimens of SB2 without treatment with chlorhexidine showed no presence of resin tags or were very scarce. This observation was different from the one of SB2 when treated with chlorhexidine before it was applied, where all the specimens showed a very clear presence of resin tags in the

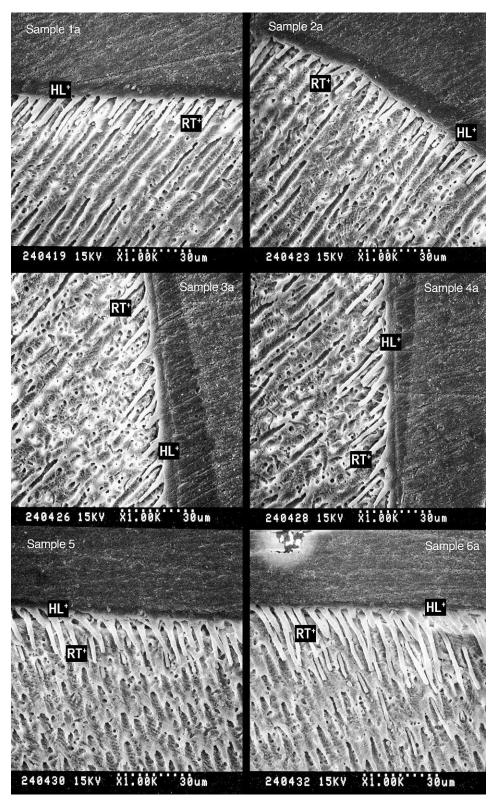


Figure 2. Scanning Electron Microscopy images of composite-dentin interface with Single Bond 2 and 2% chlorhexidine after four months of water storage of all six specimens. HL+, HL- refers to a yes or no score on the presence of a hybrid layer; RT+, RT- refers to a yes or no score on the presence of resin tags.

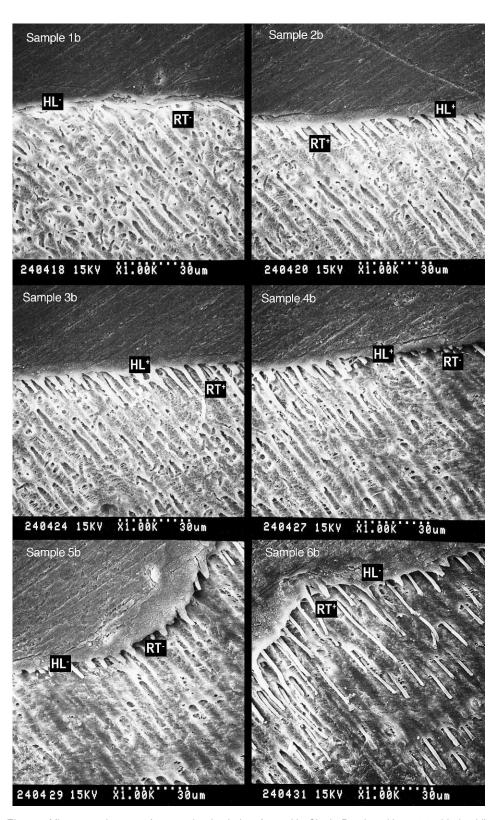


Figure 3. Scanning Electron Microscopy images of composite-dentin interface with: Single Bond 2 without 2% chlorhexidine after four months of water storage of all six specimens in this study.

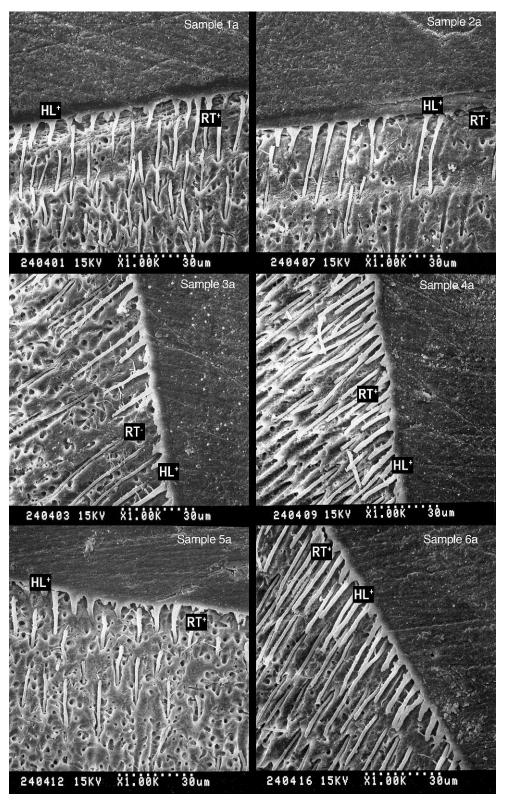


Figure 4. Scanning Electron Microscopy images of composite-dentin interface with SE Plus and 2% chlorhexidine after four months of water storage of all six specimens in this study.

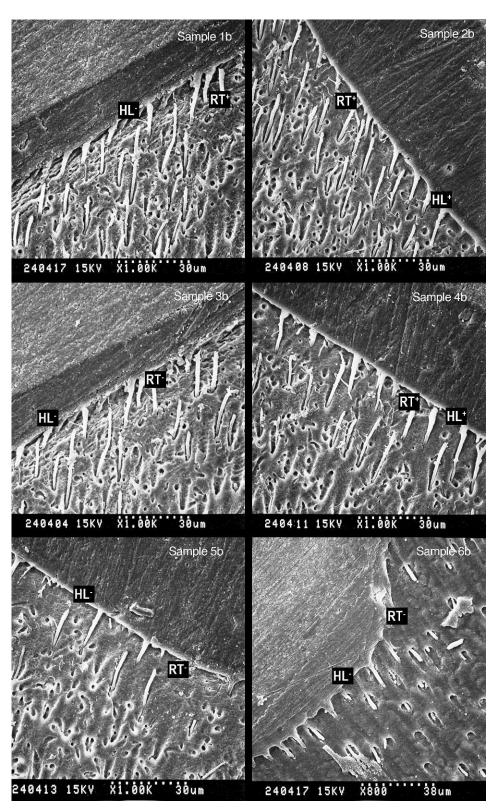


Figure 5. Scanning Electron Microscopy images of composite-dentin interface with SE Plus without 2% chlorhexidine after four months of water storage of all six specimens in this study.

dentin tubules. Regarding SEP, four of the specimens treated with chlorhexidine and three in the group not treated with chlorhexidine showed a clear presence of resin tags.

### **DISCUSSION**

The deterioration of the hybrid layer after water storage is a concept well studied and accepted. 21-23 In this study, it was clear that the effect of water storage on the hybrid layer produced a reduction in thickness. The hybrid layer was difficult to isolate in half the samples that were not pretreated with chlorhexidine. Many authors have found that the use of chlorhexidine did not produce a negative effect on the bond strength of dentin adhesives when it was used before acid etching<sup>24–27</sup> as a cavity disinfectant. Many authors also have proposed that initial bond strength of chlorhexidine-treated specimens were comparable to the control groups, 23,27-29 and some authors have shown that the bond strength of dentin adhesives after water storage is preserved with the use of 2% chlorhexidine. 23,30

The effect of protease inhibition by chlorhexidine suggests that the endogenous metalloproteinases cannot degrade the collagen fibrils left unprotected by acid etching. This effect may explain why there is a more clearly defined hybrid layer along the entire interface in all the specimens treated in this study for both adhesives. Some specimens that were not treated with chlorhexidine showed a poorly defined hybrid layer.

The negative effect of water storage on the hybrid layer can be explained. First, hydrolysis of unstable polymeric hydrogels that are less concentrated occurs as they diffuse in the acid-etched dentin. Second, the unprotected collagen fibers get degraded by the matrix metalloproteinase. 31,32 This can also explain why the effect on resin tags is not as clear as in the hybrid layer. The concentration of polymers in the resin tags is higher, and therefore less hydrolysis can occur. Specimens not treated with chlorhexidine showed a lack of resin tags in some areas of the adhesive-dentin interface, but no statistical difference was found; therefore, one cannot conclude that there could be a positive effect in the use of the chlorhexidine in terms of the presence or the length of resin tags. Resin tag formation and tag length are more dependent on the application technique and the dentin adhesive itself.<sup>33</sup> If a high number of tags can be established on application, damaging effects by water storage can be minimized, and bond strength may not be affected.

This study used a small sample size, though using the same specimens to evaluate the treated and nontreated samples increased the power of the statistical analysis. Replication with a larger sample size is recommended.

### CONCLUSION

The use of 2% chlorhexidine before the application of the DBA reduced the deterioration of the hybrid layer when exposed to water, but there was no statistical effect on its presence at the bonding agent-dentin interface or on the presence of resin tags in the tubules.

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   Abstract #468.

### Influence of Glazed Zirconia on Dual-Cure Luting Agent Bond Strength

TA Valentino • GA Borges • LH Borges

JA Platt • L Correr-Sobrinho

### Clinical Relevance

Treatment of yttrium-stabilized tetragonal zirconia ceramic surfaces with a low-fusing porcelain layer as a glaze significantly increased the bond strength of dual-cure resin cement to the ceramic surface.

### **SUMMARY**

The current study evaluated the influence of a novel surface treatment that uses a low-fusing porcelain glaze for promoting a bond between zirconia-based ceramic and a dual-cure resin luting agent. Bond strengths were compared with those from airborne particle abrasion,

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hydrofluoric acid etching, and silanizationtreated surfaces. Twenty-four yttrium-stabilized tetragonal zirconia (Cercon Smart Ceramics, Degudent, Hanau, Germany) discs were fabricated and received eight surface treatments: group 1: 110 µm aluminum oxide airborne particle abrasion; group 2: 110 μm aluminum oxide airborne particle abrasion and silane; group 3: 50 µm aluminum oxide airborne particle abrasion; group 4: 50 µm aluminum oxide airborne particle abrasion and silane; group 5: glaze and hydrofluoric acid; group 6: glaze, hydrofluoric acid, and silane; group 7: glaze and 50 µm aluminum oxide airborne particle abrasion; and group 8: glaze, 50 µm aluminum oxide airborne particle abrasion and silane. After treatment, Enforce resin cement (Dentsply, Caulk, Milford, DE, USA) was used to fill an iris cut from microbore Tygon tubing that was put on the ceramic surface to create 30 cylinders of resin cement in each treatment group (n=30). Microshear bond testing was performed at a crosshead speed of 0.5 mm/min. One-way analysis of variance, and multiple comparisons were made using Tukey's test (p < 0.5). The bond strength was affected

only by surface treatments other than silanization. The groups that utilized the low-fusing porcelain glaze with airborne particle abrasion or hydrofluoric acid showed bond strength values statistically superior to groups that utilized conventional airborne particle abrasion treatments with 50 or 110  $\mu$ m aluminum oxide (p<0.001). The treatment that utilized low-fusing porcelain glaze and hydrofluoric acid showed bond strength values statistically superior to remaining groups (p<0.001). Treatment of zirconia ceramic surfaces with a glaze of low-fusing porcelain significantly increased the bond strength of a dual-cure resin luting agent to the ceramic surface.

### INTRODUCTION

Unique mechanical properties, chemical stability, and biocompatibility make zirconia-based ceramic an attractive core material for fabrication of all-ceramic restorations. 1 Combined with CAD/CAM technology, the fabrication of complex restorations incorporating zirconia cores has become a completely digitized process and a relatively simple procedure.<sup>2,3</sup> The flexural strength and fracture toughness are considerably higher than those of other dental ceramics,<sup>4</sup> and the zirconia-based ceramic shows a distinct mechanism of stress-induced transformation toughening, meaning that the material undergoes microstructural changes when submitted to stress.4-6 Zirconia-based ceramic can actively resist crack propagation through a transformation from a tetragonal to a monoclinic phase at the tip of a crack, which is accompanied by a 3% to 4% volume increase.<sup>5</sup> Based on these characteristics, zirconia-based ceramic has been used as a prosthetic implant for medical and dental applications, posts, implant abutments, orthodontic brackets, and frameworks for crowns and bridges.<sup>2,7-10</sup>

Long-term durable bond strength to ceramic surfaces is the aim for dental clinical applications and is dependent on the micromechanical and chemical interaction between luting agent and ceramic surface. The retention and the stability of the ceramic restorations are enhanced by the adhesive bond strength, which must be strong enough to resist the expected functional loads<sup>11</sup> and hydrolytic degradation.<sup>12</sup> The luting of a zirconia restoration can be done with zinc phosphate or with modified glass ionomer cements.<sup>13</sup> However, the advantages of resin luting agents, such as marginal seal, good retention, and improvement of fracture resistance, have made them popular for use

even with high-strength ceramics. 13,14 Several studies have investigated the bond strength between resin luting agent and zirconia-based ceramic, and several methods have been proposed to promote a durable chemical and micromechanical bond with zirconia. The conventional treatments for ceramic surfaces, such as oxide airborne particle abrasion and hydrofluoric acid etching, are not able to promote a strong and stable bond with zirconia. 15 Air abrasion might affect the ceramic surface by creating microcracks that could reduce the fracture strength of the ceramic, 16 and the hydrofluoric acid etching combined with silanization, which is used with other glass and disilicate-based ceramics, has not been successful with acid-resistant and glassfree zirconia ceramics. 7,15

In recent years, the literature has shown new treatments aimed at optimizing the bond strength to zirconia-based ceramic. However, chemical bonding to zirconia is limited by the inertness of the ceramic composition and has led to the investigation of various surface-roughening methods, such as silica coating followed by silanation, <sup>17</sup> plasma spraying, <sup>13</sup> airborne particle abrasion combined with the application of phosphate ester monomer (MDP), 8,18 ceramic primers,7 Er:YAG laser,19 a selective infiltration etching technique,7 and heat-induced maturation. 11 All these treatments provide an immediate increase in bond strength. However, the association between the increased bond strength, durability, and clinical performance, as well as the development of a simple surface treatment protocol for clinicians, is still not fully defined and needs more clinical and longitudinal laboratory investigations to elucidate an optimum protocol for zirconia ceramic.

The purpose of this study was to evaluate a novel surface treatment that uses a low-fusing porcelain glaze for promoting a bond between zirconia-based ceramic and a dual-cure resin luting agent, and to analyze the association of this surface treatment combined with conventional airborne particle abrasion, hydrofluoric acid etching and silanization treatments. The null hypothesis was that there would be no difference between dual-cured resin luting agent bond strength to zirconia following conventional ceramic treatments and a novel glaze surface treatment.

### **MATERIALS AND METHODS**

### **Ceramic Surface Treatments**

Twenty-four ceramic discs  $94\%~{\rm ZrO_2}$  stabilized by  $5\%~{\rm Y_2O_3}$  (Cercon Smart Ceramics, Degudent, Ha-

nau, Germany) were fabricated to be 16 mm in diameter and 1 mm thickness. The ceramic discs were randomly assigned to eight treatment sequences and then received one of the following surface treatments:

Group 1: 110  $\mu$ m aluminum oxide particle abrasion (Renfert GbmH, Hilzingen, Germany) for 15 seconds at four-bar pressure and a distance of 4 mm from the ceramic surface. No additional treatment was applied, but the ceramic surface was washed with tap water for one minute, ultrasonically cleaned in a water bath for 10 minutes, and air-dried.

Group 2: The same treatment performed in group 1 and a silane agent (Scotchbond Ceramic Primer, 3M ESPE, Seefeld, Germany) was applied on the ceramic surface and allowed to dry for five minutes. Group 3: The ceramic surface received airborne particle abrasion with 50 µm aluminum oxide for 15 seconds at four-bar pressure (Renfert, Hilzingen, Germany). The distance of the tip from the ceramic surface was approximately 4 mm, and the tip was moved over the entire ceramic surface. The disc was washed with tap water for one minute, ultrasonically cleaned in water bath for 10 minutes, and air-dried. Group 4: The same treatment perfored in group 3, and a silane agent (Scotchbond Ceramic Primer, 3M ESPE) was applied on the ceramic surface and allowed to dry for five minutes.

Group 5: A liner ceramic (Cercon Ceram Liner, Degudent) was applied and sintered on the ceramic surface followed by a low-fusing porcelain glaze (Cercon Ceram Glaze, Degudent) with a no. 1 brush (Ney, Hanau, Germany) and sintered following the ceramic manufacturer's instructions. After that, the glaze was acid etched with 10% hydrofluoric acid (Dentsply, Milford, DE, USA) for 20 seconds, washed with tap water for one minute, and finally ultrasonically cleaned in a water bath for 10 minutes and airdried.

Group 6: The liner, glaze application, and sintering was the same as in group 5. A silane agent (Scotchbond Ceramic Primer, 3M ESPE) was applied on the ceramic surface and allowed to dry for five minutes.

Group 7: The liner, glaze application, and sintering was the same as in group 5. After that, the glazed surface received airborne particle abrasion with 50 µm aluminum oxide for five seconds at four-bar pressure (Renfert, Hilzingen, Germany). The distance of the tip from the ceramic surface was approximately 4 mm. The disc was washed with tap water for one minute, ultrasonically cleaned in a water bath for 10 minutes, and air-dried.

Group 8: Surface preparation was as described for group 7. In addition, a silane agent (Scotchbond Ceramic Primer, 3M ESPE) was applied on the ceramic surface and allowed to dry for five minutes.

### **Bonding Procedure**

The materials used in this study are listed in Table I. After surface preparation, in order to prepare the resin cement cylinder for cementation, equal lengths of Enforce resin cement (Dentsply) base and catalyst pastes were mixed for 20 seconds and then used to fill an iris that was cut from microbore Tygon tubing (TYG-030; Small Parts Inc, Miami Lakes, FL, USA) with an internal diameter and height of approximately 0.75 and 0.50 mm, respectively. The Tygon tubing was cut in a lathe using a stainless steel no. 11 scalpel blade (Scalpel SS, Miltex Instruments Co, Rietheim-Weilheim, Germany). The Tygon tubing containing resin luting agent was put on the ceramic surface and photocured for 40 seconds with 800 mW/ cm<sup>2</sup> from a halogen light curing unit (Optilux Demetron 501, Demetron Kerr, Danbury, CT, USA). In this manner, each ceramic surface was bonded at 10 different locations with the resin cylinders. The assembly of ceramic/resin luting agent was stored at room temperature (23°C ± 2°C) for one hour prior to removal of the Tygon tubing, then the specimens were immersed in distilled water at 37°C for 24 hours before microshear bond testing.

### **Microshear Bond Test**

Before the test, all the ceramic/resin cylinder interfaces were analyzed with a light microscope (Nikon Measurescope UM-2, Tokyo, Japan) for bonding defects. The cylinders with apparent interfacial gap formation, bubble inclusion, or any other defects were excluded and replaced by another one. Three sets of ceramic/resin luting agents (30 cylinders of resin cement in each treatment group) were used for each test group.

The assembly of the ceramic plate and the resin cement was adhered to the testing device using cyanoacrylate adhesive (Loctite UltraGel super glue, Loctite, São Paulo, Brazil), which in turn was placed in a universal testing machine (Instron 4411, Instron, Canton, MA, USA) for microshear bond testing. An edge of stainless steel with a thickness of 0.5 mm was fixed on the superior part of a universal testing machine and was gently adapted against the ceramic/resin luting agent interface. A microshear bond test was applied to each specimen at a crosshead speed of 0.5 mm/min until failure.

Table 1: Material Type, Brand Name, Manufacturer, and Composition					
Material Type	Brand Name	Manufacturer	Composition <sup>a</sup>		
Zirconia ceramic	Cercon	Degudent	ZrO <sub>2</sub> stabilized by Y <sub>2</sub> O <sub>3</sub>		
Low-fusion porcelain glaze	Cercon Ceram Kiss	Degudent	Vitreous porcelain and pigments		
Resin cement	Enforce	Dentsply	BisGMA, BHT, EDAB, TEGDMA, fumed silica, silanized barium, aluminum borosilicate glass (66% wt)		
Ceramic primer	Scotchbond Ceramic Primer	3M ESPE	Bisphenol A polyethoxy dimethacrylate 3-methacryloyloxypropyl trimethoxysilane		
Ceramic liner	Cercon Ceram Liner	Degudent	Metallic oxide combination		
<sup>a</sup> Manufacturer's information.					

### Statistical Analysis

The data were statistically analyzed using one-way analysis of variance, and multiple comparisons were made using Tukey's test. The statistical significance level was set at  $\alpha=0.05$ .

### **RESULTS**

The bond strength between the dual-cure resin cement and zirconia-based ceramic was affected by the ceramic surface treatments, and the silane application did not increase the bond strength values (p<0.001; Table 2). The means and standard deviations of microshear bond strength values for the groups tested are shown in Table 3.

The groups that utilized the low-fusing porcelain glaze in association with hydrofluoric acid (Figures 1 and 2) or airborne particle abrasion (Figures 3 and 4) showed bond strength values statistically superior to the groups that utilized the conventional airborne particle abrasion treatments with 50 and 110  $\mu$ m aluminum oxide particles (Figures 5 and 6, respectively), which is the treatment recommended by the zirconia manufacturer (p<0.001).

The low-fusing porcelain glaze treatment in association with hydrofluoric acid (Figures 1 and 2), independent of the silanization process, showed bond strength values statistically superior to the others groups tested (p<0.001). The groups that associated low-fusing porcelain glaze and 50  $\mu$ m aluminum oxide airborne particle abrasion (Figures 3 and 4) showed statistically intermediate bond strength values (p<0.001). The airborne particle abrasion groups (1 through 4) showed bond strength values statistically inferior to the glaze groups (5 through 8). Different oxide granulations tested did not improve bond strength values. The silane application did not increase the bond strength values within each treatment group (p<0.001; Table 3).

### **DISCUSSION**

The surface roughening methods for densely sintered zirconia ceramics are limited by the inertness and hardness of this ceramic. These characteristics inhibit the creation of grooves for microretention and chemical bond formation for optimal interaction with luting agents. The evaluated novel surface treatment that used a low-fusing porcelain glaze was able to

Table 2: Results of One-Way Analysis of Variance for Microshear Bond Test of Enforce Dual Resin Cement Bonded to Zirconia-Based Ceramic

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F	<i>p</i> -Value
Treatment	7	15367.386	5122.462	96.781	<0.001
Error	90	1026.5252220	11.4058358	_	_

Table 3: Means (SD) of Microshear Bond Strength in MPa							
110 μm Airborr Particle Abrasi		•	Airborne Abrasion		μm Airborne Abrasion	Glaze + Hydi	rofluoric Acid
No silane <sup>a</sup>	Silane	No silane	Silane	No silane	Silane	No silane	Silane
4.06 (1.36) c <sup>b</sup>	5.33 (1.58) c	3.95 (1.24) c	6.02 (1.61) c	17.45 (8.55) в	18.41 (7.47) в	20.75 (8.29) A	25.17 (8.37) A

<sup>&</sup>lt;sup>a</sup> Treatment recommended by the zirconia-based ceramic manufacturer.

promote superior bond strength values between zirconia-based ceramic and a dual-cure resin luting agent in comparison with the conventional surface treatment methods that utilized airborne particle abrasion (Figures 5 and 6) as recommended by the zirconia manufacturer (Table 3). Based on the results obtained, the proposed null hypothesis that the airborne particle abrasion treatments and a novel glaze surface treatment would not influence the bond strength of a dual-cured luting agent was not accepted.

In attempts to promote optimal bond strength to zirconia-based ceramics, several roughening methods have been investigated. T,8,11-13,17-19 The low-fusing porcelain glaze treatment was able to promote an increase in bond strength and appears to be a simple treatment that enhances traditional treatments like hydrofluoric acid or airborne particle abrasion (Figures 1 through 4).

The ceramic surface treatments should meet certain criteria, such as not compromising the integrity of the ceramic, not promoting additional problems with crown adaptation, and good interac-

Figure 1. Zirconia-base ceramic treated with low-fusing porcelain glaze and 10% hydrofluoric acid conditioning ×50 (# glazed area without etching and \* glazed area with etching).

tion with the luting agent.<sup>13</sup> Surface roughening methods can result in structural damage, material loss, grain pullout, and creation of sharp crack tips,<sup>20</sup> causing bonded restorations to become more susceptible to radial cracking under functional loads.<sup>11</sup> The low-fusion porcelain glaze forms a penetrating vitreous layer into the zirconia-based ceramic surface (Figures 1 through 4). This glaze-treated surface seems to be similar to a glass-based ceramic surface that is susceptible to air abrasion and hydrofluoric acid treatments and provides an interaction with the silane agent for promoting chemical reactivity.

The highest bond strengths were observed with the low-fusion porcelain glaze groups, and the association with hydrofluoric acid conditioning showed superior statistical bond strength values and promoted a better interaction within the cementation process (Table 3). The use of a silane agent did not improve the bond strength to zirconiabased ceramic for the groups tested, and these results are consistent with other studies that have observed that silane utilization does not enhance

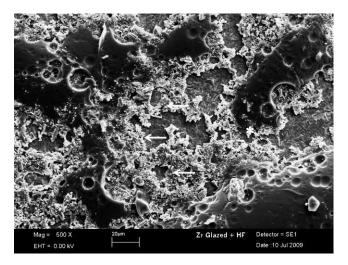


Figure 2. Zirconia-base ceramic treated with low-fusing porcelain glaze and 10% hydrofluoric acid conditioning ×500 (— glazed zirconia treated with 10% hydrofluoric acid and \* glazed zirconia).

 $<sup>^</sup>b$  Letters denote significant differences among the zirconia-based surface treatments (p<0.001; Tukey's test).

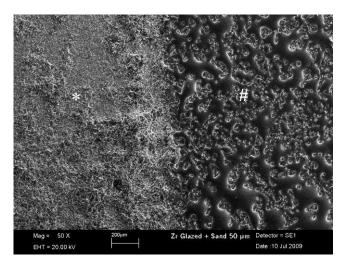


Figure 3. Zirconia-base ceramic treated with low-fusing porcelain glaze and treated with 50  $\mu$ m aluminum oxide airborne particle abrasion  $\times$ 50 (# glazed area and \* area glazed with sandblasted).

performance for high-crystalline ceramics.  $^{6,10,13,21,22}$  In addition, another reason that the silane agent did not improve the bond strength to the vitreous layer created by the glaze application can be attributed to the application of a small layer of glaze, necessary to promote an acceptable clinical marginal adaptation, and by the subsequent use of airborne particle abrasion or hydrofluoric acid etching that was responsible for partial removal of the vitreous layer (Figures 1 through 4). The zirconia airborne particle abrasion, with 50 or 110  $\mu$ m aluminum oxide, is not able to promote high bond strengths to zirconia surfaces because of the ceramic composition. Other pretreatments that aim to increase roughness or chemical bond interaction are necessary for achiev-

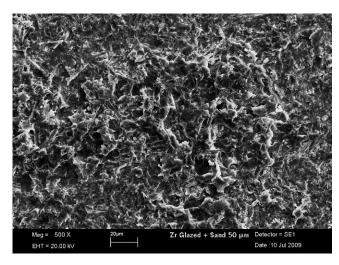


Figure 4. Zirconia-base ceramic treated with low-fusing porcelain glaze and treated with 50  $\mu$ m aluminum oxides airborne particle abrasion  $\times$ 500.

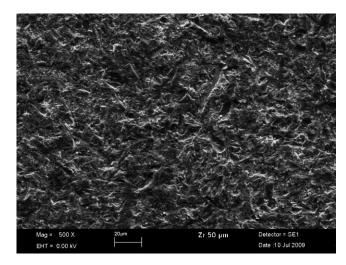


Figure 5. Zirconia-base ceramic treated with 50  $\mu$ m aluminum oxide airborne particle abrasion  $\times$ 500.

ing high immediate bond strengths and long-term clinical performance.

Optimal interaction between the ceramic surface and luting agent are necessary for the success and long-term durability of ceramic restoration. <sup>22</sup> Superb mechanical properties make zirconia-based ceramic a promising core material for fabrication of all-ceramic restorations and other dental applications. <sup>1,2,7-10</sup> A low-fusing porcelain glaze treatment seems to be a promising enhancement for resin bonding to zirconium oxide. Future investigations should assess the influence of this treatment on the overall adaptation of restorations to tooth preparations as well as degradation tests and glaze delamination of the zirconia ceramic surface.

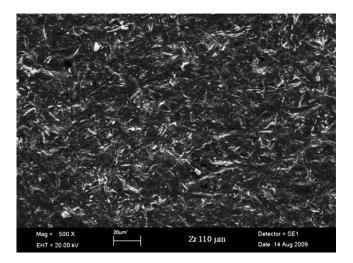


Figure 6. Zirconia-base ceramic treated with 110  $\mu$ m aluminum oxide airborne particle abrasion  $\times$ 500.

### **CONCLUSIONS**

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

- 1) Airborne particle abrasion with 50 or 110 μm aluminum oxide promotes a similar effect on the bond strength of a dual-cure resin luting agent to a zirconia-based ceramic surface.
- 2) The addition of a low-fusing porcelain glaze to a zirconia-based ceramic significantly increases the bond strength to a dual-cure luting agent.
- 3) The use of a silane coupling agent does not influence the resin bond strength to zirconia-based ceramic.

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### **Conflict of Interest Declaration**

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

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## In-Depth Polymerization of a Self-Adhesive Dual-Cured Resin Cement

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### **Clinical Relevance**

Polymerization of a dual-cured resin cement is significantly affected by ceramic thickness but not affected by activation modes. Increased irradiation times could potentially lead to higher hardness values in applications where light is not completely blocked by the overlying restoration.

### SUMMARY

The aim of this study was to assess Knoop hardness at different depths of a dual-cured self-adhesive resin cement through different

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thicknesses of Empress Esthetic® ceramic. Flattened bovine dentin was embedded in resin. The cement was inserted into a rubber mold  $(0.8 \times 5 \text{ mm})$  that was placed between two polyvinyl chloride plastic films and placed over the flat dentin and light cured by Elipar Trilight-QTH (800 mW/cm<sup>2</sup>) or Ultra-Lume light-emitting diode (LED 5; 1585 mW/cm<sup>2</sup>) over ceramic disks 1.4 or 2 mm thick. The specimens (n=6) were stored for 24 hours before Knoop hardness (KHN) was measured. The data were submitted to analysis of variance in a factorial split-plot design and Tukey's test ( $\alpha$ =0.05). There was significant interaction among the study factors. In the groups cured by the QTH unit, an increase in ceramic thickness resulted in reduced cement hardness values at all depths, with the highest values always being found in the center (1.4 mm, 58.1; 2 mm, 50.1) and the lowest values at the bottom (1.4 mm. 23.8; 2 mm, 20.2). When using the LED unit, the hardness values diminished with increased ceramic thickness only on the top (1.4 mm, 51.5; 2 mm, 42.3). In the group with the 1.4-mmthick disk, the LED curing unit resulted in similar values on the top (51.5) and center (51.9) and lower values on the bottom (24.2). However, when the cement was light cured through the 2-mm disk, the highest hardness value was obtained in the center (51.8), followed by the top (42.3) and bottom (19.9), results similar to those obtained with the QTH curing unit (center > top > bottom). The hardness values of the studied cement at different depths were dependent on the ceramic thickness but not on the light curing units used.

### **INTRODUCTION**

Resin cements have been widely used for cementation of indirect restorations, <sup>1</sup> as they have improved mechanical, physical, and adhesive properties when compared with conventional luting agents. <sup>2</sup> Furthermore, when used for cementing ceramic systems, resin cements increase the fracture strength and stability and show excellent esthetic results. <sup>3,4</sup> However, when this material is inadequately polymerized, clinical problems may arise, such as inadequate biocompatibility, <sup>5</sup> microleakage and recurrent caries, <sup>6</sup> susceptibility to degradation, marginal ditching, <sup>7</sup> discoloration, and reduction of mechanical properties. <sup>8</sup> Therefore, the clinical success, longevity, and biocompatibility of indirect restorations are largely dependent on an appropriate degree of conversion of the resin cement.

Nevertheless, in several clinical situations, the resin composite used for cementation is only partially polymerized, or adequate polymerization is impossible to achieve with visible light. Metal or ceramic restorations or even intraradicular abutments attenuate the transmission of visible light to begin the polymerization reaction of resin cement.<sup>9</sup> They can also interfere in the light spectrum transmitted. <sup>10</sup> In these cases, an appropriate degree of conversion with greater polymerization depth and hardness will be obtained by increasing the time of light exposure<sup>11</sup> or by using dual-cured resin cements with a chemical reaction that would theoretically ensure maximum conversion of monomers. However, any dual-cured resin material used for cementation is dependent on the action of visible light with adequate irradiation to obtain an optimum degree of conversion. 12-14

A resin cement with dual-cured self-adhesive components, RelyX $^{\rm TM}$  Unicem (3M ESPE, Seefeld, Germany), was introduced in 2002. According to the manufacturer, the cement contains new dimethacry-late monomers and innovative technology for initi-

ating polymerization in an acid medium by exposure to visible light or the mechanism of oxyreduction.<sup>15</sup> However, when this self-adhesive cement was polymerized by the chemical system of oxyreduction alone, a reduced degree of conversion of between 30% and 54% was shown when compared with polymerization performed by visible light and measured in the first minutes after polymerization begins. 14,16 Substantial evidence of chemically induced polymerization after light curing has been confirmed after 24 hours and more intensely after seven days. 17 Other studies conducted with different dual-cured resin cements have shown the need to wait 24 hours before fixed indirect restorations are submitted to masticatory forces when using these types of materials when they have been activated mainly by the chemical system. <sup>18-20</sup>

Until recently, most studies conducted with this self-adhesive cement have analyzed its adhesive properties on different substrates (enamel, dentin, ceramic, and intraradicular abutments, among others). Some of the chemical and physical properties were also analyzed, and the degree of conversion was assessed by Fourier transform spectrophotometry. However, those studies did not examine the influence of different light curing units and the interference of ceramic materials in the polymerization efficiency of RelyX Unicem.

Therefore, the aim of this study was to investigate the influence of ceramic thickness and light curing units on the dual-cured resin cement  $\operatorname{RelyX^{TM}}$  U100 polymerization at different depths by means of the Knoop hardness test. It should be remembered that, according to the manufacturer, the only difference between the self-adhesive cements  $\operatorname{RelyX^{TM}}$  Unicem and  $\operatorname{RelyX^{TM}}$  U100 is the distribution system. While Unicem requires an activator, triturator, and applicator, U100 can be manipulated manually because of its clicker system.

### **MATERIALS AND METHODS**

### **Fabrication of Ceramic Disks**

Empress Esthetic® covering ceramic (Ivoclar Vivadent, Schaan, Liechtenstein) was used to make two disks measuring 8 mm in diameter and 1.4 mm thick or 2 mm thick in shade ETC2. Metal matrixes were used to make the disks. The ceramic powder was mixed with the modeling liquid on a glass plate using a flexible ceramic spatula (KOTA) until the mixture was homogeneous and a pasty, shiny consistency was obtained. The ceramic was inserted into the

matrix with the use of a no. 3 hair paint brush (Ivoclar Vivadent) wet with deionized water. The ceramic condensation process was carried out under vibration, the excess material was removed, and sintering was performed in an EP600 furnace (Ivoclar Vivadent) in accordance with the temperatures recommended by the manufacturer. The ceramic disks were stored in a dry place at room temperature.

### Substrate Preparation

To simulate the condition of cementing an indirect restoration using dentin reflectance, a bovine tooth was used. The root was removed using a diamond disk mounted on a bench lathe (Nevone, São Paulo, Brazil), making it possible to remove the coronal pulp tissue with a no. 5 probe (Duflex, São Paulo, Brazil). The incisal and proximal surfaces of the crown remnants were ground using a universal polishing machine APL-4 (Arotec, Cotia, Brazil) with 120- and 200-SiC abrasive paper (Carborundum Saint-Gobain, Recife, Pernambuco, Brazil), in sequence, under water cooling, in order to fit the specimens in the three-quarter-inch polyvinyl chloride (PVC) matrixes. The bovine incisors were embedded in polypropylene resin with the buccal surface facing out, which was ground flat using 200-, 400-, and 600-SiC abrasive papers under water cooling.

### **Fabrication of Samples**

The samples were fabricated from self-adhesive dual-cured resin cement RelyX<sup>TM</sup> U100 (3M ESPE), which was light cured with either a halogen light appliance (QTH, Elipar Trilight, 3M ESPE) that emitted a light intensity at 800 mW/cm² and using a light curing time of 40 seconds or a blue light-emitting diode (LED) fitted with additional ultraviolet lamps (5 Ultralume, Ultradent Products, South Jordan, UT, USA) emitting light intensity at 1585 mW/cm² and using a light curing time of 20 seconds. It should be noted that both light curing units produced nearly equivalent light energy (LED = 31.7 J/cm² and QTH = 32 J/cm²).

On the dentin surface, a PVC packaging film (PVC Film, Goodyear do Brasil Produtos de Borracha Ltda, São Paulo, Brazil) was seated, and over this a rubber mold (0.8-mm height and 5-mm diameter) was bulk filled with the cement prepared following the manufacturer's recommendations. Another plastic film was seated over this set, and a ceramic disk of one of the predetermined thicknesses (1.4 or 2 mm) was digitally compressed to promote extrusion

and removal of excess material. The PVC films were used to prevent the cement samples from adhering to the substrate and to the ceramic. Next, the cement was light activated with either a QTH or an LED appliance, resulting in cement specimens  $800~\mu m$  thick. The experimental groups were formed by combinations between the factors of ceramic disk thickness and light curing appliance, totaling four groups with n=6. Figure 1 shows the experimental setup of the study.

After light curing, the samples were stored in a dry oven at 37°C for 24 hours. To measure the Knoop hardness of the cement, a water-cooled diamond disk was used to section the samples longitudinally (Extec model 12205, Extec Corp, Enfield, CT, USA). The cut surface resulting from sectioning was polished under water cooling in a universal polishing machine model APL-4, using 400-, 600-, and 1200-grit silicon carbide abrasive paper for 15, 30, and 60 seconds, respectively.

### Knoop Hardness Measurement Data and Statistical Analysis

After polishing, indentations were made on the samples with a microhardness tester model HMV-2 (Shimadzu, Tokyo, Japan). A 50-gf load was applied for 15 seconds. Three equidistant indentations were made at each predetermined depth from the surface, which was in contact with the PVC film closest to the ceramic disk (top, 50  $\mu m$ ; center, 400  $\mu m$ ; bottom, 750  $\mu m$ ) in the center and periphery of the longitudinal section. For each sample, the direction in which the indentations began was inverted so that no region would be favored.

A mean hardness was obtained for each depth in each sample (three measurements). The data were submitted to analysis of variance applied in a splitplot factorial design. The plots represented the light

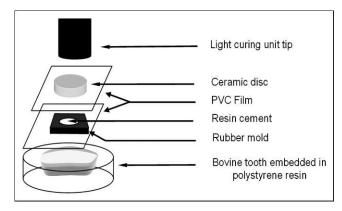


Figure 1. Schematic representation of specimen preparation.

Table 1: Mean Hardness Values (Knoop Hardness Test) of RelyX<sup>TM</sup> U100 with Regard to the Light Curing Unit, Thickness of the Interposed Ceramic, and Cement Depth Assessed (Standard Deviation)<sup>a</sup>

Light Curing Unit	Depth	Thickness		
O		1.4 mm	2 mm	
QTH	Тор	42.0 (3.5) Ab	40.6 (5.4) Bb	
	Center	58.1 (9.1) Aa	50.1 (5.7) Ba	
	Bottom	23.8 (4.4) Ac	20.2 (2.7) Bc	
LED	Тор	51.5 (7.6) Aa	42.3 (5.5) Bb	
	Center	51.9 (4.8) Aa	51.8 (6.6) Aa	
	Bottom	24.2 (2.3) Ab	19.9 (3.3) Ac	

<sup>&</sup>lt;sup>a</sup> Means followed by different letters differ within each light curing unit by analysis of variance and Tukey tests ( $p \le 0.05$ ); capital letters in the horizontal and lowercase letters in the vertical. There was no significant difference between the curing modes (p = 0.4484).

curing unit  $\times$  ceramic thickness, and subplots represented the measured depths. The level of significance considered was 5%.

### **RESULTS**

The hardness results are shown in Table 1. Statistical analysis showed that the factors "ceramic thickness" (p=0.0057) and "measurement depths" (p<0.0001) were significant, while the factor "light curing unit" (p=0.4484) was not. There was a significant interaction among the three factors (p=0.0109). Table 1 shows the result obtained by breaking down the interactions.

In the groups light cured with the halogen lamp, an increase in ceramic thickness reduced the hardness values (KHN) of the cement at all depths, with the highest values always being found in the center (1.4 mm, 58.1; 2 mm, 50.1) and the lowest values at the bottom (1.4 mm, 23.8; 2 mm, 20.2). When the LED appliance was used, an increase in ceramic thickness resulted in diminished hardness values only on the top (1.4 mm, 51.5; 2 mm, 42.3). In the group in which the 1.4-mm disk was used, LED induced similar values at the top (51.5) and center (51.9) and lower values at the bottom (24.2). However, when the 2-mm disk was used, the highest hardness value was obtained in the center (51.8).

followed by the top (42.3) and bottom (19.9), results that were similar to those obtained with the halogen light curing unit (center > top > bottom).

### **DISCUSSION**

A hardness test is commonly used as a simple and reliable method for indirectly indicating the degree of conversion of resin cements<sup>6</sup>, and the Knoop microhardness test is the most indicated for polymeric materials since the dimensions used as reference for this calculation do not undergo elastic recovery after removing the load.<sup>27</sup> The degree of conversion in a polymerization reaction depends on the energy supplied during light polymerization, characterized by the product of light intensity and exposure time. 11 Additionally, the location of the indentation made in laboratory tests is extremely important since hardness values are usually higher in the center of the material than at its extremities or edges. This can be explained by the fact that, in the center of the material, the free radicals of the monomer are tridimensionally surrounded by possible reactive partners, whereas a free radical located at the extremity of the test specimen will find reactive partners located on only one side of the hypothetical sphere in which the free radical is the center.<sup>28</sup> This study assessed the polymerization depth of an 800-µm film in order to indicate its conversion potential, although the film thickness obtained in accordance with the ISO 9917 specification for RelvX<sup>TM</sup> Unicem cement was 23.2 um.<sup>29</sup>

When cured by QTH unit, the 2-mm-thick ceramic disk decreased the hardness values of  $RelyX^{TM}$  U100 at all depths studied. The lowest hardness values and, consequently, the lowest degree of conversion are usually attributed to the attenuation of light caused by the distance from curing light tip<sup>30,31</sup> and by the increase in opacity of the material resulting from the increase in thickness of the prosthesis.  $^{8,10,14,19,32}$  The translucence of ceramics is related to their thickness, microstructure (crystalline content), number of firing cycles carried out in their processing, and the presence of porosities. 33 Empress Esthetic, used in this current study, is a leucitereinforced vitreous ceramic, with higher translucence, smaller grain size, and more homogeneously distributed leucite crystals than its predecessor, Empress.<sup>34</sup> Pazin and others<sup>32</sup> found that the Empress Esthetic ceramic, at the thicknesses of 1.4 and 2 mm, showed no influence on the emission spectrum of halogen and LED curing units (including the same brands used in this present study), maintaining the peak wavelength of the curing units

in the same position in the curve. However, this ceramic was capable of reducing the level of irradiance of a QTH unit to approximately 50% at the same thicknesses as those used in the current study. Turther studies are necessary to assess the activation of RelyX U100 through other ceramics of different colors and opacities and with the use of other levels of irradiance of light curing appliances.

Regardless, all the test specimens light cured by the halogen light curing unit through 1.4- and 2-mmthick disks showed higher hardness values in the center region (400 um) followed by the top (50 um) and finally the bottom of the material (750 µm). The lower hardness values found in the bottom region may be attributed to two possible causes: 1) the attenuation of incident light, resulting from the absorption and scattering promoted by the ceramic spacer<sup>33</sup> and the organic and inorganic components of the resin cement, 11 and 2) the impossibility of the polymerization reaction continuing because of the increase in viscosity of the resin caused by the initial polymerization and entrapment of radicals and chemical promoters in the polymer network. 36 Thus, development of the polymer network could be affected both by the reduction in the conversion of monomers and by an interference in the type and degree of cross-linking.37

In places where the light arrives with less intensity, a lower number of polymer growth centers is generated, and this is unfavorable to the formation of polymer networks that are more densely composed of cross-links.<sup>37</sup> Therefore, lower hardness values in the bottom region of the resin cement could also be related to its lower cross-link density. Thus, debonding of the prosthesis could occur clinically as a result of a cohesive failure of the luting agent.<sup>38</sup> In addition, one must consider that the present study analyzed the hardness values after 24 hours of dry storage, while the setting reactions via oxyreduction and acid base present in RelyX<sup>TM</sup> Unicem have been shown to be capable of modifying the values of the degree of conversion seven days after light activation.<sup>17</sup> Further investigations are necessary to determine how important the chemical setting of this dual-cured cement in longer periods is with regard to the durability of indirect restorations cemented with RelyX<sup>TM</sup> U100.

Since the layer inhibited by oxygen usually does not exceed 20  $\mu m,^{39,40}$  it was not a factor with the topmost region evaluated in the current study (50  $\mu m).$  The intermediate hardness values observed on the top region for QTH curing, compared to the center and bottom regions, could be attributed to the migration of

the organic polymer to the top of the luting agent, which occurs because of the compression exerted on the ceramic disk while seating it on the tooth preparation and light activation, promoting a superficial layer rich in resin both in modified ionomer materials and in resin materials.<sup>41</sup> Clinically, one should be concerned about the additional use of light at the restoration margins in order to minimize the oxygen inhibition on the resin cement surface.

When the cement RelyX<sup>TM</sup> U100 was light cured by a LED light curing unit, one could observe two different situations with regard to the polymerization depth of the cement: 1) the hardness values on the top were similar to those found in the center when light cured through the 1.4 mm thick ceramic disk, and 2) hardness values on the top were dependent on the disk thickness, which does not occur with the center and bottom values. Therefore, the top region of RelyX<sup>TM</sup> U100 would be more dependent on the level of irradiance during light curing/activation/polymerization by LED. Ultra-Lume LED 5 is a high-powered, third-generation LED (1585 mW/cm<sup>2</sup>), with a broader spectrum band than the second-generation LEDs.<sup>32</sup> It is known that high-intensity light emission during the first seconds of light activation causes the rapid formation of the polymer network on the top layer of the composite, characterized by the formation of cross-links, 42 which could increase the hardness values found under the previously mentioned conditions, including values at the top similar to those found in the center. Thus, it is expected that the reduction in mobility of the monomers in the body of the specimens would occur, making polymerization of the center and bottom regions dependent on the additional acid-base setting reactions, that is, the reaction between the metal ions of the vitreous particles of nonsilanized aluminum fluorine silicate and methacrylate phosphate radical.<sup>15</sup>

It is also known that the polymerization reactions via free radical and the acid base present in hybrid materials, such as in resin-modified glass ionomers and even RelyX<sup>TM</sup> U100, compete with and inhibit one another.<sup>43</sup> This is most likely the reason why the thickness of the ceramic disk did not influence the hardness values of the center and bottom regions when light cured by the LED curing unit in this current study. Therefore, an increase in light intensity could improve the polymerization efficiency of the luting agent and shorten the irradiation time, contributing to patient comfort and reducing clinical time for professionals. The additional polymerization reactions present in RelyX<sup>TM</sup> U100 will occur slowly,

but they will contribute to the formation of a polymer network with a high molecular weight. Therefore, the patient must be instructed to take care when exerting masticatory effort in the first 24 hours (or even seven days) after prostheses have been cemented with this cement.

### **CONCLUSIONS**

Based on the results of the present study and considering its limitations, it was concluded that the thickness of the ceramic restoration negatively influenced the polymerization of the self-adhesive cement RelyX<sup>TM</sup> U100 when cured by QTH at all analyzed depths. However, only the top region of the specimens cured by the LED unit was affected by the ceramic thickness.

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# Selective Enamel Etching: Effect on Marginal Adaptation of Self-Etch LED-Cured Bond Systems in Aged Class I Composite Restorations

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### **Clinical Relevance**

Selective enamel etching was shown to be an effective approach to reduce gap formation in Class I composite restorations for one-step self-etch adhesives.

### **SUMMARY**

The aim of this study was to evaluate the influence of previous enamel etch and light emitting diode (LED) curing on gap formation of self-etch adhesive systems in Class I composite restorations after thermomechanical aging (TMA). Thus, on 192 human molars, a box-shaped Class I cavity was prepared maintaining enamel margins. Self-etch adhesives (Clearfil SE and Clearfil S3) were used to restore the preparation with a microhybrid composite. Before application of the adhesives, half of the teeth were enamel etched for 15

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seconds with 37% phosphoric acid; the other half were not etched. For the photoactivation of the adhesives and composite, three lightcuring units (LCUs) were used: one polywave (Ultra-Lume LED 5, UL) and two single-peak (FlashLite 1401, FL and Radii-cal, RD) LEDs. After this, epoxy resin replicas of the occlusal surface were made, and the specimens were submitted to TMA. New replicas were made from the aged specimens for marginal adaptation analysis by scanning electron microscopy. Data were submitted to Kruskal-Wallis and Wilcoxon tests ( $\alpha$ =0.05). Before TMA, when enamel was etched before the application of S3, no gap formation was observed; however, there were gaps at the interface for the other tested conditions, with a statistical difference ( $p \le 0.05$ ). After TMA, the selective enamel etching previous to the S3 application, regardless of the LCU, promoted higher marginal adapta-

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tion compared to the other tested groups  $(p \le 0.05)$ . Prior to TMA, higher marginal integrity was observed, in comparison with specimens after TMA ( $p \le 0.05$ ). With regard to Clearfil SE and Clearfil Tri-S cured with FL, no differences of gap formation were found between before and after aging (5.3  $\pm$  3.8 and  $7.4 \pm 7.5$ , respectively), especially when the Clearfil Tri-S was used in the conventional protocol. When cured with RD or UL and not etched, Clearfil Tri-S presented the higher gap formation. In conclusion, additional enamel etching promoted better marginal integrity for Clearfil Tri-S, showing it to be an efficient technique for Class I composite restorations. The two-step self-etch adhesive was not influenced by selective enamel etching or by the LED-curing unit.

### **INTRODUCTION**

Composite restorations have been widely used in clinical practice, due to esthetic and some biomechanical properties similar to dental hard tissues. For the bonding procedure of these adhesive restorations, self-etching bond systems were introduced to decrease the number of bonding technical steps, since the presence of acidic monomers in their composition yields simultaneous etching and priming of the dental hard tissues. A partial removal of the smear layer is promoted and, consequently, the formation of smear plugs left undisturbed, decreasing tooth postoperative sensitivity and leaving the adhesive protocol less time-consuming. 4-6

Self-etching adhesives are known to exhibit a good bonding performance to dentin and a poor bonding behavior to enamel.<sup>3,4</sup> In order to remove the smear layer created by instrumentation, demineralize the enamel substrate, and increase bond quality and durability, a selective enamel phosphoric acid etching before the application of a self-etch adhesive has been proposed. 7-11 Some studies demonstrated that this additional enamel etching decreases gap formation when self-etch adhesives are used. 5,6,12-16 However, this additive step for direct composite restoration procedures was only evaluated in Class II, III, and V restorations. 5,6,13-16 Since the Class I preparation exhibits a high Configuration factor with enamel surrounding the superficial margins, it is considered the best model for understanding the real effects of enamel etching and the stress development on the tooth/adhesive interface. 14,17

An adequate bond system cure is another important factor to consider in restoring Class I cavities to

ensure a good bond performance and marginal integrity. An efficient light-curing unit (LCU) should guarantee a satisfactory adhesive and composite degree of conversion, which may improve its physical and mechanical properties, yielding a good marginal seal. 18-22 Quartz-tungsten-halogen (QTH) lamps have been largely used in restorative procedures; however, they present some drawbacks like bulb, filter, and reflector degradation over time and a lifetime of approximately 40 to 100 hours. 18-20 In this sense, light emitting diodes (LEDs) have been shown to overcome these problems, promoting an adequate cure of resin composites and dental adhesives through emission of unfiltered blue light. 18-20 These LCUs generate a narrow spectral range that targets the absorption wavelength of camphorquinone, vielding low amounts of wasted energy and minimum heat generation, with a longer lifetime and less decrease of light intensity. 18-20 Thus, the curing potential of current LED curing units has been shown to be similar to that presented by conventional QTH light units. 23-25

It is known that some resinous materials, like resin cements, and some adhesive systems are not well cured with conventional single-peak LEDs due to alternative photoinitiator content.<sup>26</sup> Because of this, LEDs with additional wavelengths (polywave third generation LEDs) were developed, emitting light wavelengths within a spectral region that targets the absorption peak of camphorquinone and within the UV-VIS region (400-415 nm). 23-25 This polywave behavior is expected to yield an adequate cure of adhesives and composites that contain alternative photoinitiator systems, such as phenyl propanedione, bis-alkyl phosphinic oxide, and trimethylbenzoyl-diphenyl-phosphine oxide. 25 Since manufacturers do not state all photoinitiator content, is it important to choose an adequate LCU that would polymerize all resinous materials, <sup>25-27</sup> enhancing the marginal seal and improving the composite restorations' durability.

Thus, this study aimed to investigate the influence of prior enamel etching and LED curing lights on gap formation of self-etch bond systems in Class I composite restorations. The first tested hypothesis was that selective enamel etching would improve the marginal adaptation of the adhesive systems in Class I composite restorations. The second hypothesis was that the third generation polywave LED would present lower gap formation for the tested bond systems. Moreover, the third hypothesis was that the thermomechanical fatigue would increase

Table 1: Composition, Application Mode, and Manufacturers' Information for the Adhesive Systems Tested						
Adhesive Systems	Composition	Application Mode				
Clearfil SE Bond (Kuraray Medical Inc, Tokyo, Japan)	Primer (batch 00896A): water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate.	Apply primer for 20 seconds. Mild air stream. Apply bond. Gentle air stream. Light cure at an energy density of 11 J.				
	Adhesive (batch 01320A): MDP, bis-GMA, HEMA, camphorquinone, hydrophobic dimethacrylate, N,N-diethanol p-toluidine bond, colloidal silica.					
Clearfil Tri-S Bond (Kuraray Medical Inc, Tokyo, Japan)	Adhesive (batch 00116A): MDP, bis-GMA, HEMA hydrophobic dimethacrylate, dl-camphorquinone, silanated colloidal silica, ethyl alcohol, and water.	Apply the bond system for 20 seconds. Gentle air stream for 10 seconds. Light cure at an energy density of 11 J.				
Charisma (Heraeus Kulzer, Hanau, Germany)	Composite (batch 010080): Bis-GMA, TEGDMA, barium aluminum boro silicate glass and pyrogenic silicon dioxide, photoinitiators (64% filler),	Light cure at an energy density of 22 J.				

interfacial debonding, promoting higher gaps at the superficial margins.

### **MATERIALS AND METHODS**

One hundred ninety-two healthy human third molars were selected. The teeth were collected after obtaining the patient's informed consent under a protocol approved by the State University of Campinas ethical review board (057/2009). The teeth were cleaned, embedded in polystyrene resin, and their occlusal surfaces were wet polished with 320-grit SiC paper under running water (Politriz, AROTEC, São Paulo, Brazil) to expose a flat enamel surface area without exposing dentin. Then, typical Class I cavities were prepared using no. 56L carbide burs (KG Sorensen, Barueri, Brazil) at high-speed, under air/water cooling. After five-cavity preparations, the bur was replaced. Preparations had a standard size, with cavity dimensions of 5 mm mesial-distally, 5 mm buccal-lingually, and a 3 mm depth, maintaining all cavity margins on enamel substrate. Two selfetch adhesive systems were used for the bonding protocols: Clearfil Tri-S Bond (one-step self-etch, pH = 2.7, Kuraray Medical Inc, Okayama, Japan) and Clearfil SE Bond (two-step self-etch, pH = 2, Kuraray Medical Inc). The composition, application mode, and batch number of the adhesive systems are presented in Table 1. After cavity preparation, teeth were assigned to 12 groups (n=16) according to the three studied factors: selective enamel etching, bond system, and curing light (two conditioning protocols  $\times$  two adhesives  $\times$  three LEDs).

For the photoactivation procedure three LEDs were tested: FlashLite 1401 (FL) (Discus Dental,

Culver City, CA, USA), Radii-cal (RD) (SDI Limited, Victoria, Australia), and Ultra-Lume LED 5 (UL) (Ultradent, South Jordan, UT, USA). Prior to the restorative procedure, the output power (mW) of each LCU was measured with a calibrated power meter (Ophir Optronics, Har, Hotzvim, Jerusalem, Israel). Then, irradiance (mW/cm²) was determined by dividing the output power by the tip end area. Spectral distributions were measured with a calibrated spectrometer (USB2000, Ocean Optics, Dunedin, FL, USA). Beam distribution and irradiance data were integrated using the Origin 6.0 software (OriginLab, Northampton, MA, USA). The spectral range distribution of each LCU is shown on Figure 1, and the characteristics of the LCUs are presented in

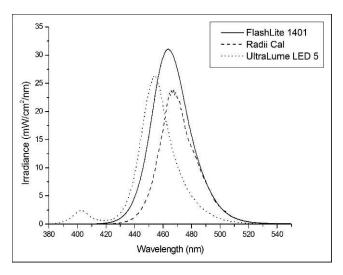


Figure 1. The spectral range distribution of each LCU used in this study.

Table 2: Characteristics of the Light-Curing Units (LCUs) Used in This Study						
LCU	Manufacturer	Туре	Tip Diameter, mm	Irradiance, mW/cm <sup>2</sup>	Composite Radiant Exposure, J/cm <sup>2</sup>	Adhesive Radiant Exposure, J/cm <sup>2</sup>
FlashLite 1401	Discus Dental, Culver City, CA, USA	Single-peak	7	1077	22	11
Radii-cal	SDI Limited, Victoria, Australia	Single-peak	7	731	22	11
Ultra-Lume LED 5	Ultradent Products Inc, South Jordan, UT, USA	Polywave	11 × 7	800	22	11

Table 2. Also, energy density was standardized to approximately 11 J for curing the bonding system and 22 J for each composite increment photoactivation. For RD, due to the fact that it mandatorily operates in ramp mode for 5 seconds, these initial seconds were discarded, and only a continuous light was delivered to keep the irradiance standardized. Consequently, an equal energy density was obtained for each of the LCUs.

All groups were restored with B1-shade Charisma microhybrid composite (Heraeus Kulzer, Hanau, Germany), using an incremental oblique technique with six increments of approximately 2 mm thick. The first layer was applied horizontally and light cured, followed by two oblique layers. Next, another three layers were placed in the same way as described before, until the cavity was completely filled. Then, finishing and polishing procedures were performed with medium-, fine-, and extra fine-grit aluminum oxide disks (SoftLex, 3M/ESPE, St Paul, MN, USA), respectively. After polishing, impressions with a low viscosity vinyl polysiloxane material (Express XT, 3M ESPE) of the teeth were taken and a first set of epoxy resin replicas (Epoxicure Resin, Buehler, Lake Bluff, IL, USA) was made for scanning electron microscopy (SEM) evaluation. In sequence, specimens were submitted to 200,000 mechanical loading of 40N (2 Hz) and 500 thermal cycles (ranging from 5°C to 55°C with a dwell time of 60 seconds in each bath with an interval of 5 seconds) in a thermomechanical device ER-11000 (ERIOS, São Paulo, Brazil) to simulate aging of the composite restorations in oral environment conditions.

New impressions of the teeth were made and another set of replicas was made for each restoration. All replicas were mounted on aluminum stubs, sputter coated with gold, and evaluated with a scanning electron microscope (JEOL, JSM-5600LV, Tokyo, Japan) at 200× magnification. SEM analysis of the composite/enamel marginal adaptation was performed by one operator having experience with quantitative margin examination and who was blinded to the restorative procedures. The marginal integrity between resin composite and enamel was expressed as a percentage of the entire superficial margin length.

### **Enamel Etching Patterns**

Eighteen half-teeth (n=3 for the two self-etch adhesives with and without prior enamel etch and negative and positive controls) were ground and randomly assigned into 12 groups. For the positive control, a 37% phosphoric acid treatment was realized and for the negative control, the enamel surface did not receive any treatment. The experimental groups were treated with the two tested bond systems using manufacturer's instructions with or without prior enamel etching. The treated surfaces were thoroughly rinsed with alternate baths of acetone (20 seconds) and ethanol (20 seconds) in an attempt to remove the self-etch primers and the monomer components. All specimens were dehydrated in ascending grades of ethanol (25%, 50%, 75%, and 90%) for 10 minutes each and immersion in 100% ethanol for 30 minutes. After dry storage at 37°C for 24 hours, specimens were sputter coated with gold and analyzed by SEM.

### **Statistical Analysis**

For the statistical analysis, as the data did not exhibit normal data distribution (Kolmogorov-Smirnov test), nonparametric tests were used (Kruskal-Wallis for groups' comparison and Wilcoxon matched-pairs signed-rank tests for pairwise com-

Table 3: Results of the Gap Formation Analysis (% and SD) of Enamel Margins Before and After Thermomechanical Aging)<sup>†</sup>

Tested Conditions	Before Aging, % (SD)	After Aging, % (SD)
FlashLite/no etch/Clearfil SE	2.6 (4.7)B	5.6 (4.5)AB*
FlashLite/etch/Clearfil SE	0.6 (1.3)B	3.0 (2.8)AB*
FlashLite/no etch/Clearfil Tri-S	5.3 (3.8)B	7.4 (7.5)AB
FlashLite/etch/ Clearfil Tri-S	0A	2.4 (4.9)A*
Radii-cal/no etch/Clearfil SE	3.2 (3.9)B	13.1 (14.7)AB*
Radii-cal/etch/Clearfil SE	3.6 (8.2)B	6.2 (8.6)AB
Radii-cal/no etch/ Clearfil Tri-S	3.0 (3.1)B	12.4 (10.7)B*
Radii-cal/etch/ Clearfil Tri-S	0A	2.9 (5.7)A
Ultra-Lume/no etch/Clearfil SE	3.5 (4.0)B	3.9 (4.4)AB
Ultra-Lume/etch/Clearfil SE	3.7 (3.7)B	4.8 (4.9)AB
Ultra-Lume/no etch/ Clearfil Tri-S	3.1 (4.3)B	8.6 (7.5)B*
Ultra-Lume/etch/ Clearfil Tri-S	0A	0.9 (2.61)A

 $<sup>^{\</sup>dagger}$  Same letters within column indicate no statistically significant difference (p $\leq$ 0.05, Kruskal-Wallis and Dunn comparison). Asterisks stand for p $\leq$ 0.05; Wilcoxon matched-pairs and signed-rank tests.

parisons before and after thermomechanical aging) with a pre-set alpha of 0.05.

### **RESULTS**

### **Marginal Adaptation Analysis**

The results of the marginal adaptation analysis are shown in Table 3. No gap formation was observed before thermomechanical loading when enamel was etched before the application of Clearfil Tri-S; however, there were gaps at the interface of the Clearfil SE Bond regardless of the energy source, with statistical difference (Kruskal-Wallis test;  $p \le 0.05$ ). After aging, selective etching prior to the Clearfil Tri-S application, when RD or UL were used, promoted higher marginal adaptation compared to the other tested groups (Kruskal-Wallis test;  $p \le 0.05$ ). Figure 2 shows examples of marginal

integrity and marginal gaps of Class I composite restorations that were observed in this study.

Prior to thermomechanical loading, higher marginal integrity was observed in comparison with after aging (Wilcoxon test;  $p \leq 0.05$ ). When the bonding systems were cured with FL and only when Clearfil Tri-S was used in the conventional way were there no differences between before and after aging (5.3  $\pm$  3.8 and 7.4  $\pm$  7.5, respectively). When cured with RD or UL and not etched, Clearfil Tri-S presented the higher gap formation.

### **Enamel Etching Patterns**

The SEM enamel etching patterns are shown in Figures 3 (A,B), 4 (A,B), and 5 (A,B,C).

### DISCUSSION

An important factor to promote clinical success of Class I resin composite restorations is a satisfactory enamel marginal adaptation. The presence of gaps is considered as the first sign of restoration failure, clinically evidenced by marginal staining. Also, when detectable marginal disruption is present, these interface defects could lead to interfacial leakage. In the oral environment, many pulpal sensitivities and responses are related to when bacterial leakage occurs along the tooth/composite bonding interface.

In the present work, the first hypothesis was partially validated, since only the gap formation for the one bottle all-in-one self-etch adhesive Clearfil Tri-S was affected by the selective enamel etching procedure, regardless of the LCU. This may be explained by some characteristics of this mild onestep adhesive, like its pH and etching potential.<sup>3,4</sup> As shown by SEM evaluation of the enamel etching patterns, Clearfil Tri-S alone promotes a smooth enamel demineralization, not increasing the surface free energy and bond penetration. Its pH of approximately 2.6 did not promote an adequate demineralization of the enamel surface and resulted in poor bond strength. This weak bond interface is easily affected by composite shrinkage stress during the photocuring procedure. 7,8,28 In this sense, the selective enamel etch procedure could have promoted a deeper dissolution of prism cores and boundaries in a type III etching pattern, increasing the surface free energy of this substrate and consequently, increasing the percentage of gap-free margins.

For Clearfil SE Bond, the previous acid etching did not affect the gap formation of the composite restorations. This may have occurred because of

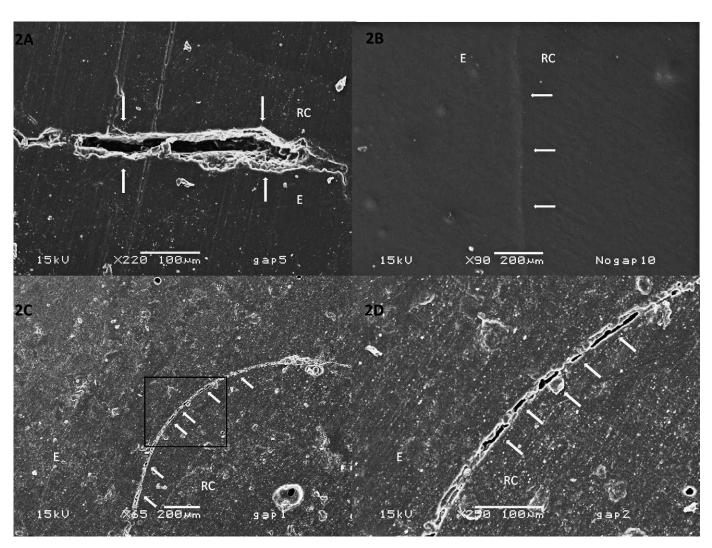


Figure 2. (A): The white arrows point to interfacial gap formed between resin composite and enamel. (B): The white arrows indicate a perfect marginal seal between enamel and composite. (C): Note the composite/resin interface presenting some interfacial gaps. The white arrows point to the correct location of the marginal gaps. (D): A high magnification showing the interfacial gaps between composite and dental enamel.

the lower pH of the adhesive system, approximately 2.0, which could have promoted higher enamel demineralization compared to Clearfil Tri-S alone, regardless of the curing light. Also, the studied two-step bond system contains a separate hydrophobic resin that is applied after the acidic primer. This hydrophobic resin coat can improve bond durability, especially due to the structural polymer network that is not hydrophilic and can maintain optimal bonding behavior after fatigue stress. 8,30,31

The second hypothesis of this study was rejected because the polywave third-generation LED did not improve the marginal adaptation of the resin composite restorations. The photoinitiator content of the composite and adhesive resin formulations seems to be camphorquinone, as informed by manufacturers. Therefore, the polywave LED for camphorquinonebased resin exhibited a similar behavior as the singlepeak second generation LED. This was demonstrated by the fact that both adhesive systems and the microhybrid composite with camphorquinone did not have interfacial integrity affected. Also, they may have presented a similar degree of cure and consequently, less marginal shrinkage stress, preserving the superficial marginal adaptation between composite and enamel.

Another fact to discuss is the morphology of the Ultra-Lume LED 5 tip, in which there is a central LED that emits visible light at the peak spectra of camphorquinone and four accessory LEDs in the corner of the tip that emit UV-VIS wavelengths. In a Class I preparation, with 25 mm², as the condition presented in this work, light emitted by the accessory LEDs would not reach the adhesive resin

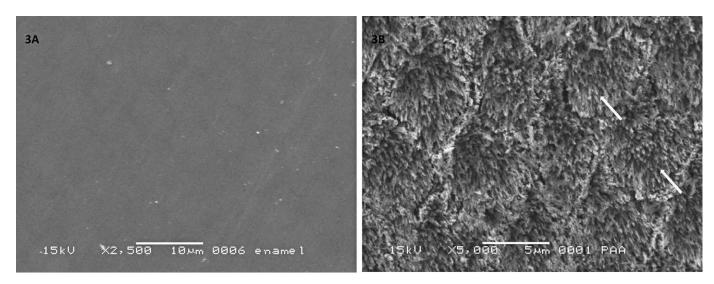


Figure 3. (A): SEM photomicrograph showing the dental enamel without any acid treatment. (B): Phosphoric acid (37%) etched enamel, showing a type II etching pattern, with the dissolution of the prism cores.

since the tip dimensions are higher than the area of the tooth preparation.<sup>24</sup> Consequently, only the central LED with the absorption peak of camphorquinone may have irradiated the adhesive resin, promoting similar gap formation as FlashLite and Radii-cal. This fact is not in agreement with a previous study, which found that polywave LEDs promote better resin mechanical properties compared to single-peak LEDs, even when the light curing tip end is located at a long distance.<sup>22</sup>

For half of the tested groups the thermomechanical loading promoted higher gap formation, partial-

ly validating the third hypothesis. When specimens were restored using Clearfil Tri-S without selective enamel etching, the thermomechanical aging promoted less marginal integrity, except when FL was used for photopolymerization. The previous enamel etch may have guaranteed a higher bond performance due to the increase in the enamel surface free energy caused by a deeper enamel demineralization. A,5,14 The etching pattern of Clearfil Tri-S with previous acid treatment shows a deeper demineralization of enamel cores and boundaries, favoring the penetration of the adhesive resin. Consequently, the

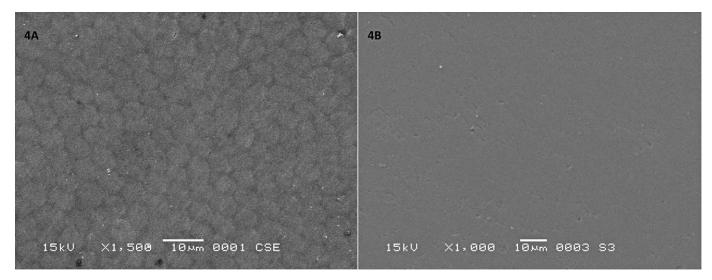


Figure 4. (A): SEM photomicrograph showing the smooth enamel etching promoted by Clearfil Tri-S, with no dissolution of prism cores and boundaries. (B): SEM photomicrograph showing the smooth primer etching of Clearfil SE, with a type I etching pattern, with only dissolution of prism boundaries.

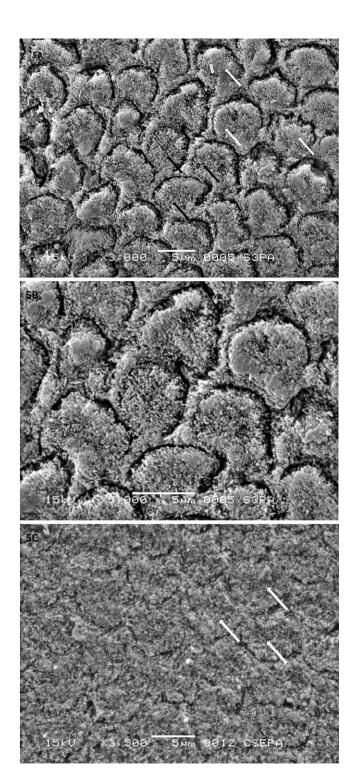


Figure 5. (A): SEM photomicrograph of Clearfil Tri-S with previous phosphoric acid etching, showing a deep dissolution of prism cores and boundaries, with a type III etching pattern (characteristics of the type I and II). The white arrows point to the characteristics of type I etching pattern, with intact prism core, while the black arrows show characteristics of the type II etching pattern, with higher dissolution of prism peripheries. (B): SEM photomicrograph of Clearfil Tri-S with higher magnification 5000×. (C) SEM photomicrograph of Clearfil SE with previous acid etching, with a type III etching pattern, pointed out by the white arrows.

thermomechanical effect was not capable of inducing higher gaps at the interface, preserving the enamel/composite bonding. For FL, the aging may not have affected bonding to enamel because this LCU emitted higher light irradiance compared to the other curing devices, even if the energy dose was standardized. This higher irradiance may have caused a bond disruption at the interface both before and after thermomechanical fatigue. 32-34

For Clearfil SE, Ultra-Lume curing light promoted the maintenance of marginal integrity even after thermomechanical aging, regardless of additional enamel etching. This may be explained by the hydrophobic layer of this bond system which may have not been influenced by the composite shrinkage stress and consequently maintained the bond interface with no alterations. This additional layer can promote a higher monomer conversion and allow marginal integrity after thermomechanical loading. Also, when RD was used with enamel etching, the aging loading did not influence the percentage of marginal gaps.

Although some *in vitro* studies exist, final conclusions regarding the role of enamel selective acid etching for self-etch adhesives in Class I composite restorations will depend on the outcomes of clinical trials. Clinical long-term studies and investigations of the retention ability of this approach for bond systems in the oral environment can best evaluate the quality and durability of these restorations.

### CONCLUSION

Selective enamel etching promotes better marginal integrity for Clearfil Tri-S, showing itself to be an efficient additional step for Class I composite restorations. The two-step self-etch adhesive was not influenced by selective enamel etching or by the LED-curing unit. In general, the mild one-step self-etch bond system preserved the marginal adaptation integrity when enamel was previously etched, except when the single-peak LED FlashLite, with higher irradiance, was used.

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# Influence of Volumetric Shrinkage and Curing Light Intensity on Proximal Contact Tightness of Class Il Resin Composite Restorations: *In Vitro* Study

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### **Clinical Relevance**

Volumetric polymerization shrinkage and curing light intensity should be considered when restoring proximal contact of class II cavities with resin-based materials.

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### SUMMARY

Background: Proximal contact tightness of class II resin composite restorations is influenced by a myriad of factors. Previous studies investigated the role of matrix band type and thickness, consistency of resin composite, and technique of placement. However, the effect of volumetric shrinkage of resin and intensity of curing light has yet to be determined. Thus, the aim of this study was to identify the influence of these factors on the proximal contact tightness when restoring class II cavity preparations in vitro.

Methods: Sixty artificial molars were restored with either a low-shrinkage (Filtek Silorane, 3M ESPE) or a conventional (Z100, 3M ESPE) composite and polymerized with low-intensity (Smartlite IQ2, Dentsply) or high-intensity light curing units (Demi<sup>TM</sup>, Kerr). Proximal

contact tightness was measured using the Tooth Pressure Meter. Data were statistically analyzed using one-way analysis of variance and Tukey *post hoc* test.

Results: Use of low-shrinkage composite (Filtek Silorane) resulted in significantly tighter proximal contacts compared to the use of conventional composite (Z100) when cured with the same polymerization unit (p<0.001). Moreover, the low-intensity curing unit (IQ2) resulted in significantly tighter contacts than the high-intensity unit when material is constant (p<0.001).

Conclusions: Low-shrinkage resin composite and low curing light intensity is associated with tighter proximal contact values.

### INTRODUCTION

The reconstruction of tight and anatomically correct proximal contacts in class II composite restorations remains an issue for most general practitioners. The difficulty in achieving tight proximal contacts has been attributed to elasticity and thickness of the matrix<sup>1,2</sup> as well as lack of condensability and shrinkage of resin composite.<sup>3</sup> Several techniques and instruments have been proposed to obtain tight proximal contacts,<sup>1,4</sup> and the general consensus is that with the use of sectional matrix bands in combination with separation rings, adequate proximal contacts can be achieved.<sup>2,3,5-8</sup>

Along with the effect of the matrix system and separation technique on the proximal contact tightness, volumetric shrinkage that occurs during setting of the composite material might also have a significant effect. The volumetric shrinkage for traditional methacrylate-based composites ranges from 2% to 6%. Recently, a low-shrinkage composite was introduced in which the methacrylate matrix is replaced by so-called siloranes. The ring-opening chemistry of this resin significantly reduces the volumetric shrinkage below 1%. 10 The main sequel of polymerization shrinkage is the development of internal contraction stress that can damage the marginal seal of the bonded restorations. Contraction stresses can lead to interfacial gap formation and produce postoperative sensitivity, marginal staining, or recurrent caries. Moreover, cusp displacement may occur, resulting in postoperative sensitivity, crack, or fracture development. 11-14 However, it is unclear if shrinkage has an effect on the obtained proximal contact tightness.

The introduction of new high-power polymerization units has initiated a debate on their effect on the properties of the resin composite material. Volumetric shrinkage, for example, was found to be lower with low-intensity curing units compared to high-intensity ones, with similar irradiation times. Moreover, reduced intensity slowed down the rate of polymerization but did not reduce the conversion as long as an irradiation time of 60 seconds was employed. On the basis of obtaining optimal conversion and adaption, it was demonstrated that the irradiation time was more effective than irradiation energy and that high intensity light curing does not necessarily lead to optimal quality. <sup>16</sup>

The effect of variables such as the thickness and elasticity of the matrix band, different separation techniques, and consistency of the resin composite, have already been investigated in several studies. However, the exact effect of the volumetric shrinkage on the obtained proximal contact tightness remains unknown. Therefore, the aim of this study was to investigate, *in vitro*, the effect of two resin composites with different volumetric shrinkage values and the effect of two polymerization units with different light intensities on the obtained proximal contact tightness of class II composite restorations.

The null hypothesis  $(H_0)$  of this study was that the amount of volumetric shrinkage of resin composite and the intensity of the curing light will have no statistical significant effect on the proximal contact tightness when restoring class II cavity preparations with resin composite.

### **MATERIALS AND METHODS**

A class II MO-cavity preparation was prepared in an artificial ivorine lower left first molar (Kilgore International, Coldwater, MI, USA). The cavity dimensions for the proximal portion were  $5.0 \times 4.0$ × 2.0 mm buccolingual, occlusogingival, and mesiodistal, respectively, while for the occlusal portions, the dimensions were  $4.0 \times 2.5 \times 3.0$  mm buccolingual, occlusopulpal, and mesiodistal, respectively. This preparation was the master model and was duplicated by the manufacturer (Kilgore). resulting in 60 identical preparations. Teeth were placed in a manikin model (Kilgore) and phantom head (KaVo Dental, Biberach, Germany) during all restorative procedures in order to simulate clinical conditions. A copper-zinc alloy cast of the second premolar was used to prevent wear of the distal surface of this tooth during the restorative procedures and proximal contact tightness measurements (Figure 1).<sup>6,7</sup>



Figure 1. Cavity design used for all test groups

Specimens were equally divided over four groups according to type of composite and polymerization unit used (n=15). All restorative procedures were performed by one operator (HS). Composite materials used were a low-shrinkage silorane-containing composite material with a volumetric shrinkage of <1% (Filtek Silorane, 3M ESPE, St. Paul, MN) and a conventional methacrylate-based composite material, with a volumetric shrinkage of 2.26-2.61% 17 (Z100, 3M ESPE). Each composite material was polymerized using two light polymerization units: a low-power LED unit (Smartlite IQ2, Dentsply, York, PA, USA, light intensity 700 mW/cm<sup>2</sup>) and a highpower LED-unit (Demi<sup>TM</sup>, Kerr Corp, Orange, CA, USA, light intensity 1100-1300 mW/cm<sup>2</sup>). Both polymerization units had a light guide tip of 8 mm in diameter. The light intensity was measured with the Demetron L.E.D. Radiometer (Detection range: 0-2000 mW/cm<sup>2</sup>, Kerr Corporation) at the beginning of the study and every 15 specimens thereafter.

For all groups, a circumferential precontoured matrix (1101c, KerrHawe SA, Bioggio, Switzerland) placed in a Tofflemire retainer (Kerr Corporation) was used. A wedge (Premier Dental Products Co, Plymouth Meeting, PA, USA) was placed from the lingual aspect followed by the application of the separation ring (V-ring, TrioDent, Katikati, New Zealand). The matrix band was lightly burnished with a hand instrument (PFI 49, Dentsply Ash, Weybridge, Surrey, United Kingdom) until no visual space was left between the matrix and adjacent tooth. Also, an explorer was used to check the fit of the matrix band at the gingival margin of the proximal box.

The composite materials were used in combination with the manufacturer-recommended adhesive system. For the low-shrinkage material (Filtek Silor-

ane), a special adhesive system (Silorane System Adhesive Self-Etch Primer and Bond, 3M ESPE) was used. For the conventional composite material, a three-step etch-and-rinse adhesive system (Scotchbond Multi-Purpose, 3M ESPE) was employed. Both adhesive systems were applied and polymerized according to the manufacturer's instructions. In all groups, the composite material was placed in three increments of less than 2 mm using a strategic incremental technique to minimize the C-factor. 18 The first increment was applied to the gingival seat, and the second and third increments were applied in an oblique manner to the buccal and lingual cavity walls, respectively. Each increment was cured individually after placement for 20 seconds. After removal of the matrix band, the restorations were postcured for an additional 20 seconds from the buccal and lingual sides. Restorations were not finished or adjusted in order to prevent changes of the proximal surface.

After restoration, the model was removed from the phantom head and placed in a custom-made setup to standardize proximal contact measurement procedures (Figure 2). A second investigator (MS), blinded to the material and curing technique, was asked to perform the proximal contact measurements. Proximal contact tightness was measured using the Tooth Pressure Meter, a device that is constructed at the University of Technology Delft in the Netherlands and was previously used in



Figure 2. Custom-made setup to standardize measurement using the Tooth Pressure Meter

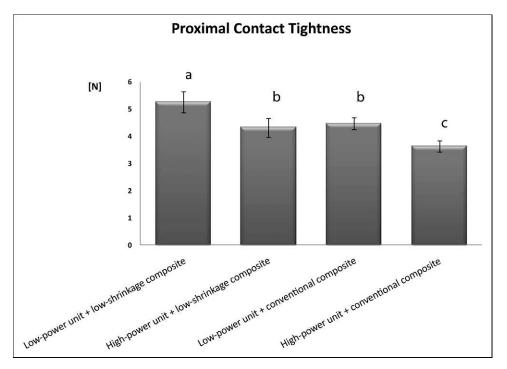


Figure 3. Mean proximal contact tightness (N) with standard deviations for the four groups tested. Different symbols (a, b, and c) indicate statistically significant differences between groups (analysis of variance; p<0.05).

other in vitro studies. 6-8,19 This device uses a 0.05mm-thick metal strip that is inserted interdentally from an occlusal direction. The tightness of the proximal contact is quantified as the maximum frictional force (N) when the strip is removed in the occlusal (vertical) direction. To obtain a standardized measurement of all proximal contact areas, the manikin model and the Tooth Pressure Meter were mounted in a special device allowing a standardized insertion and removal of the metal strip, as shown in Figures 2 and 3. To minimize variations in proximal contact measurements due to repositioning of the tooth in the manikin model, a special protocol for proximal contact measurements was applied. The final result for a measuring site was the mean value of three consecutive measuring procedures. Each procedure included the removal and repositioning of the tooth in the manikin model followed by three consecutive contact measurements using the Tooth Pressure Meter. A measurement failed when the outcome exceeded the maximum (preset) range of 0.5 N between the three measurements, such as due to deformations of the strip or a nonparallel removal of the strip from the interdental area. This measurement was then excluded from the analysis and repeated. Customwritten software in Excel (MS Office 2000 for Windows) was used for data acquisition and for construction of diagrams relating force to seconds.

To determine differences in proximal contact tightness between groups, a one-way analysis of variance followed by Tukey *post hoc* test was performed. The level of significance was set at p < 0.05.

### **RESULTS**

Figure 3 shows means and standard deviations of the proximal contact tightness. The use of the lowshrinkage composite (Filtek Silorane) resulted in statistically significant tighter proximal contacts compared to conventional composite (Z100) when polymerized using the same curing light (p < 0.001). Moreover, the low-intensity curing light (IQ2) resulted in statistically significant tighter proximal contacts than the high-intensity one when used with the same composite material (p<0.001). The tightest proximal contacts were obtained when the lowshrinkage composite (Filtek Silorane) was cured with low-intensity polymerization unit (IQ2) (5.25 ± 0.39 N), while the weakest contacts were observed when the conventional composite was polymerized with the high-intensity unit (3.63  $\pm$  0.20 N). In the case of low-shrinkage composite cured with the highintensity curing unit (DEM), the obtained proximal contact tightness was 4.32 ± 0.34 N, which was not statistically significant different from the proximal contacts measured in the conventional composite

cured with the low-power unit (4.47  $\pm$  0.22 N; p=0.71).

### **DISCUSSION**

Based on the statistical results, the low-shrinkage resin composite provided significantly higher proximal contact tightness between the materials, while among the polymerization units, the low-power unit resulted in significantly higher proximal contact tightness (IQ2). Thus, the null hypothesis of the study  $(H_0)$  was rejected.

The magnitude of volumetric shrinkage experienced by a composite is determined by its filler volume fraction and the composition and degree of conversion of the resin matrix. 20 Volumetric shrinkage is an inherent property specific to each monomer. Studies have shown that silorane-based resin composites exhibit less shrinkage than methacrylate-based resin composites. 10 In methacrylatebased resins, when monomers in proximity react to establish a covalent bond, the distance between the two groups of atoms is decreased, translating into volumetric shrinkage.<sup>20</sup> However, as silorane-based resin polymerizes, ring monomers connect by opening, flattening, and extending toward each other (cationic ring opening process), resulting in less volumetric shrinkage compared to methacrylate-based resins. 10 This difference in curing dynamic is a possible explanation for the difference in proximal contact tightness observed between the two materials. However, this difference in contact tightness could be attributed to differences in the consistency and handling characteristics of the resin composites used in this study. Also, operator bias regarding the material could be a possible influencing factor. The limitations of the study precluded identifying a direct cause-and-effect relationship.

Additionally, the light intensity of the polymerization unit was found to have a statistically significant effect on the proximal contact tightness. Previous studies have shown that low-intensity polymerization units resulted in less volumetric shrinkage than the high-intensity units. <sup>21,22</sup> The volumetric shrinkage of resin composite has been shown to be proportional to its degree of conversion. <sup>23,24</sup> In photoactivated materials, the degree of conversion depends on radiant exposure (J/cm<sup>2</sup>), which is the product of light irradiance (intensity) and exposure time. <sup>25</sup> Thus, lower degree of conversion is obtained with low-power intensity at a fixed exposure time, resulting in less volumetric shrinkage. Moreover, the slower curing process will delay

the gel point. This allows stress relaxation to occur within the resin and at the interface, reducing the volume of the shrinkage. These phenomena may contribute to the greater proximal contact tightness values observed when a low-power unit was used.

In the current study, an *in vitro* model was used to compare the various systems. The validity of this model was demonstrated by Loomans and others<sup>6</sup> and Saber and others, <sup>19</sup> where it was concluded that the *in vitro* model is representative of the clinical situation. Natural tooth movement cannot be reproduced in a laboratory setting. However, the standardization of tooth movement in the current model enabled the authors to determine the influence of the experimental variables on proximal contact tightness.

Finally, it must be pointed out that the provision of interdental separation is the most important consideration when restoring proximal contact of class II resin composite restorations. This can be best achieved by using separation rings. <sup>5-7,19</sup> Nevertheless, this study showed that type of composite, possibly volumetric shrinkage, and intensity of polymerization light influence proximal contact tightness. However, a direct cause-and-effect relationship between shrinkage and contact tightness was not established.

### CONCLUSION

The volumetric shrinkage of resin composite and intensity of the curing light should be considered influencing factors during restoration of proximal contact with resin-based materials.

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### A Simplified Technique for Restoring Interproximal Root Surface Lesions

AS Mennito • WG Renne

### **PURPOSE**

Interproximal root surface caries can often be very difficult to restore due to their lack of direct access and location to the gingiva. This article provides a technique to simplify restoring interproximal root surface lesions. This technique conserves tooth structure, utilizes glass ionomer as the definitive restorative material, and requires very little finishing once the material is fully set.

### **CLINICAL TECHNIQUE**

For all procedures involving bonding of any nature, the authors recommend placement of a rubber dam in order to obtain proper isolation. However because these types of lesions are often subgingival, a split dam technique may be required.

First, application of a rubber dam or comparable isolation system is performed. Next, the interproximal decay is removed from either a buccal or lingual approach, depending on which allows easier access. Typically, these lesions are found within 1-2 mm of the cementoenamel junction (CEJ)<sup>1</sup> (Figure 1) and will extend the entire buccal-lingual width of the tooth. The finished preparation will usually resemble a half circle once completed (Figures 2 and 3).

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Figure 1. Preoperative radiograph showing interproximal root surface lesion on the distal of tooth #18.



Figure 2. Mock-up of location and shape of typical interproximal root surface lesion on typodont tooth #13.



Figure 3. Actual preparation shape and size of tooth #18.

Once the decay has been removed, the preparation is conditioned using Cavity Conditioner (20% polyacrylic acid, GC America, Alsip, IL, USA). This is placed in the preparation for 10 seconds and then rinsed with water. The excess water is then removed using a single puff of air, leaving the preparation slightly moist. Next, a Tofflemire band (Henry Schein, Melville, NY, USA) is placed around the tooth making sure that the band extends past the gingival margin of the lesion to healthy tooth structure. Either a number 1 or a number 2 band will be used depending on how far gingivally the lesion is located. The band is trial fitted and a scratch mark is placed at the desired access location using a bur (Figure 4). The band is then removed from the mouth and a hole is made using a small bur, in this case, a 330 carbide bur (Brasseler USA, Savannah, GA, USA). This hole should be between 2.5 and 3 mm in diameter. This is just large enough

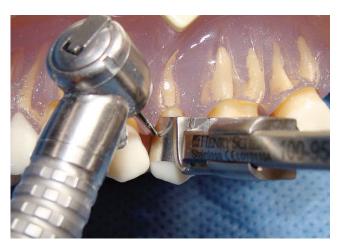


Figure 4. Making a hole in the matrix band to allow access for restorative material.



Figure 5. Proper size of hole in matrix band to allow penetration of syringe tip.

to allow the tip of the glass ionomer syringe to pass through (Figure 5).

At this point, the band is reinserted (Figure 6). A wedge may be placed in order to ensure proper adaptation of the band to the gingival margin. Often, the wedge is inserted only to the point of passive binding. Forcing the wedge into place can cause the band to bulge into the cavity preparation, leaving the final restoration with a less than ideal anatomic form.

Fuji IX GP Extra (GC America) is then activated and triturated for 10 seconds at ±4000 rpm and immediately placed into the applicator. The tip of the syringe is placed just into the opening in the matrix band, and the material is discharged aggressively until it begins to extrude from the hole (Figure 7). When syringing the material, do so forcefully to allow the material to spread into the preparation and become well adapted. Wait about one minute and



Figure 6. The band with the hole in place allowing access to the preparation.



Figure 7. Backflow of glass ionomer through access hole means preparation has been adequately filled.

then begin to clean up the excess material while it is still in the gelatinous phase, particularly around the access hole. Removing the excess at this point helps to minimize finishing once the material is set and will ensure that the matrix does not get locked into place. Often, excess material will creep occlusally along the inside of the band, and cleanup will likely be required adjacent to the marginal ridge as well. This is easily done with the tip of an explorer.

Once the material is set (in the case of Fuji IX GP Extra, around 3 minutes after trituration), the band is removed and an ET-6 bur (Brasseler USA) is used to remove any remaining flash. The restoration is then coated using G-Coat Plus (GC America) to protect the glass ionomer from moisture fluctuations, as well as to give it a smoother surface finish.



Figure 8. Appearance of final restoration of typodont after trimming and coating.



Figure 9. Appearance of final restoration of tooth #20 after trimming and coating.

The final restorations should have proper anatomic contour and have a shiny surface finish (Figures 8 and 9). Bitewings of completed restorations can be taken to confirm excellent results.

### **DISCUSSION**

According to the 2010 census, the number of people over the age of 62 years in the United States increased 21% over the past 10 years. As the average age of the population increases, so does the number of geriatric patients who require dental care. Older patients have several challenges to overcome regarding the maintenance of their oral health. These patients are often on multiple medications that can cause them to produce less saliva, which increases the likelihood of caries formation. Likewise, older patients tend to have gingival recession and exposed root surfaces. These surfaces are more prone to decay than the enamel covered coronal portion of the tooth. When compounded with reduced manual dexterity and the inability to properly manipulate floss, these patients are more likely to form interproximal root surface decay.<sup>2</sup>

Root surface lesions can often be difficult to restore. Typically, they are found at the level of the gingiva and often extend subgingivally. Interproximal lesions offer an even greater challenge since they often cannot be easily accessed. Sometimes accessing these lesions from the occlusal is the only way to restore them. However, this method requires removing large amounts of what is often healthy tooth structure, which is less than ideal. When the caries can be accessed and removed by direct access from the buccal or lingual, the challenge is then finding the best material and method of isolation in order to restore it.

When choosing a restorative material for these lesions, one must take into account the strengths and weaknesses of each potential material and how they will affect the longevity of any restoration. When accessing interproximal root surface lesions from the buccal or lingual, it can be difficult sometimes to position the bur and handpiece in such a way as to create the proper mechanical retentive features required to place an amalgam restoration. Likewise, the banding of these lesions to create a proximal wall against which an amalgam can be condensed is extremely challenging. When the band is in place, the cavity cannot be accessed. Placing composite would address the problem of needing mechanical retention in the preparation; however, the proximity to the gingiva and the inability to isolate the preparation from moisture would create problems with bond strength and restoration longevity.3 Likewise, the bonding effectiveness of today's bonding systems on cementum is unpredictable at best.4

Conventional glass ionomer restorative materials are, by the nature of their composition, self-adhering.<sup>5</sup> These materials form a chemical bond with the mineral content of the tooth and only require minimal conditioning of the tooth.<sup>6</sup> Studies have shown that conditioning with 20% polyacrylic acid prior to placing Fuji IX GP Extra increases the bond strength.7 These materials also release fluoride in significant enough quantity and longevity to drastically reduce the incidence of recurrent caries. Conventional glass ionomer has also been shown to possess the ability for the fluoride reservoir to be "recharged" by means of topical application of fluoride gels or pastes.8 Root surface lesions are often found at or below the level of the gingiva, making isolation from moisture difficult. Glass ionomers are hydrophilic materials that actually require water as part of the acid/base setting reaction that they undergo.9 Therefore, if moisture contamination occurs during placement of your restoration, it will not be as catastrophic as it would be for a composite restoration. Furthermore, conventional glass ionomer has shown excellent biocompatibility<sup>10</sup> and gingival response to restorations placed subgingivally. 11 A downside of this material is its lack of overall strength. 12 However, this is of little consequence interproximally where the restoration will be subject to minimal stress. Likewise, the relative poor esthetics of these materials should not be a factor due to the location of these lesions.

Fuji IX GP Extra was the material used for this technique. Directly after trituration, the material has

a slightly flowable viscosity, which allows it to be syringed into the preparation and adapted to the internal walls. It is important to syringe the material quickly after trituration to ensure it is still in this slightly flowable state. If allowed to begin setting up, it will not adapt as well and voids may form. Because this technique relies on the pressure of the material against the walls for adaptation, it is important to initially syringe more material than can fit into the preparation. This back fill pressure will help get excellent adaptation of the material into the cavity preparation but will also cause some overflow of the material past the margins. This overflow typically occurs within the band in an occlusal direction and is easily removed with an explorer tip while the material is setting. Good adaptation of the band along the gingival portion of the preparation limits movement of excess material in this direction. Being able to band these lesions actually reduces the amount of finishing required compared to freehand placing of these restorations. Furthermore, having the band in place assures the material only bonds to the intended tooth and not the adjacent tooth as well.

Advantages of this technique include, most importantly, preservation of tooth structure. By providing a good technique to restore interproximal lesions, practitioners no longer have to access these lesions through the marginal ridge in order to be able to place a matrix band. This preserves the strength of the tooth while still being able to provide a sound restoration. Another positive outcome associated with this technique is the lack of finishing required. Often the gingival margins require no finishing at all due to tight adaptation of the band to the tooth at this point. Since no condensation force is placed on the glass ionomer after it is syringed, the material stays within the confines of the matrix band. The flash that is produced is in areas that are easily accessible and is simple to remove.

There are several potential problems associated with this technique. In some parts of the mouth, interproximal root surface lesions cannot be accessed directly. For example, individuals with third molars can sometimes get these lesions on the distal root surfaces of the second molars. In these instances, it may be easier to access the decay from the occlusal with a class II type preparation. Another difficulty with this technique involves placement of the band. It can sometimes be difficult to ensure that your band is seated gingival to the margin of the cavity preparation because this area cannot be visualized as directly as it can be when doing a class II restoration. It is imperative that the gingival margin of these restora-

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tions be smooth, not only for the health of the tissue in that area, but also for the longevity of the restoration. An uneven, subgingival margin can be mistaken for calculus by dental hygienists who can end up chipping or dislodging these fillings with scalers during prophylactic appointments.

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## Verifying Occlusal Reduction During Tooth Preparation

ST McGill • JR Holmes

Obviously, it is important to obtain and verify adequate occlusal reduction during tooth preparation. Failure to do so can lead to a number of potential problems. The resultant restoration may be difficult for the technician to fabricate easily with an appropriate functional and esthetic occlusal surface. The restoration may be thin and/or weak as a result of the thickness itself or attempts by the clinician to equilibrate high spots prior to luting. Over time, inadequate thickness in excursive areas may lead to occlusal wear and loss of integrity (ie, holes).

Attempts by clinicians to verify adequate occlusal reduction usually rely on visual assessment, which can be inaccurate. Occlusal guides such as leaf gauges or tabs of known thickness are sometimes used (Figure 1) but do not always easily reveal the exact location of inadequate reduction on the occlusal surface. Since many clinicians like to accomplish occlusal reduction as a first step in crown preparation, this can be a problem as it is not as easy to judge the reduction when the axial surfaces have not been prepared.

The technique described here is quick and reliable and requires no special equipment or instrumentation but allows the clinician to verify adequate occlusal reduction in centric occlusion as well as excursive movements.

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### OCCLUSAL REDUCTION AND VERIFICATION TECHNIQUE

Proper occlusal reduction is accomplished by clinicians in a variety of ways with the aid of depth cuts, hemi-prep techniques, and reduction matrices. Once the initial occlusal reduction is made, a visual inspection with the teeth in centric occlusion is used to verify the result. Unfortunately, limited access intraorally can lead to a discrepancy between what actually exists and the amount of reduction the technician needs. Once the occlusal reduction is finished, the remainder of the preparation is completed (axial reduction and margination) without further verification of the occlusal reduction. Therefore, it is important to verify the reduction at the initial stage of preparation or as the last step in the process.

Using blue periphery wax (Sturgident Periphery Wax, Heraeus Kulzer LLC), a small ball of wax is



Figure 1. Various products that can be used to aid in the assessment of occlusal reduction.

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Figure 2. Through a functionally generated wax index, a periodontal probe is used to check for minimally reduced areas.



Figure 3. Through the wax index, a diamond bur is used to create a divot in the exact spot on the prepared tooth deemed to have inadequate occlusal reduction.

placed over the tooth and the patient is instructed to close together into centric occlusion. Many clinicians use this (or similar techniques) to check centric occlusion clearance. The wax is removed from the mouth to inspect for thin spots. An approximation is made regarding the area needing the reduction, and the process is repeated. An inherent problem is trying to estimate exactly where this under-reduced area is once the wax is out of the mouth.

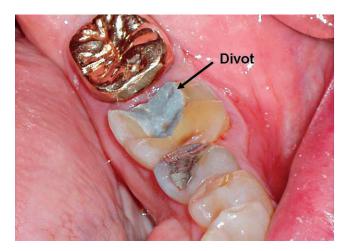


Figure 4. Wax index is removed and the area needing more reduction is clearly marked.

In the technique advocated here, after closing into the wax in centric occlusion, the patient is then guided through excursive movements, which produces a functionally generated wax index over the teeth (Figure 2). Without removing the wax from the mouth, a periodontal probe (Williams periodontal probe - Hu-Friedy) with millimeters marked in black is used to test and identify areas of minimal reduction. A rounded-end diamond burr (Brassler 856-018) that is used for occlusal reduction is used (through the wax) to create a divot approximately the depth needed for additional reduction (Figure 3). The wax is removed from the mouth, and the exact spot is identified for further reduction (Figure 4). Once accomplished, the remainder of the preparation can be completed with the assurance that excess adjustment of the restoration will not need to be done at the delivery appointment nor will the opposing dentition need to be altered.

(Accepted 8 September 2011)

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