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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 Research

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# Clinical Evaluation of Lithium Disilicate Veneers Manufactured by CAD/CAM Compared with Heat-pressed Methods: Randomized Controlled Clinical Trial

IBL Soares-Rusu • CA Villavicencio-Espinoza • NA de Oliveira • L Wang • HM Honório  
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### Clinical Relevance

Lithium disilicate veneers for esthetic restorations show great accuracy and similarity, regardless of the type of fabrication technique.

### SUMMARY

**Objectives:** This study aimed to evaluate and compare the clinical performance of two different ceramic veneer methods: CAD/CAM (IPS e.max CAD) and heat-press (IPS e.max Press) at 6 and

12 months of follow-up, and the level of patient satisfaction after treatment.

**Methods and Materials:** Patients were selected according to eligibility criteria, with a minimum of two and a maximum of six veneers per patient, for

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a total of 178 veneers randomized in two groups. A split-mouth, longitudinal, interventional, double-blind and single-center study was carried out according to the fabrication technique. Scores were attributed to the veneers according to the criteria of the United States Public Health Service (USPHS) regarding marginal adaptation, color change, marginal discoloration, restoration fracture, tooth fracture, restoration wear, antagonist tooth wear, presence of caries, and postoperative sensitivity. All patients answered a satisfaction questionnaire using the Visual Analogue Scale (VAS). Statistical significance was determined using two-way ANOVA and Tukey test, with a significance level of 5%.

**Results:** The marginal adaptation criterion showed statistical difference between periods ( $p=0.017$ ), regardless of the processing method (baseline means: CAD=1.056, PRESS=1.067, 6- to 12-month follow-up: CAD=1.089, PRESS=1.078). The other evaluated criteria showed no statistical differences between baseline and after 6 to 12 months. The level of satisfaction assessed by the VAS before and after treatment was 7.06 and 9.5, respectively.

**Conclusions:** The two methods presented similar clinical performance after 12 months, and the patient's level of satisfaction was considered high.

## INTRODUCTION

Lithium disilicate ceramics have two different initial presentations: ingots for heated-pressed fabrication and blocks for milling using CAD/CAM technology. Ingots (IPS e.max Press; Ivoclar Vivadent, Shaan, Liechtenstein) and blocks (IPS e.max CAD; Ivoclar) largely replace the IPS Empress 2 system. Ingots of leucite-reinforced ceramics for pressing present flexural strength between 120 and 180 MPa and fracture toughness between 1.03 and 1.3 MPa m<sup>1/2</sup>. The lithium disilicate ceramics have shown flexural strength (IPS e.max Press 400 MPa and IPS e.max CAD 360 MPa) and fracture toughness (between 2.8 and 3.5 MPa m<sup>1/2</sup>), and their optical properties contribute to esthetic balance.<sup>1, 2, 3, 4</sup>

Lithium disilicate ceramics provide monolithic restorations.<sup>5</sup> However, the two fabrication methods are different. The milling process prevents inaccuracies resulting from waxing, investment, and improper manipulation during injection, pickling, finishing, and polishing. This technique requires fewer finishing procedures and only surface polishing.<sup>6</sup> The injection of

the liquid material in the pressing technique may assure greater marginal flow, resulting in better adaptation of the veneers on preparations with a smaller width in the marginal area.

According to Guess and others,<sup>6</sup> and Willard and others,<sup>7</sup> the milling technique results in a material with fewer defects and more uniform distribution of the crystals. The milled ceramics' disadvantages are inferior marginal adaptation because the parameters of preparation marginal finishing, cement space, and marginal adaptation depend on each software and operator. Some studies demonstrated that pressed lithium disilicate veneers showed better marginal adaptation, thinner cement layers, and greater resistance to marginal leakage than those manufactured by a milling process.<sup>8,9</sup> However, finishing in the two methods requires compensation for customized characterizations.<sup>4</sup>

The ceramic veneer blocks available include feldspathic ceramics reinforced by leucite, lithium disilicate-based,<sup>10,3</sup> and, recently, zirconium-reinforced lithium disilicate.<sup>11</sup> Lithium disilicate blocks are manufactured in the metasilicate state, that is, 40% lithium in metasilicate crystals and vitreous matrix.<sup>12,13</sup> In this state, the material has a bluish color and can be easily milled. After the milling process, the veneer undergoes a final thermal treatment to increase the crystalline phase, resulting in lithium disilicate with the maximum optical and mechanical properties.<sup>14</sup> This crystallization process takes about 25 minutes at 830°C, and the dimensional alteration is about 0.2%, which does not affect marginal and proximal adaptation or occlusion. The physical properties of the lithium disilicate ceramics depend on different parameters, including microstructure, which plays an important role in determining the flexural strength, flexural toughness, modulus of elasticity, and optical properties.<sup>4</sup>

The long-term clinical success of feldspathic ceramics<sup>15</sup> is that of a 93.5% survival rate over 10 years.<sup>16</sup> Taking into consideration marginal adaptation and marginal discoloration, a study revealed that after seven years, only 2.5% and 4.2% of the indirect feldspathic restorations, respectively, exhibited poor adaptation and discoloration with a 97.5% rate of success.<sup>17</sup> Studies have reported the clinical success<sup>18</sup> and marginal adaptation of milled lithium disilicate veneers,<sup>19</sup> but little is known about the effect of the restorative material on the clinical behavior of the CAD/CAM system, mainly regarding the marginal adaptation of veneers.<sup>20</sup> Therefore, this study aimed to evaluate and compare the clinical performance of two different ceramic veneer fabrication methods:

CAD/CAM with CEREC inLab (IPS e.max CAD) and heat-pressed (IPS e.max Press) fabrication, at 6 and 12 months of follow-up, and the level of the patient's satisfaction after treatment. The null hypotheses were: (1) the ceramic veneers manufactured by heat-pressed and CAD/CAM methods would not have statistically significant differences in clinical performance, and (2) the two different methods would show a similar level of patient level satisfaction before and after treatment.

## METHODS AND MATERIALS

### Study Design

This split-mouth, prospective, interventional, double-blinded (patients and examiners), longitudinal, randomized controlled trial compared two factors: fabrication method – (IPS e.max CAD and IPS e.max Press) and time (baseline, 6 months, and 12 months). The veneers were randomized in pairs with the consideration of the manufacturing process. Two examiners evaluated the veneers using the modified United States Public Health Service (USPHS) method.<sup>21</sup> Prior to the study, the examiners were calibrated and Kappa agreement assessed to ensure the standardization and interpretation of the results.<sup>22</sup>

The parameters for the study design followed the Consolidated Standards of Reporting Trials (CONSORT).<sup>23</sup> This study was submitted and approved by the Institutional Review Board. All the participants were instructed about the study and signed an informed consent form.

### Selection of the Participants

The inclusion criteria were as follows: individuals of both genders, good general health, aged between 18 and 60 years, referral for veneers on the anterior teeth,<sup>24</sup> requiring at least two and at most six maxillary anterior veneers, willing to undergo radiographic examination, good maxillo-mandibular relation, occlusal stability, and being willing to sign an informed consent. The exclusion criteria was comprised of the following: smokers, individuals with large restorations on anterior teeth, dark colored teeth (shades VITA A3.5 and C4), teeth with fluorosis, teeth with periodontitis, teeth with severe gingival bleeding, poor oral hygiene, high caries rates, history of allergy to any of the materials, pregnancy, use of drugs known to interfere with the oral environment, systemic or malignant diseases, inability to be submitted to any specific techniques of the study, lack of space for the proper installation of the veneers, and presence of parafunctional habits (eg, bruxism). Selection of the participants was done according to the

CONSORT 2010 Flow Diagram (Figure 1).

According to the inclusion and exclusion criteria, 33 individuals (27 females and 6 males), aged between 18 and 52 years were selected. Each individual received at least two and at most six veneers on the anterior teeth, manufactured according to the study groups: CAD (experimental) – milling process (CAD/CAM Cerec In Lab, Sirona, Bensheim, Germany); and PRESS (control) – heat pressed process (Table 1). The total number of veneers delivered were 178.

### Treatment Planning and Tooth Preparation

Digital smile design was performed using Apple's Keynote Software to obtain veneer proportions, and to enable communication and increase predictability. Standardized extraoral photographs were taken with a Nikon D5300 digital reflex camera using the following parameters: manual mode 1/125, f22, and ISO 125, coupled with Sigma Macro 105mm DX lens; twin manual 1/1 flash (Nikon R1C1 Wireless Close-UP Speedlight System) equipped with four AA Mignon batteries (Energizer Ultimate Lithium, + AA 1,5 V, 3000 mAh); and a flash holder (Agnòs, Italy). The analysis of the face thirds was made according to the participant's smile. Initial impressions were taken using a heavy and light condensation silicone (Xantopren/Optosil, Heraeus Kulzer, Germany) one-step technique. The study casts were obtained and the wax-up was performed according to digital planning.

Intraoral photographs were taken prior to tooth preparation to select the color under polarized light, with a gray card and VITA scale. A mock-up with bisacrylic resin (Protemp 4; 3M ESPE, St. Paul, Minnesota, USA) was made to predict the final esthetic outcome (Figure 2A and 2B), checking the tooth shape and size. At the same appointment, after the participant's evaluation and approval, the teeth were prepared through the mock-up in order to guide facial and incisal reduction.

Tooth preparations were standardized, ranging from a 0.5–1.0 mm reduction on the labial surface and a 1.0 mm reduction on the incisal.<sup>25</sup> All preparations were performed by the same practitioner (IBLSR). Parallel guide grooves were made on the labial surface with a diamond bur (#4141; KG Sorensen, São Paulo, Brazil) under water irrigation and, from cervical to incisal regions (#2135; KG Sorensen), positioned at two inclinations (Figure 3A and 3B). The incisal reduction was executed following the cervical-incisal guide groove (#3053; KG Sorensen). The preparation of the labial surface was carried out (#2135; KG Sorensen) following the guiding grooves with the aid of a red pencil, the gingival margin was marked, and a retraction cord



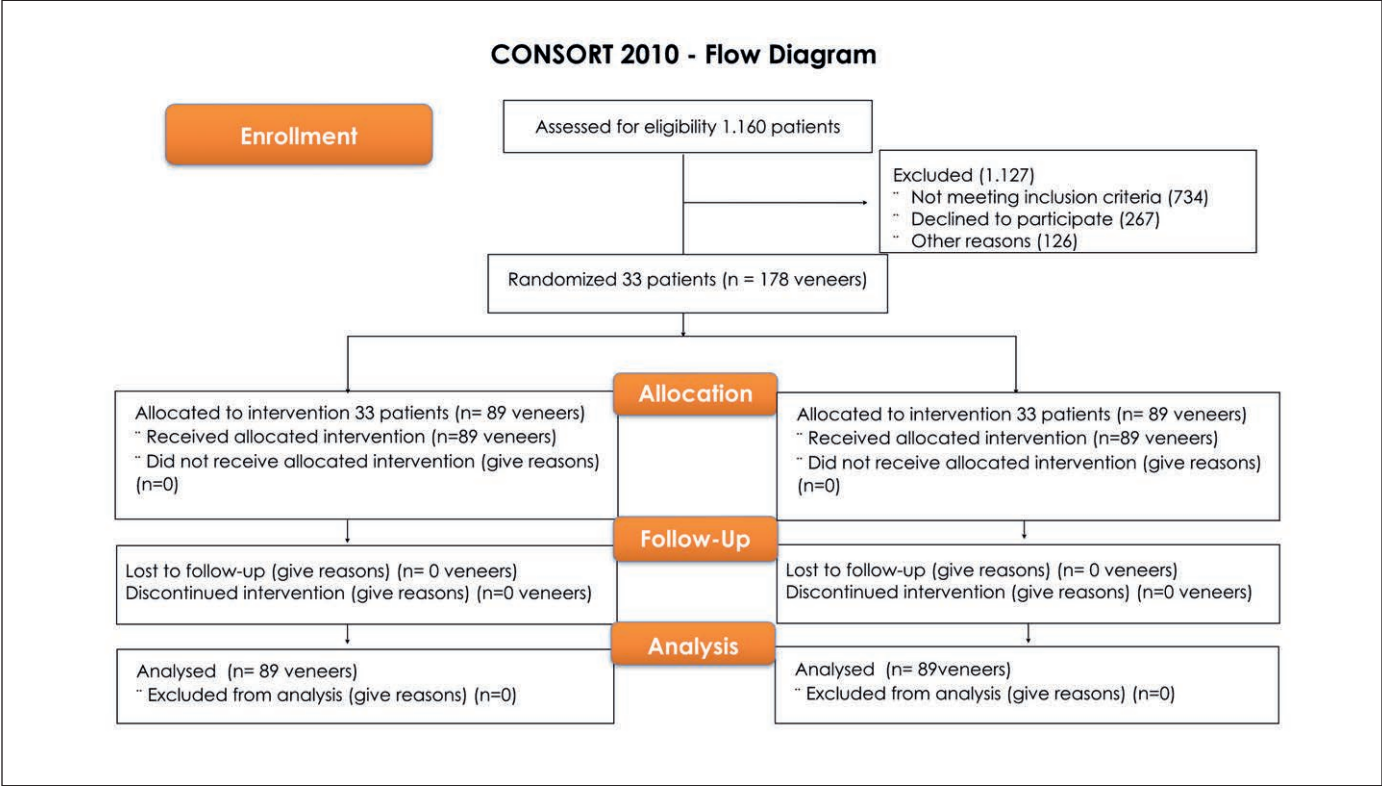


Figure 1. CONSORT 2010 Flow Diagram.

was inserted (Ultrapack 000; Ultradent, South Jordon, Utah, USA). The finishing procedure was done with a bur (#2135FF; KG Sorensen). Polishing was accomplished with Soflex discs (3M ESPE) and rubber polishing burs (Astropol, Ivoclar Vivadent) (Figure 4A). Prior to the impression, a retraction cord (Ultrapack 000; Ultradent) was inserted (Figure 4B) and the impression was performed with heavy and light body Polyvinyl siloxane (PVS) (Virtual, Ivoclar Vivadent) to

obtain the working casts. The provisional restorations were made with bisacrylic resin (Protemp 4; 3M ESPE).

**Preparation of the Veneers**

The working casts were sent to the laboratory to obtain veneers made according to the following manufacturing methods:

Table 1: Study Groups				
Groups	Ceramic	Color	Follow-up	Composition <sup>a</sup>
CAD (n=89)	Lithium disilicate	IPS e.max CAD (HT, LT)	Baseline, 6 and 12 months	Components: SiO <sub>2</sub> Other Components: Li <sub>2</sub> O, K <sub>2</sub> O, MgO, ZnO, Al <sub>2</sub> O <sub>3</sub> , P <sub>2</sub> O <sub>5</sub> , and other oxides.
PRESS (n=89)	Lithium disilicate	IPS e.max Press (HT, LT)	Baseline, 6 and 12 months	Components: SiO <sub>2</sub> Other Components: Li <sub>2</sub> O, K <sub>2</sub> O, MgO, ZnO, Al <sub>2</sub> O <sub>3</sub> , P <sub>2</sub> O <sub>5</sub> , and other oxides.
Abbreviations: Al <sub>2</sub> O <sub>3</sub> , aluminum oxide; Li <sub>2</sub> O, lithium oxide; K <sub>2</sub> O, potassium oxide; MgO, magnesium oxide; P <sub>2</sub> O <sub>5</sub> , phosphorus pentoxide; ZnO, zinc oxide. <sup>a</sup> Manufacturers' information.				



Figure 2. (A): Intraoral initial photograph. (B): Mock-up with bisacrylic resin.



Figure 3. Tooth preparation. (A): Mesial-distal guide grooves on labial surface. (B): Cervical-incisal guide grooves.

**CAD/CAM:** Titanium oxide powder was applied, and the working casts were digitally scanned (inEos Blue scanner; CEREC 3D, Sirona). The images were processed by the CEREC InLab (SW15.0) software to enable preparation analysis and veneer design. All veneers were designed with the aid of the Biocopy tool, which uses the initial wax planning as a guide (Figure 6A). Other software tools were also used to adjust tooth morphology, marginal adaptation, texture, incisal edge, and the cast position to obtain the insertion axis. The adjustments were performed on both labial and palatal surfaces (Figure 5A and 5B). After that, the ceramic block (IPS e.max CAD; Ivoclar Vivadent) information was introduced in the software to initiate milling (MXCL; Sirona). After milling, each veneer was tested on the model to check adaptation (Figure 6B) and submitted to the specific crystallization cycle in a Programat oven (Ivoclar Vivadent), following the manufacturer's instructions.

**PRESS:** The veneers were made using injectable ingots (IPS e.max Press) following the manufacturer's instructions. The press technique is based on the lost wax technique, followed by



Figure 4. (A): Tooth preparation after polishing procedure. (B): Insertion of the retraction cord for impression.

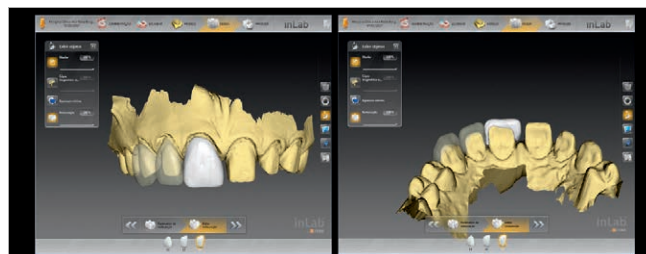


Figure 5. Software tools for marginal adjustment. (A): labial surface. (B): Palatal surface.

investing, wax elimination in an oven, press injection of the ceramic ingot in an appropriate oven, and a pickling and finishing procedure. Regardless of the manufacturing process, the veneers were tested on the working cast (Figure 7) and on the prepared tooth to verify the adaptation. The veneers were then finished with pigments and glazed.

### Cementation of the Veneers

First, the cement color was selected with the respective Variolink N Try-in resin cement (Ivoclar Vivadent). Next, the teeth were submitted to prophylaxis with pumice (Maquira; Maringá, PR, Brazil) and Robinson brush (Injecta, São Paulo, SP, Brazil), followed by absolute isolation. All teeth were acid etched with 37% phosphoric acid (Dentsply), for 30 seconds, followed by rinsing and air drying. On the etched tooth surface, a layer of the adhesive of Adper Scotchbond Multi-Purpose (3M ESPE) was applied without light-curing. In areas of exposed dentin, the Adper Scotchbond Multi-Purpose primer (3M ESPE) was applied for 15 seconds, followed by the adhesive agent application, and was air dried for 5 seconds.

The veneers' internal surfaces were prepared by conditioning with 10% hydrofluoric acid (Condac Porcelana; FGM, Joinville, SC, Brazil) for 20 seconds, followed by rinsing and air drying. On the conditioned ceramic surface, a layer of silane (Monobond Plus; Ivoclar Vivadent) was applied for 60 seconds, followed by a layer of adhesive system Adper Scotchbond



Figure 6. (A): Initial waxed working cast. (B): Checking the veneers' adaptation on the working cast.



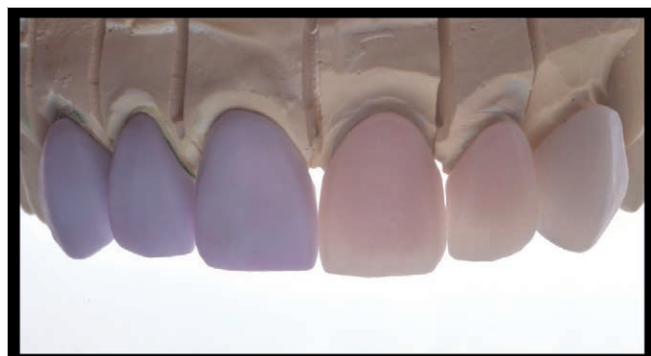


Figure 7. Test of the veneers on the working cast before and after crystallization.



Figure 9. Final photograph after cementation.

Multi-Purpose (3M ESPE). Then, the selected light-cured resin cement Variolink N (Ivoclar Vivadent) (Figure 8A) was applied on the internal surface of the veneer, and the veneer was seated with mild digital pressure on the tooth, starting with the central incisors, followed by the lateral incisors, and then the canine teeth. The cement was light-cured with a multiple-peak LED light-curing device (VALO Cordless, Ultradent) with a PointCure lens ( $\varnothing$  2.5 mm, VALO Cordless; Ultradent), to provide a localized beam on a reduced area, for 3 seconds (Figure 8B). This is the time period for the initial light-curing of the cement in the middle third. The excess resin cement was removed with a thin brush on all the veneer margins. The final light-curing (maximum power of  $1400 \text{ mW/cm}^2$ ) was performed in the labial and incisal surfaces for 60 seconds each (VALO Cordless; Ultradent). The polishing procedure was carried out 24 hours after final light-curing, with the aid of abrasive rubber polishing points (Astropol; Ivoclar Vivadent) (Figure 9).

### Clinical Assessment

The veneers were scored according to the modified USPHS method<sup>21</sup> for: marginal adaptation, color alteration, marginal discoloration, restoration fracture, tooth fracture, restoration wear, antagonist tooth

wear, presence of caries, and postoperative sensitivity (Table 2).<sup>26</sup> For the analysis of the evaluation criteria, the clinical examination performed was tactile and visual. During the examination, a clinical mirror and an exploratory probe were used to check marginal integrity, adaptation, discrepancies, stains, and surface texture of the veneers. The clinical assessments were performed by two calibrated and blinded examiners at the study periods: baseline, 6 months, and 12 months of follow-up.

*Assessment of the Patient's Level of Satisfaction* – To record the level of esthetic satisfaction, all patients were asked to answer a questionnaire with a VAS before and after treatment. This questionnaire comprised 10 questions, in which the answers were registered by a horizontal line scored from 0 (very unsatisfied) to 10 (very satisfied). The questions were:

- Are you satisfied with the aesthetics of your smile?
- Are you satisfied with the color of your teeth?
- Are you satisfied with the shape of your teeth?
- Are you satisfied with the size of your teeth?
- Regarding chewing, how do you feel?
- Regarding comfort, how do you feel?
- Regarding phonetics, how do you feel?
- Are you satisfied with the appearance of your gums?
- Are you satisfied with the shape of your lips?
- Are you satisfied with the alignment of your teeth?

*Statistical Analysis* – Values were assigned to each score as follows: Alpha=1, Bravo=2, Charlie =3. Repeated measures two-way ANOVA and Tukey test were used to compare the modified USPHS method values ( $\alpha=0.05$ ). All statistical analyses were performed with STATISTICA 10.0 software and SIGMAPLOT 12.0 software.



Figure 8. **A:** Application of the resin cement and placement of the veneer. **B:** Initial light-curing (3 seconds) with the point cure method.

Table 2. *Criteria of the Modified United States Public Health Service Method*

Topics (acronym)	Score	Criteria
Marginal adaptation (MARA)	Alpha	Margin continuity (without prominence or crack)
	Bravo	Little discontinuity detectable by explorer, but it does not require replacement
	Charlie	Prominence or crack; require replacement
Color alteration (COA)	Alpha	No color alteration close to the tooth structure
	Bravo	Little color alteration, clinically acceptable
	Charlie	Esthetically unacceptable
Marginal discoloration (MARD)	Alpha	No marginal discoloration
	Bravo	Marginal discoloration
	Charlie	Deep discoloration
Restoration fracture (RESF)	Alpha	No fracture
	Bravo	Small fracture fragments (1/4 of the restoration)
	Charlie	Severe fracture (3/4 of the restoration)
Tooth fracture (TFRA)	Alpha	No tooth fracture
	Bravo	Small fracture fragments of tooth fracture (1/4)
	Charlie	Severe tooth fracture (1/2)
Restoration wear (RESW)	Alpha	No wear
	Bravo	Wear
Antagonist tooth wear (ANTW)	Alpha	No wear
	Bravo	Wear
Caries presence (CARP)	Alfa	Absent
	Charlie	Present
Postoperative sensitivity (POSTS)	Alpha	Absent
	Charlie	Present

## RESULTS

### Clinical Assessment

At the follow-up visits, a 100% response rate was achieved; therefore, the 178 veneers placed were accounted for.

Regardless of the manufacturing process, the marginal adaptation (MARA) exhibited statistically significant greater means at the following-up periods of 6 and 12 months (CAD = 1.089; PRESS = 1.078) than at baseline (CAD = 1.056; PRESS = 1.067) ( $p=0.017$ ), without statistical difference between the groups ( $p=0.923$ ) and with interaction of group vs time periods ( $p=0.362$ ) (Figure 10).

The assessment of the restoration fracture (RESF) showed no statistically significant differences between periods ( $p=0.097$ ), groups ( $p=0.343$ ), and without the

interaction of group versus time periods ( $p=0.715$ ) (Figure 10). No statistically significant differences occurred for the postoperative sensitivity (POSTS) between periods ( $p=0.081$ ), groups ( $p=0.556$ ), and without interaction of group vs time periods ( $p=0.081$ ) (Figure 10).

Regardless of the manufacturing process and the study period, the following topics were scored as Alpha at all assessments, and no statistical analysis was possible: color alteration, marginal discoloration, tooth fracture, restoration wear, tooth antagonist wear, and caries presence.

*Assessment of the Patient's Level of Satisfaction* – All participants ( $n=33$ ) returned for the assessment and answered the questionnaire before and after treatment. The level of satisfaction before treatment was  $7.06 \pm 1.5$  and  $9.5 \pm 0.49$  after treatment (Figure 11).



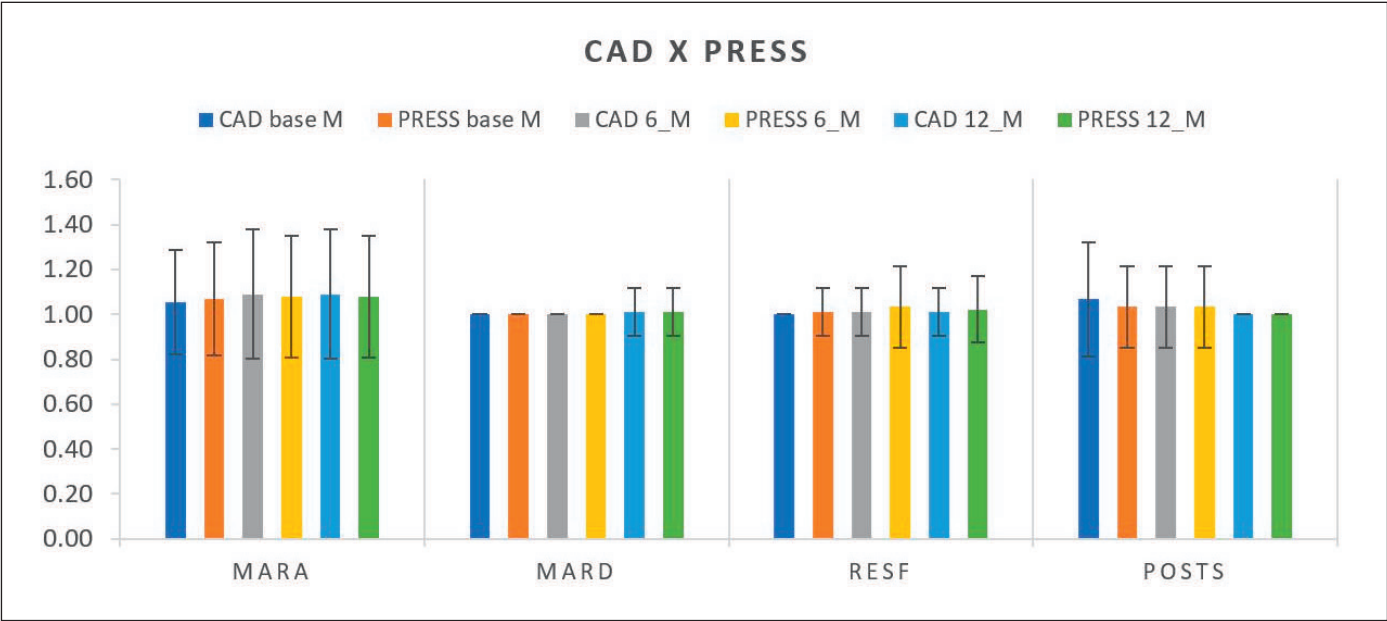


Figure 10. Means and standard deviation of the manufacturing process at the study periods (baseline, 6 months, and 12 months of follow-up) for the topics marginal adaptation (MARA), restoration fracture (RESF), and postoperative sensitivity (POSTS).

DISCUSSION

These study results revealed no statistically significant differences in the clinical performance of the two manufacturing processes (CAD/CAM milling or heat-pressed); thus, the first null hypothesis was accepted. Other laboratory studies analyzed possible differences between the manufacturing methods of lithium disilicate veneers, and they did not find statistically significant differences by reporting that both processes had marginal gap values lower than 120  $\mu$ m, which is within the clinically acceptable rate.<sup>27</sup>

According to the analysis of this study's modified USPHS data, the marginal adaptation exhibited statistically significant differences between study periods, regardless of the manufacturing process. This

agrees with a study reporting that the manufacturing process did not affect the marginal adaptation of veneers.<sup>5,6</sup> Nevertheless, recent laboratory studies showed that lithium disilicate veneers manufactured by the heat-pressed method demonstrated marginal spaces that were significantly smaller than those manufactured by a CAD/CAM system, although both results were within the clinically acceptable limits.<sup>28,29,30</sup> Further studies are necessary on marginal adaptation because this is one of the determining factors for long-term success, due to its impact on esthetics, resistance, gingival health, and caries risk.<sup>31,32</sup>

The tooth preparation type is one of the factors that can influence the marginal adaptation of the veneers. In this study, the preparation width ranged from 0.5–1.0 mm, with a flat incisal edge, and no palatal bevel. The rationale behind this choice was that incisal reductions with a palatal bevel are more prone to ceramic fracture.<sup>25</sup>

The tooth preparations used in this study were minimally invasive and were based on the literature reporting the best results of resistance to fracture of veneers, which contributes to the long-term success of the restorations.<sup>33-37</sup> We performed the tooth preparation on the mock-up because of the advantages of making the diagnosis and communication easier, by providing treatment predictability, aiming at greater control of the preparation, and resulting in enough room for the proper adaptation of the veneers. This technique is based on the final volume of the restoration and enables most of the preparation to stay in the enamel. The study

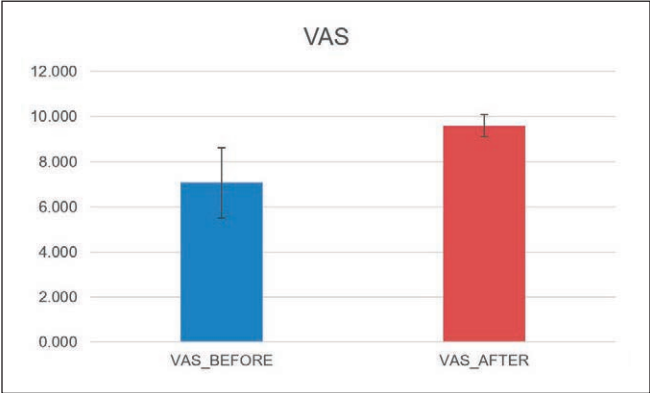


Figure 11. Mean and standard deviation of the patient's level of satisfaction.

by Gurel and others<sup>36</sup> demonstrated that this technique increases the number of restorations over enamel (80.5%) and has a high index of marginal adaptation (100%), significantly increasing the performance of the veneers and decreasing postoperative sensitivity. Knowing that the bond strength to enamel is higher than that to dentin,<sup>38</sup> marginal gaps occur due to the wear of the cement, leading to microleakage, staining, and postoperative sensitivity.<sup>15</sup> Interproximal tooth reduction was performed to enable the adjustments required to change the tooth shape and position.<sup>24</sup> This proximal surface preparation had clinical and laboratory advantages that overcome significantly the removal of the tooth structure.<sup>39</sup>

Other topics scored in this study by the modified USPHS method were color alteration, marginal discoloration, restoration fracture, tooth fracture, restoration wear, wear of the antagonist tooth, caries presence, and postoperative sensitivity, with no statistical significance between study periods and groups. Clinical studies on the applicability of lithium disilicate veneers in many clinical situations show success rates ranging from 93.5%–100% for follow-up periods from 1 to 6 years.<sup>40, 41</sup>

Although the 12-month clinical analysis of this study did not reveal any color changes of the restorations, regardless of the material, the examiners scored some marginal discoloration observed as pigmentation points on the cement line, in the cervical area, but without statistical differences between the manufacturing methods. Neither restoration fracture nor fracture of the antagonist tooth was observed. This may be justified because of the high five-year survival rate and clinical success of lithium disilicate veneers of 99.0% and 96.4%, respectively.<sup>42</sup> In this study, all participants had satisfactory oral hygiene and no caries lesions were detected.

The second null hypothesis was rejected because of the difference between the patient's level of satisfaction before and after treatment. The level of satisfaction is an important topic in clinical trials.<sup>43</sup> The VAS is the most used method for measuring tooth and facial esthetics.<sup>44</sup> All participants returned for the second assessment, and the mean values increased from 7.05 (before) to 9.5 (after treatment). This result agrees with the literature reporting high levels of satisfaction after ceramic laminates.<sup>42</sup> The reason behind the high level of esthetic satisfaction was the treatment planning process, which plays an important role in building rapport between dentists and patients, who may initially disagree about what is important and significant from an esthetic point of view.<sup>45</sup>

Considering a clinically acceptable maladaptation of 120 µm reported in laboratory studies (ISO 6872:2015) and that IPS e.max CAD had higher flexural resistance and better internal adjustment,<sup>46</sup> we consider that it is possible to obtain a satisfactory outcome of the veneers produced by a CAD/CAM system. We emphasize the important role of communication between the dentist and the laboratory technician, and performing the try-in of the veneers on the working cast in clinic prior to cementation. These steps enable the detection of possible failures in marginal adaptation, and improve the understanding about the veneers' characterization. This will consequently improve the esthetic outcome.

Regardless of the manufacturing process, the clinical success can be achieved by proper treatment planning, knowledge on proper bonding techniques, clinical and laboratory expertise, and clinical optimization and patient satisfaction.<sup>47</sup>

## CONCLUSION

The different manufacturing methods of lithium disilicate veneers (milling or pressing) had similar clinical performance after a 12-month follow-up period, with a high level of patient esthetic satisfaction.

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## Conflict of Interest

The authors have no financial interest in any of the companies or products mentioned in this article.

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# Clinical Performance of Enamel Microabrasion for Esthetic Management of Stained Dental Fluorosis Teeth

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## Clinical Relevance

Enamel microabrasion is an effective first-line esthetic treatment for the removal of tooth stains due to fluorosis, with an improvement in the appearance of teeth that is associated with a high level of patient acceptance.

## SUMMARY

**Objective:** To assess the immediate postoperative clinical efficacy of an enamel microabrasion procedure for the management of stained dental fluorosis.

**Methods and Materials:** A total of 103 maxillary and mandibular teeth exhibiting fluorosis from 21 patients assessed according to the Thylstrup-Fejerskov (TF) index were treated

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using enamel microabrasion. All teeth were subjected to enamel microabrasion using Opalustre (Ultradent Products Inc, South Jordan, UT, USA). Pretreatment and 24-hour posttreatment photographs were taken using a digital single-lens reflex camera. A visual analog scale (VAS) with scores ranging from 1 to 7 was used to assess improvement in appearance and change in brown stains followed by photographic  $\Delta E$  assessment using the CIEDE2000 formula. Patient satisfaction and tooth sensitivity were recorded on a VAS ranging from 1 to 5. Data were analyzed using parametric and nonparametric tests ( $\alpha=0.05$ ).

**Results:** There was a significant difference ( $p<0.001$ ) between the pretreatment appearance/brown stain scores and the posttreatment appearance/change in staining scores. A significant difference ( $p<0.05$ ) was noted in the posttreatment  $L^*$  and  $\Delta E$  values, and 80% of patients were satisfied with the treatment. No patients reported sensitivity.

**Conclusion:** The results of the present study show the efficacy of microabrasion for the esthetic management of stained dental fluoro-

**sis with a high level of patient acceptance and absence of tooth sensitivity. The drawback of microabrasion in the posttreatment result is influenced by the preoperative severity of the initial fluorosis.**

## INTRODUCTION

Dental fluorosis is a condition of enamel demineralization that is caused by excessive intake of fluoride. It results in white, opaque areas or discolorations ranging from yellow to brown, with or without porosities in the enamel surface.<sup>1</sup> Fluorosis staining of the anterior teeth is an esthetic problem that has been shown to have a psychological impact on the affected individuals.<sup>2</sup> Thus, conservative esthetic management of dental fluorosis not only improves the smiles but also greatly enhances the self-esteem of those afflicted.

The geological crust of India, especially South India, contains fluoride-rich minerals that can contaminate underground drinking water.<sup>3</sup> Tamil Nadu, Madurai, the district from where this article originates, is an endemic fluorosis area that has fluoride levels in drinking water of about 1.5 to 5.0 ppm.<sup>4</sup>

Dental fluorosis stain management is accomplished by three strategies: 1) removing the stained enamel, 2) bleaching the enamel surface, and 3) covering the stained enamel surface.<sup>5</sup> These strategies are done either separately or in combination.

Enamel microabrasion is method of management that removes the outer subsurface enamel as well as the entrapped stains by using a gel with hydrochloric acid.<sup>1</sup> Enamel microabrasion is the suggested first-line treatment for the management of dental fluorosis stains because it, along with stain removal, also improves the surface texture of the enamel surface.<sup>6</sup> Celik and others<sup>1</sup> showed the efficacy of enamel microabrasion in the improvement of appearance in dental fluorosis stains. They also concluded that the efficacy of microabrasion is limited by the severity of the present stains.

With the exception of a few case reports, no studies have been conducted to assess objectively the efficacy of microabrasion for the management of dental fluorosis in India.<sup>7,8</sup> The present study was planned with the primary aim to assess objectively the immediate postoperative efficacy of microabrasion for the management of dental fluorosis. Secondary objectives were to assess the factors influencing

the outcome of the treatment, patient satisfaction, and occurrence of tooth sensitivity.

## METHODS AND MATERIALS

Approval from the Ethics Committee of the home institution was obtained, and 21 patients consented to participate in the study. A sample size of 100 teeth was calculated to be sufficient to detect the clinical difference in outcome (alpha error = 0.05, power = 95%, effect size = 0.3; G power 3.1.9.2. software, Germany).<sup>9</sup> The patients (and, where appropriate, parents or guardians) were informed about the nature of the treatment, study, and photographs to be taken, and they were asked to sign an informed consent. All stained incisors and canines included in the study were managed by enamel microabrasion.

### Patient Selection

A total of 103 teeth of 21 patients (7 males and 14 females) with a mean age of  $23.9 \pm 6.21$  years were included. Maxillary and mandibular incisors and canines of these patients were evaluated using the Thylstrup-Fejerskov (TF) index (Figure 1)<sup>10</sup> by the operator and an experienced, calibrated faculty member. Questionable or normal teeth were not included in the study. The inclusion and exclusion criteria for the study were as follows.

#### Inclusion criteria:

- Patients who wanted to change the appearance of their stained teeth
- Patients with three or more stained incisors and canines
- Teeth free of caries or restorations
- Patients willing and able to attend periodic follow-up visits

#### Exclusion criteria:

- Hypersensitive teeth
- Smoking habit
- Poor oral hygiene
- Previous treatment for the stained teeth
- Any history of allergies to dental treatment

### Enamel Microabrasion

All maxillary and mandibular incisors and canines with fluorosis stains visible upon smiling, laughing, or speaking were treated in the study. All teeth were photographed initially prior to treatment using a Canon EOS Rebel T6 (Canon, Tokyo, Japan) camera under controlled lighting and at the same distance from the maxillary incisors using a tripod. The same light source, camera, and exposure settings were



TF score	
0	The normal translucency of the glossy creamy-white enamel remains after wiping and drying of the surface
1	Thin white lines are seen running across the tooth surface. Such lines are found on all parts of the surface. The lines correspond to the position of the perikymata. In some cases, a slight 'snowcapping' of the cusps/incisal edges may also be seen
2	The opaque white lines are more pronounced and frequently merge to form small cloudy areas scattered over the whole surface. 'Snowcapping' of the incisal edges and cusp tips is common
3	Merging of the white lines occurs, and cloudy areas of opacity occur spread over many parts of the surface. In between the cloudy areas, white lines can also be seen
4	The entire surface exhibits a marked opacity, or appears chalky white. Parts of the surface exposed to attrition or wear appear to be less affected
5	The entire surface is opaque, and there are round pits (focal loss of the outermost enamel) that are less than 2 mm in diameter
6	The small pits may frequently be seen merging in the opaque enamel to form bands that are less than 2 mm in vertical height. In this class are also included surfaces where cuspal and facial enamel has chipped off, and the vertical dimension of the resulting damage is less than 2 mm
7	There is a loss of the outermost enamel in irregular areas, and less than half the surface is so involved. The remaining intact enamel is opaque
8	The loss of the outermost enamel involves more than half the enamel. The remaining intact enamel is opaque
9	The loss of the major part of the outer enamel results in a change of the anatomical shape of the surface/tooth. A cervical rim of opaque enamel is often noted

Figure 1. *Thylstrup-Fejerskov (TF) index scoring.*

used for posttreatment photos. A focal length of 55mm with flash and auto white balance was used as the camera settings for all photographs. The patient position was adjusted to ensure the maxillary incisors were in the plane of focus. Ambient lighting conditions were difficult to control in the department outpatient environment, and efforts were made to standardize the lighting by excluding daylight and keeping 16 light tubes constant throughout the procedures in the examination room.<sup>11,12</sup> The teeth were kept moist with saliva and water to prevent dehydration during photographic exposures. All clinical procedures, photography, and objective color analysis were performed by the same operator.

Light-cure resin gingival barrier (SDI, Victoria, Australia) was used to protect the gingival tissues. Microabrasion was conducted using Opalustre (Ultradent Products Inc, South Jordan, UT, USA) applied onto the stained areas of the teeth. The microabrasion technique was performed according to the manufacturer's instructions. A fine-grit diamond abrasive bur was used initially for 5 to 10 seconds on the stained areas of the tooth to help the microabrasion slurry penetrate into the enamel. This was followed by drying of the teeth and application of the microabrasion slurry of approximately 1- to 3-mm thickness onto the stained regions of the teeth. The surfaces to which slurry was applied were microabraded using rubber prophyl cups (OpalCups, Ultradent Products Inc) attached to a gear reduction handpiece at a speed of approximately 4000 rpm with a slight pressure for 60 seconds. The teeth were rinsed with water between slurry applications. Depending on the stain reduction, microabrasion was repeated up to six times during the same

appointment to achieve the desired result. No more than a single syringe of microabrasion slurry was used for any one patient. Enamelast (Ultradent Products Inc), a flavored fluoride varnish sweetened with xylitol, and 5% sodium fluoride in a resin carrier were applied onto the tooth surfaces and left for three minutes. Postoperative photographs were taken 24 hours after the treatment using the same settings from before the treatment (Figure 2). All patients were instructed to use a GC Tooth Mousse (GC Corp, Tokyo, Japan) application twice daily for 20 days according to the manufacturer's instructions.

### Subjective Photographic Evaluation

The photographs were evaluated by two independent, calibrated examiners. Five pairs of pre- and posttreatment photographs were randomly selected for determining inter- and intraexaminer reliability. Posttreatment photographs were evaluated for improvement in appearance and change in brown stains using a seven-point visual analog scale (VAS; Table 1). Improvement in appearance was assessed based on the smoothness achieved as compared with the pretreatment stage. Tooth sensitivity and patient satisfaction were assessed using a five-point VAS (Table 1).

### Objective Photographic Color Analysis

Objective analysis of photographs was performed by modifying the method described by Bengel.<sup>9,10</sup> Only the maxillary incisors were evaluated, as these were in the plane of focus when the images were acquired. The pre- and posttreatment photographs were

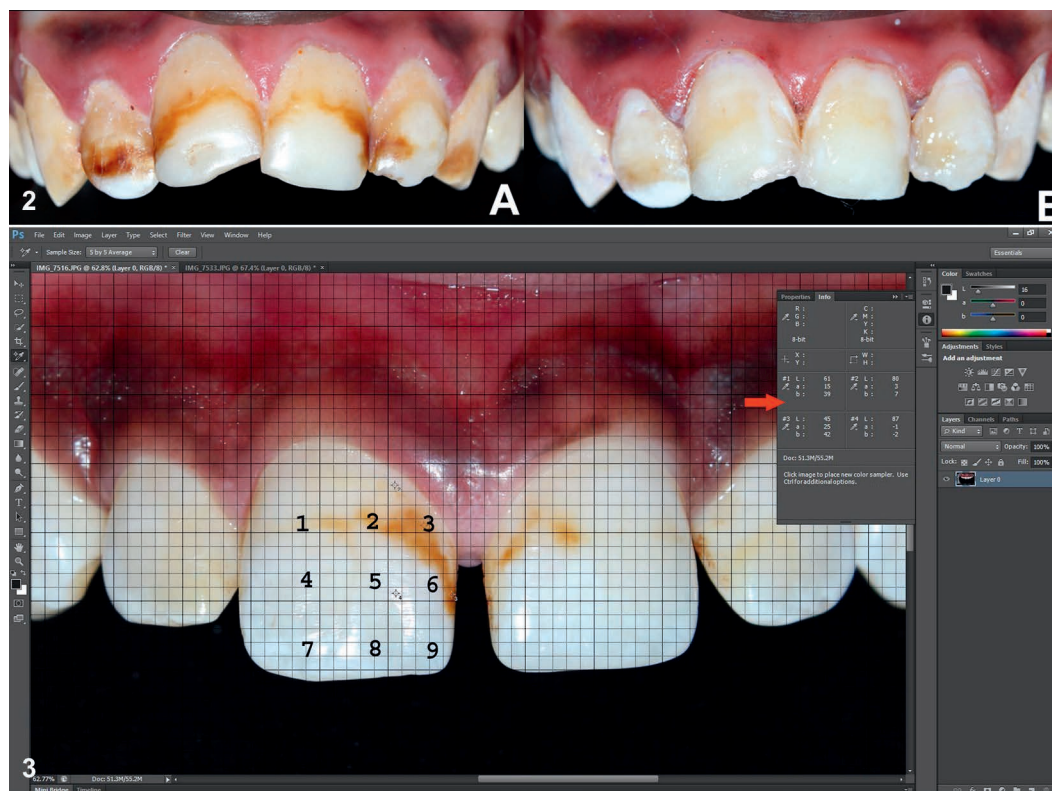


Figure 2. (A): Pretreatment photograph of fluorosed maxillary incisors TF index=2 and moderate-intensity brown stains. (B): Posttreatment photograph.

Figure 3. Screenshot of Adobe Photoshop software showing different stained points marked on the maxillary central incisor. The arrow mark shows the measured L\*a\*b\* values in subdivision 6 of the grid.

opened in Adobe Photoshop CS5 ("Ctrl + O"; Adobe Inc, San Jose, CA, USA). "View>show>grid" was used to superimpose a grid on the photographs. "Edit>preferences>guides>grids," and "slices>grid line every" were chosen, and values of 70 mm and 5 were input into the "gridline every" and "subdivision," respectively, to change the size of the grid to 70 × 70 mm. This grid size was chosen to enable the maxillary central and lateral incisors to be incorporated into 3 × 3 and 2 × 2 grids, respectively (Figure 3). Each of these grids was numbered from left to right, as shown in Figure 3. The layer panel was made visible by selecting "windows>layers," and the layers were unlocked by double-clicking the lock symbol to the right of the "background." The image was "zoomed in" using the "zoom tool (Z)" to have only the maxillary incisors in the viewing window. All photographs were analyzed using similar settings. To minimize the errors due to different ambient lighting conditions, the photographs were taken with a gray card.<sup>9</sup> From each of the grids in the incisors, two points were selected: 1) most stained and 2) an unstained or least stained in the pretreatment photographs. If a grid did not have any

staining or had no difference in stain color, the grid was not used for color measurement. From the "windows" menu, the "info" tab was selected, and the pointer was moved to the selected points to obtain the "x" and "y" coordinates and CIE L\*a\*b\* values. CIE L\*a\*b\* values were calculated using the "color sampler (I)" tool by right-clicking on selected points and choosing the "lab color" option with a dimension of 1 × 1 pixels. To estimate the ΔE values (color difference between most stained and unstained), the points selected in the pretreatment photographs were precisely relocated in the post-treatment images using the reference "x" and "y" coordinates.

The ΔE values were obtained using the CIEDE2000 formula with an online delta E calculator (<http://www.colormine.org/delta-e-calculator/Cie2000>).

### Statistical Analysis

Statistical analyses were performed using SPSS 23.0 (IBM Corp, Armonk, NY, USA). The normality of the data was assessed using the Shapiro-Wilk test, as

Table 1: Visual Analog Scales						
Pretreatment presenting appearance scores						
1 Highly roughened surface	2 Roughened surface	3	4 Moderately smooth surface	5	6	7 Evenly smooth surface
Pretreatment brown stain scores						
1 Dark intensity	2	3 Moderate intensity	4	5	6 Mild intensity	7 No brown stains
Posttreatment improvement in appearance scores						
1 No improvement	2 Mild improvement	3	4 Moderate improvement	5	6	7 Exceptional improvement
Posttreatment change in brown stain scores						
1 No change	2 Mild change	3	4 Moderate change	5	6	7 Totally removed
Tooth sensitivity scores						
0 No sensitivity	1 Mild sensitivity	2	3 Moderate sensitivity	4	5 Severe sensitivity	
Patient satisfaction scores						
1 Extremely dissatisfied	2 Dissatisfied	3 Neither satisfied or dissatisfied	4 Satisfied	5 Extremely satisfied		

data could not be assumed to be distributed normally. Therefore, improvement in appearance and change in brown stains were tested using the Wilcoxon signed-rank test. The influence of the TF index score and tooth type on the improvement in appearance and change in brown stains was assessed using the Kruskal-Wallis test. The  $L^*$  color values followed a normal distribution, and differences in  $L^*$  values were analyzed with a paired  $t$ -test. The  $\Delta E$  (color differences) between pre- and post-treatment images was compared using the Wilcoxon signed-rank test. The  $\Delta E$  differences between the maxillary central and lateral incisors were assessed using the Kruskal-Wallis test. The Friedman test compared the posttreatment  $\Delta E$  differences between the subdivisions in the grid. For all tests, the probability level for statistical significance was at  $\alpha=0.05$ .

## RESULTS

### Subjective Photographic Evaluation

Cohen's kappa statistic values were 0.80 and 0.79, respectively, for inter- and intra-examiner agreement. Table 2 describes the distribution of tooth type; TF index scores; means for presenting appearance, brown stain scores, and improvement in appearance; and change in brown stain scores. There was a significant difference ( $p=0.00$ ; Wilcoxon signed-rank test) between the presenting appear-

ance/brown stain scores and the posttreatment improvement in appearance/change in brown stain scores (Table 2). As the TF index scores increased, there was a significant decrease ( $p<0.001$  and  $0.007$ , respectively; Kruskal-Wallis test) in posttreatment improvement in appearance and change in brown stain scores (Table 2). Table 3 details the significant ( $p<0.001$ ; Kruskal-Wallis) difference in posttreatment scores in comparison with the pretreatment scores of presenting appearance and brown stains. With higher pretreatment scores, the posttreatment performance of microabrasion also significantly improved.

Table 4 shows the patient satisfaction scores and sensitivity incidence. Of the patients, 80% (17 patients) were either satisfied or extremely satisfied with the treatment outcome. None of the patients in the present study experienced tooth sensitivity during the procedure. No significant association (Kruskal-Wallis test) was evident between the patient treatment satisfaction and the number of teeth treated, presenting appearance, brown stains, improvement in appearance, or change in stain scores.

### Objective Photographic Analysis

Of the total 103 teeth, 78 maxillary central and lateral incisors were assessed for CIE  $L^*a^*b^*$  values. Table 5 presents the  $L^*$  values of the stained points



Table 2: Distribution of Tooth Type and Visual Analog Scores for Different TF Index Scores

	TF Index <sup>A</sup>										Total Teeth		
	1		2		3		5		6			7	
	Count	Mean ± SD	Count	Mean ± SD	Count	Mean ± SD	Count	Mean ± SD	Count	Mean ± SD		Count	Mean ± SD
Tooth type													
Maxillary central incisor	18		7		11		1		0		2		20
Maxillary lateral incisor	17		9		8		3		2		0		19
Maxillary canine	9		0		2		5		0		0		20
Mandibular central incisor	1		2		0		0		1		0		19
Mandibular lateral incisor	1		1		0		1		0		1		8
Mandibular canine	1		0		0		0		0		0		9
Total	47		19		21		10		3		3		103
Presenting appearance		5.55 ± 0.73 <sup>B</sup>		4.87 ± 0.81 <sup>B</sup>		4.79 ± 0.73 <sup>B</sup>		4.05 ± 0.50 <sup>B</sup>		4.00 ± 1.00 <sup>B</sup>		4.17 ± 0.29 <sup>B</sup>	
Brown stain score		5.40 ± 0.62 <sup>C</sup>		4.89 ± 0.86 <sup>C</sup>		5.24 ± 1.22 <sup>C</sup>		4.40 ± 0.70 <sup>C</sup>		4.17 ± 2.02 <sup>C</sup>		4.83 ± 0.76 <sup>C</sup>	
Improvement appearance		6.21 ± 0.52 <sup>a,b</sup>		6.11 ± 0.68 <sup>a,b</sup>		6.21 ± 0.46 <sup>a,b</sup>		5.45 ± 0.37 <sup>a,b</sup>		5.50 ± 0.50 <sup>a,b</sup>		5.17 ± 0.29 <sup>a,b</sup>	
Change in brown stains		6.44 ± 0.40 <sup>a,c</sup>		6.50 ± 0.44 <sup>a,c</sup>		6.43 ± 0.46 <sup>a,c</sup>		5.95 ± 0.55 <sup>a,c</sup>		5.50 ± 1.00 <sup>a,c</sup>		5.67 ± 0.58 <sup>a,c</sup>	
Abbreviation: TF index, Thystrup-Fejerskov index.													
<sup>Aa</sup> Kruskal-Wallis test results showed a significant association (p<0.001 and 0.007, respectively) between the TF index scores and improvement in appearance and change in brown stain scores.													
<sup>BbC,c</sup> Wilcoxon signed-rank test results showed a significant difference (p<0.001) in scores between presenting appearance, brown stain scores to improvement in appearance, and change in brown stain scores.													

Abbreviation: TF index, Thylstrup-Fejerskov index.

<sup>Aa</sup> Kruskal-Wallis test results showed a significant association ( $p < 0.001$  and  $0.007$ , respectively) between the TF index scores and improvement in appearance and change in brown stain scores.

<sup>BbC,c</sup> Wilcoxon signed-rank test results showed a significant difference ( $p < 0.001$ ) in scores between presenting appearance, brown stain scores to improvement in appearance, and change in brown stain scores.

in pre- and posttreatment stages from each subdivision of the grid. Paired  $t$ -test showed a significant difference ( $p < 0.05$ ) between the pre- and posttreatment  $L^*$  values of the stained areas in all of the subdivisions except for subdivision 8. This subdivision was the least evaluated in the present study (only 9 teeth). The color difference ( $\Delta E$ ) between the stained and unstained areas for pre- and posttreatment stages in each of the subdivisions showed a significant difference ( $p < 0.05$ ) using the Wilcoxon signed-rank test (Table 6). Mean pre- and posttreatment  $\Delta E$  values are depicted in Table 6. Subdivisions 7, 8, and 9 had higher posttreatment  $\Delta E$  values. Between the maxillary central and lateral incisors,

there was no significant difference in  $\Delta E$  values (Kruskal-Wallis test). The Friedman test did not show any difference in  $\Delta E$  values between the different subdivisions of the grid for each tooth.

## DISCUSSION

In the present investigation, the VAS used was similar to the one used by the Celik group.<sup>1,13</sup> Subjective color evaluations were followed up with objective color change assessment using imaging software (Adobe Photoshop CS5). The TF index scoring criteria were used to classify the fluorosed teeth, as they are based on the histopathological features of the degree of subsurface enamel porosity in dental fluorosis and are more precise in recording the early signs as well as severe grades of fluorosis.<sup>14</sup>

Enamel microabrasion was repeated a maximum of six times per tooth to achieve the desired outcome.

Table 3: Mean Posttreatment Improvement in Appearance and Brown Stain Scores in Comparison With Pretreatment Scores

Pretreatment Present Appearance	Posttreatment Improvement in Appearance Scores, Mean $\pm$ SD	n
Moderately smooth	5.8284 $\pm$ 0.53337 <sup>a</sup>	67
Evenly smooth	6.5139 $\pm$ 0.40508 <sup>a</sup>	36
Brown stains		
Dark intensity	4.5000 $\pm$ 0 <sup>b</sup>	1
Moderate intensity	6.1917 $\pm$ 0.47916 <sup>b</sup>	60
Mild intensity	6.5789 $\pm$ 0.33944 <sup>b</sup>	38
No brown stains	7.0000 $\pm$ 0 <sup>b</sup>	4

<sup>a</sup> Kruskal-Wallis test results showed a significant difference ( $p < 0.001$ ) in posttreatment scores in comparison with the pretreatment scores.

<sup>b</sup> Kruskal-Wallis test results showed a significant difference ( $p < 0.001$ ) between the posttreatment change in stain score in comparison with pretreatment brown stain score.

Table 4: Patient Satisfaction and Tooth Sensitivity Scores

	Count n	%	Mean
Patient satisfaction			
Dissatisfied	1	4.8	
Neither satisfied or dissatisfied	3	14.3	
Satisfied	13	61.9	
Extremely satisfied	4	19.0	
Patient satisfaction score			3.95
Tooth sensitivity			
No sensitivity	21	100.0	

	Mean	Minimum	Maximum	SD	p-Value (Paired t-test)
Pretreatment L*1 a	71.66	46.00	88.00	10.06	
Posttreatment L*1 A	82.70	65.00	94.00	6.55	0.000
Pretreatment L*2 b	75.11	46.00	89.00	10.33	
Posttreatment L*2 B	85.78	67.00	98.00	5.90	0.000
Pretreatment L*3 c	68.43	26.00	90.00	12.80	
Posttreatment L*3 C	81.19	18.54	94.00	11.65	0.000
Pretreatment L*4 d	67.10	21.00	87.00	14.76	
Posttreatment L*4 D	82.55	62.00	97.00	8.24	0.000
Pretreatment L*5 e	81.69	67.00	93.00	7.86	
Posttreatment L*5 E	89.55	80.00	96.00	4.38	0.001
Pretreatment L*6 f	67.81	30.00	84.00	13.43	
Posttreatment L*6 F	83.26	64.00	97.00	7.37	0.013
Pretreatment L*7 g	66.80	41.00	86.00	14.51	
Posttreatment L*7 G	81.40	63.00	97.00	9.30	0.001
Pretreatment L*8 h	76.56	67.00	87.00	6.31	
Posttreatment L*8 h	88.33	81.00	96.00	4.95	0.086
Pretreatment L*9 i	62.67	31.00	85.00	15.25	
Posttreatment L*9 I	76.83	52.00	91.00	11.34	0.001

<sup>a</sup> A difference in letter case (lowercase vs uppercase) indicates a statistically significant difference, and the same letter case represents no statistically significant difference. Similar letters denote paired t-tests performed between these two groups.

Sundfeld and others showed that microabrasion application for a maximum of five to 10 times results in enamel removal of up to 10 to 200  $\mu\text{m}$ , which is clinically acceptable.<sup>15</sup> Only one syringe of Opalustre was used for each patient, and this was done to observe what level of posttreatment change a single syringe can bring for a patient. None of the patients in the current study required more than one syringe. In agreement with the previous investigation by Celik and others,<sup>1</sup> the results of the current trial show that the posttreatment scores for enamel microabrasion were better when the severity of fluorosis was mild and also when the smoothness of enamel and brown stain scores were less severe. As explained by Celik and others,<sup>1</sup> the brown stains in fluorosed teeth are acquired external stains, and the depth to which these stains have penetrated is amenable to microabrasion removal, as seen in the significant change in post-treatment scores of the present study. Only one patient in the present study had a severe form of fluorosis with loss of enamel surface (TF index score=7). This patient required a composite restoration but was satisfied with the outcome of enamel microabrasion. Patients' acceptance or satisfaction with an esthetic procedure is highly subjective and depends on their social, cultural, and economical background. Nearly 80% of patients in the present study were satisfied with the treatment outcome, and none of the patients opted for any further

improvement by any other means. Furthermore, no patients in the present observation reported any tooth sensitivity, supporting the safety of the procedure.

A literature search yielded numerous case reports on the efficacy of microabrasion in fluorosed teeth and recommended this method as an effective and minimally invasive procedure.<sup>16-19</sup> Only very few clinical studies have evaluated the color change achieved with microabrasion in stained fluorosis teeth.<sup>1,13</sup> Adobe Photoshop CD5 software was used for CIE L\*a\*b\* assessment, which is similar to other reports evaluating color change in treated fluorosed teeth.<sup>12,20</sup> Objective color evaluation results revealed that the L\* value increased significantly in all areas of the tooth except in subdivision 8. This might be because this subdivision was the least assessed in the present report. On average, the posttreatment L\* value of stained points increased by 10 values in all of the areas of the tooth. Pretreatment  $\Delta E$  values between the stained and unstained points on the tooth were significantly halved after microabrasion mean  $\Delta E \leq 3.7$  units is considered to be a clinically acceptable color match in the oral cavity.<sup>21-23</sup> Despite the fact that the postmicroabrasion mean  $\Delta E$  was  $>3.7$  units for all areas of the teeth assessed, none of the patients in the present report opted for any further intervention. As stated earlier, the esthetic

Table 6: Pre- and Posttreatment Mean  $\Delta E$  Values for Maxillary Incisors Assessed in Each Subdivision and Compared With Wilcoxon Signed-Rank Test<sup>a</sup>

	Tooth Type, Mean $\pm$ SD		Mean $\pm$ SD	p-Value (Wilcoxon Signed-Rank Test)
	Maxillary central incisor (n=40)	Maxillary Lateral Incisor (n=38)		
Pretreatment $\Delta E$ 1 a	12.08 $\pm$ 5.59	16.01 $\pm$ 8.74	14.24 $\pm$ 7.69	0.000
Posttreatment $\Delta E$ 1 A	8.45 $\pm$ 4.58	8.57 $\pm$ 5.02	8.52 $\pm$ 4.79	
Pretreatment $\Delta E$ 2 b	11.56 $\pm$ 6.00	15.72 $\pm$ 9.26	13.67 $\pm$ 8.04	0.000
Posttreatment $\Delta E$ 2 B	6.85 $\pm$ 4.23	6.83 $\pm$ 3.54	6.84 $\pm$ 3.86	
Pretreatment $\Delta E$ 3 c	16.91 $\pm$ 8.68	22.81 $\pm$ 14.56	20.28 $\pm$ 12.63	0.000
Posttreatment $\Delta E$ 3 C	7.84 $\pm$ 4.50	11.36 $\pm$ 8.99	9.79 $\pm$ 7.49	
Pretreatment $\Delta E$ 4 d	22.16 $\pm$ 13.79	21.79 $\pm$ 14.93	21.98 $\pm$ 14.23	0.000
Posttreatment $\Delta E$ 4 D	10.22 $\pm$ 6.18	9.02 $\pm$ 5.84	9.66 $\pm$ 6.00	
Pretreatment $\Delta E$ 5 e	14.29 $\pm$ 10.08	—	14.29 $\pm$ 10.08	0.000
Posttreatment $\Delta E$ 5 E	7.01 $\pm$ 3.98	—	7.01 $\pm$ 3.98	
Pretreatment $\Delta E$ 6 f	20.54 $\pm$ 10.84	—	20.54 $\pm$ 10.84	0.000
Posttreatment $\Delta E$ 6 F	9.04 $\pm$ 6.12	—	9.04 $\pm$ 6.12	
Pretreatment $\Delta E$ 7 g	26.18 $\pm$ 11.62	—	26.18 $\pm$ 11.62	0.000
Posttreatment $\Delta E$ 7 G	12.40 $\pm$ 5.72	—	12.40 $\pm$ 5.72	
Pretreatment $\Delta E$ 8 h	21.24 $\pm$ 11.52	—	21.24 $\pm$ 11.52	0.008
Posttreatment $\Delta E$ 8 H	11.18 $\pm$ 7.48	—	11.18 $\pm$ 7.48	
Pretreatment $\Delta E$ 9 i	28.12 $\pm$ 15.18	—	28.12 $\pm$ 15.18	0.004
Posttreatment $\Delta E$ 9 I	15.98 $\pm$ 9.96	—	15.98 $\pm$ 9.96	

<sup>a</sup> A difference in letter case (lowercase vs uppercase) indicates a statistically significant difference; a similar letter case denotes Wilcoxon signed-rank test performed between these two groups.

acceptance of tooth color is dependent on a patient's social, cultural, and economic background, and the current postmicroabrasion mean  $\Delta E$  of  $>3.7$  units was acceptable for patients in this study. No direct comparison of the amount of color change was possible, as a literature search indicated no objectively evaluated data following microabrasion in stained fluorosed teeth.<sup>1,13</sup>

The significant improvement in posttreatment appearance and brown stain scores obtained by microabrasion in the present investigation demonstrates the efficacy of this treatment in the esthetic management of stained dental fluorosis. Results of the current study show that even though the performance of enamel microabrasion is limited by the severity of fluorosis, the pretreatment smoothness of the enamel, and the intensity of brown stains, this procedure is an effective first treatment choice to improve esthetics and is a minimally invasive procedure that, when supplemented with other options such as vital-tooth bleaching, can still enhance the success of treatment. To achieve  $\Delta E$  values  $<3.7$  units, microabrasion procedures should be followed up with bleaching and resin infiltration, as suggested in various case reports.<sup>24-26</sup> The objective evaluation after bleaching and resin infiltration in

microabraded teeth should be reported in future studies.

The patients' acceptance of this treatment in the current work shows that in a district such as Madurai, an area with endemic fluorosis, microabrasion can be an economically feasible first-line treatment option compared with other esthetic procedures for the management of stained fluorosed teeth. Clinicians should be informed about the effectiveness and limitations of this procedure for the management of stained fluorosed teeth and also about the patients' perception regarding the treatment outcome.

Long-term follow-up of the patients included in the present study is planned to evaluate the stability of change produced by microabrasion. Furthermore, the authors have requested sponsorship by dental products manufacturers for further treatment of these patients with home bleaching and resin infiltration and objective assessment of the color change following these procedures.

## CONCLUSIONS

The results of the present study show the efficacy of microabrasion for the esthetic management of



stained dental fluorosis teeth, with a high level of patient acceptance and no tooth sensitivity. Thus, microabrasion, a minimally invasive esthetic procedure, should be the first line of treatment in improving the appearance of the smile in patients with stained fluorosed teeth.

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#### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the CSI College of Dental Sciences. The approval code issued for this study is CSIDSR/12/2018.

#### Conflict of Interest

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

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# Long-term Clinical Performance of Composite Resin or Ceramic Inlays, Onlays, and Overlays: A Systematic Review and Meta-analysis

J Fan • Y Xu • L Si • X Li • B Fu • M Hannig

## Clinical Relevance

Composite resin or ceramic inlays, onlays, and overlays can achieve high long-term survival and success rates.

## SUMMARY

**Objective:** This study evaluated the long-term clinical performance and complications of composite resin or ceramic inlays, onlays, and overlays, as well as identified the factors that might influence the clinical outcome of the restorations.

**Method:** A systematic literature search was conducted in the Pubmed, Embase, Cochrane Central Register of Controlled Trials, and Web of Science databases until April 30, 2019, without language restrictions. Randomized clinical trials, clinical retrospective, and prospective cohort studies with a mean follow-up period of five years

were included. Two reviewers extracted the study data independently. Newcastle-Ottawa Scale was applied for quality assessment. Meta-analysis was performed by the random-effects model and fixed-effects model.

**Results:** After removal of duplicates, 2818 studies were identified. Finally, 13 observational studies were included in the meta-analysis based on retrospective and prospective cohort studies. The cumulative survival rate and success rate of composite resin inlays, onlays, and overlays were 91% and 84% after five years of follow-up, respectively. The survival rates of ceramic inlays and onlays were 90% at 5 years, 89% at 8 years

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and 85% at 10 years, while the success rates of ceramic inlays and onlays were 88% at 5 years and 77% at 10 years. Secondary caries and endodontic complications were the predominant failures for composite resin inlays, onlays, and overlays, while restoration fractures and endodontic complications were the main failures for ceramic inlays and onlays. No direct association between parafunctional habits and bruxism and the fractures of restorations was found. Nonvital teeth and multiple-surface restorations tended to increase the risk of failure. Regarding other factors influencing the clinical outcome, no definite conclusion could be drawn due to inconsistent results.

**Conclusions:** The long-term clinical outcomes have been demonstrated to achieve high survival and success rates based on 10-year data for ceramic inlays and onlays, as well as 5-year data for resin inlays, onlays, and overlays.

## INTRODUCTION

With the rapid development of the dental bonding technology by means of micro- and nanomechanical interlocking, indirect adhesive restorations for posterior teeth have been widely used in contemporary restorative dentistry.<sup>1</sup> Most common types of posterior indirect adhesive restorations include inlays, onlays, and overlays.<sup>2-4</sup> Numerous clinical trials have demonstrated that the preservation of sound tooth structure is an important factor for the durability of the restorations.<sup>5-10</sup>

An inlay is a dental restoration without cusp coverage and made outside of the oral cavity to correspond to the form of the prepared cavity and tooth morphology, which is cemented or adhesively bonded into the tooth (Figure 1A).<sup>11</sup> Inlays could be used for restorations of teeth having medium- to large-size class-II cavities with well-preserved buccal and lingual walls.<sup>12</sup> Christensen<sup>13</sup> recommended the application of inlays when the width of the isthmus is confined within one-third to half of the distance between the buccal cusp tip and lingual cusp tip. An onlay is a partial coverage restoration of a tooth that restores one or more cusps as well as the partial or entire occlusal surface, which is retained by conventional and resin cements (Figure 1B and 1C).<sup>11</sup> Christensen<sup>13</sup> suggested the use of onlays when the width of the isthmus is larger than half the distance from buccal cusp tip to lingual cusp tip and/or when a weak cusp exists. An overlay, a special type of an onlay with entire cusp coverage, is an adhesively bonded restoration (Figure 1C and 1D).<sup>14</sup> Ferraris<sup>1</sup> suggested the main indications of onlays and

overlays are as follows: (1) medium- to large-size cavities with one or more cusps missing; (2) morphological modification of the occlusal surface and/or an increase of the occlusal vertical dimension for full-mouth oral rehabilitations rather than aggressive interventions such as full-crown restorations; and (3) seeking preservation of pulp vitality and minimal invasive intervention of the cracked tooth. Besides traditional restorations such as inlays, onlays, and overlays, newer adhesive restorations such as additional overlays, occlusal veneers, overlay veneers, long-wrap overlays, and adhesive crowns are being increasingly applied with the development of adhesive dentistry.<sup>12</sup>

Compared with direct filling restorations, indirect restorations possess the advantages of easily restoring occlusal morphology and proximal contacts as well as reducing polymerization shrinkage.<sup>15</sup> Although crown or post-and-core crown restorations have achieved reliable results, crown preparations need to remove a large amount of sound tooth structure.<sup>16</sup> Recently, Vagropoulou and others<sup>17</sup> reported that the five-year survival rates for crowns, inlays, and onlays were more than 90%. Li and others<sup>18</sup> reported that the success and survival rates of the mildly defective endodontically treated premolars were 96.3% and 98.1%, respectively (after three years of restorations with quartz fiber posts and crowns), while the success and survival rates of severely defective nonvital premolars were 88.5% and 96.2%, respectively. However, the success and survival rates of the mildly defective nonvital premolars were both 96.6% after three years of restorations with ceramic onlays, while those of the severely defective nonvital premolars were 94.1% and 100%.<sup>18</sup> Though no significant difference was found, the ceramic onlays seemed to reveal higher success rates to restore medium or severe defects of endodontically treated premolars as a result of fewer debonding failures.<sup>18</sup>

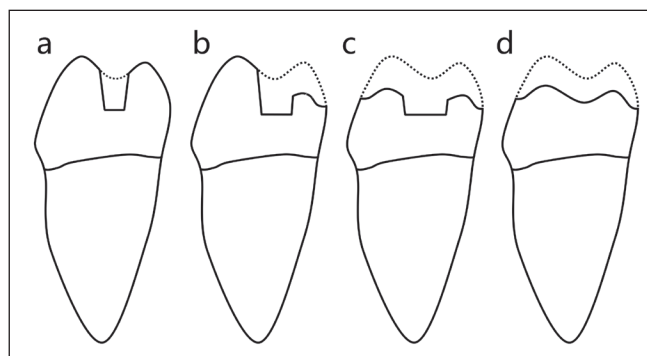


Figure 1. Types of restorations: inlay (A), onlay (B, C), overlay (C, D). Conventional cement and resin cement can be used for inlays and onlays. Overlays should be adhesively bonded.

Considering the esthetic demand, inlays, onlays, and overlays are often made of tooth-colored materials such as composite resins and ceramics.<sup>19</sup> Composite resins are composed of a resinous matrix and reinforced fillers with different sizes of particles.<sup>20</sup> Great improvements of the mechanical properties of tooth-colored composite resins enable their applications to restore large-size cavities using indirect restorations.<sup>21</sup> Compared with composite resins, ceramic restorations possess higher wear resistance and compressive forces.<sup>22, 23</sup> Nevertheless, ceramics are brittle and more prone to fracture under tensile stresses than composite resins. Based on the data of two randomized clinical trials (RCTs), Fron Chabouis and others<sup>24</sup> concluded that indirect ceramic restorations performed better than indirect composite resin restorations within the first six months, but this may not be valid in long-term clinical service. Furthermore, composite resin inlay restorations reveal survival rates of 79.3% to 92.0% after 5 to 10 years in clinical service,<sup>25-30</sup> while the survival rates of ceramic inlay restorations have been reported in a wide range from 51.4% to 96.0% after 5 to 15 years of follow-up periods.<sup>10,28,31-38</sup> Mangani et al<sup>39</sup> reported that the weighted average success rate of composite resin inlays was 92.8% after 2.6 years of the mean observation period, while the success rate of ceramic restorations reached 96.3% after 5.9 years of the mean observation period. This data demonstrated that indirect restorations functioned excellently in the treatment of both Class I and II cavities in posterior teeth.<sup>39</sup> Recently, Morimoto and others<sup>40</sup> reported that ceramic inlays, onlays, and overlays performed very well after long-term clinical service, although data greater than five years on composite inlays, onlays, and overlays is lacking.

With the increasing use of indirect aesthetic restorations, the systematic review of the long-term clinical performance of composite resin and ceramic restorations such as inlays, onlays, and overlays needs to be updated. The aim of the present review and meta-analysis was to (1) systematically evaluate the cumulative survival and success rates of composite resin or ceramic inlays, onlays, and overlays after 5-, 8-, and 10-year follow-up periods; (2) analyze the main complications of failures; and (3) identify the factors that may influence the clinical outcome of restorations based on RCTs and observational studies with five years of a mean follow-up period.

## METHODS AND MATERIALS

The systematic review protocol was registered at the PROSPERO database under #CRD42018100783 and carried out in accordance with the Preferred Reporting

Items for Systematic Reviews and Meta-Analyses statement protocol.<sup>41</sup> Survival indicates that restorations are considered to be clinically acceptable according to the clinical criteria during the follow-up period. Once the restoration is debonded, it is considered as a failure, regardless of whether the restorations can be rebonded or not. Success indicates that restorations function well without any complications and don't need any clinical interventions during the follow-up period.

## Eligibility Criteria

The search strategy conducted for the systematic review and meta-analysis was based on the following elements:

- P (population): Population included permanent posterior teeth restored with ceramic or composite resin inlays, onlays, and overlays.
- I (intervention): Intervention indicated that patients received the treatments of ceramic or composite resin inlays, onlays, and overlays.
- O (outcome): Outcome included the survival and success rates of ceramic or composite resin inlays, onlays, and overlays, analysis of the biological and mechanical complications, as well as identification of the factors that may influence the survival rates of composite resin or ceramic restorations.
- S (study): Study designs included RCTs, clinical retrospective and prospective cohort studies.

Follow-up period: The period of mean clinical observation was at least five years.

## Exclusion Criteria

1. Case reports, reviews, protocols, letters, laboratory studies, animal studies, and meeting abstracts.
2. Studies that did not report dropout rate, survival rate, and complete data or incongruous data for analysis.
3. Studies that had dropout rates of restorations higher than 30% during 5 years of follow-up.
4. Studies that had sample sizes either less than 30 restorations or less than 15 patients.
5. Studies that did not define the clinical evaluation criteria.
6. In the case that two or more studies involved the same population, only the most recent one or complete one was included.

## Information Sources and Search Strategy

The electronic databases including Pubmed, Embase, Cochrane Central Register of Controlled Trials and

Web of Science were searched until April 30, 2019. We also manually identified unpublished and ongoing clinical trials related to the topic of review on the website ClinicalTrials.gov ([www.clinicaltrials.gov](http://www.clinicaltrials.gov)). The literature search strategy was employed using Mesh terms and keywords. Details are summarized in Supplementary Table 1.

The relevant articles were imported into Endnote X7 software to eliminate duplicates. Two reviewers independently assessed all titles and abstracts. The potential articles and abstracts without sufficient information were screened via reading full-text.

### Data Extraction and Data Items

Two reviewers independently extracted the necessary data from the selected papers. For each identified study, the following items were obtained by two reviewers: authors, materials, country, evaluation criteria, follow-up period, setting/operator, ages (means), number of patients, number of restorations, dropout rate, study type, survival rate, success rate and score. Any disagreements were resolved by discussion and data rechecking. A third examiner was invited to check the process and settle discrepancies when the two reviewers did not agree.

### Quality Assessment

The quality analysis of the identified observational study was conducted independently by two reviewers using the Newcastle-Ottawa Scale (NOS).<sup>42</sup> The studies were dichotomized into high quality and low quality according to the aspects of the quality of selection, comparability and outcome. A study scoring no less than 6 was considered to be of high methodological quality, while a study scoring less than 6 was considered to be low quality. Any disagreement between two reviewers was resolved by discussion.

### Measures and Statistical Analysis

The data concerning the clinical performance of composite resin or ceramic inlays, onlays and overlays with a mean follow-up period ranging from 5 to 10 years were assessed. Descriptive and statistical analyses were performed to estimate survival rates, success rates and complication rates. Survival rates, success rates and complication rates were calculated through logit transformation. Heterogeneity was analyzed with inconsistency index ( $I^2$ ) statistic and Q statistic. The random-effects model was adopted when heterogeneity of the eligible studies was obvious ( $I^2 > 50\%$ ); the fixed-effects model was used when heterogeneity was not significant ( $I^2 < 50\%$ ). Subgroup analysis was employed

to explain the source of heterogeneity. Funnel plots were used to explore the bias of publication. All analysis was performed using R software version 3.4.0 and the Meta package (R Foundation, Vienna, Austria).

## RESULTS

### Literature Search

Initial searches using Mesh terms and keywords obtained a total of 4757 articles in the aforementioned databases. No additional study was added by manual search, and 1939 studies were eliminated due to duplicates. After the titles and abstracts were screened, 2640 articles were excluded and 178 were considered for full-text evaluation. Finally, 165 studies were ruled out, and 13 studies<sup>28-31,33,36,43-49</sup> were adopted for meta-analysis in this study. The screening process of the literature is summarized in Figure 2.

### Characteristics of the Studies

The main characteristics of the included studies are presented in Table 1. Thirteen studies adopted in this study were retrospective or prospective cohort studies with 5 to 10 years of follow-up. Nine studies investigated ceramic restorations, and three studies investigated composite resin restorations. Only one study involved both ceramic and composite resin restorations. The earliest data in this study were published in 2000,<sup>31,43,44</sup> and the most recent one was in 2015.<sup>49</sup>

Some studies<sup>25,27,37,50-52</sup> were excluded in this study due to incongruous statistical data. In addition, one study investigated overlays on average after 93 months, and the dropout of patients/restorations and the failures of restorations at different years were not reported<sup>53</sup>. Therefore, the data were not extracted for statistical analysis in the present study. Another two studies<sup>54,55</sup> were excluded, because the numbers of dropout patients were higher than those of the dropout restorations.

### Quality Assessment

Of 13 studies, 11 studies with NOS scores  $\geq 6$  were considered as high quality; only two studies<sup>28,46</sup> with NOS scores = 5 were considered to have a high risk of bias. The mean value of quality for the 13 observational studies was score 6.77. (Supplementary Figure 1)

### Survival Rate

*Composite Resin Inlays, Onlays, and Overlays* – The cumulative survival rate of the composite resin inlays, onlays and overlays in the included 4 studies was 91%



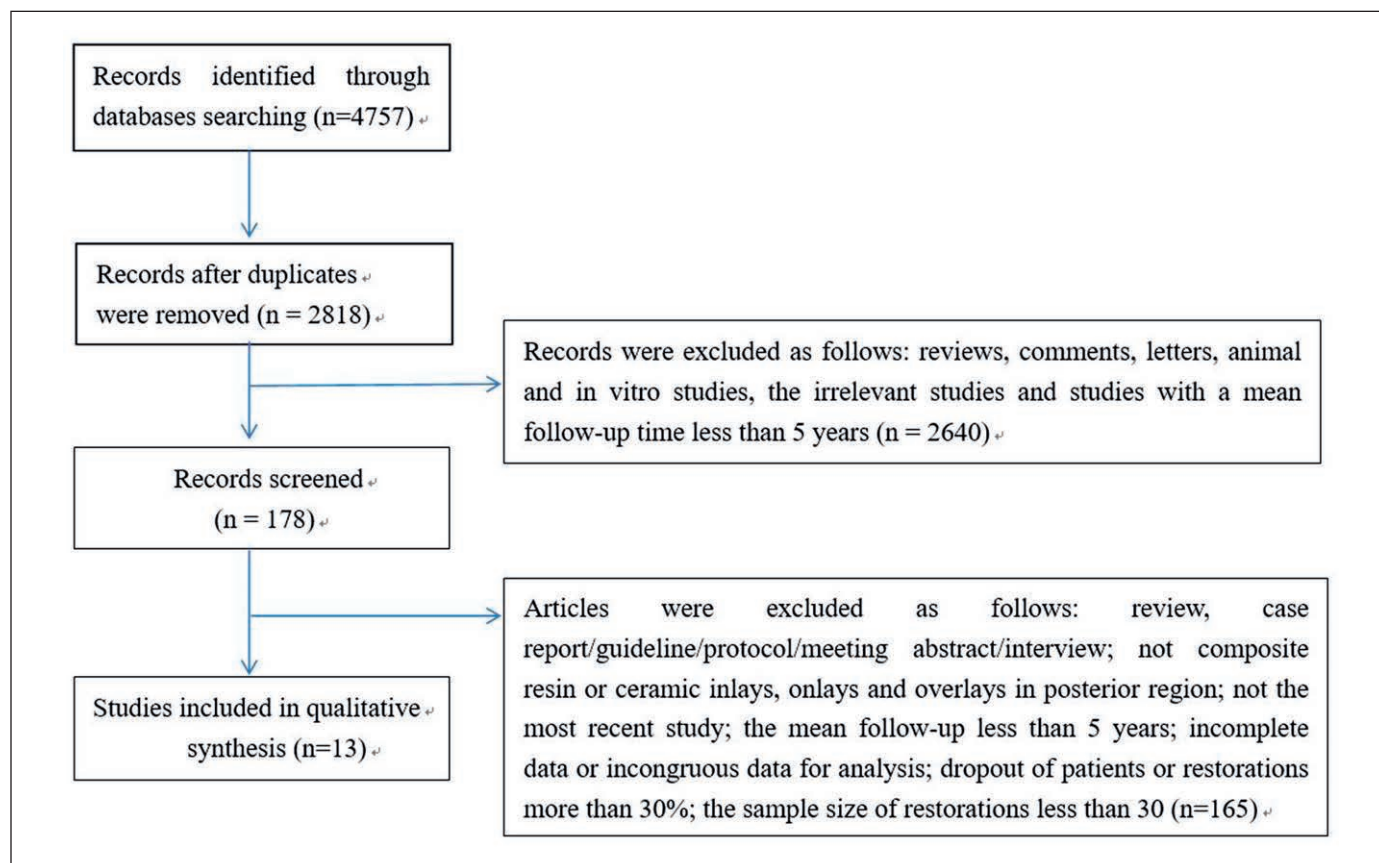


Figure 2. Flowchart of the search strategy.

(95% CI: 86~94%,  $I^2=47\%$ ,  $p=0.13$ ) after 5 years of follow-up (Figure 3A). Only one study reported that the 10-year survival rate of indirect composite resin restorations was 79.2%.<sup>28</sup>

**Ceramic Inlays and Onlays** – Nine studies reported on the survival rate of ceramic inlays and onlays. The 5-year cumulative survival rate reached 90% (95% CI: 86-93%,  $I^2=51\%$ ,  $p=0.04$ ) (Figure 4A). According to subgroup analysis, the survival rate of feldspathic porcelain inlays and onlays was 90% (95% CI: 86-93%,  $I^2=0\%$ ,  $p=0.57$ ) and that of glass ceramic restorations was 86% (95% CI: 73-94%,  $I^2=78\%$ ,  $p=0.01$ ) after 5 years of clinical service (Figure 5A). Dropouts of patients and restorations may be the main reason for the heterogeneity of the 5-year cumulative survival rate of the ceramic restorations (Supplementary Figure 2).

The 8-year survival rate of ceramic inlays and onlays was 87%-91% according to three studies.<sup>31,43,48</sup> Based on the fixed effect model, the pooled survival rate of the ceramic restorations was 89% (95% CI: 83-93%,  $I^2=0$ ,  $p=0.80$ ) (Figure 4B), in which the 8-year survival rates of feldspathic porcelain and glass ceramic restorations were

88% (95% CI: 77-94%,  $I^2=0$ ,  $p=0.46$ ) and 90% (95% CI: 81-95%,  $I^2=0$ ,  $p=0.74$ ), respectively (Figure 5B).

The 10-year survival rate of the ceramic restorations was 85% (95% CI: 76-91%,  $I^2=32\%$ ,  $p=0.22$ ) (Figure 4C).

### Success Rate

**Composite Resin Inlays, Onlays, and Overlays** – Based on data from 3 studies,<sup>29,30,47</sup> the cumulative 5-year success rate of composite resin inlays, onlays and overlays ranged from 83.6-88.0% and the pooled success rate was 84% (95% CI: 78-89%,  $I^2=34\%$ ,  $p=0.22$ ) (Figure 3B). Whereas, Thordrup and others<sup>28</sup> reported that the 10-year success rate of composite resin restorations was 66.7%.

**Ceramic Inlays and Onlays** – The 5-year cumulative success rate of ceramic inlays and onlays was 88% (95% CI: 82-92%  $I^2=24\%$ ,  $p=0.27$ ) (Figure 6A). One study reported that the 8-year success rate of ceramic inlays was 80%.<sup>43</sup> The 10-year success rate of ceramic inlays and onlays ranged from 67.96% to 83.6% and the pooled estimate for the 10-year success rate was 77% (95% CI: 59-89%  $I^2=64\%$ ,  $p=0.10$ ) (Figure 6B).<sup>28,33</sup>

Table 1: *Characteristics of the Studies*

No.	Author (year)	Materials	Country	Investigation Period	"Evaluation Criteria"	"Follow-up Period (y)"
1	Sjogren et al. (2004) [32]	Vita Mark	Sweden	NR	Modified USPHS	10
2	Schulz et al. (2003) [44]	Mirage ceramic	Sweden	1988-1997	CDA	6.3
3	Hayashi et al. (2000) [42]	G-cera Cosmoteh	Japan	1990.10-1991.3	Modified USPHS	8
4	"Pallesen & van Dijken (2000) [30]"	"Vita Mark Dicor MGC"	Denmark	NR	Modified USPHS	8
5	"Molin & Karlsson (2000) [43]"	"Cerec Mirage Empress"	Sweden	NR	CDA	5
6	Najatidanesh et al. (2015) [48]	"CEREC blocks Empress CAD blocks"	Iran	2009.3-2009.9	CDA	5
7	Cetin et al. (2013) [28]	"Estenia Tescera ATL"	Turkey	2005-2006	Modified USPHS	5
8	Zhang et al. (2008) [46]	3M Vitremer	China	2001.3-2001.10	Modified USPHS	5
9	D' Arcangelo et al. (2014) [29]	Enamel Plus HFO	Italy	2005.4-2007.1	Modified USPHS	5
10	Thordrup et al. (2006) [27]	"Cerec.cos2.0 Brilliant DI Vita Dur N Estilux Kulzer"	Denmark	NR	CDA	10
11	van Dijken (2003) [45]	IPS Empress	Sweden	NR	Modified USPHS	5
12	Kramer et al. (2008) [47]	IPS Empress	Germany	NR	Modified USPHS	8
13	Santos et al. (2013) [35]	"Duceram IPS Empress"	Brazil	NR	Modified USPHS	5.5

*Abbreviations: CDA, California Dental Association; NR, not reported; PC, prospective cohort; RC, retrospective cohort; Score, the value of the Newcastle-Ottawa Scale (NOS); USPHS, United States Public Health Service.*

Setting/ Operator	"Age Range (mean)"	"No. of Patients"	"No of Restorations"	"Dropout (%) (Patient/ Restoration)"	Study Type	"Survival Rate (%)"	"Success Rate (%)"	Score
University / NR	26-73 (48)	27	66	8% / 7%	PC	88.5	83.6	6
"Private Practice / 1"	28-79 (54)	52	109	1.9% / 1.8%	RC	84.1	65.4	7
University / NR	NR	25	45	0	RC	86.7	80	6
NR / 1	24-58 (40)	16	32	0	RC	90.6	NR	7
NR / 1	23-56	20	60	0	PC	86.7	NR	7
"Private Practice / 1"	18-70 (45.5)	109	159	5.5% / 3.8%	RC	95.4	NR	8
University / 1	20-28 (23)	54	41	0	RC	97.6	NR	8
University / 1	20-60	NR	100	9%	RC	95.6	87.9	8
University / 1	18-51	47	79	9.7% / 7.5%	RC	87.7	83.6	8
NR / 1	23-69	37	58	10.8% / 10.3%	PC	78.8	67.3	5
University / NR	22-68 (45.5)	29	79	10.3% / 11.3%	RC	88.7	81.7	5
University / 6	NR	31	94	25.8% / 27.7%	PC	89.7	NR	6
NR / 1	25-44 (33)	35	86	25.7% / 27.9%	RC	79	NR	7

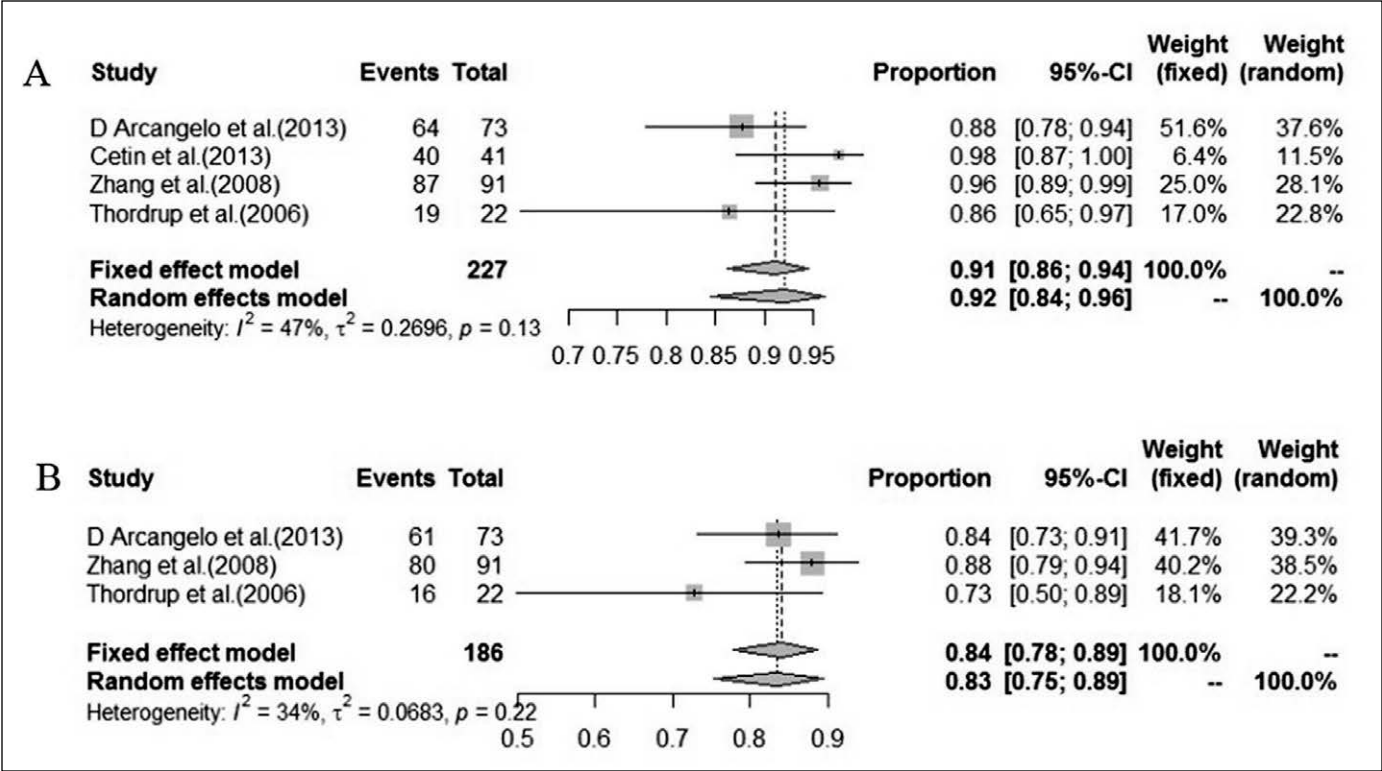


Figure 3. Five-year cumulative survival rate (A) and success rate (B) of composite resin inlays, onlays, and overlays.

Publication Bias Analysis

The shape of funnel plots does not reveal any obvious asymmetry (Supplementary Figure 3). Moreover, there was no publication bias on the basis of Egger's test ( $p > 0.05$ ) in the composite resin and ceramic groups.

Complications

According to the identified studies of composite resin inlays, onlays, and overlays, the reasons for failures included endodontic complications such as post-operative sensitivity, pulpitis, and pulp necrosis, secondary caries, fracture of restorations, debonding and severe restoration discolorations. Secondary caries and endodontic complications were the most frequent reasons leading to failures, with the pooled proportions of 47% (95% CI: 26 - 70%) and 27% (95% CI: 11 - 54%) (Figs. 7A, B).

For the ceramic restorations, fracture of restorations, endodontic complications and secondary caries were the main reasons of failures. During 5 years of follow-up, a total of 34 fracture failures among 62 failures occurred with a pooled proportion of 54% (95% CI: 40-67%) (Figure 8A). Five studies revealed that 20% (95% CI: 11-33%) of failure cases were caused by endodontic complications including post-sensitivity, pulpitis

and pulp necrosis (Figure 8B). The overall estimated proportion of secondary caries was 14% (95% CI: 7-26 %) (Figure 8C). In addition, marginal defects, open contacts, dull surfaces, and tooth fractures were also reported as failures. After 8 years of follow-up, the proportions of the fractures of restorations and the endodontic complications were 54% (95% CI: 28-78%) and 34% (95% CI: 7-78%), respectively (Figs. 9A-B). In addition, the pooled proportion of fracture of restorations among failures increased to 61% (95% CI: 34-83%) after 10 years of follow-up (Figure 10). All the data of complications are summarized in Table 2.

Factors Influencing the Survival Rate of Restorations

*Molar and Premolar Regions* – Five studies<sup>33,36,43,45,49</sup> investigated the clinical performance of restorations in molar and premolar regions. Schulz and others<sup>45</sup> reported that inlays in the molar region were three times more likely to be fractured than those in premolar region. Furthermore, three studies<sup>33,36,49</sup> demonstrated that fracture of restorations occurred only in the molar region. Contrarily, Hayashi and others<sup>43</sup> reported that restorations in premolars were 2 times more prone to fracture than those in molars.



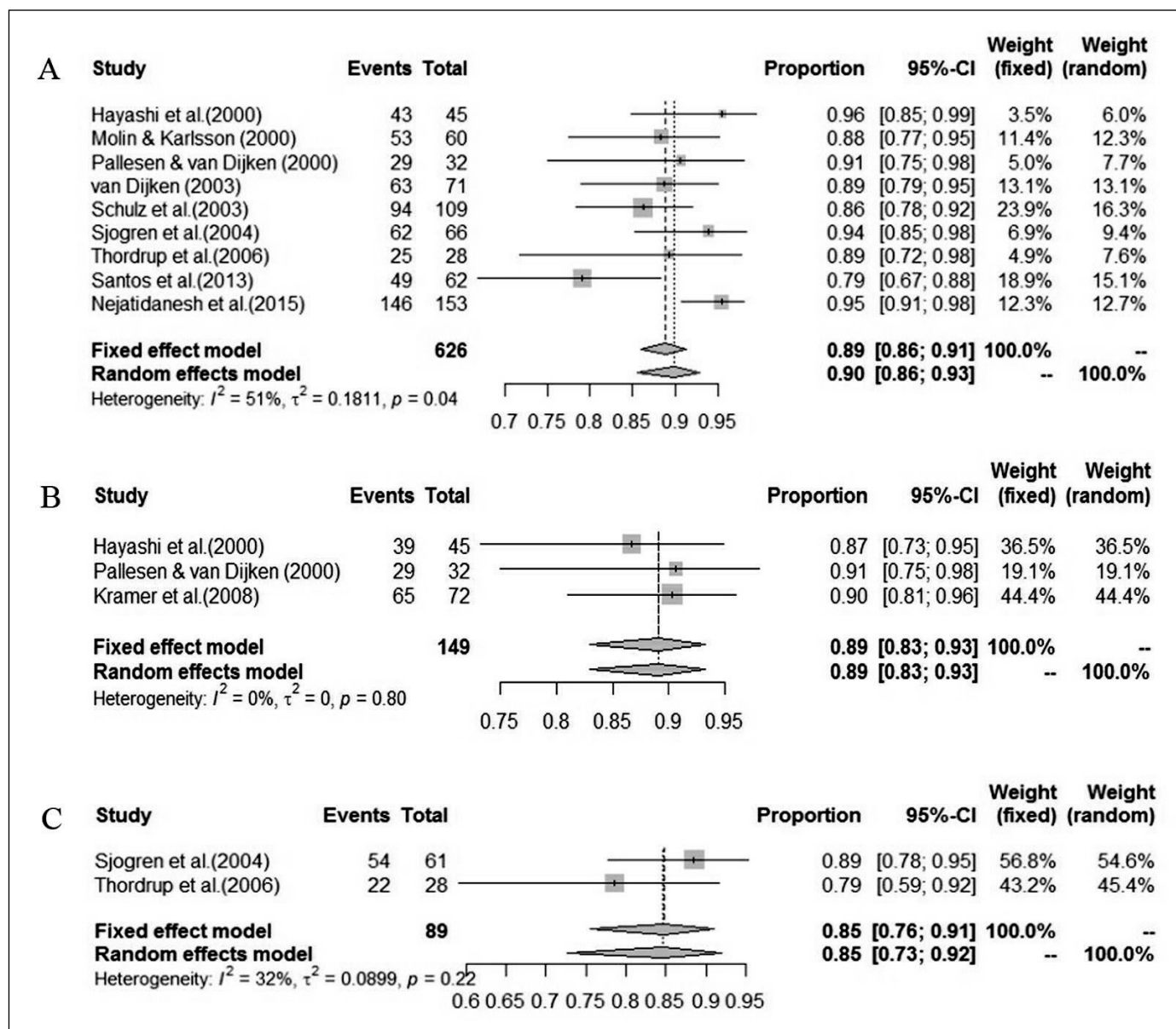


Figure 4. Cumulative five year (A), eight-year (B), and ten-year (C) survival rate of ceramic inlays and onlays.

**Types of Restorations** – Hayashi and others<sup>43</sup> placed 43 inlays and 2 onlays in 25 patients. They reported that 4 inlays fractured while the two onlays functioned well after 8 years of follow-up. However, Santos and others<sup>36</sup> concluded that the fracture rate of onlays was 1.6 times larger than that of inlays when they investigated 53 inlays and 33 onlays after 5 years in service, and two of them were fractured in each type of restoration. In addition, Sjogren and others<sup>33</sup> reported that three-surface inlays were 2.88 times more susceptible to fracture than two-surface inlays. Nejatidanesh and others<sup>49</sup> reported that no failure occurred in two-surface inlays, 1.72% of three-surface restorations failed as a result of dentin hypersensitivity, 8.16% of four-

surface restorations failed due to fracture, retention loss and hypersensitivity, and 5.26% of more than four-surface restorations failed owing to fracture and hypersensitivity. All the fractures occurred in four- or more-surface restorations.<sup>49</sup>

**Luting Cements** – Three studies<sup>33,46,48</sup> reported the implementation of different adhesives and luting cements for inlays made by the same restorative materials. Two studies reported that no different clinical outcomes of 5-8 years in service were found ( $p > 0.05$ ) after resin-modified glass ionomer cement (Fuji Plus, GC Dental Industrial Corp., Tokyo, Japan) and self-cure composite resin cement (Panavia 21, Kuraray-Noritake, Tokyo, Japan) were compared,<sup>46</sup>

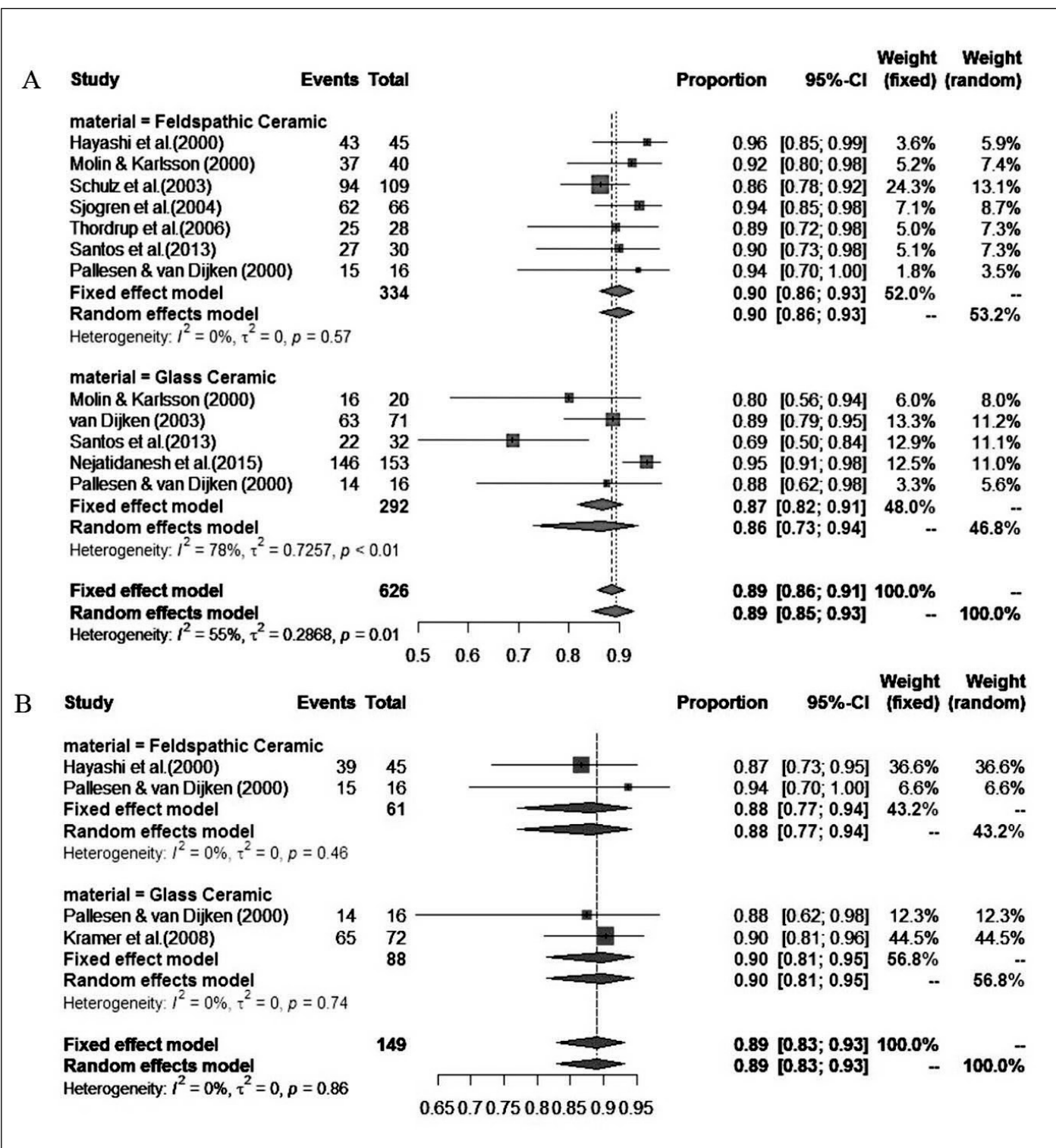


Figure 5. Five-year (A) and eight-year (B) cumulative survival rate of feldspathic and glass ceramic inlays and onlays.

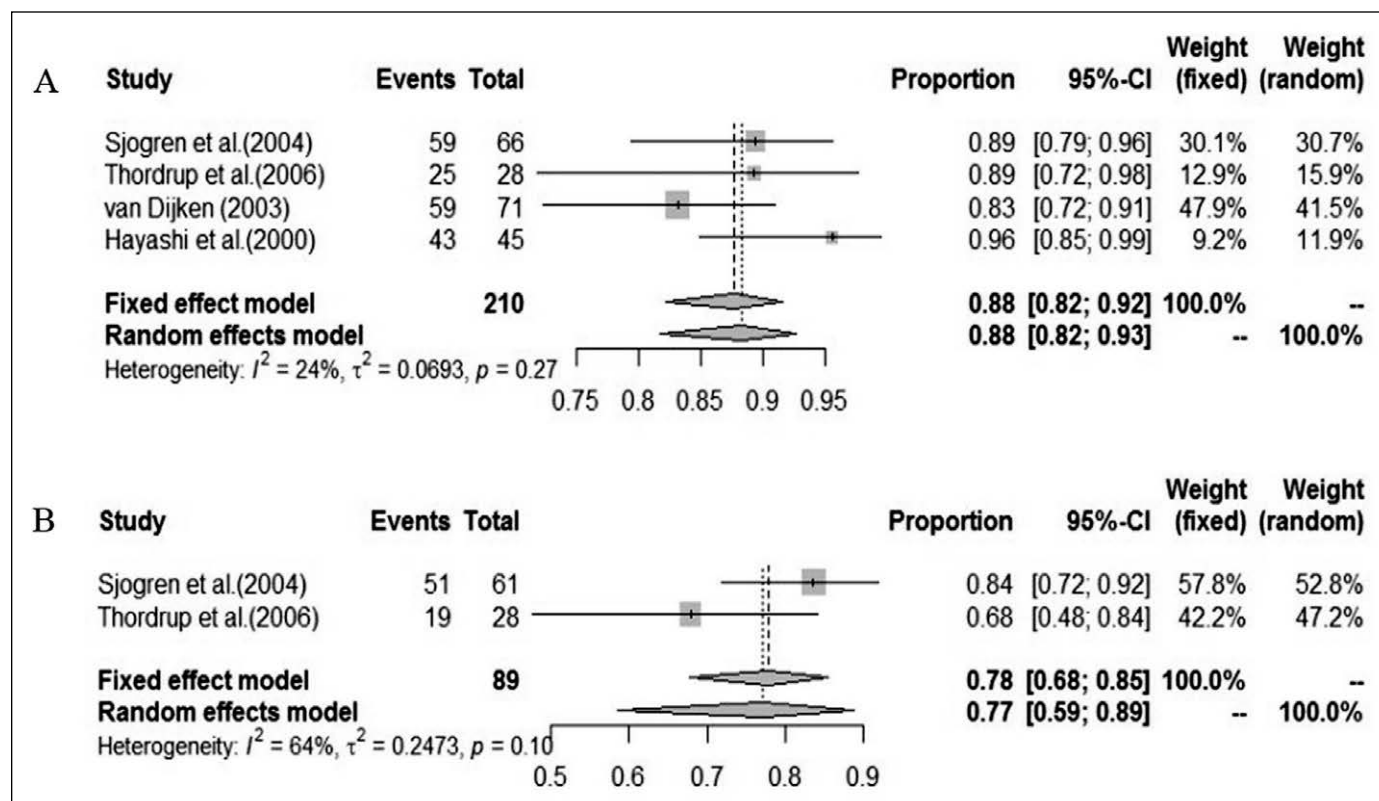


Figure 6. Five-year (A) and ten-year (B) cumulative success rate of ceramic inlays and onlays.

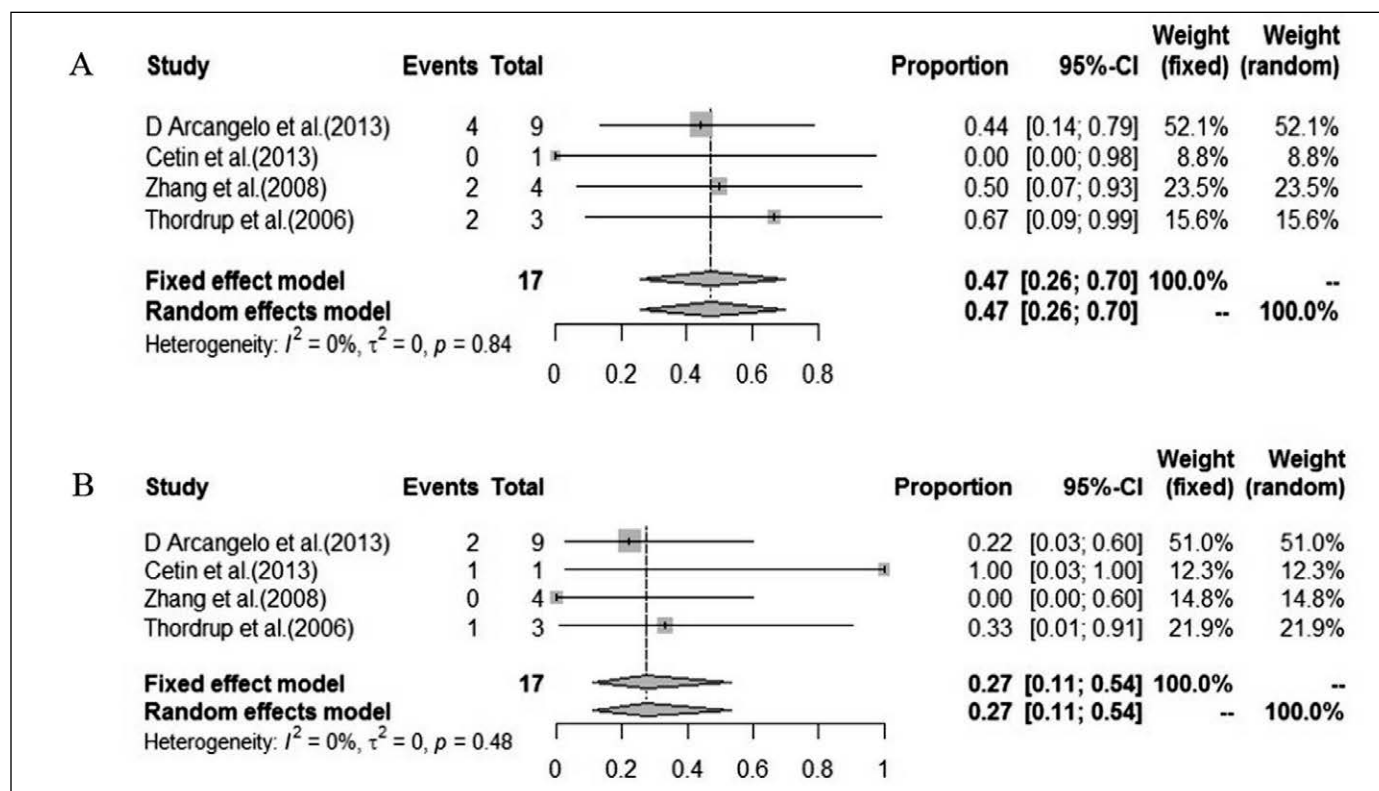


Figure 7. Five-year pooled proportion of complications of composite resin inlays, onlays and overlays. (A): secondary caries; (B): endodontic complications.

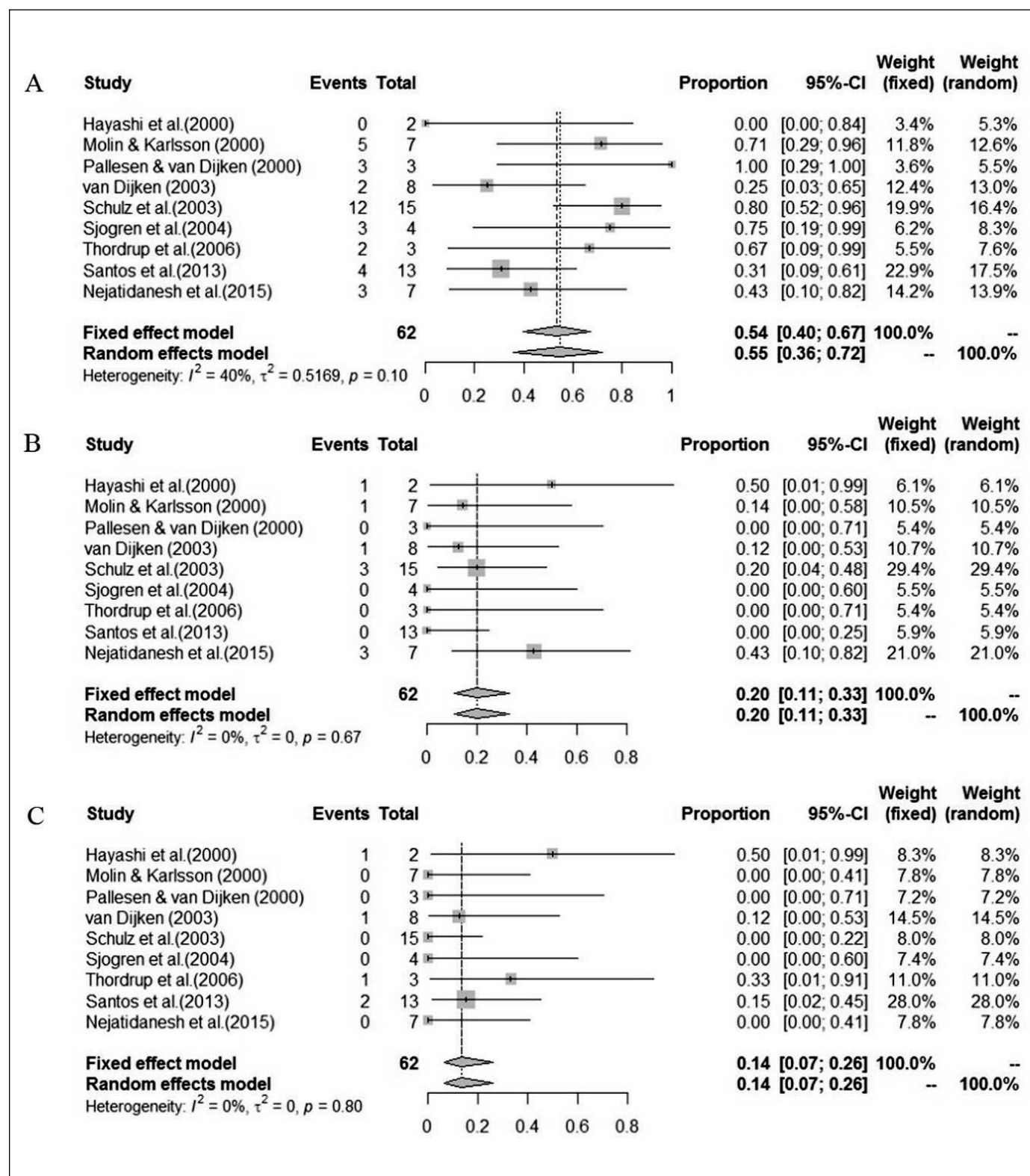


Figure 8. Five-year pooled proportion of complications of ceramic inlays and onlays. (A): Restoration fractures; (B): Endodontic complications; (C): Secondary caries.



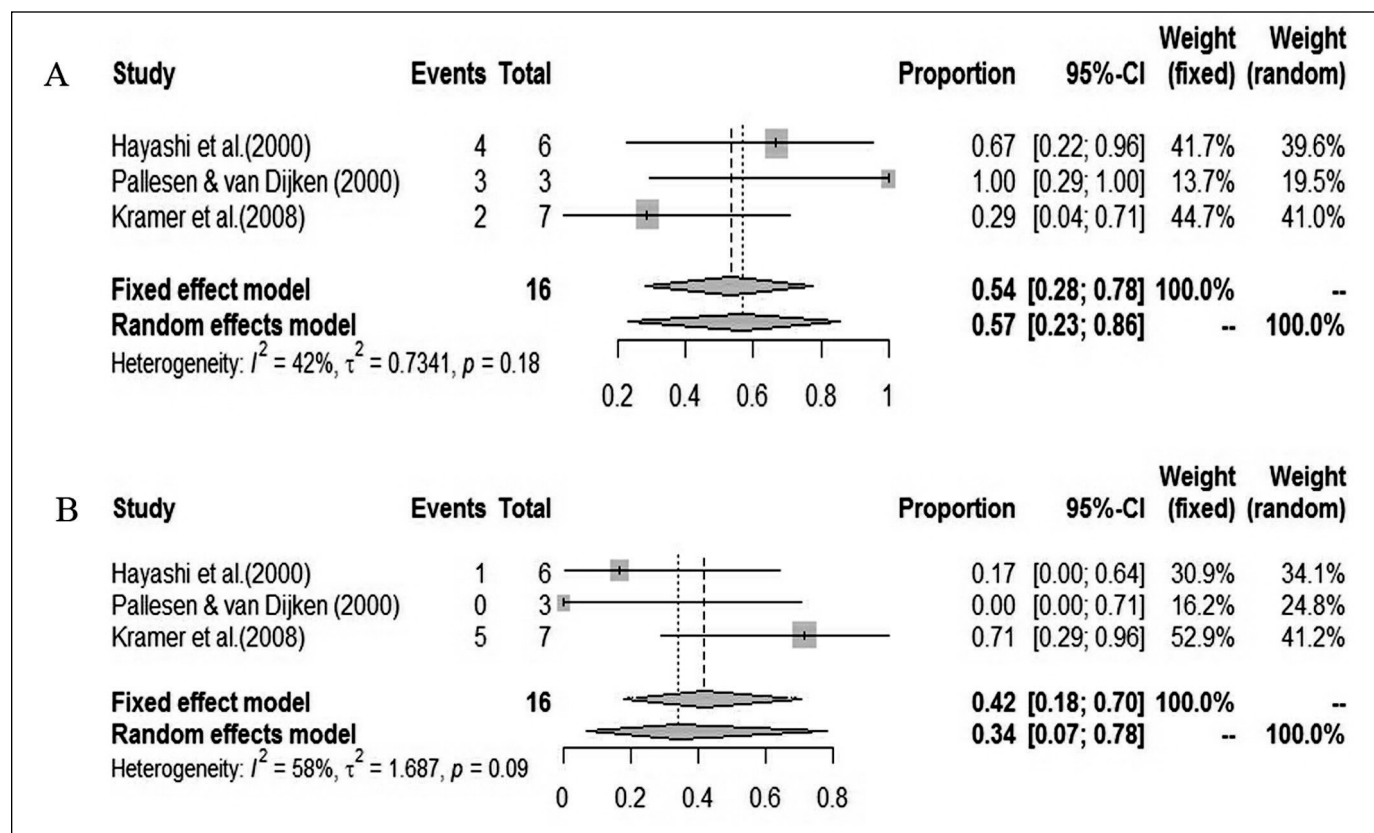


Figure 9. Eight-year pooled proportion of complications of ceramic inlays and onlays. A: Restoration fractures; B: Endodontic complications.

as well as two light-cure resin cements (EBS Multi + Compolute, 3M ESPE, Seefeld, Germany; Syntac + Variolink II, Ivoclar Vivadent, Schaan, Liechtenstein) were investigated.<sup>48</sup> Sjogren and others<sup>33</sup> reported that the estimated survival rate of Vita Mark II (Vita Zahnfabrik, Bad Säckingen, Germany) inlays fabricated by CAD/CAM after 10 years was 89%. The survival rate (77%) of the dual-cure composite resin (Vita Cerec Duo Cement, Coltene-Whaledent, Altstetten, Switzerland) was significantly lower than that (100%) of the chemically cure composite resin (Cavex Clearfil F2, Cavex, Haarlem, the Netherlands) ( $p < 0.05$ ).<sup>33</sup>

**Materials and Fabrication Methodology** – Pallesen & van Dijken<sup>31</sup> reported that the fracture rate of machinable Dicor MGC (Dentsply, Konstanz, Germany) inlays was 12.5% after 8 years of follow-up, while that of sintered Vita Mark II was 6.25%. Molin & Karlsson<sup>44</sup> indicated that the 5-year failure rate of Cerec (Vita Cerec, Vita Zahnfabrik, Bad Säckingen, Germany) inlays fabricated by CAD/CAM was 10% as a result of restoration fracture and debonding, and that of pressed IPS Empress (Ivoclar Vivadent, Schaan, Liechtenstein) inlays was 20%. On the contrary, 5% of sintered Mirage (Chameleon Dental Products, Kansas City, USA) inlays

failed because of endodontic complications. In a study by Santos and others,<sup>36</sup> 18.75% of pressed Empress inlays and 10% of sintered Duceram (Dentsply-Degussa, Dentsply International Inc., PA, USA) inlays failed due to mechanical and biological complications after 5 years of follow-up. Based on these studies, it is difficult to reveal which material or fabrication method is superior to the others.

**Tooth Vitality** – Six of the included studies investigated the clinical outcome of inlay and onlay restorations for vital teeth,<sup>30,31,33,36,46,47</sup> while the other six studies<sup>28,29,43-45,48</sup> did not mention the vitality of teeth. Only Nejatidanesh and others<sup>49</sup> investigated the effect of tooth vitality on the clinical outcome of ceramic inlays and onlays. They reported that no fracture of ceramic inlays or partial coverage occurred in 92 vital teeth after 5 years of follow-up, while three restorations were fractured in 67 nonvital teeth. Nonvital teeth had a significantly higher risk of fracture ( $p < 0.05$ ). Vital teeth tend to achieve better clinical performance with ceramic restorations.

**Tooth Preparation** – Clinical performance associated with the tooth preparation or different thickness of restorations was not involved in this study.

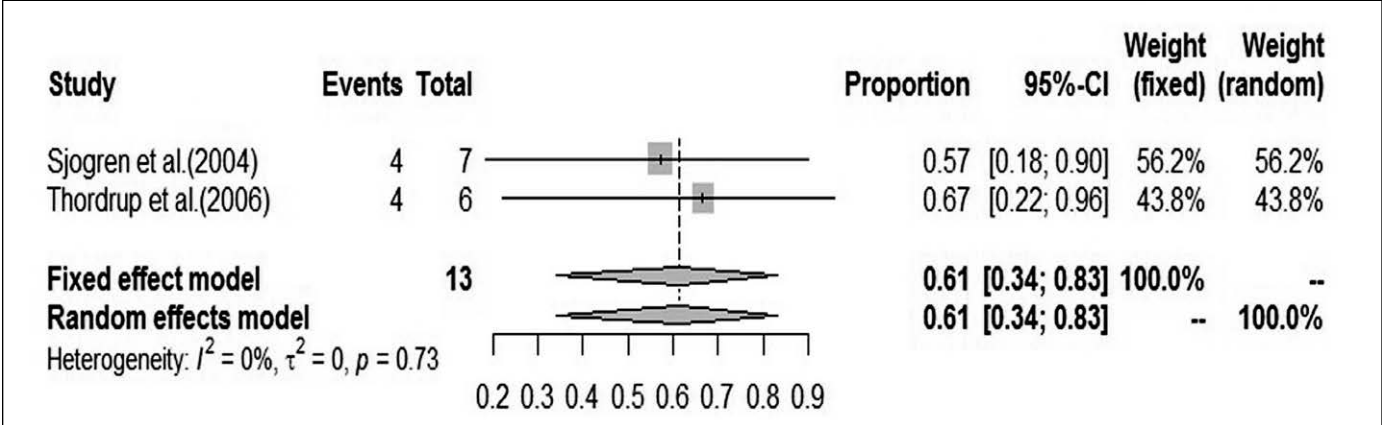


Figure 10. Ten-year pooled proportion of restoration fractures of ceramic inlays and onlays.

**Parafunctional Habits and Bruxism** – Parafunctional habits and bruxism were not mentioned in the majority of the previous publications.<sup>29,31,43-46,48</sup> Four studies<sup>28,30,36,47</sup> clearly stated patients who suffered from temporomandibular disorders or had signs of bruxism or clenching habits were excluded. Two studies included the patients with parafunctional habits and bruxism.<sup>33,49</sup> Sjogren and others<sup>33</sup> and Nejatidanesh and others<sup>49</sup> indicated that no association between the parafunctional habits and fractures of restorations was found.

DISCUSSION

The present study evaluated the long-term clinical survival and success rates of composite resin and ceramic inlays, onlays, and overlays, as well as the main failure reasons of restorations and the factors influencing the survival rate of restorations.

Several systematic reviews concerning composite resin and ceramic inlays, onlays, and overlays have been published.<sup>24,39,56,57</sup> Recently, Morimoto and others<sup>40</sup> reported that the survival rate of ceramic inlays, onlays and overlays was 95 % at 5 years and 91% at 10 years. More recently, Abduo & Sambrook<sup>58</sup> systematically evaluated the longevity of ceramic onlay restorations for at least 2 years of clinical follow-up and identified the

factors that influence the survival of a ceramic onlay. They reported that the survival rate of the ceramic onlay restorations was 71%-98.5% after more than 5 years of observation. They also indicated that tooth preparation, tooth vitality and occlusal force tended to influence the clinical outcome of ceramic onlays while different materials, manufacturing techniques and luting cements had minimal effects on the survival of ceramic onlays.<sup>58</sup> Compared to their meta-analysis results, data in the present meta-analysis appeared to be ultra-conservative due to the long clinical evaluation (at least 5 years) and low restoration dropout (less than 30%). Besides the ceramic restorations, this meta-analysis covered the long-term survival and success rates of indirect composite resin restorations as well. According to the data in this review, we hold the view that the multiple-surface restorations and nonvital teeth may have a negative effect on the longevity of restorations. With respect to the other factors potentially influencing the clinical outcome, no definite conclusion could be drawn owing to lacking consistent data.

Survival Rate of Composite Resin Inlays, Onlays, and Overlays

With the advance of esthetic and mechanical properties of composite resins, these materials have been

Table 2: Summary of Data of Complications				
Complications	Estimated Pooled Proportions			
	5-yr		8-yr	10-yr
	Composite resin	Ceramic	Ceramic	Ceramic
Fracture	24% (9-51%)	54% (40-67%)	54% (28-78%)	61% (34-83%)
Endodontic complications	27% (11-54%)	20% (11-33%)	34% (7-78%)	-

recommended to be used for inlays or onlays even in large cavities.<sup>21,59,60</sup> The 5-year cumulative survival rate of composite resin inlays, onlays and overlays in this review was 91%, indicating an excellent clinical outcome. Pallesen & Qvist<sup>27</sup> reported that 88% of the composite resin inlays performed clinically acceptable even after 11 years in service. This is in agreement with the study performed by Barabanti and others<sup>37</sup> They stated that approximately 90% of indirect composite resin inlays and onlays used for restoring large tooth defects still functioned well after 10 years of clinical service.<sup>37</sup> However, Thordrup and others<sup>28</sup> reported that the 10-year cumulative survival rate of composite resin inlays decreased to 79.2% due to secondary caries and endodontic complications. Furthermore, van Dijken and others<sup>25</sup> reported that the cumulative survival rate of direct composite resin inlays and onlays was 82.3% after 11 years of clinical service. Most recently, Derchi and others<sup>61</sup> reported that the failure rate of the composite resin inlays was 12% at 12 years. However, these data were excluded in the present meta-analysis due to inconsistent results in respect to the number of failures<sup>25</sup> and high dropout rate.<sup>61</sup> This disparity might be attributable to the limited number of RCTs and the high dropout rate. In the meanwhile, Ravasini and others<sup>62</sup> revealed that the survival rate of indirect posterior composite restorations was 81% at 10 years according to the Kaplan-Meier plot. This is completely in accordance with the previous report.<sup>28</sup> They also reported that the probability of survival rate will rapidly decrease to 57% after 20 years in service.<sup>62</sup> In contrast to the mechanical failure of ceramic restorations, the main failures of composite resin restorations were biological complications, including secondary caries and endodontic complications.

### Survival Rate of Ceramic Inlays, Onlays, and Overlays

In this study, the cumulative survival rate of ceramic inlays and onlays reached 90% at 5 years, and slightly decreased over time, 89% at 8 years and 85% at 10 years. These findings are in agreement with the previous studies.<sup>63-65</sup> Arnelund and others<sup>63</sup> indicated that the overall survival rate of ceramic inlays and onlays was 92% after a 60 month follow-up period. Federlin and others<sup>65</sup> reported that 88.8% of the ceramic inlay and onlay restorations were judged as clinically acceptable after 5.5 years of follow-up. Zimmer and others<sup>64</sup> reported that Class I and II CAD/CAM ceramic inlays/onlays fabricated with feldspathic porcelain (Vita Mark II) and glass ceramic blocks (Dicor, Corning Dentsply, Konstanz, Germany) had a survival rate of 85.7% at 10 years. This is in line with the cumulative survival rate

in this study. To date, there are not sufficient RCT data concerning inlays and onlays for more than 10 years of clinical follow-up. Santos and others<sup>38</sup> evaluated the 12-year clinical performance of sintered (Duceram) and pressable (IPS Empress) ceramic inlays and onlays with a total cumulative survival rate of 47.92%. Contrarily, Frankenberger and others<sup>35</sup> reported that 84% of IPS Empress inlays and onlays functioned well over 12 years. This is attributable to the high restoration dropout rates of 44.19%<sup>38</sup> and 39.59%<sup>35</sup> in both studies. Reiss and others<sup>66</sup> reported that the success rate of 1011 Cerec inlays was 84.4% after 16.7 years in service according to the Kaplan-Meier plot. These data were excluded in this study due to the unreported failures and dropout rates. However, Arnetzl<sup>53</sup> reported that the estimated survival rate of nonretentive ceramic overlays was 96.5% after 93 months in average. Most recently, Edelhoff and others<sup>67</sup> reported that occlusal onlays made of monolithic lithium disilicate for full-mouth oral rehabilitation presented a 100% survival rate in seven patients with severe toothwear up to 11 years.

### Which Restoration Is the Best Among Inlays, Onlays, and Overlays?

Inlay, onlay, and overlay restorations can function well even in the case that the tooth hard structures suffer from extensive loss.<sup>10</sup> However, it has not been clarified whether onlay or overlay restorations could perform better than inlays.<sup>36,43,54,55,68-70</sup> Recently, several in vitro studies investigated the fracture resistance of indirect restorations related to different cavity designs of posterior teeth.<sup>68-70</sup> However, their results were not always consistent. For example, Cubas and others<sup>68</sup> reported that MOD inlays presented fracture resistance similar to sound teeth while the restorations with partial or complete coverage (onlays and overlays) exhibited significantly lower fracture resistance than inlays and sound teeth. Therefore, they concluded that less invasive preparation of inlays should be preferred.<sup>68</sup> Contrarily, Alshiddi & Aljinbaz<sup>69</sup> reported that the endodontically treated teeth restored with composite resin inlays had a significant increase in fracture resistance when compared to the teeth restored with onlays. Therefore, they pointed out that tooth fracture up to the root was prone to occur in teeth with inlay restorations, whereas, most of the fractures are confined to the restorations when onlays are placed.<sup>69</sup> Furthermore, Harsha and others<sup>70</sup> reported that the fracture resistance of partial coverage (onlays) seemed to be higher than MOD inlays, but both were not significantly different, and complete coverage (overlays) could reinforce the tooth structure at maximum. There were some conflicting results in previous clinical studies concerning inlays

and onlays. Otto and others<sup>54,55</sup> reported that the fracture rates of inlay and onlay restorations were 4.1% and 6.7% at 10 years, and 7.2% and 6.7% at 15 years, respectively. Hayashi and others<sup>43</sup> reported that the fracture rate of inlay restorations was higher than that of onlay restorations, since four of 43 inlays and none of a total of 2 onlays fractured during 8 years of follow-up. Contrarily, Santos and others<sup>36</sup> reported that the fracture rate of onlays was 1.6 times larger than that of inlays after 5 years. Therefore, further RCTs investigating the influence of different cavity designs on the clinical success rate should be performed.

### Feldspathic Porcelain versus Glass Ceramic

Usually, flexural strength of glass ceramic is much stronger than that of feldspathic porcelain.<sup>71,72</sup> Based on the flexural strengths, the failure rate of glass ceramic restorations might be lower than that of feldspathic porcelain restorations. However, in the present study, the cumulative survival rates of feldspathic porcelain and glass ceramic inlays were recorded as 90% and 86%, respectively. Santos and others<sup>36</sup> reported a comparatively low survival rate of glass ceramic restorations due to the severe restoration discolorations.<sup>36</sup>

### Tooth Preparation and Stronger Materials

Regarding ceramic restorations, fractures occurred as a principal complication with the pooled proportion of 54% at 5 years, 54% at 8 years and 61% at 10 years, indicating that the more hard tooth structure lost due to cavity preparation, the stronger material needed. In order to resist the mastication force in posterior teeth, the thickness of ceramics has been suggested to be at least 1.5 to 2 mm for functional cusps and 1-1.5 mm for non-functional cusp.<sup>73-75</sup> Concerning the thickness of ceramic restorations, the results of laboratory research and clinical trials were entirely inconsistent.<sup>10,19,76-78</sup> Laboratory studies indicated that the thickness of inlays and onlays may not be an important factor influencing the fracture risk of restorations.<sup>77,78</sup> Holberg and others<sup>77</sup> analyzed a finite element model of inlays and reported that the thinner inlay would not significantly increase the risk of fracture. This is in agreement with a laboratory report,<sup>78</sup> indicating that cusp coverages of 1.5 and 2.5 mm had similar fracture rates when endodontically treated teeth were restored with composite resin onlays.<sup>78</sup> In contrast, clinical trials draw the opposite conclusion and suggest at least 2 mm thickness for feldspathic porcelain and leucite-reinforced ceramics.<sup>10,19,76</sup> Murgueitio and others<sup>19</sup> discovered that the thickness of the occlusal surface of the failed restorations was less than 2 mm in their

clinical research. Based on two clinical trials, van Dijken and others<sup>10,76</sup> also insisted that the cusp fracture of IPS Empress restorations can be effectively prevented when the thickness of the ceramic reached 2 mm. IPS e.Max (Ivoclar Vivadent, Schaan, Liechtenstein) ceramic made of lithium disilicate has been widely used for its high flexural strength (up to 400 MPa).<sup>79</sup> High short-term survival rates of IPS e.Max inlay and onlay restorations have been reported.<sup>75,80,81</sup> Moreover, the long-term performance of IPS e.max Press partial and entire coverage restorations demonstrated no failures after 7-11 years of follow-up.<sup>67,82</sup>

### Vital Teeth Versus Nonvital Teeth

In this analysis, only one study<sup>49</sup> indicated that ceramic fracture more frequently occurred in nonvital teeth than in vital teeth. Generally, a large amount of dentin removal by root canal therapy leads to lower fracture resistance of the residual tooth. Removal of pulp tissues may also increase the brittleness of dentin.<sup>83</sup> Several studies revealed that posterior indirect adhesive restorations in nonvital teeth functioned well after 2-4 years of clinical service.<sup>15,84,85</sup> Nevertheless, two previous publications showed that the vital tooth had a more favorable outcome and were less likely to fail than the nonvital teeth.<sup>19,76</sup> After 3 years of IPS Empress onlays and partial coverage restorations, 85.7% of failures took place in the restorations of nonvital teeth.<sup>19</sup>

### Molar Versus Premolar

The restorations in molar regions are subjected to larger masticatory forces than those in premolars. They are presumed to be more prone to be fractured. Numerous previous publications revealed a significantly higher rate of failure of restoration in molars than that in premolars.<sup>19,21,40,86</sup> Contrarily, Collares and others<sup>3</sup> reported that no differences of the success and survival rates were found between inlays and onlays, as well as between molars and premolars after they analyzed 5791 ceramic inlays and onlays.

### Parafunctional Habits

Numerous previous reports showed that composite resin and ceramic inlay and onlay restorations were more prone to be fractured when subjects had parafunctional habits.<sup>7,55,87,88</sup> It might be attributed to parafunctional habits playing a significant role in material fatigue leading to fracture after long-term clinical service.<sup>87</sup> Contrarily, some researchers insist that direct evidence between parafunctional habits and the fracture of composite resin and ceramic restorations had not been found.<sup>58,89</sup>



### Limitation of This Systematic Review and Meta-analysis

Most of the data in the included studies reveal homogeneity. Nevertheless, when the 5-year survival rate of ceramic inlays was calculated, the included studies showed moderate heterogeneity ( $I^2=51\%$ ). After a subgroup analysis was carried out to explain the origin of heterogeneity, the heterogeneity was obviously associated with dropout of patients and restorations, which may lead to a high risk of bias. Thirteen observational studies included in this study were prospective or retrospective cohort studies with their evidence levels being lower than that of RCTs. Hence, further RCTs are needed to investigate the long-term performance of composite resin or ceramic inlays, onlays, and overlays.

### CONCLUSION

The cumulative survival and success rates of composite resin inlays, onlays and overlays were 91% and 84% at 5 years, respectively. The survival rates of ceramic inlays and onlays were 90% at 5 years, 89% at 8 years and 85% at 10 years, while the success rates of ceramic inlays and onlays were 88% at 5 years and 77% at 10 years. Biological complications, including secondary caries and endodontic complications with the respective pooled proportion of 47% and 27%, were the main reasons leading to the failure of composite resin restorations. In contrast, mechanical complications were the principal issue of ceramic restorations, accounting for 54% of the restoration failures. Nonvital teeth and multiple-surface restorations had negative effects on the longevity of the inlay and onlay restorations. Based on the data in this study, no direct association between parafunctional habits and bruxism and the fracture of restorations was found. Due to inconsistent data or lack of sufficiently evident data in this study, a definite conclusion could not be drawn regarding factors influencing the clinical outcome, such as restorations in premolar and molar regions, luting cements, fabrication method of the restorations, tooth preparation and parafunctional habits. Hence, further RCTs should be conducted to investigate clinical performance of composite resin and ceramic inlays, onlays and overlays for tooth restorations.

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### Conflict of Interest

The authors have no financial interest in any of the companies or products mentioned in this article.

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# Repair Bond Strength of High-viscosity Glass-ionomer Cements Using Resin Composite Bonded with Light- and Self-cured Adhesive Systems

HA El-Deeb • EH Mobarak

## Clinical Relevance

High-viscosity glass-ionomer cements (HVGICs) used with atraumatic restorative treatment can be repaired with light- or self-cured adhesive systems; however, the repair bond strength of two-step, self-etching and one-step adhesives in the light-cure mode surpass one-step self-cure adhesives. Working on a feasible self-cure approach in the absence of such in rural areas as well as in war zones is of prime importance.

## SUMMARY

**Objectives:** Despite the success rate of high-viscosity glass-ionomer cements (HVGICs) used in atraumatic restorative treatment (ART) restorations, partial or bulk fracture of the proximal portion has been recorded to be one of the main causes of proximal restoration failures. Repair

of these restorative materials requires a practical solution, especially in cases where there is a lack of electricity. Thus, the purpose of this study was to evaluate the repair microshear bond strength ( $\mu$ SBS) of three HVGICs using a resin composite in association with adhesive systems having different curing modes (ie, light- vs self-curing mode).

**Methods and Materials:** A total of 105 discs (12 mm in diameter and 2 mm thick) of three HVGICs: GC Fuji IX GP Fast (GC Corporation, Tokyo, Japan); Fuji IX GP glass-ionomer cement containing chlorhexidine (GC Corporation, Tokyo, Japan); and ChemFil Rock zinc-reinforced HVGIC (Dentsply De-Trey GmbH, Konstanz, Germany) were prepared. Each specimen was divided into three horizontal sections, according to the tested adhesive system or curing mode: Clearfil SE Bond 2 (two-step, self-etch adhesive); (Kuraray Noritake Dental Inc., Tokyo, Japan) in light-cure mode; Clearfil Universal Bond (one-step, self-etch

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adhesive); (Kuraray Noritake Dental Inc., Tokyo, Japan) in light-cure mode; or Clearfil Universal Bond (one-step, self-etch adhesive); (Kuraray Noritake Dental Inc., Tokyo, Japan) in self-cure mode, mixing it with Clearfil DC Activator (Kuraray Noritake Dental Inc., Tokyo, Japan). A resin composite microcylinder was bonded to each horizontal section of each specimen using starch tubes. The bonded discs were stored in artificial saliva at 37°C for 24 hours. A  $\mu$ SBS test was conducted using a universal testing machine, while failure modes were determined using scanning electron microscopy. Data were statistically analyzed using two-way analysis of variance (ANOVA), one-way ANOVA, and Bonferroni post hoc tests ( $\alpha=0.05$ ).

**Results:** Two-way ANOVA revealed a statistically significant effect for the adhesive systems ( $p<0.01$ ) and not for the HVGICs ( $p=0.05$ ) nor their interactions ( $p=0.99$ ). When using Clearfil SE Bond 2 and Clearfil Universal in a light-cure mode, significantly higher  $\mu$ SBS values were found when compared with Clearfil Universal in a self-cure mode.

**Conclusions:** The three tested HVGICs can be successfully repaired using two-step or one-step self-etch adhesive systems. The one-step self-etch adhesive system in light-cure mode is preferred when compared with the self-cure mode.

## INTRODUCTION

Minimal tooth preparation and the application of adhesive therapeutic restorations are among the targets in minimal-intervention dentistry. The atraumatic restorative treatment (ART) approach fulfills these goals where the carious tooth is prepared using special hand instruments and high-viscosity glass-ionomer cements (HVGICs) are used. Underserved communities that lack electricity can also benefit from this approach when using the hand-mixed version of HVGICs because the need for an amalgamator to mix the activated capsules is removed. Also, a light curing unit is not required in contrast with many resin-modified glass-ionomer restorative materials. For primary teeth, a 93% success rate after two years has been recorded for single-surface ART restorations<sup>1</sup> and a 62% success rate was found for the compound or complex proximal restorations.<sup>2</sup>

When used in permanent teeth, ART recorded a 97% and 85% success rate for single-surface restorations after two and three years of clinical service, respectively;

and a 95% and 85% success rate for proximal restorations after one and two years of clinical service, respectively.<sup>1,3,4</sup> Partial or bulk fracture of the proximal part of the restoration was reported to be one of the main causes of failure of proximal restorations.<sup>5</sup>

Marginal fracture of ART restorations can occur after a short time of clinical service, even within 24 hours, due to improper isthmus carving (especially if the restoration was placed by a less experienced operator), the presence of an unobserved plunger cusp, the induction of a crack during the removal of the proximal band, or inadvertent biting on a hard object (particularly within the first 24 hours of clinical service). Additionally, ART proximal restorations can fracture during clinical use, since the strength of the glass ionomer cement (GIC) materials (including HVGICs) cannot compete with the strength of direct resin composite materials.<sup>6-8</sup>

Repair, rather than replacement, of defective restorations is more conservative and cost effective. The immediate bonding of GICs to resin composite was reported to be acceptable.<sup>9</sup> A study was recently performed to evaluate the bonding ability of aged HVGIC using resin composite bonded using etch-and-rinse and self-etch adhesives.<sup>10</sup>

Two complications that may be encountered during the repair of defective HVGICs by direct resin composite is (1) the lack of electricity to cure the resin material, or (2) to have a fractured ditch deep enough to cause concern over the depth of cure with the light curing unit. In these cases, the use of adhesive systems and resin materials with a self-curing mode could be a solution.

To date, there are no studies regarding the repair potential of the different types of HVGICs used for the ART approach in conjunction with resin composite that is bonded using the universal one-step adhesive system in either the light-cure or self-cure mode.

The null hypotheses of the current study were: (1) there is no difference in the repair  $\mu$ SBS among the different HVGICs, and (2) there is no difference between the different adhesive systems/curing modes on the repair  $\mu$ SBS values of the tested HVGICs.

## METHODS AND MATERIALS

Materials used in this study, and their batch numbers, manufacturers, and compositions are listed in Table 1.

### Specimen Preparation

A stainless-steel flat washer, 20 mm in diameter and 2-mm thick, was used as a mould. The mould provided

Table 1: Tested Material Names, Batch Numbers, Manufacturers, and Chemical Compositions

Material/Batch No.	Manufacturer	Composition
GC Fuji IX GP Fast (radiopaque posterior glass-ionomer restorative cement in capsules; #0804141)	GC Corporation, Tokyo, Japan	Alumino-fluoro-silicate glass, polyacrylic acid, distilled water, polybasic carboxylic acid.
Fuji IX GP containing chlorhexidine HVGIC (radiopaque posterior glass-ionomer restorative cement in powder/liquid)	GC Corporation, Tokyo, Japan	Powder: Alumino-fluoro-silicate glass to which 1% chlorhexidine diacetate was incorporated.  Liquid: polyacrylic acid, distilled water, polybasic carboxylic acid.
ChemFil Rock (advanced glass-ionomer restorative material in capsules; #K79200030-03)	Dentsply De-Trey GmbH, Konstanz, Germany	Calcium-aluminium-zinc-fluoro-phosphor-silicate glass, polycarboxylic acid, iron oxide pigments, titanium dioxide pigments, tartaric acid, water.
Dentin conditioner (#280739GC)	GC Corporation, Tokyo, Japan	20% polyacrylic acid, 3% aluminum chloride hexahydrate component.
Clearfil SE Bond 2 (two-step, self-etch adhesive system; dental universal self-etch adhesive; primer: #3282KA; bond: #3281KA)	Kuraray Noritake Dental Inc., Tokyo, Japan	Primer: MDP, HEMA, hydrophilic dimethacrylate, dl-camphorquinone, N,N-diethanol-ptoluidine, water.  Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dlcamphorquinone, N,N- diethanol-p-toluidine, silanated colloidal silica.
Clearfil Universal Bond (single component adhesive; #6B0016)	Kuraray Noritake Dental Inc., Tokyo, Japan	Bis-GMA, HEMA, ethanol, 10-MDP, hydrophilic aliphatic dimethacrylate, colloidal silica, dl- camphorquinone, silane coupling agent, accelerators, initiators, water.
Clearfil DC Activator (#3250KA)	Kuraray Noritake Dental Inc., Tokyo, Japan	Activator, ethanol, catalysts, accelerators.
Clearfil DC Core Plus Dual-cure, radiopaque two-component core build-up material (#2942KA)	Kuraray Noritake Products Corporation, Tokyo, Japan	Paste: Bis-GMA, hydrophilic aliphatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate, silanized barium glass filler, silanized colloidal silica, colloidal silica, chemical- initiator, photo-initiator, pigments.  Paste: TEGDMA, hydrophilic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate, silanized barium glass filler, silanized colloidal silica, aluminum oxide filler, photo-accelerator, chemical-accelerator.
Abbreviations: Bis-GMA, bis-phenol A diglycidylmethacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethylene-glycol dimethacrylate.		

an internal hole with a 12 mm diameter to allow for the packing of the tested material. A total of 105 discs of the three tested HVGICs: Fuji IX GP Fast capsules (GC Corporation, Tokyo, Japan), Fuji IX GP glass-ionomer cement containing chlorhexidine (GC corporation, Tokyo, Japan), and ChemFil Rock zinc-reinforced capsules (Dentsply De-Trey GmbH, Konstanz, Germany) were prepared. The mould was placed on a celluloid strip and a dry glass slab. Then, each tested HVGIC was mixed according to the manufacturer's instructions and packed into the internal hole of the mould. Another glass slide was placed over the packed disc, with pressure to compact the material until it was completely set.

Specimens were left to set at room temperature (23°C) and at 100% humidity for 20 minutes. After removal of the glass slab, each HVGIC disc was checked for any pitting or defects to be discarded. Vaseline was applied and the discs were stored at 100% humidity in an incubator with a 37°C adjusted temperature for 24 hours.

### Grouping of the Specimen

Two equidistance horizontal notches were made to divide the HVGIC disc into three horizontal sections. Each section received one of the tested repair adhesive systems: light-cured Clearfil SE Bond 2 (two-step, self-etch adhesive; Kuraray Noritake Dental Inc., Okayama, Japan) or Clearfil Universal Bond (one-step, self-etch adhesive; Kuraray Noritake Dental Inc., Okayama, Japan) in light-cure mode or Clearfil Universal Bond (one-step, self-etch adhesive) in self-cure mode, with Clearfil DC Activator (Kuraray Noritake Dental Inc., Okayama, Japan). A dual-cured Clearfil DC Core Plus (Kuraray Noritake Dental Inc., Okayama, Japan) with an automix delivery system was used as the repair material. A build-up microcylinder was bonded with each adhesive system on each HVGIC disc, providing 315 microcylinders (n=35 per group).

### Restorative Procedures

All HVGIC discs were wet-ground flat using 600-grit silicon carbide paper to obtain a smooth, matte surface, and then etched using Scotchbond Etchant gel (3M ESPE, St Paul, Minnesota, USA) for 15 seconds, rinsed with oil-free water from an air/water syringe for 15 seconds, and blotted dry using gauze to prevent desiccation of the cement.

Starch tubes (pasta ZARA, Brescia, Italy) with a 0.96-mm internal diameter were cut to a height of 1 mm to be used to build-up the Clearfil DC Core Plus microcylinders.<sup>11</sup> The tip of the automix syringe of the

Clearfil DC Core Plus was used to inject the material into the starch tubes. After the application of each adhesive system (according to the manufacturer's instructions), a filled starch tube was randomly placed onto each horizontal section and light cured for 20 seconds using the LED Curing Light (GC America Inc., Alsip, Illinois, USA), with a wavelength range of 440-490 nm and an energy output of 650 mW/cm<sup>2</sup>. The light intensity of the curing unit was checked using an LED radiometer (Kerr Dental Specialties, Orange, California, USA) at the beginning of the study and every week during the study period.

For the first horizontal section, Clearfil Bond 2 Primer was applied for 20 seconds and dried with a mild air flow for 5 seconds. Clearfil Bond 2 Bond was applied and gently air-thinned using oil-free air for 2 seconds, and then light cured for 10 seconds. For the middle horizontal section, Clearfil Universal Bond was rubbed for 10 seconds, gently air-dried using oil-free air for 5 seconds, and light cured for 10 seconds.

One drop of Clearfil Universal Bond was mixed with one drop of Clearfil DC Activator, and the mixed adhesive was applied to the third horizontal section of each HVGIC disc. The adhesive was rubbed for 10 seconds and dried using mild air flow for 5 seconds. A Clearfil DC Core Plus build-up-filled starch tube was placed onto the third section of the disc. This section, that received the self-cure mode repair system, was allowed to dark-cure for 20 minutes. Afterward, all bonded HVGIC discs were immersed in artificial saliva for 4 hours at 37°C to soften the starch tubes. The softened starch tubes were carefully removed using a #11 sharp lancet (Wuxi Xinda Medical Device Co., Jiangsu, China), leaving the resin composite microcylinders bonded to the HVGIC discs. Resin composite microcylinders were checked using a magnifying lens (Bausch and Lomb, Co. Rochester, New York, USA) at 6x magnification to detect interfacial gaps, bubble inclusions, or other defects, which were excluded. Bonded discs were stored in artificial saliva in a 37°C incubator for 24 hours.

### Microshear Bond Strength Testing

To avoid bias, the bonded discs were coded by a person other than the authors, thus blinding the testing and statistical analysis.<sup>12</sup> Each bonded HVGIC disc was secured in the lower part of a specially designed attachment jig to hold the specimens to the testing machine.<sup>13</sup> The attachment jig was in turn screwed into the lower fixed and the upper movable compartments of the testing machine (Model LRX-plus; Lloyd Instruments Ltd., Ferham, UK), with a load cell of 5 kN. A wire loop prepared from a 180 µm orthodontic



stainless-steel ligature wire (G&H Orthodontics, Franklin, Indiana, USA) was wrapped around the bonded microcylinder as close as possible to its base and touching the HVGIC surface. A tensile load was applied via the testing machine at a crosshead speed of 0.5 mm/minute. Data were recorded using computer software (Nexygen-MT; Lloyd Instruments, UK). The calculation of the  $\mu$ SBS value was done by dividing the load at failure by the bonding area to express the bond strength in MPa.

### Statistical Analysis

Data were statistically presented in terms of mean  $\pm$  standard deviation (SD). In the present study, the repair bond strengths of the different adhesives were considered as the dependent variables, while the HVGICs were the independent variables. Normal distribution of the data was verified using the Kolmogorov-Smirnov test. A two-way ANOVA test was performed to determine the effect of the adhesive systems and the HVGICs on the repair bond strength. Two-way ANOVA was also used to detect any significant interactions between these two variables. One-way ANOVA was used to detect any significant differences among the  $\mu$ SBS repair values of each tested adhesive system applied with the different HVGICs and among the  $\mu$ SBS repair values of each tested HVGIC repaired with the different adhesive systems. Bonferroni test was used for pairwise comparisons. Statistical calculations were done using the computer program SPSS for Microsoft Windows version 15 (Statistical Package for the Social Science; SPSS Inc., Chicago, Illinois, USA).

### Mode of Failure

After measuring the bond strength, each HVGIC disc was examined using an environmental scanning electron microscope (Quanta 200; FEI Company, Philips, Netherlands) at 25 Kv to determine the failure modes of the detached microcylinders. The failure mode was categorized as follows:

Type I: Adhesive failure at the HVGIC interface.

Type II: Cohesive failure in the adhesive layer.

Type III: Cohesive failure in HVGIC.

Type IV: Mixed failure (involving both adhesive and cohesive failures).

Representative photomicrographs for the failure modes were captured at various magnifications.

### RESULTS

The mean and SD for each experimental group are listed in Table 2. Two-way ANOVA revealed a statistically significant effect for the adhesive systems ( $p < 0.01$ ) but not for the HVGICs ( $p = 0.05$ ) and their interactions ( $p = 0.99$ ). Based on this, the first null hypothesis failed to be rejected, while the second null hypothesis was rejected.

For each HVGIC (GC Fuji IX GP Fast, Fuji IX GP containing chlorhexidine, and ChemFil Rock), one-way ANOVA revealed a statistically significant difference among the three adhesive systems ( $p = 0.02$ ), as shown in Table 2. The light-cured modes of Clearfil SE Bond 2 and Clearfil Universal presented significantly higher  $\mu$ SBS values when compared to Clearfil Universal in self-cure mode (Table 2).

Table 2: Repair Microshear Bond Strength Values (Mean [SD]) in MPa of the Tested Adhesives to the Different High-viscosity Glass-ionomer Cements

HVGICs	Adhesive Systems			
	Clearfil SE Bond 2	Clearfil Universal Bond light-cure mode	Clearfil Universal Bond self-cure mode	p-value
GC Fuji IX GP Fast	23.45 (7.4) <sup>aA</sup> [Ptf/tnt=0/35]	21.06 (6.7) <sup>aA</sup> [Ptf/tnt=0/35]	15.75 (5.8) <sup>aB</sup> [Ptf/tnt=3/35]	0.025
Fuji IX GP- CHX	27.66 (6.5) <sup>bA</sup> [Ptf/tnt=0/35]	25.69 (8.5) <sup>aA</sup> [Ptf/tnt=0/35]	19.29 (8.0) <sup>aB</sup> [Ptf/tnt=3/35]	0.025
ChemFil Rock	25.96 (6.3) <sup>aA</sup> [Ptf/tnt=0/35]	24.47 (7.3) <sup>aA</sup> [Ptf/tnt=0/35]	18.48 (6.8) <sup>aB</sup> [Ptf/tnt=4/35]	0.028
p-value	0.32	0.31	0.40	

Different uppercase letters denote significant differences within rows. Different lowercase letters denote significant differences within a column.

Abbreviations: CHX, chlorhexidine; HVGICs, high-viscosity glass-ionomer cements; Ptf, pretest failure; SD, standard deviation; tnt, total number of tested specimens.

Regarding the failure modes, when GC Fuji IX GP Fast, Fuji IX GP containing chlorhexidine, and ChemFil Rock HVGICs were bonded using the light-cured Clearfil SE Bond 2 and Clearfil Universal, they presented predominately mixed failures followed by cohesive failures in the adhesive layer. Alternatively, Clearfil Universal in self-cure mode mainly presented cohesive failures in the adhesive layer. Figure 1 depicts the percentages of the recorded failure modes. Representative scanning electron micrographs for some failure modes of the tested HVGIC specimens are presented in Figure 2.

DISCUSSION

Previous studies have mainly focused on the reparability of the resin-modified GICs.<sup>14-16</sup> Additionally, the bond strength of earlier versions of conventional GICs to resin composite has been investigated.<sup>17-21</sup> Nevertheless, no published study has considered the reparability of the recent types of HVGICs, which are used as final restorations in ART treatment, and using resin composite with different curing modes.

Based on the results of this current study, all tested HVGICs could successfully be repaired. Researchers showed that successful bonding to GIC is based on two components: inherent microporosities of the material and the use of an interfacial bonding agent.<sup>17</sup>

When evaluating the structure, the relatively large glass particles embedded in the HVGIC matrix provides porosities, which could act as undercuts capable of retaining additional material that can penetrate within those porosities. The relatively higher, but not significant, repair bond strength of all tested adhesive systems to chlorhexidine containing HVGIC could validate this hypothesis. As it is, a hand-mixed powder and liquid product, chlorhexidine containing HVGIC could present an increase in microporosities when compared with the other tested capsulated HVGICs, which could allow for an increase in the microretention of the intermediate materials.<sup>22</sup> Meanwhile, the minor difference in porosity between restorative cements mixed with both methods<sup>23</sup> might explain the nonsignificant differences in the results recorded among the tested HVGICs in the present study.

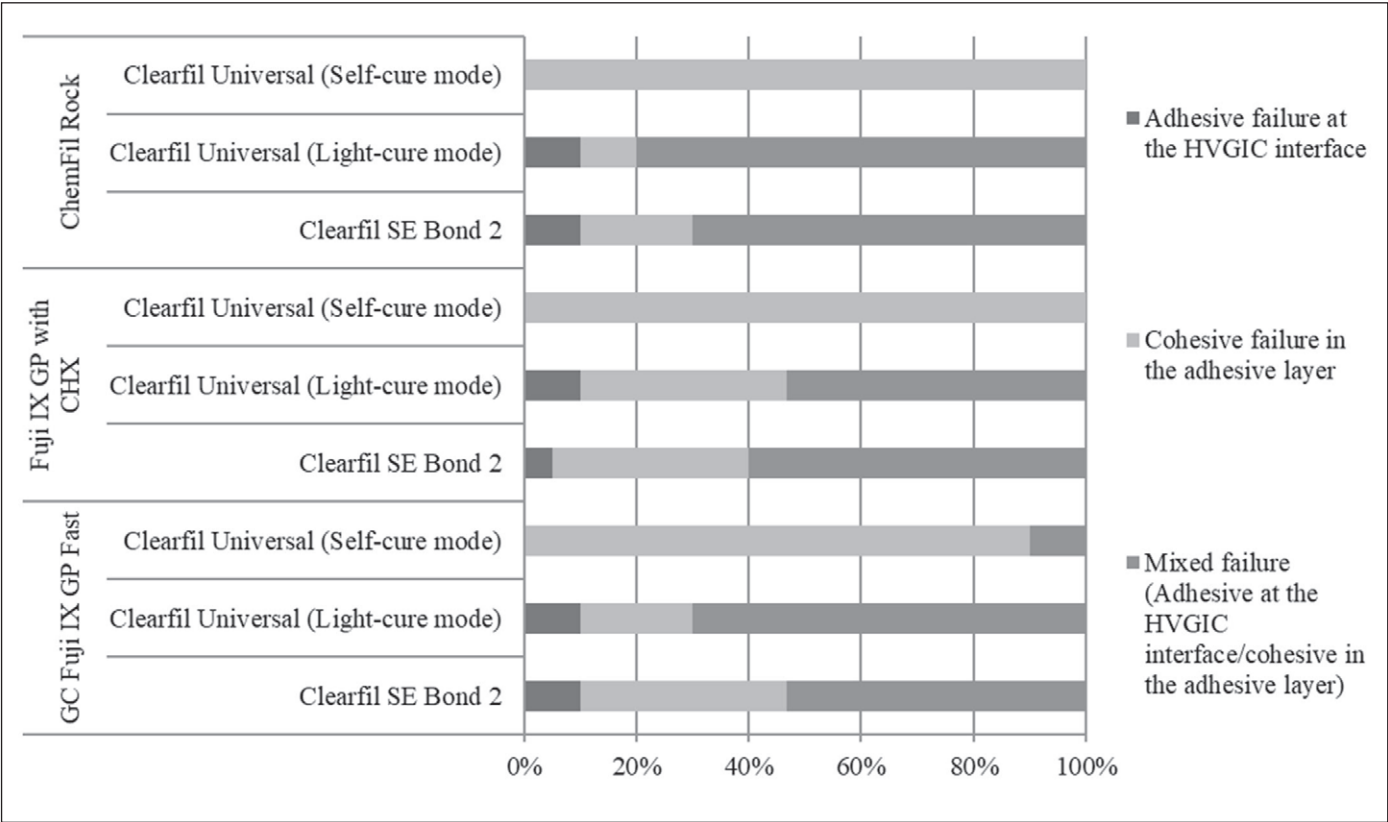


Figure 1. The percentages of the recorded modes of failure in the tested groups.

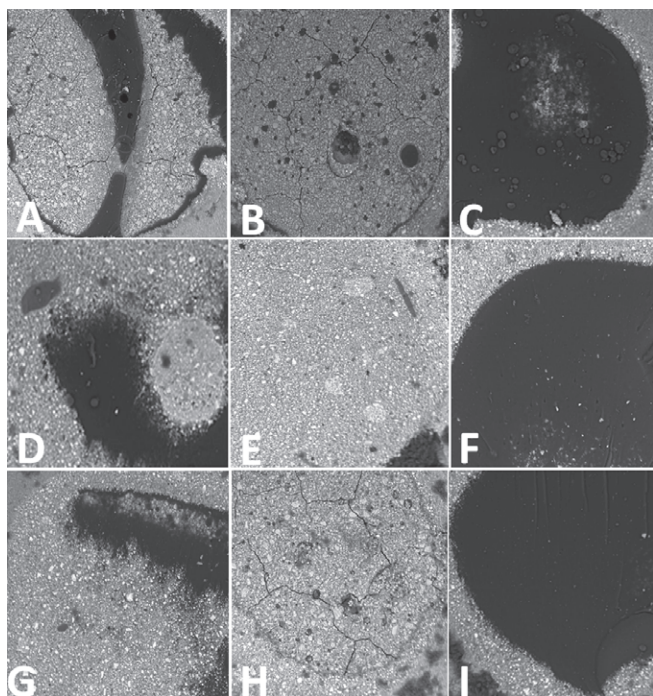


Figure 2. SEM photomicrographs showing the failure modes of GC Fuji IX GP Fast (A–C), Fuji IX GP containing chlorhexidine (D–F), and ChemFil Rock (G–I). (A, D, G): Represent mixed failure; (adhesive failure at the HVGIC interface and cohesive failure in the adhesive layer). (B, E, H): Represent adhesive mode of failure at the HVGIC interface. (C, F, I): Correspond to the cohesive mode of failure in the adhesive layer. Abbreviation: SEM, scanning electron microscope.

Etching prior to performing a repair is a point of controversy. Some authors found that etching the material had no significant effect on the bond strength,<sup>20,21</sup> with some authors proving that it weakened the cohesive strength of the material,<sup>24</sup> and others demonstrating that it improved the bond strength.<sup>19,25,26</sup>

Despite the differences in the experimental methods, those studies concluded that etching could be a practical step provided it was done after complete setting of the material (24 hours), utilized 37% phosphoric acid concentration for 15 seconds, and was followed by 15 seconds of rinsing.<sup>9</sup> Etching of the material to be repaired is clinically recommended, as it acts as a microscopic cleaning procedure and helps to expose fresh glass particles, which could enhance the chemical interaction with some self-etch adhesives. Moreover, the evaluated HVGICs proved to have high physicomechanical properties<sup>27,28</sup> that could allow them to withstand the minor effect of etching.

Reliable bonding between GICs and resin composites should always be done in conjunction with an effective intermediate bonding agent.<sup>17</sup> Self-etch adhesive

systems are user friendly, less technique sensitive, and require less clinical application time; therefore, they are frequently used to achieve the equivalent bond strength to etch-and-rinse adhesives. To date, one study discussed the bond strength of light-cured self-etch adhesive systems to one HVGIC.<sup>10</sup>

It has been suggested that some self-etch adhesives are able to form a chemical bond to the calcium content of the tooth structure; therefore, they also could chemically bond to the calcium and strontium present in the HVGICs.<sup>10</sup> This could explain the high bond strength of the two-step Clearfil SE Bond 2 or one-step Clearfil Universal adhesive, as both contain 10-methacryloyloxydecyl dihydrogen phosphate, which can bond to the calcium and strontium contents of HVGICs.<sup>10</sup>

Nonetheless, it should be noted that universal adhesives have an increased hydrophilicity and high acidic monomer concentration. Water is required to ionize the acidic monomer, dissolve the smear layer, and demineralize the substrate. High acidic monomer concentrations could lead to water sorption and osmotic blistering, resulting in a decrease of the marginal integrity of the adhesive and creating a weakened adhesive area. The repair bond durability of HVGICs with universal adhesives is still under investigation.

This present study showed a significantly low bond strength of the Clearfil Universal adhesive in self-cure mode when compared with the light-cured approach. Although no published study investigated the performance of the universal adhesive in the light-versus self-cure modes bonded to HVGIC, the results of Foxton and others reported that light exposure of the dual-cured adhesive improved its bonding to root dentin.<sup>29</sup>

Previous studies have reported an incompatibility problem between the residual acidic monomers present at the oxygen-inhibited layer of the simplified adhesive, which react with the initiator component (aromatic tertiary amine) in the dual-cured composite core; this reaction hinders polymerization of the material. The use of a self-cured activator has been suggested to eliminate this incompatibility. The latter has a sodium salt of aryl sulfinic acid, which reacts with the acidic monomers to produce phenyl- or benzene-sulfonyl free radicals to initiate the self-cured composite polymerization. However, the use of a self-cured activator did not achieve a comparable result to the use of the universal adhesive in either the light-cured or dual-cured modes.<sup>30</sup> It is important to know that Clearfil DC Core Plus, which was used in the present study, has a “slow” setting, self-cure mechanism. This is due to the reduction in its camphorquinone content,



which minimizes its sensitivity to ambient light. Further research is required to determine whether the slow setting mechanism inhibits polymerization due to the diffusion of the acidic monomer from the adhesive system to the resin composite.<sup>30, 31</sup>

The results of this current study add to the clinical knowledge of the repair of ART restorations. The idea of using a dual-cured resin composite core build-up to overcome some of the limitations of a light-cured resin composite could be of value. The dual-cured resin composite provides proper handling characteristics, extended working time, and it eliminates the depth-of-cure problem faced in critically inaccessible areas. Manufacturers have claimed that these materials will satisfactorily cure in 5 to 7 minutes without light exposure. It has been suggested that a 20- to 60-second delay in light-curing dual-cured core build-up materials could minimize the interference of the self-curing mechanism of these materials, allowing initial conversion and decreasing the polymerization shrinkage stresses of the materials.<sup>32</sup> However, this delay might allow for moisture contamination or added time during clinical procedures that might complicate the case. Further research on the curing performance of dual-cured resin composite is recommended.

Analysis of the mode of failure as a complementary step for bond strength testing was performed in the present study. All HVGICs bonded with the light-cured Clearfil SE Bond 2 and Clearfil Universal light-cured mode specimens showed a predominantly mixed type of failure. On the other hand, the specimens of Clearfil Universal in self-cure mode predominantly had cohesive failures in the adhesive layer, which is a concern regarding the quality of bonding. These results were in agreement with the bond strength results.

Finally, the present study demonstrates that the use of a dual-cure resin composite material in combination with light-cured, self-etch adhesive systems could be a successful repair approach for defective or undercontoured HVGICs used in ART restorations. The use of the self-cure mode in repairing defective restorations requires further study to enhance its bond strength values and quality of bonding. ART is a minimally invasive approach to dentistry, representing an optimum treatment option for geriatric patients, patients with special needs or rare diseases, and patients with dental anxiety. The use of ART can be extended to various restorative techniques, such as the delayed sandwich technique and protective restorations. Moreover, working on self-cure approach to be feasible in the absence of eccentricity in rural areas as well as in war zones is of a prime request.

## CONCLUSIONS

The three tested HVGICs could be successfully repaired using two-step/one-step self-etch adhesive systems. The one-step self-etch adhesive system in light-cure mode is preferred when compared with the self-cure mode.

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## Conflict of Interest

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

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# Masking of High-Translucency Zirconia for Various Cores

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## Clinical Relevance

Various core materials with different shades affect the final color of high-translucency monolithic zirconia restorations. The blue core shows the greatest color difference in final zirconia restorations followed by metal, A3 dentin-shade resin core, and white core.

## SUMMARY

The purpose of this study was to evaluate the masking ability of high-translucency monolithic zirconia for various core materials. A computer-aided design-computer-aided manufacturing system was used to design a zirconia disc with a diameter of 10 mm and a thickness of 1.0 mm. Four groups of cores (n=15 each) were fabricated with blue-colored dual-cure resin, white-colored dual-cure resin, A3 dentin-shade composite resin, and titanium block with 10-mm diameter and 5-mm thickness.

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Dual-cure, self-adhesive resin cement discs with a thickness of  $25.0 \pm 0.02 \mu\text{m}$  were fabricated. The color was measured using a handheld spectrophotometer. Color measurements of all specimens were performed on a white background. To assess the masking ability of zirconia, the difference between the values measured with zirconia on a white background and the values measured with zirconia on each of the four types of core material as a background with the cement specimens interposed (zirconia + cement + core) was determined. To enhance the optical connection between the specimens, distilled water was applied between each layer during each measurement.

The results showed that the value of  $\Delta E$  was highest for the blue core followed by metal, A3 dentin-shade resin core, and white-resin core. No significant differences were observed between the metal core and the A3 dentin-shade resin core or between the A3 dentin-shade resin core and the white core. The blue core had the significantly highest  $\Delta E$  value based on Tukey's honest significant difference test.

Different core materials affect the final color of high-translucency monolithic zirconia restorations. Thus, our study showed that the final color of high-translucency monolithic

**zirconia restorations could be affected by the type of core material used.****INTRODUCTION**

The use of all-ceramic restorations has considerably increased since the introduction of zirconia in dentistry. Ceramics have become a universally accepted material of choice for the restoration of anterior as well as posterior teeth, because of the adequate mechanical characteristics and outstanding esthetics.<sup>1</sup>

To reduce the risk of veneer fracture and to simplify the process of restoration, manufacturers have recently introduced monolithic zirconia restorations.<sup>2</sup> The use of monolithic zirconia restorations is increasing in restorative dentistry because of their biocompatibility, superior esthetics, simple clinical technique, and low cost relative to cast gold restorations.<sup>3</sup> Additional characteristics of monolithic zirconia restorations include natural toothlike appearance, low corrosion potential, and low thermal conductivity.<sup>4-6</sup>

Zirconia restorations do not require excessive tooth preparation such as in glass-based all-ceramic crowns<sup>7</sup> owing to their strong mechanical properties (flexural strength of 900 to 1500 MPa).<sup>8</sup> However, monolithic zirconia restorations have compromised esthetics because of lower translucency as compared with that of glass ceramic restorations.<sup>9</sup> This could be attributed to the increased size of crystalline particles, which induce greater light scattering and reduced translucency because of the decreased passage of light through the material.<sup>10</sup>

Dental manufacturers and laboratories have gradually overcome this weakness and have recently been marketing high-translucency monolithic zirconia restorative materials that have high esthetics and excellent strength properties.<sup>9</sup> Such monolithic zirconia materials are decent alternatives to conventional materials used in esthetic restorations and meet the requirements of both patients and dentists by providing higher translucency without sacrificing strength properties.<sup>9</sup>

Of the high-translucency monolithic zirconia products recently launched in the market, Lava Esthetic Fluorescent Full-Contour Zirconia is 5Y-PSZ (5 mol% yttria partially stabilized zirconia) and consists of cubic-phase zirconia in concentrations greater than 50%.<sup>11</sup> Lava Esthetic is aimed at not only improving the translucency but also reproducing the fluorescence of the tooth itself, and it has been reported to have higher fluorescence than Lava Plus.

The maximum value of the fluorescence spectrum of Lava Esthetic is about 450 nm (blue), a value very close to the fluorescence spectrum of bovine dentin. Lava Esthetic shows dentin-like fluorescence even in darker shades, such as A3.5, whereas Lava Plus is noticeably fluorescent only in lighter shades such as A1.<sup>12</sup>

The importance of the core color increases as the translucency of zirconia increases. Zirconia is used not only for the restoration of prepared teeth but also for the prosthetic restoration of implants, wherein it may cover a titanium abutment, as well as for the restoration of blue-colored cores when blue-colored core materials are used for accurate distinction of the margins in a wide range of restorations. As discussed, zirconia should possess a certain ability of masking the core, and hence, information about the type and thickness of zirconia with optimal masking ability and translucency will be continuously required.

Basso and others<sup>13</sup> stated that monolithic glass ceramics can mask C4-shade cores but not metal cores, and they have a lower color-masking ability compared with that of glass-ceramic-layered zirconia. Moreover, it was reported that the  $\Delta E$  value decreased as the thickness of the monolithic substructure increased from 0.7 to 2 mm.<sup>13</sup>

Kim and Kim<sup>14</sup> compared the optical properties of precolored monolithic zirconia ceramics, veneered zirconia, and lithium disilicate glass ceramics and found that the amount of color change was beyond the acceptability threshold. They concluded that precolored monolithic zirconia ceramics may cause color mismatch because of high  $L^*$  and low  $a^*$  and  $b^*$  values.<sup>14</sup>

Tabatabaian and others<sup>15</sup> stated that if the treatment option is monolithic zirconia restoration on an A4 shade core material or a prepared tooth (with dentin color), the thickness of the restorative material should be at least 0.9 mm to attain an acceptable final shade, considering the size of the core and/or the possible amount of tooth removal required.<sup>15</sup>

However, monolithic zirconia used in several studies was a low-translucency block with high opacity, unlike those used in recent times, with enhanced translucency. Hence, the results showed considerable limitations for its use in esthetic restorations. Furthermore, with the growing demands of zirconia for implant restorations, knowledge about the possible degree of masking of titanium abutments has become increasingly important to enhance the outcome of esthetic restorations.

Therefore, the purpose of this study was to evaluate the masking ability of high-translucency monolithic zirconia for various core materials.

The null hypothesis was that different core materials would not affect the final color of high-translucency monolithic zirconia restorations.

## METHODS AND MATERIALS

### Zirconia Specimen Preparation

A computer-aided design–computer-aided manufacturing system (Rhinoceros 5 CAD program, Rhinoceros 5 SR 13, Robert McNeel & Associates, Seattle, WA, USA) was used to design a zirconia disc (Lava Esthetic Fluorescent Full-Contour Zirconia Discs [LE], 3M ESPE, St Paul, MN, USA) with a diameter of 10 mm and thickness of 1.0 mm. Shade A2 zirconia blocks were milled using the Roland milling machine (Roland DWX-52D, Roland DGA Corporation, Irvine, CA, USA), which was calibrated by the CAM software (hyperDENT, Open Mind Technologies AG, Wessling, Germany). After the completion of the milling process, specimens were sectioned from the sprue and were trimmed. Specimens were contained in a sintering box and were sintered according to the manufacturer's recommendations. Since they were manufactured in the A2 shade, the dipping process was not performed. The specimens were sintered at a maximum temperature of 1520°C for 12 hours in a sintering furnace.

Zirconia discs were sequentially polished with 600-, 800-, 1000-, and 1200-grit silicon carbide abrasive papers in a polishing machine accompanied by water cooling to obtain the predetermined thickness ( $1.0 \pm 0.02$  mm). A digital micrometer (293 MDC-MX Lite, Mitutoyo Corp, Tokyo, Japan) with an accuracy of 0.002 mm was used to measure the thickness. Only one surface of the disc was polished to simulate clinical conditions.

### Core Disk Preparation

Four groups of cores ( $n=15$ , each) were fabricated with blue-colored dual-cure resin (Core-flo DC, Bisco Inc, Schaumburg, IL, USA), white-colored dual-cure resin (Core-flo DC, Bisco Inc), A3 dentin-shade composite resin (Filtek Z350 A3 dentin, 3M ESPE), and titanium block (Osstem TS premilled abutment, Osstem Implant, Seoul, Korea). Acrylic plates were prepared with a hollow space of 10-mm diameter and 5-mm height to fabricate a mold for the resin core. The blue-colored and white-colored resin were added to the mold with the help of a Mylar strip (SKY Striproll 10, Suki Dental Co,

Goyang, Gyeonggi-do, South Korea). When the mold was filled up to 5 mm, the Mylar strip was placed on the top of the resin. The core material was polymerized with a light-polymerizing unit (Smart-Lite Pen Style LED curing light, Dentsply DeTrey, Konstanz, Germany) for 40 seconds with an intensity of 800 mW/cm<sup>2</sup>. An A3 dentin-shade composite resin was added in increments of 2 mm and light-cured with the curing unit as described previously. The resin cores were sequentially polished with 600-, 800-, 1000-, and 1200-grit silicon carbide abrasive papers in a polishing machine. The titanium core was custom fabricated using a titanium abutment block (Osstem TS premilled abutment) with 10-mm diameter and 5-mm thickness. The same micrometer was used to measure the thickness of the titanium core ( $5.0 \pm 0.02$  mm). If the thickness was less than the intended thickness, the core was discarded.

### Cement Disk Preparation

Dual-cure, self-adhesive resin cement (3M RelyX U200 A2, 3M ESPE) was used (Table 1). Cement was mixed according to the manufacturer's recommendations. The mixture of resin cement was applied between two polyester strips, and the strips were kept below a hard transparent plate under pressure of 9.8 N.<sup>16</sup> The cement was polymerized with a polymerizing light unit (SmartLite Pen Style LED curing light, Dentsply DeTrey) at an intensity of 800 mW/cm<sup>2</sup> for 20 seconds from each side.

The cured cement was trimmed with a blade to conform to the shape of the resin core disc. The thickness of the cement disc was adjusted with polishing. Until the intended thickness was obtained ( $25.0 \pm 0.02$   $\mu$ m), the cement specimen was polished and subsequently measured by digital micrometer.

### Color Measurement

The color was measured using a handheld spectrophotometer (Vita EasyShade V, Vita Zahnfabrik, Bad Säckingen, Germany). A silicon putty index (3M ESPE Express STD, 3M ESPE) was fabricated to maintain similar conditions in all the specimens despite different materials being used and to avoid external light.<sup>17,18</sup> Color measurements of all the specimens were performed on a white background. To assess the masking ability of zirconia, the difference between the values measured with zirconia on the white background and the values measured with zirconia on each of the four types of



Table 1: Materials Used in This Study

Table 1: <i>Materials Used in This Study</i>						
Material		Manufacturer	Lot No	Diameter, mm	Thickness, mm	
Zirconia						
LE 1.0	Lava Esthetic Fluorescent Full-Contour Zirconia	3M ESPE, St Paul, MN, USA	3994896	10	1	
Core						
Blue	Core-Flo DC Blue	Bisco Inc, Schaumburg, IL, USA	1800002442	10	5	
Metal	Osstem TS premilled abutment	Osstem, Seoul, Korea	PTA18F234			
White	Core-Flo DC	Bisco Inc, Schaumburg, IL, USA	170003296			
	Opaque white					
A3 shade resin	Filtek Z350	3M ESPE, St Paul, MN, USA	N718626			
Cement						
U200A2	RelyX U200 Automix self-adhesive resin cement	3M Deutschland GmbH	3722465	10	0.025	

core material as a background with the cement specimens interposed (zirconia + cement + core) was determined. To enhance the optical connection between the specimens, distilled water was applied between each layer during each measurement.

The data were presented in  $L^*$ ,  $a^*$ , and  $b^*$  values according to the Commission International de l'Eclairage or International Commission on Illumination.<sup>19</sup>

$\Delta E$  values were calculated using the following formula:

$$\Delta E_{ab}^* = [(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2]^{1/2}$$

Vichi and others<sup>20</sup> provided three different ranges to differentiate between color shifts. A  $\Delta E$  value less than 1.0 is considered undetectable by the human eye, and a  $\Delta E$  value between 1.0 and 3.3 is considered visible by skilled operators but clinically acceptable. A  $\Delta E$  value greater than 3.3 is not clinically acceptable, because it is appreciable by a nonskilled person.

Accordingly, in the present study, the clinically acceptable limit was set at a  $\Delta E$  value of 3.3, the threshold used in several studies.

### Statistical Analysis

Statistical analyses were performed using R statistical software, version 3.5.1 (R Development Core Team, R Foundation for Statistical Computing, Vienna, Austria).

Significant differences between the groups were determined using the Tukey's honest significant difference (HSD) test and one-way analysis of variance ( $\alpha=0.05$ ).

### RESULTS

The experimental study protocol is summarized in Figure 1. The  $\Delta E$  values of color change were computed by using the above-mentioned formula with the  $L^*$ ,  $a^*$ , and  $b^*$  values for LE 1.0 alone and the corresponding values for LE 1.0 combined with each of the four types of cores. All measurements were performed with a white background. Figure 2 shows the  $L^*$ ,  $a^*$ , and  $b^*$  values with zirconia on the white background and zirconia combined with cement and the four different core materials.

The results showed that the value of  $\Delta E$  was highest for blue core, followed by metal, A3 dentin-shade resin core, and white-resin core (Table 2). No significant differences were observed between the metal core and the A3 dentin-shade resin core or between the A3 dentin-shade resin core and the white core. The blue core had the significantly highest  $\Delta E$  value based on Tukey's HSD test.

### DISCUSSION

According to the results, the highest change in color was evident with LE 1.0 mm combined with RelyX U200 A2 cement and the blue core. In contrast, no significant differences were observed in color between the metal core and A3 dentin-shade resin core, which is considered to have a similar color to a prepared tooth.

The zirconia and the cement used in this study were of the A2 shade; however, the shade of the core material was darker or lighter than A2, which made it impossible to mask the color of the core with zirconia and cement. Particularly, the zirconia used in this study showed high translucency, and hence, its masking ability was not optimum. However, with the increasing esthetic demands, efforts

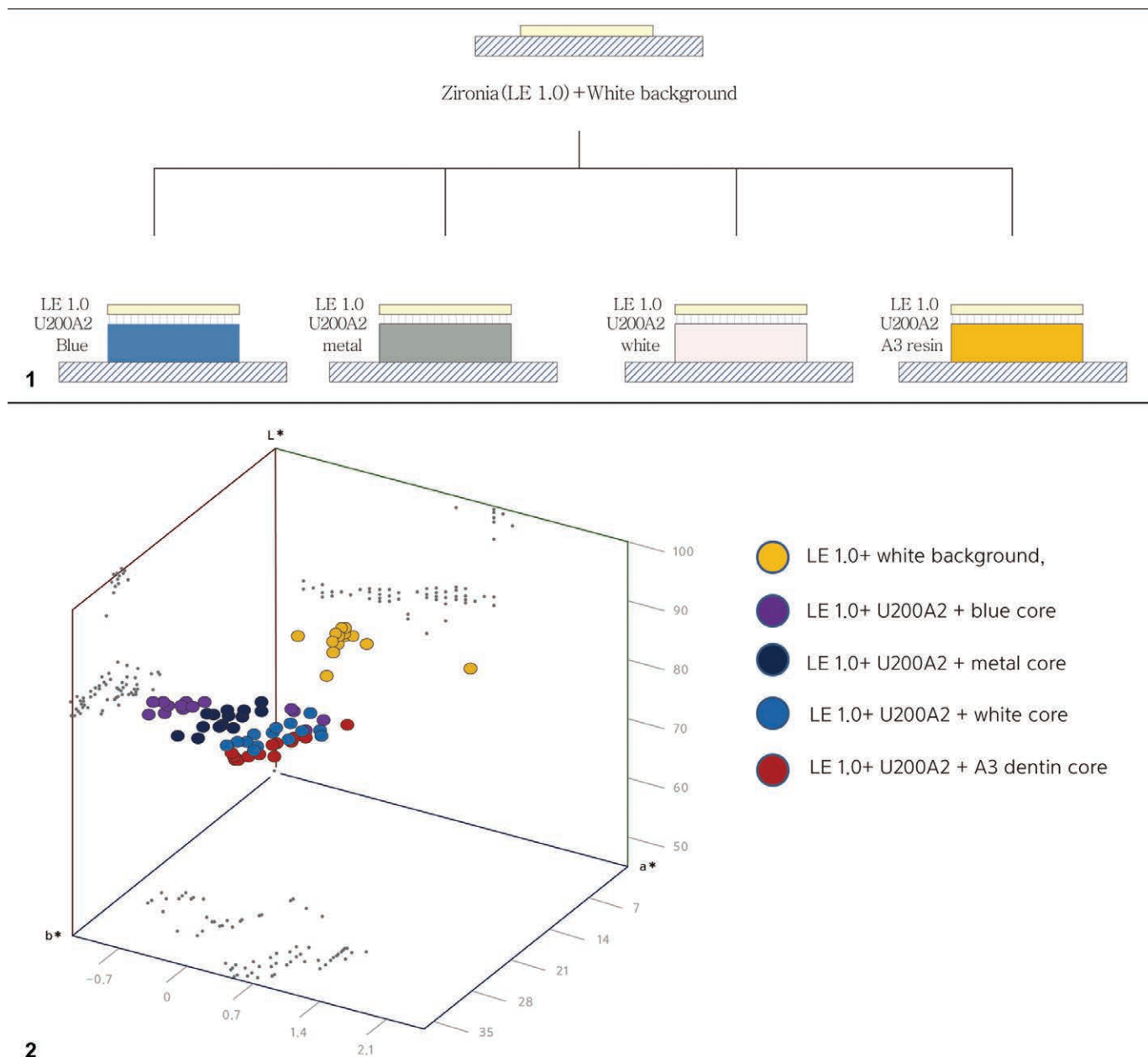


Figure 1. Flowchart of the study. Color measurements were performed with zirconia (Lava Esthetic 1.0) laid on white background and zirconia combined with cement (U200A2) and four different core materials.

Figure 2. Three-dimensional plot of color distributions, which represent the  $L^*$ ,  $a^*$ , and  $b^*$  values of zirconia (Lava Esthetic [LE]) on white background and zirconia combined with cement (U200A2) and four different core materials. Yellow, LE 1.0 on white background; purple, LE 1.0 + U200A2 + blue core; navy blue, LE 1.0 + U200A2 + metal core; blue, LE 1.0 + U200A2 + white core; red, LE 1.0 + U200A2 + A3 dentin core.

should be directed to determine the optimal methods for using monolithic zirconia with improved translucency.

According to our results, color change in the final restoration was not significant between the metal (titanium) core and A3 dentin-shade resin core. This suggests that there is no significant difference in the

final color when titanium, the material commonly used for implant abutments, or A3 dentin-shade resin, which is the shade of a prepared tooth, are restored with zirconia.

The translucency of zirconia has been studied extensively in the literature. According to Church and others,<sup>9</sup> even the most translucent zirconium

Table 2: Measurements of Color Difference<sup>a</sup>

Core	Blue (n=15)	Metal (n=15)	A3 Dentin (n=15)	White (n=15)
$\Delta E$	$21.5 \pm 2.0$ A	$19.0 \pm 1.6$ B	$17.3 \pm 2.1$ BC	$15.8 \pm 2.0$ C

<sup>a</sup> Differences were measured between LE 1.0 on a white background and LE 1.0 combined with each of the four core materials and resin cement. All specimens were measured against a white background. The different uppercase letters indicate differences between the core materials (in the rows;  $p < 0.05$ ).

oxide material is not as translucent as lithium disilicate, but high-translucency zirconia material at a clinically acceptable minimal thickness is as translucent as lithium disilicate. Moreover, flexural modulus and flexural strength are significantly greater in high-translucency zirconia materials compared with that in lithium disilicate. Considering this, the level of translucency comparable with that of lithium disilicate can be achieved by the use of zirconia with minimum removal of tooth structure. Therefore, in the present study, it was determined to study the translucency of zirconia while maintaining the thickness of zirconia specimens within a range of statistical insignificance.

The increased translucency of LE, in particular, is assumed to be achieved by controlling the proportions of the crystalline phases. Translucency increases if the amount of the cubic phase increases and that of the tetragonal phase decreases, because the cubic phase prevents the scattering of light from the grain boundary.<sup>21</sup> The amount of the cubic phase increases as the level of yttria increases, consequently improving translucency.<sup>22</sup>

Increased translucency in zirconia is achieved from the structural change that occurs when increasing the yttria content from 3 to 5 mol%. The tetragonal zirconia phase reduces the concentration of cubic phase particles, resulting in decreased flexural strength (600-800 MPa).<sup>23</sup> Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) is zirconia stabilized with 5.18 wt% yttria and a tetragonal phase in a concentration of 90% or more; however, the yttria concentration should increase to 7 wt% or higher to achieve adequate translucency.<sup>24</sup> It has been reported that the combination of a mean grain size less than 80 nm and a 75% tetragonal–25% cubic phase proportion, with a porosity content less than 0.01%, can produce a translucent zirconia ceramic.<sup>24</sup> Moreover, translucency can be increased to an ultra-level, if the cubic phase is increased to 50%. Reduction in grain size and increase in the cubic phase may, however, decrease the flexural strength and fracture toughness of zirconia.<sup>25</sup> Translucency decreases as reflection increases. According to

Zhang,<sup>24</sup> internal light scattering is influenced by porosity, additives, defects, grain size and their boundary, crystalline phase, and thickness. High porosity increases light scattering and reduces translucency, as the refractive index between air ( $n=1$ ) and zirconia ( $n=2.1-2.2$ ) is different.<sup>26</sup> Porosity can be controlled by increasing a sintering parameter such as temperature, cycle, and/or time.<sup>27</sup>

In the study conducted by Yu and others,<sup>28</sup> the translucency parameter (TP) represented the color difference between a material over a black and a white background. The TP of human dentin was found to be 16.4 and that of enamel to be 18.1 at a thickness of 1.0 mm, similar to the TP of glass ceramic (14.9-19.6). According to Wang and others,<sup>29</sup> the TP of monolithic zirconia was 5.5 to 13.5 at a thickness of 1.0 mm; in particular, the TP of Lava Plus high-translucency zirconia was 13.5, which is lower than that of human dentin. Sulaiman and others<sup>10</sup> reported that TP values of 1.0 mm zirconia in the specimen group were 11.16 to 15.3, lower than the TP values of enamel and dentin. They concluded that several improvements would be required for zirconia to match the translucency of natural teeth optimally. However, in the present study, the TP values of zirconia of thickness 1.0 mm were higher, with 14.91 for Lava Plus and 17.36 for LE. Thus, the TP of LE was higher compared with that of human dentin of the same thickness but lower compared with that of human enamel. Changes in the crystalline structure of zirconia are believed to have contributed to the improved translucency.

Tabatabaian and others<sup>17</sup> investigated the thickness of zirconia coping required to mask the color of a variety of restorative materials and reported that the optimum thickness for achieving an ideal masking ability was 0.4 mm for A1 and A3.5 shade composite resin, A3 shade zirconia, and nonprecious gold alloy, whereas it was 0.6 mm for amalgam and 0.8 mm for nickel-chromium alloy.<sup>17</sup> In clinical situations with existing cores, various options are available to compensate for the effect of the background, such as using an opaque cement, increasing the thickness of veneering porcelain, or fabricating a zirconia coping with a proper thickness.<sup>17</sup>

The best possible luting agent should be used to achieve high bond strength after cementation. Resin cement is often preferred for the cementation of all-ceramic restorations because of its low solubility, good esthetics, and high bond strength.<sup>30</sup> Moreover, it is used to modify the final color of the restoration and mask the color of the substructure.<sup>31</sup> The self-cure resin cement does not require curing by the use

of visible light and hence has an advantage in deep cavities or if a thick restorative material is used. However, manipulation of self-curing resin cement has a risk of entrapment of air bubbles and resultant formation of voids on the adhesive interface. In addition, the color of the resin cement can have a slightly yellowish tinge if a tertiary amine catalyst is used. A major advantage of light-cure resin cement is the ease of use. It does not have a limitation of working time, and excessive luting material can be easily removed prior to curing. However, the amount of light reaching the floor of the cavity is decreased in deep cavities in the case of a ceramic- or resin-based composite restoration, thus negatively affecting light activation of the resin cement. Dual-cure resin luting agents have been developed in an attempt to combine the ideal properties of self-cure and light-cure resin cements. The chemical curing components guarantee complete polymerization in the floor of deep cavities, while photo-activation ensures immediate finishing after exposure to curing light.<sup>32</sup>

Rosenstiel and others<sup>33</sup> reported that the film thickness of the luting agent can directly affect long-term clinical success. According to the guidelines by the American Dental Association (ADA), a maximum film thickness of 25  $\mu\text{m}$  is allowed for a type I cement, which is designed for the accurate seating of precision attachments and for other uses. ADA type II materials, which are recommended for uses except the cementation of precision attachments, can have a maximal film thickness of 40  $\mu\text{m}$ .<sup>34</sup> Leinfelder and others<sup>35</sup> suggested that the interfacial gap should not exceed 100  $\mu\text{m}$ , particularly on the occlusal surface, since wider gaps commonly result in extensive wear of the composite resin luting agent. Therefore, in this study, the resin cement thickness was determined as for type I cement (25  $\mu\text{m}$ ).

There are a few limitations of this study. First, variables associated with aging-induced color changes were not considered. With aging, zirconia may show a change in translucency, which could be attributed to tetragonal-to-monoclinic phase transformation. Incremental change in the microstructure of Y-TZP with aging could be related to a change in light reflection of the monoclinic and tetragonal crystals. Furthermore, surface porosities in the region of phase transformation can change (micro-cracks) because of a change in the volume of the monoclinic crystal, influencing translucency.<sup>36</sup> Further, colors of the cement and resin core are expected to change with aging. Specifically, it is expected that the color of resin-based materials may shift toward

yellow because of water absorption by components such as triethyleneglycol dimethacrylate and 2,2-bis (4-[2-hydroxy-3-methacryloyloxy] phenyl) propane and that the color may change as a result of the concentration of uncured camphorquinone depending on the polymerization rate.

Second, the specimens were not directly cemented in this experiment. With direct cementation, light reflection and refractive index would have been different, exerting differing influences on translucency and color changes and producing differing outcomes than the current laboratory experiment.

Third, the cement thickness used in the experiment was relatively less. The typical resin cement thickness of 100  $\mu\text{m}$  has been used in several previous studies, which is thicker than the thickness used in the present experiment. The effect on color change may have been smaller in this study because the resin cement discs were thinner. The thickness used in the current experiment, 25  $\mu\text{m}$ , is the thickness required for more precise restorations such as inlays and onlays. Accordingly, it is speculated that if the cement is thicker, the masking effect may be stronger, and the masking ability of different cement types may differ.

Lastly, the thickness of zirconia considered in this experiment was 1.0 mm, which is greater than the minimal thickness (0.8 mm) recommended for LE by the manufacturer. If we had used different thicknesses of zirconia, we might have obtained different results. Therefore, comparisons of a larger range of thicknesses, up to 2.0 mm, would have produced more clinically useful findings.

## CONCLUSION

Different core materials would affect the final color of high-translucency monolithic zirconia restorations. The blue core showed the greatest color difference in final zirconia restorations followed by metal, A3 dentin-shade resin core, and white core. Metal core and A3 dentin-shade resin core did not show a significant color difference in the final zirconia restoration.

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## Conflict of Interest

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



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

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# Effect of LED Light-Curing Spectral Emission Profile on Light-Cured Resin Cement Degree of Conversion

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## Clinical Relevance

The use of multippeak LED light-curing guarantees efficiency on light activation of Ivocerin-containing light-cured resin cement.

## SUMMARY

**Objectives:** This study evaluated the degree of conversion (DC) of an Ivocerin-containing light-cured resin cement activated through different thicknesses of a lithium disilicate glass ceramic using two LED light-curing units (LCUs). It also evaluated the influence of the glass ceramic interposition on irradiance and the spectral emission profile of the LED LCUs.

**Methods and Materials:** Medium-translucency lithium disilicate glass ceramic specimens of 0.3-, 1.0-, and 2.0-mm thickness were heat pressed. A single-peak and a multippeak LED LCU were selected. Irradiance and spectral emission profile were assessed, the light trans-

mittance was calculated, and the translucency parameter was determined for each thickness. DC was calculated after 20, 40, or 60 seconds of light activation by attenuated total reflection/Fourier-transform infrared spectroscopy. DC data were analyzed using three-way analysis of variance (ANOVA) and the Tukey honestly significant difference (HSD) test, irradiance and light transmittance data were analyzed using two-way ANOVA and the Tukey HSD. Spearman's correlation test was performed between the translucency parameter and light transmittance ( $\alpha=0.05$ ).

**Results:** DC ranged from 71.1% to 80.1%, increasing significantly from light activation of 20 to 60 seconds. Irradiance ranged from 186.1 to 2013.5 mW/cm<sup>2</sup>. Multippeak LED LCU showed higher DC and irradiance than single-peak LED LCU. Light transmittance ranged from 13.3% to 61.5%. Irradiance and light transmittance decreased as lithium disilicate glass ceramic thickness increased. The translucency parameter and light transmittance showed a significant correlation.

**Conclusions:** Multippeak LED LCU allows higher C=C conversion with shorter light activation time of Ivocerin-containing light-cured resin cement with an interposed medium-translucency lithium disilicate glass ceramic.

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## INTRODUCTION

The demand for ceramic esthetic restorations has increased over the past years.<sup>1-4</sup> The continuous development of ceramic materials improves their indications of use and esthetics.<sup>3,5-7</sup> Among ceramic options, lithium disilicate glass ceramics are often the restorative material of choice since they combine fracture resistance and esthetics.<sup>3,8,9</sup> An innovative manufacturing technology was introduced in 2016, designed to further enhance the properties of lithium disilicate glass ceramics.<sup>8,10</sup> Described as high-density micronization, this process results in smaller crystals that are better distributed and in higher density in the glassy matrix, leading to a ceramic (GC Initial LiSi Press, GC Co, Tokyo, Japan) with better physical properties, superior esthetics, lower wear potential to antagonist teeth, and higher polishability compared to any other lithium disilicate glass ceramic.<sup>8,10</sup>

However, ceramic materials with excellent mechanical properties do not necessarily imply better clinical performance.<sup>11,12</sup> The success of ceramic restorations relies on the adhesion of the luting agent to tooth structure.<sup>13,14</sup> Resin-based cements are used to lute glass ceramic restorations.<sup>11</sup> The polymerization process of resin cements can be initiated by the application of a light source (light-cured resin cements), by a chemical redox reaction (chemical-cured resin cements), or by the combination of these two processes (dual-cured resin cements).<sup>13,15,16</sup> Light-cured resin cements have two major advantages: working time is controlled by clinicians, and better color stability is attained compared to both dual- and chemical-cured resin cements.<sup>7,17</sup> However, ceramic type<sup>12,17-21</sup> and its thickness,<sup>4,12,13,17,22,23</sup> translucency,<sup>2,7,12,24</sup> and color<sup>23</sup> may attenuate the light emitted by a light-curing unit (LCU). Thus, the light source must be powerful enough to be transmitted through the ceramic restoration and reach the resin cement with sufficient irradiance to carry out the polymerization process appropriately.<sup>7,17,19,21,25,26</sup>

One way of quantifying the polymerization of resin cements is to measure their degree of conversion (DC), which represents the percentage of monomers converted into polymers.<sup>11,15</sup> A high DC of resin cements has usually been linked to better physical and mechanical properties,<sup>7,19,21,27</sup> clinical performance,<sup>7,23,27</sup> biocompatibility,<sup>16,28</sup> color stability,<sup>13</sup> and adhesion to tooth structure<sup>25</sup> in addition to lower solubility in the oral environment.<sup>13,24</sup> Light activation can be achieved by many light sources,<sup>1,29,30</sup> but LED LCUs are currently the

“gold standard” to perform this process.<sup>29</sup> Camphorquinone is the most common photoinitiator present in resin cements, with a light absorption spectrum ranging from about 425 to 495 nm and thus compatible with the spectral emission profile of LED LCUs.<sup>26,29,30</sup> However, materials formulated with camphorquinone tend to be slightly yellowish.<sup>26,30,31</sup> The search for resin composite materials with lighter colors has led manufacturers to develop alternative photoinitiators to circumvent this limitation,<sup>26</sup> the most common being Lucirin TPO, PPD, and Ivocerin.<sup>26,30</sup> The counterpoint to using alternative photoinitiators is the occasional need for a shorter light wavelength than that emitted by a single-peak LED LCU (420 to 480 nm), also classified as a first- or second-generation LED LCU.<sup>26,30</sup> Recently, third-generation LED LCUs (also classified as multi-peak LED LCUs) have been developed that have a broader spectral emission profile (390 to 490 nm) compared to single-peak LED devices.<sup>26,29-31</sup>

Ivocerin is a patented photoinitiator considered more reactive than camphorquinone with a light absorption spectrum ranging from about 390 to 445 nm (absorption peak at 408 nm).<sup>30,32</sup> It can be used alone, that is, without any additional coinitiator; this ensures better color stability of the materials that use it.<sup>32,33</sup> An example of a light-cured resin cement that contains this photoinitiator is Variolink Esthetic LC (Ivoclar Vivadent, Schaan, Liechtenstein). For reasons of industrial protection, its manufacturer does not describe the entire formulation of this light-cured resin cement, omitting the types and concentrations of the photoinitiators. Therefore, the characteristics that the LED LCU should have to light activate this material are unclear.<sup>30,34</sup> To date, literature is scarce regarding the use of different LED LCUs in Ivocerin-containing light-cured resin cements as well as the interaction of the light produced by these LED LCUs with lithium disilicate glass ceramics. Thus, the objective of the present study was to evaluate the DC of an Ivocerin-containing light-cured resin cement light activated with progressive exposure times (20, 40, or 60 seconds) through different thicknesses of a lithium disilicate glass ceramic using two LED LCUs and to evaluate the influence of the interposition of glass ceramic on irradiance and on spectral emission profile of the light emitted by the LED LCUs. The null hypotheses were that DC would not be influenced by the interposition of the glass ceramic, exposure time, or the LED LCU.



## METHODS AND MATERIALS

### Preparation of Glass Ceramic Specimens

A castable CAD/CAM acrylic resin block (Vipi Block VBS, Dentsply Sirona, York, PA, USA) was sectioned with a double-faced diamond disk (IsoMet Blade 15LC, Buehler, Lake Bluff, IL, USA) coupled to a precision cutting machine (IsoMet 1000, Buehler) at a speed of 350 rpm under constant water cooling. The cuts were performed perpendicular to the outer surface of the block, yielding rectangular specimens (19×16 mm) with 0.3-, 1.0-, and 2.0-mm thicknesses. The specimens were reduced to quadrangular plates with square bases measuring 10 mm using a diamond disk at low speed. All the dimensions were checked with a digital caliper (Mitutoyo CD-6" CSX, Mitutoyo, Kawasaki, Japan).

A wax cylinder (sprue #2.5, Kota, Cotia, Brazil) was attached to the center of one side of each specimen of acrylic resin. Groups of six specimens, two of each thickness, were positioned on an investment ring system (IPS Investment Ring System, 300 g, Ivoclar Vivadent) for investing (Bellavest SH, Bego, Bremen, Germany). After setting for 20 minutes, the investment ring was placed into a burnout furnace (3000 10P, EDG, São Carlos, Brazil). The furnace was heated from room temperature to 600°C at a heating rate of 20°C/min, and left at this temperature for 10 minutes. Then it was heated to 900°C and cooled to 850°C and left at this temperature for 30 minutes. Following this, the investment ring was placed in a press furnace (EP 5000, Ivoclar Vivadent) to press the glass ceramic ingot (GC Initial LiSi Press, MT-A1, GC Co; initial temperature 700°C, heating rate 60°C/min, remaining at 917°C for 25 minutes and then pressed). After completing the press cycle, the investment ring was sectioned with a sintered diamond disk at low speed; the specimens were then divested using air abrasion at 1.5 bar. Sprues were cut with a diamond disk at low speed. Both bases of glass ceramic specimens were wet ground manually with #180 grit SiC abrasive papers (231Q, 3M Corp, St Paul, MN, USA), obtaining five glass ceramic specimens of each thickness ( $0.3 \pm 0.01$ ,  $1.0 \pm 0.01$ , and  $2.0 \pm 0.01$  mm).

Ceramic glaze (IPS Ivocolor Glaze Powder Fluo, Ivoclar Vivadent) was brushed on one side of each glass ceramic specimen to fill any irregularities. The specimens covered with glaze were positioned on a firing tray and fired in a ceramic furnace (P510, Ivoclar Vivadent; initial temperature of 403°C, heating rate of 60°C/min, remaining at 770°C for 90 seconds). Then all glazed surfaces were abraded with

#600 grit SiC abrasive papers (211Q, 3M Corp) until they had exactly 0.3-, 1.0-, and 2.0-mm thickness and checked with a digital caliper. The nonglazed surfaces were etched with 9% hydrofluoric acid gel (Porcelain Etch, Ultradent, South Jordan, UT, USA) for 20 seconds and then washed with water spray for 30 seconds. Any residue was removed by ultrasonic cleaning for 10 minutes in distilled water. After air drying for 30 seconds, one coat of a silane coupling agent (Silane, Ultradent) was applied to the etched surface for 60 seconds and air-dried for 15 seconds.

### Silicone Guide Preparation

The following procedure was used to standardize the thickness and obtain a resin cement film thickness ( $\sim 50 \mu\text{m}$ ) representative of a luted ceramic restoration in a clinical situation.<sup>35</sup> A Mylar strip was positioned on a glass plate; a small portion of the light-cured resin cement (Variolink Esthetic LC, shade Light+, Ivoclar Vivadent) was placed on the Mylar strip, and the glazed surface of a glass ceramic specimen was positioned on the resin cement. A load of 250 gf was applied for two minutes at the center of the glass ceramic specimens with a custom device containing a flat rubber point 10 mm in diameter.<sup>22</sup> Excess resin cement was removed with a small brush, and the resin cement was light activated for 40 seconds through the glass ceramic specimen. Afterward, equal parts of vinyl polysiloxane (VPS) base and catalyst putty impression material (Express XT, 3M Oral Care, St Paul, MN, USA) were hand mixed for 30 seconds. The mixture was positioned on the stack, and another glass plate was placed on the VPS impression material. Manual pressure was exerted on the latter glass plate until it came into full contact with the glass ceramic specimen. After VPS polymerization was complete, the upper glass plate was removed, the excess silicone guide was cut with a knife, and the resin cement in contact with the glass ceramic specimen was removed with a #12 scalpel blade. This procedure was performed for one glass ceramic specimen of each thickness.

### Degree of C=C Conversion

The DC of the light-cured resin cement (Variolink Esthetic LC, shade Light+) was measured at room temperature by attenuated total reflection/Fourier-transform infrared (ATR-FTIR) spectroscopy (IR-Prestige-21, Shimadzu, Kyoto, Japan). The silicone guide was positioned on the center of the ATR module (DuraSamplIR II, Smiths Detection Inc, Edgewood, MD, USA), leaving the diamond crystal

at the center of a space corresponding to the glass ceramic specimen. The light-cured resin cement was applied directly from its application syringe to the diamond crystal. A Mylar strip was positioned on the resin cement, and the glass ceramic specimen was placed on the stack with its glazed surface facing upward. Specimen thickness was standardized by positioning a microscope slide on the stack and pressing gently until it made full contact with the silicone guide. The microscope slide was removed, and the resin cement was light activated for 20, 40, or 60 seconds through the different glass ceramic specimens of 0.3-, 1.0-, or 2.0-mm thickness. Light activation was performed with the tip of the LED LCU immediately above the glass ceramic specimens ( $n=5$ ). Control groups were evaluated by positioning the resin cement directly on the diamond crystal, being light activated for 20, 40, or 60 seconds without the interposition of the glass ceramic specimens.

The infrared spectra collected between 1500 and 1800  $\text{cm}^{-1}$  in absorbance mode at 4  $\text{cm}^{-1}$  spectral resolution over 12 scans was plotted on a software program (IRsolution, v1.60, Shimadzu) and analyzed. The DC of each specimen was calculated using the standard baseline method, which is based on changes in the ratios between the absorbance peak heights corresponding to the aliphatic (1637  $\text{cm}^{-1}$ ) and aromatic (1608  $\text{cm}^{-1}$ ) C=C prior to and after resin cement light activation. The absorbance intensity of aromatic C=C was used as an internal reference, as its intensity does not change during the polymerization reaction.<sup>12</sup> The DC was evaluated immediately after light activation of the light-cured resin cement and calculated according to the following equation:

$$\text{DC}(\%) = \left[ 1 - \frac{\left( \frac{\text{abs}(\text{C}=\text{C}_{\text{aliphatic}})}{\text{abs}(\text{C}=\text{C}_{\text{aromatic}})} \right)_{\text{cured}}}{\left( \frac{\text{abs}(\text{C}=\text{C}_{\text{aliphatic}})}{\text{abs}(\text{C}=\text{C}_{\text{aromatic}})} \right)_{\text{uncured}}} \right] \times 100,$$

where  $\text{abs}(\text{C}=\text{C})_{\text{aliphatic}}$  refers to the aliphatic absorbance peak and  $\text{abs}(\text{C}=\text{C})_{\text{aromatic}}$  refers to the aromatic absorbance peak for both cured and uncured resin cements.

### Irradiance, Spectral Emission Profile, and Light Transmittance Through Ceramic

Two LED LCUs with similar irradiance of approximately 1000  $\text{mW}/\text{cm}^2$ , measured by a curing radiometer (Demetron L.E.D. Radiometers, KaVo Kerr, Brea, CA, USA), were selected: a second-generation LED LCU, also classified as a single-

peak LED LCU,<sup>30,31,36</sup> with spectral emission between 440 and 480 nm, presenting only one emission peak (Radii Plus, SDI, Melbourne, Australia),<sup>37</sup> and a third-generation LED LCU, also classified as a multipeak LED LCU,<sup>30,31,36</sup> with spectral emission between 395 and 480 nm, presenting two distinct emission peaks (VALO on its standard power mode, Ultradent).<sup>38</sup> The irradiance and the spectral emission profile of each LED LCU through glass ceramic specimens with different thicknesses were evaluated using a light spectrometer (MARC-RC, BlueLight Analytics, Halifax, NS, Canada). The LED LCUs were held by a clamp (benchMARC, BlueLight Analytics), and their tips were positioned at the center of the top surface sensor. The measurements were performed with and without interposition of the glass ceramic specimens between the sensor and the LED LCU tip ( $n=5$ ). All glass ceramic specimens of each thickness were evaluated with their glazed surfaces facing the LED LCU tip. The LED LCUs were activated for 20 seconds in all evaluations, and both irradiance and spectral emission profile data were recorded in a software program (MARC, v4.01, BlueLight Analytics). The light transmittance (%) was calculated as the percentage ratio between light irradiance through glass ceramic specimens and light irradiance without interposition of glass ceramic specimens.

### Translucency Parameter

The translucency parameter of glass ceramic specimens was evaluated with a sphere spectrophotometer (SP60, X-rite, Grand Rapids, MI, USA). The spectrophotometer was calibrated with a ceramic disk for white calibration measurements and with the trap opening for black calibration measurements, as recommended by the manufacturer. Color data were represented by CIE  $L^*a^*b^*$  coordinates: the  $L^*$  parameter represents the lightness, where 100 is pure white and 0 pure black; the  $a^*$  parameter represents red-green coordinates, where positive values represent red and negative values green; and the  $b^*$  parameter represents yellow-blue coordinates, where positive values represent yellow and negative values blue. Three consecutive readings were performed over a white background ( $L^*=94.7$ ,  $a^*=-0.89$ , and  $b^*=-0.38$ ) and over a black background ( $L^*=0.20$ ,  $a^*=0.23$ , and  $b^*=-0.94$ ) at the center of the glazed surface of each glass ceramic specimen, and the average was considered. The mean translucency parameter of each glass ceramic specimen was calculated by the following equation:<sup>39</sup>

Table 1: Degree of C=C Conversion (%) of the Light-Cured Resin Cement According to Glass Ceramic Thickness and Exposure Time for Each LED Light-Curing Unit (LCU) (Mean  $\pm$  SD) (n=5)<sup>a,b</sup>

Thickness (mm)	Exposure Time					
	Multipeak LED			Single-Peak LED		
	20 s	40 s	60 s	20 s	40 s	60 s
0.0 (control)	74.0 $\pm$ 1.0 Aa	80.1 $\pm$ 0.9 Ab*	80.1 $\pm$ 0.6 Ab*	74.3 $\pm$ 1.6 BCa	75.5 $\pm$ 1.2 ABb*	78.0 $\pm$ 1.7 ABb*
0.3	76.6 $\pm$ 1.0 Ba*	79.9 $\pm$ 0.2 Ab*	80.0 $\pm$ 0.4 Ab	73.4 $\pm$ 1.7 Aba*	77.4 $\pm$ 1.7 Bb*	79.4 $\pm$ 1.0 Bb
1.0	75.8 $\pm$ 1.2 ABa	77.2 $\pm$ 1.1 Ba	79.9 $\pm$ 0.9 Ab	76.4 $\pm$ 1.8 Ca	78.1 $\pm$ 2.1 Bb	79.4 $\pm$ 2.0 Bb
2.0	74.0 $\pm$ 0.9 Aa*	76.8 $\pm$ 0.5 Bb*	77.0 $\pm$ 0.9 Bb	71.1 $\pm$ 1.2 Aa*	74.6 $\pm$ 0.5 Ab*	75.7 $\pm$ 1.0 Ab

<sup>a</sup> Different letters (uppercase for rows, lowercase for columns) indicate statistical differences for a specific LED LCU ( $p < 0.05$ ).

<sup>b</sup> Asterisk (\*) indicates statistical differences between LED LCUs at each exposure time ( $p < 0.05$ ).

$$TP = [(L^*_w - L^*_b)^2 + (a^*_w - a^*_b)^2 + (b^*_w - b^*_b)^2]^{1/2},$$

where w refers to color values of each specimen over a white background and b to the values over a black background.

### Statistical Analysis

All the data were submitted to the Kolmogorov-Smirnov test to confirm normal distribution. DC (%) data were submitted to three-way analysis of variance (ANOVA) (glass ceramic thickness  $\times$  exposure time  $\times$  LED LCU) and the Tukey honestly significant difference (HSD) test. Irradiance and light transmittance data were submitted to two-way ANOVA (glass ceramic thickness  $\times$  LED LCU) and the Tukey HSD test. The Spearman correlation was performed for the translucency parameter and light transmittance. All analyses were performed using a statistical software program (SPSS, v21.0, IBM, Armonk, NY, USA) with  $\alpha = 0.05$ .

## RESULTS

### Degree of C=C Conversion

DC showed a statistically significant difference for LED LCU ( $p < 0.001$ ), glass ceramic thickness ( $p < 0.001$ ), and exposure time ( $p < 0.001$ ). The interaction between LED LCU and glass ceramic thickness ( $p < 0.001$ ) and the interactions between the three factors were statistically significant ( $p < 0.001$ ). Results for DC are shown in Table 1. DC increased significantly from 20 to 60 seconds of light activation in all ceramic thicknesses for both LED LCUs ( $p < 0.05$ ). Increasing light activation from 40 to 60 seconds was statistically significant only when the multipeak LED LCU was used on a 1.0-mm-thick ceramic specimen ( $p < 0.05$ ). For the multipeak LED LCU, DC for 20 seconds of light activation was statistically higher in the 0.3-mm-thickness group than the control and the 2.0-mm-thickness groups ( $p < 0.05$ ). Regarding 40 seconds of light activation,

the control and the 0.3-mm-thickness groups presented the highest DC. As for 60 seconds of light activation, the lowest DC was for the 2.0-mm-thickness group. For the single-peak LED LCU, DC for 20 seconds light activation was statistically higher in the 1.0-mm-thickness group than the 0.3- and the 2.0-mm-thickness groups ( $p < 0.05$ ). Regarding 40 and 60 seconds of light activation, DC for the control and the 2.0-mm-thickness groups were statistically similar.

Comparing the LED LCUs, multipeak LED LCU showed higher DC than single-peak LED LCU. The multipeak LED LCU showed higher DC for 20 seconds of light activation for both the 0.3- and the 2.0-mm-thickness groups ( $p < 0.05$ ). Regarding 40 seconds of light activation, the multipeak LED LCU showed higher DC for the control, the 0.3-mm-thickness, and the 2.0-mm-thickness groups ( $p < 0.05$ ). As for 60 seconds of light activation, the multipeak LED LCU showed higher DC only for the control group ( $p < 0.05$ ).

### Irradiance, Spectral Emission Profile, and Light Transmittance

Irradiance showed a statistically significant difference between LED LCUs and thicknesses ( $p < 0.001$ ). The interaction between the factors was also statistically significant ( $p < 0.001$ ). Irradiance results are shown in Table 2. The irradiance of both LED LCUs decreased significantly as the glass ceramic specimen thickness increased ( $p < 0.05$ ). The multipeak LED LCU showed the highest irradiance values ( $p < 0.05$ ) for all thicknesses. The spectral emission profiles of the single-peak LED LCU and the multi-peak LED LCU are shown in Figure 1 and Figure 2, respectively. The multipeak LED LCU showed a spectral emission profile with two distinct peaks at 394 and 459 nm; the single-peak LED LCU had only one peak at 457 nm.



Table 2: LED Light-Curing Unit Irradiance According to Glass Ceramic Thickness (Mean $\pm$ SD; $n=5$ ) <sup>a</sup>				
Thickness (mm)	Multipeak		Single-Peak	
	Irradiance (mW/cm <sup>2</sup> )	Attenuation (%) <sup>b</sup>	Irradiance (mW/cm <sup>2</sup> )	Attenuation (%) <sup>b</sup>
0.0 (control)	2013.5 $\pm$ 10.7 Aa	—	1400.7 $\pm$ 3.1 Ab	—
0.3	1168.6 $\pm$ 46.2 Ba	42.0	861.9 $\pm$ 22.6 Bb	38.5
1.0	662.3 $\pm$ 7.3 Ca	67.1	428.8 $\pm$ 18.3 Cb	69.4
2.0	298.8 $\pm$ 9.7 Da	85.2	186.1 $\pm$ 8.2 Db	86.7

<sup>a</sup> Different letters (uppercase for rows, lowercase for columns) indicate statistical differences ( $p < 0.05$ ).

<sup>b</sup> Attenuation of irradiance compared with the control.

No statistically significant difference in light transmittance was observed for the LED LCUs ( $p=0.894$ ), only for the different glass ceramic specimen thicknesses ( $p < 0.001$ ). The light transmittance mean values are shown in Table 3. The light transmittance for both LED LCUs decreased significantly as the glass ceramic specimen thickness increased ( $p < 0.05$ ).

### Translucency Parameter

The glass ceramic translucency parameter according to thickness was 0.3 mm ( $27.2 \pm 4.2$ ), 1.0 mm ( $12.9 \pm 0.5$ ), and, 2.0 mm ( $7.9 \pm 0.2$ ). The translucency parameter and light transmittance showed a significant nonlinear correlation ( $p < 0.05$ ) for multipeak LED LCU ( $r_s=0.887$ ) and single-peak LED LCU ( $r_s=0.900$ ).

### DISCUSSION

The null hypotheses were rejected. DC was influenced by the interposition of glass ceramic, exposure time, and LED LCU.

The glass ceramic ingot translucency selected in the present study was medium translucency due to its vast indications for use (thin veneers, veneers, inlays, onlays, crowns, and three-unit bridges).<sup>8</sup> Following the preparation design guidelines for this ceramic material, the following minimum thicknesses are recommended: 0.3 mm for thin veneers; 1.0 mm for table tops, inlays, and onlays; and 1.5 mm for incisal/occlusal crown surfaces.<sup>8</sup> This is the ultimate reason for evaluating the interposition of glass ceramic specimens of 0.3- and 1.0-mm thickness

and its effect on the DC of the light-cured resin cement (Variolink Esthetic LC). This light-cured resin cement is indicated for luting glass ceramic restorations up to 2.0 mm in thickness.<sup>33</sup> This indication led glass ceramic specimens with 2.0-mm thickness to be included in the present study. The Light+ shade of light-cured resin cement was chosen due to its higher opacity among the five available shades.<sup>33</sup> Leloup and others<sup>40</sup> found that an increased opacity of a light-cured composite may reduce its DC. Thus, selecting the Light+ shade to lute a 2.0-mm glass ceramic thickness restoration would represent an extreme indication for the studied light-cured resin cement. A limitation of the present study was the evaluation of only one level of translucency of a single glass ceramic brand and only one shade of an Ivocerin-containing light-cured resin cement. Future investigations should consider comparing other glass ceramic shades and opacities as well as light-cured resin cements with other alternative photoinitiators.

The selection of the LED LCUs to be tested in the present study was performed by measuring the irradiance of LED LCUs by a handheld curing radiometer (Demetron L.E.D. Radiometers), a device used by clinicians and some researchers to monitor the output from their LCUs.<sup>41</sup> Two LED LCUs with similar irradiance of approximately 1000 mW/cm<sup>2</sup> were selected. However, according to the results of the present study, a discrepancy was noticed between the irradiance measured by the handheld curing radiometer to those measured by the MARC-RC device; thus, the selection of the LED LCU was a major limitation of the present study, as ideally both

Table 3: Light Transmittance According to Glass Ceramic Thickness for Each LED Light-Curing Unit (Mean $\pm$ SD; $n=5$ ) <sup>a</sup>				
LED LCU		Thickness (mm)		
		0.3	1.0	2.0
Light transmittance (%)	Multipeak	58.0 $\pm$ 2.3 Aa	32.9 $\pm$ 0.9 Ab	14.8 $\pm$ 0.5 Ac
	Single-peak	61.5 $\pm$ 1.6 Aa	30.6 $\pm$ 1.3 Ab	13.3 $\pm$ 0.6 Ac

<sup>a</sup> Different letters (uppercase for rows, lowercase for columns) indicate statistical differences ( $p < 0.05$ ).



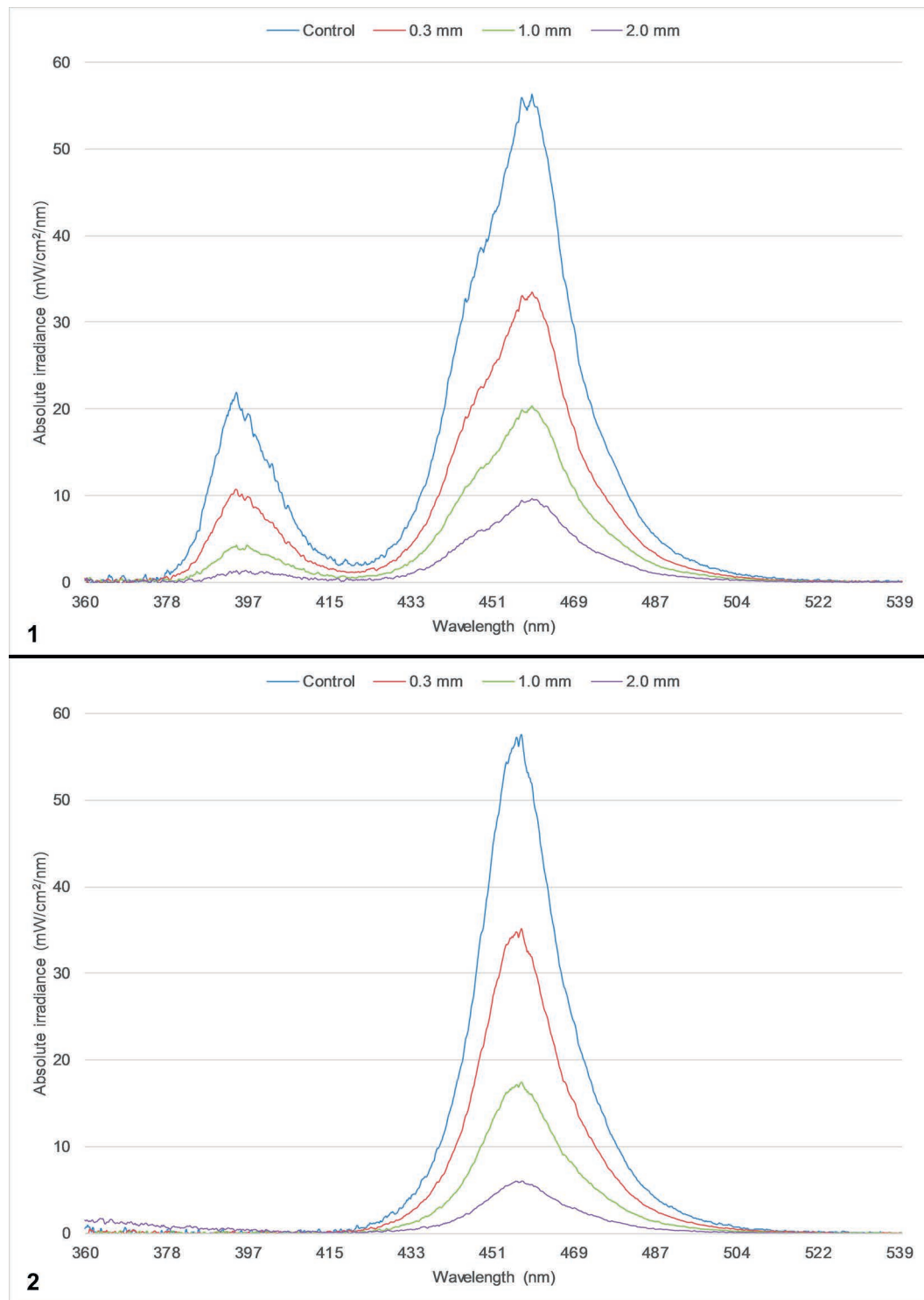


Figure 1. Spectral emission profile of multipeak LED light-curing units according to wavelength for each glass ceramic thickness ( $n=5$ ).

Figure 2. Spectral emission profile of the single-peak LED light-curing units according to wavelength for each glass ceramic thickness ( $n=5$ ).

LED LCUs should have the same irradiance measured by a MARC-RC device.

Several studies have shown that interposition of a restorative material may<sup>4,12,17,19,42,43</sup> or may not<sup>15,17,24,27,42</sup> be able to reduce the DC of light-cured resin cements. In the present study, there was no consensus for the influence of glass ceramic interposition on DC since the increase in the glass ceramic specimen thickness did not necessarily result in worse results. It is noteworthy to mention that there was a situation in which the interposition of a glass ceramic specimen with 0.3-mm thickness did not reduce but actually increased the DC compared to the control. This apparent abnormality may be explained by slower and smaller formation of free radicals due to light absorption by and diffusion into the structure of glass ceramic, reducing the polymerization rate, delaying the light-cured resin cement viscosity, and extending time for free radical diffusion, thus allowing greater conversion of monomers into polymers.<sup>26,42</sup> A similar result was found by Faria-e-Silva and Pfeifer,<sup>42</sup> where a 0.5-mm-thick glass ceramic interposition tended to present higher DC than the control (without glass ceramic interposition).

A 40-second light activation (except in one group) was enough to obtain the highest DC for both LED LCUs. The increase in exposure time of a light-cured resin cement yielded similar results in other studies.<sup>2,18</sup> Alshaafi and others<sup>18</sup> showed an increase in DC by increasing light activation not only from 20 to 40 seconds but also from 40 to 60 seconds. This can be partially explained by the greater thickness of the light-cured resin cement specimen (0.5 mm) and by the use of another light-cured resin cement (Variolink II base past, shade A1, Ivoclar Vivadent) in that study. Archegas and others<sup>2</sup> tested different exposure times (40, 80, and 120 seconds) and did not find any significant difference between 40 and 80 seconds, whereas differences between 40 and 120 seconds were significant. That study also used another light-cured resin cement (RelyX Veneer, shade A3, 3M Oral Care) specimens with 0.5-mm thickness.

When the light from the LED LCU reaches the surface of a glass ceramic, a considerable portion of its energy is lost (absorbed by and diffused into the structure of the material) and another portion transmitted.<sup>44</sup> It was observed that light transmittance of different glass ceramic thicknesses was statistically similar between LED LCUs, showing that there is no relation between light transmittance and emission profile of LED LCU. Thus, the tested

multipeak LED LCU presented higher irradiance in all thicknesses since the value of its irradiance without glass ceramic interposition was higher than that of the single-peak LED LCU. The irradiance transmitted by the multipeak LED LCU through glass ceramic in the present study as well as that by Faria-e-Silva and Pfeifer<sup>42</sup> was measured by the same light spectrometer (MARC-RC; however, the light transmittance that they found was slightly higher than that of the present study, probably because they evaluated a glass ceramic with higher translucency (IPS Empress Esthetic, shade ET1, Ivoclar Vivadent).

The silicone guides made for DC measurement ensured that only light transmitted through the glass ceramic specimen would reach the light-cured resin cement. Thus, an interesting finding was that DC was not always related to the irradiance received by the resin cement. There were situations in which the DC was not reduced compared to the control despite a significant reduction in light irradiance. This indicates that the irradiance received by the resin cement was sufficient to perform an acceptable monomeric conversion. Similar results have been found in other studies.<sup>15,21,42</sup>

The visible light absorption spectrum of Ivocerin corresponds approximately to a wavelength ranging between 390 and 445 nm with an absorption peak at 408 nm.<sup>30,33</sup> Both LED LCUs emitted light with a spectrum within this range. Since the multipeak LED LCU has a spectral emission profile with two distinct peaks (at 394 and 459 nm), it was able to activate the photoinitiator better.<sup>26,30</sup> However, it did not necessarily result in higher DC. In some groups, the single-peak LED LCU—with only one emission peak at 457 nm and transmitting lower irradiance to the light-cured resin cement—presented results statistically similar to those of the multipeak LED LCU. This can probably be explained by the higher reactivity of Ivocerin than camphorquinone, thus optimizing the polymerization reaction.<sup>30,33</sup> Therefore, a lower irradiance—even though not at Ivocerin's absorption peak—would be able to result in enough DC. Another possible explanation may be the presence of other photoinitiators with an absorption spectrum about 457 nm, such as camphorquinone or PPD.<sup>26</sup> However, the manufacturer classifies the resin cement used in the present study as amine free,<sup>32,33</sup> and the CQ-amine combination is the most commonly used photoactivation system in light-cured composites.<sup>45,46</sup>

The glass ceramic used in the present study behaved as a filter for neutral density, meaning that

the spectral emission profile of LED LCUs though the three glass ceramic thicknesses did not change. Alshaafi and others<sup>18</sup> and Faria-e-Silva and Pfeifer<sup>42</sup> found similar results. However, LED LCU spectral emission profiles did not prove to be a decisive factor to improve Ivocerin-containing light-cured resin cement DC. AlQahtani and others<sup>1</sup> also evaluated the influence of different LED LCUs on DC. These authors found similar results regarding the spectral emission profile effect on DC using another light-cured resin cement (Variolink II base paste, shades A1 and A4).

Light transmittance and the translucency parameter presented a near perfect positive nonlinear correlation for both LED LCUs; that is, increased translucency parameter of the tested glass ceramic was associated with increased light transmittance. However, since this correlation is nonlinear, the value of one of these parameters cannot be established when the other is known. This finding differs from that of the study by Oh and others,<sup>12</sup> in which the evaluated glass ceramics presented a near perfect linear positive correlation between these parameters. Thus, in that case, it would be possible to determine the value of one parameter knowing the value of the other. However, it is worth mentioning that the methodology used by Oh and others<sup>12</sup> to evaluate light transmittance and the translucency parameter differs from that used in the present study.

Although DC was evaluated at room temperature, a higher temperature, such as human body temperature (37°C), would probably not result in higher DC. Another study demonstrated that increasing the temperature from 23°C to 54°C did not result in statistically higher DC for a light-cured resin cement (RelyX Veneer).<sup>47</sup> In the present study, the DC was measured immediately after resin cement light activation. No new DC measurement was performed 24 hours after light activation because the specimens could not be repositioned on the diamond crystal of the ATR module as they were for the initial measurement. In spite of that, in another study,<sup>12</sup> it was reported that there was no significant increase in DC of a light-cured resin cement (Variolink N base paste, Ivoclar Vivadent) when measured immediately and 24 hours after light activation, probably because the resin cement specimens were about 50 µm thick (same thickness as in the present study), thus allowing enough light irradiation to reach maximal DC.

The success of ceramic restorations depends not only on the mechanical properties of the ceramic

system but also on the resin cement's mechanical properties and adequate polymerization reaction.<sup>7,13,16,19,21,23,27,28</sup> Thus, since mean DC ranged from 71.1% to 80.1%, Ivocerin-containing light-cured resin cements can be selected for luting lithium disilicate glass ceramic restorations of medium translucency using either a single- or multipeak LED LCU. Several studies have evaluated light- and/or dual-cured resin cements DC through ceramic;<sup>a</sup> however, methodological differences (such as the use of another resin cement, ceramic type, LED LCU, DC measurement method, and/or spectrometer to measure light transmittance and light irradiance) do not allow an adequate comparison with the present study.

A recently published systematic review and meta-analysis selected 13 clinical trials of laminate veneers by methodology quality with a median follow-up period of nine years. Laminate veneers resulted in a high estimated overall cumulative survival rate (89%).<sup>48</sup> Laminate veneers resulted in low complication rates; the following clinical outcomes of interest were debonding (2%), ceramic fracture/chipping (4%), secondary caries (1%), severe marginal discoloration (2%), and endodontic problems (2%).<sup>48</sup> The present article's authors speculate that most of these complication outcomes might be partially attributed to ineffective light activation during the luting procedure that could have resulted in low DC. Light-activation luting procedure protocols still need to be investigated in further laminate veneer clinical trials.

Although the multipeak LED LCU proved to be superior to the single-peak LED LCU in the present study, it is difficult to state whether differences between mean DC using these two LED LCUs could have clinical implications. However, the use of a multipeak LED appears to reduce light-activation time of Ivocerin-containing resin cement, reducing chair time.

## CONCLUSIONS

The results of this laboratory study seem to indicate that the interposition of medium translucency lithium disilicate glass ceramic has a minor effect on degree of conversion for the analyzed light-cured resin cement. In spite of that, using a multipeak LED light-curing unit seems to achieve a higher degree of conversion in a shorter light-activation time.

<sup>a</sup> References 1, 2, 11, 12, 15, 17-21, 24, 25, 27, 42.

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### Conflict of Interest

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

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# The Influence of Dentin Wall Thickness and Adhesive Surface in Post and Core Crown and Endocrown Restorations on Central and Lateral Incisors

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## Clinical Relevance

Post and core crowns and endocrowns perform similarly on fracture strength, but endocrowns have more repairable fractures.

## SUMMARY

**Purpose:** The main purpose of this study was to determine the influence of dentin wall thickness (DWT) and adhesive surface on the fracture strength and failure mode in maxillary incisors restored with post and core crowns or endocrowns.

**Methods and Materials:** Forty-eight sound maxillary incisors were selected and randomly divided into four groups (n=12): lateral incisor endocrown, lateral incisor post and core, central incisor endocrown, and central incisor post and core. All specimens obtained an endodontic treatment and were decoronated (2 mm ferrule remained). Chamfer outlines ended at the cemento-enamel junction (outline in dentin). Dentin wall thickness (mm) was measured on 12 points per sample using a modified digital calliper. Fiber posts and cores were placed in two groups, and an immediate dentin sealing was applied on exposed dentin in all groups before taking digital impressions. Digital impressions were analyzed and the adhesive surface (mm<sup>2</sup>) was measured. Indirect restorations were made of lithium disilicate (IPS e.max, computer-aided design/computer-aided manufacturing). The restorations were luted after surface conditioning the crowns and teeth. Thermocyclic aging was performed (10,000 times in baths of 5°C and 55°C) and the specimens were loaded until fracture. Fractures were specified

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on failure mode and repairability, and they were analyzed with one-way ANOVA,  $\chi^2$ -test, and linear regression analysis in SPSS ( $\alpha=0.05$ ).

**Results:** There was no significant difference in fracture strength and failure mode between all groups. Endocrown restorations on central incisors had significantly more repairable fractures than the post and core crowns. Regression analyses showed a statistically significant positive correlation between DWT/adhesive surface and fracture strength in the post and core groups.

**Conclusions:** Both endocrowns and post and core crowns on the central and lateral incisors obtained clinically applicable fracture strengths. In the central incisor groups, the endocrown restorations had significantly more repairable failures. When the walls were thicker, or when the adhesive surface was larger, higher fracture strengths were obtained in the post and core groups.

## INTRODUCTION

Severe coronal loss of tooth tissue complicates the restoration of anterior teeth. This coronal destruction gives a higher chance of tooth fracture during function.<sup>1</sup> If there is a ferrule left, the tooth restoration complex is stronger.<sup>2-4</sup> To improve retention and fracture strength when there is less ferrule (<2 mm) on anterior teeth, post and cores are applied. The subsequent loss of tissue due to the preparation for a post weakens the root.<sup>5-7</sup> Metal posts were related to higher root stresses, thereby leading to irreparable fractures.<sup>8</sup> Therefore, nowadays, more resilient glass fiber posts are used. The use of glass fiber posts results in mechanical characteristics more like that of dentin.<sup>9</sup> In a review study by Zhou and others,<sup>8</sup> it was shown that more often glass fiber posts led to loosening of the post instead of a fracture of the root.

With improvements in adhesive technology and controversy about the use and function of posts, an alternative treatment was offered: monoblock restorations called endocrowns.<sup>10</sup> When comparing posterior endocrowns to the post and core treatment, a systematic review concluded that there was no difference in fracture strength ( $p=0.07$ ,  $n=8$  articles).<sup>11</sup> In the last 10 years some articles have been published on the *in vivo* application of endocrowns, but the amount of clinical evidence is limited.<sup>12-14</sup> The type of teeth used in most articles about endocrowns are (pre)molars; there is a lack of evidence for the application of endocrowns in the anterior region. The highest bite forces (400-800

N) are found in the posterior region.<sup>15,16</sup> Ferrario and others<sup>17</sup> concluded that the maximum single tooth bite force on the central and lateral incisors was between 94-150 N (40%-48% of the maximum on molars). Little is known about the lateral forces on incisors, though on molars it was found to be around 200 N.<sup>18,19</sup> However, it is evident that the loading of incisors is different than that of molars.

The first publications on endocrowns in anterior teeth were with metal ceramic crowns using conventional cementation. The conclusions were mostly that there was no significant difference between post and no post.<sup>20-22</sup> Ramirez-Sebastian and others<sup>23</sup> studied the use of ceramic endocrowns using adhesive application on central incisors. This *in vitro* study concluded that endocrowns are sufficient for restoring anterior teeth with a ferrule of at least 2 mm. The recent systematic review published by Naumann and others,<sup>24</sup> which analysed eight articles, concluded that the ferrule effect (and maintaining cavity walls) are the most important factors in the survival of endodontically treated teeth (ETT). A recent study compared the fracture resistance of endocrowns using different preparation depths (3 and 6 mm);<sup>25</sup> no significant difference between the groups was found ( $p>0.05$ ). Deeper preparation results in more tissue loss, and thereby the chance of perforation of the root. The remaining dentin wall thickness (DWT) could influence the survival of indirect restorations on ETT. In a systematic review of *in vitro* studies, it was recommended to use smaller post diameters to retain more DWT, which improved the fracture resistance of post-restored teeth.<sup>26</sup> The ability of teeth to survive forces and resist fracture is in direct positive correlation with the amount of DWT surrounding the post. In a study by Farina and others<sup>27</sup> on remaining DWT, the results showed that the groups with 1 and 2 mm DWT had significantly higher fracture strength values than the 0.5 mm DWT group ( $p<0.05$ ). In conjunction with wall thickness, the total amount of the adhesive surface of dentin could be of influence; however, there is no evidence concerning the relation of the amount of dentin and fracture strength in endocrowns. Therefore, the objectives of this study were to compare (1) the fracture strength, (2) mode of failure, and (3) determine the possible correlation between the variables (DWT and adhesive surface), and (3) fracture strength in endocrowns and post and core crowns on central and lateral incisors. The tested null hypotheses were that there would be no significant differences in fracture strength, failure mode, and repairability between ceramic endocrowns and post and core crowns, and that there would be no significant correlation between surface/DWT on fracture strength.



## METHODS AND MATERIALS

Sound human central (n=24) and lateral (n=24) maxillary incisors, free of cervical restorations and root canal treatment, were selected from a pool of recently extracted teeth. An Institutional Review Board statement of “no permission needed” was received for this study. Sound teeth with complete and straight roots, and without fractures, were included. Both central and lateral incisors were randomly divided into two groups, as shown in Table 1.

Each tooth was endodontically treated following a standard protocol under 5-7.5× magnification (OPMI pico, Zeiss, Oberkochen, Germany). An opening was made using diamond burs and was manually prepared using #15 and #20 K-Files (Dentsply Sirona, York, PA, USA). Thereafter, Ni-Ti rotary instruments (Wave-One Gold Primary 25/.07; Dentsply Sirona) were used, following the manufacturer’s instructions. In between each file (hand or rotary) the canal was rinsed with sodium hypochlorite (3%). Gutta percha (Wave One Gold Primary; Dentsply Sirona) was fitted. The canal was dried using paper points, and after applying the sealant (AH-plus Jet; Dentsply Sirona) the gutta percha was applied. Gutta percha was removed until 4 mm for the central endocrown (CE) and lateral endocrown (LE) groups or 9 mm for the central post (CP) and core group and lateral post (LP) and core group, underneath the cemento-enamel junction (CEJ) using hot instruments and Gates Glidden drills (Nr. 3, ø 0.9 mm; Dentsply Sirona). The pulp chamber was cleaned with alcohol and the samples were closed using Teflon tape. Following the endodontic treatment, the samples were embedded 2 to 3 mm underneath the CEJ in a self-curing PMMA (ProBase Cold; Ivoclar Vivadent, Schaan, Liechtenstein) using a mould.

The brands, types, chemical compositions, manufacturers, and batch numbers of the materials used for the study are listed in Table 2. Preparations were performed by one calibrated operator. Each tooth was reduced until 2 mm remained above the CEJ. The pulp chamber preparation of CE and LE samples was prepared tapered (to prevent undercuts for digital scanning), with an apical diameter of 1.5 mm. A chamfer was prepared, resulting in a 2-mm high and 1-mm deep ferrule (the dimensions are shown in Figure 1A). For CP and LP, the fiber post system (Rebilda; VOCO, Cuxhaven, Germany) was used without further preparation of the root canal. The smallest post (red; apical diameter, 0.5 mm; coronal diameter, 10 mm; length, 19 mm) was used for both LP and CP. Before luting the post, a chamfer of 1 mm was prepared around the ferrule (the dimensions are shown in Figure 1B). The remaining DWT of all samples was measured

using a modified digital caliper (Kreator KRT705004; Varo, Lier, Belgium) at 12 places, as noted in Figure 2. The DWT was divided into three categories: incisal, cervical, and outline for analysis. In each category four measurements (mesial, buccal, distal, and palatal) were made per sample. This led to 12 measurements per sample.

Immediately after preparation and measurements, immediate dental sealing (IDS) was applied on all exposed dentin. The preparation was etched for 15 seconds with 35% phosphoric acid (Ultra-etch; Ultradent, South Jordan, UT, USA), following 15 seconds of water rinsing and air drying for 3 seconds. Optibond FL Primer (Kerr Dental, Orange, CA, USA) was applied for 15 seconds using a microbrush and air dried for 15 seconds. Optibond FL Adhesive (Kerr Dental) was applied, the excess removed, and light-cured for 20 seconds (>1000 mW/cm<sup>2</sup>, Bluephase 20i; Ivoclar Vivadent). The irradiant light was polywave and was measured before use in this study.

After preparation, the posts of the post core groups were placed. The post was cut to the right length (15 mm, 4 mm above preparation) using a diamond bur, cleaned with 70% alcohol, and silanized using Ceramic Bond (VOCO). Debris was removed from the inside of the root canal with 70% alcohol, rinsed with water, and dried with air and paper points. The root canal was etched for 15 seconds using 35% phosphoric acid (Ultra-etch), followed by 15 seconds of water rinsing. The root canal was dried using paper points and Optibond FL Primer (Kerr Dental) was applied for 15 seconds. Optibond FL Adhesive (Kerr Dental) was applied, the surplus removed, and light cured for 20 seconds (>1000 mW/cm<sup>2</sup>, Bluephase 20i; Ivoclar Vivadent). Rebilda DC Core (Quickmix Syringe; VOCO) was applied to the bottom of the root canal and the post was inserted. Surplus cement was removed and photopolymerized for 40 seconds. Core build-up was done with Clearfil AP-X

Table 1: Description of the Study Groups

Abbreviation	Description
LE	Lateral, 6-mm deep endocrowns
CE	Central, 6-mm deep ceramic endocrowns
LP	Lateral, 11-mm post, composite core, ceramic crown
CP	Central, 11-mm post, composite core, ceramic crown
Abbreviations: CE, central endocrown; CP, central post endocrown; LE, lateral endocrown; LP, lateral post endocrown.	

Table 2: Brands, Types, Chemical Compositions, Manufacturers, and Batch Numbers of the Materials Used for the Experiments

Brands/Type	Chemical Composition	Manufacturer	Batch Number
Phosphoric etch	38% H <sub>3</sub> PO <sub>4</sub> (phosphoric acid)	Ultradent	BFCVX BFKSJ
Prime	HEMA, GPDM, PAMM, ethanol, water, photo initiator	Kavo Kerr	5638300
Adhesive	TEGDMA, UDMA, GPDM, HEMA, bis-GMA, filler, photo initiator	Kavo Kerr	
Fiber post	Solid composite of glass fibers, inorganic fillers, and polydimethacrylates	VOCO	1809049 1812391 1723008
Ceramic Bond	Mixture of ingredients with >50% acetone	VOCO	1748245
Dual cure resin cement	Bis-GMA 2.5%–5%, UDMA 10%–25%, DDDMA 5%–10%	VOCO	1802340
Light cure composite	Bis-GMA, TEGDMA, silanated barium glass filler, silanated silica filler and colloidal silica, di-Camphorquinone, catalysts, accelerators, pigments	Kuraray Noritake	AT0722
Silica coating particles	Aluminium trioxide particles coated with silica (particle size 30 µm)	3M ESPE	
Ceramic etching gel	9% hydrofluoric acid	Ultradent	B9QRL
Silane coupling agent	Ethanol, 3-(trimethoxysilyl)propyl-2-methyl-2-propenoic acid	Bisco	1800002460
Silane coupling agent	Ethanol, 3-trimethoxysilylpropylmethacrylate, methacrylated phosphoric acid ester	Ivoclar Vivadent	
Light curing composite	1,4-Butandiol dimethacrylate, urethane dimethacrylate, diurethane dimethacrylate, iso-propylidene-bis (2(3)-hydroxy-3(2)-4(phenoxy) propyl)-bis(methacrylate), glass filler: mean particle size 0.7 µm; highly dispersed silicone dioxide	Micerium	2017004722
Glycerin gel	Purified water, glycerin, methylparaben, propylparaben, propylene glycol, hydroxyethylcellulose, disodium phosphate, sodium phosphate, tetrasodium, EDTA	Johnson & Johnson	B189231
Lithium disilicate	97% SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , P <sub>2</sub> O <sub>5</sub> , K <sub>2</sub> O, Na <sub>2</sub> O, CaO, F, 3% TiO <sub>2</sub> , pigments, water, alcohol, chloride	Ivoclar Vivadent	W45123
Abbreviations: Bis-GMA: bisphenol-glycidyl methacrylate; GPDM, glycerophosphate dimethacrylate; HEMA, hydroxyethyl methacrylate; PAMM, phthalic acid monoethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.			

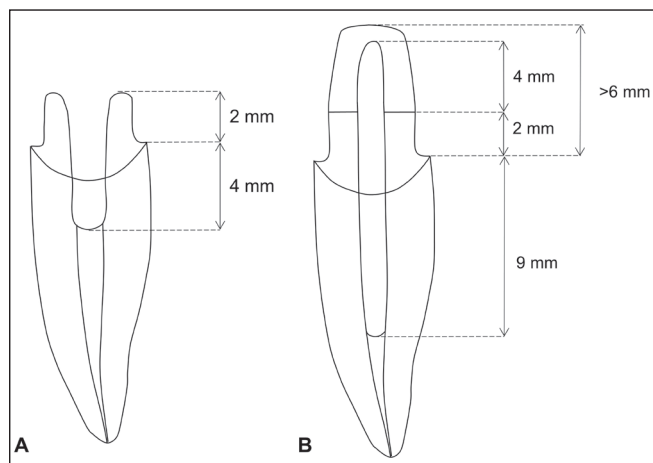


Figure 1. Dimensions of endocrown (A) and post and core samples (B).

PLT A3 (Kuraray Noritake Dental Inc., Tokyo, Japan). The fiber post was completely covered with composite. The preparation was finished using diamond burs and silicone rubbers (Shofu, Kyoto, Japan).

The prepared and measured samples were then scanned with an intraoral scanner (Omnicam; Dentsply Sirona). After scanning the preparations, the crowns were designed using Cerec SW 4.4.4 software. The height dimensions of the crowns were made the same for the LE and LP samples (9 mm) as well for the CE and CP samples (11 mm). To increase the fit of the endocrowns, the designed space for the cement spacer was reduced from 120  $\mu$ m to 30  $\mu$ m in the Cerec software. The lithium disilicate crowns (IPS e.max; Ivoclar Vivadent) were made using the Sirona MC-XL milling unit (Dentsply Sirona). The burs used were

Cylinder Bur 12 EF, Cylinder Pointed Bur 12 EF, Step Bur 12S, and the Cylinder Pointed Bur 12S (Dentsply Sirona). After crystallization, the crowns were glazed (IPS e.max Ceram Glaze Paste FLUO; Ivoclar Vivadent). The inner side of the crowns was analyzed for surplus of glaze paste and, if present, the surplus glaze was removed using sandblasting.

All indirect restorations were luted using a heated resin composite material (Enamel HFO UD2; Micerium, Avigno, Italy). The lithium disilicate crowns were conditioned with 9% hydrofluoric acid ceramic etch (Ultradent, Cologne, Germany) for 60 seconds, rinsed in water with neutralizing agent, and then air dried. Phosphoric acid (35%, Ultra-etch; Ultradent) was applied for 1 minute to clean the gross amount of glass particles on the intaglio, and after rinsing the crowns they were ultrasonically cleaned (Emag, Valkenswaard, The Netherlands) in distilled water for 5 minutes. The crowns were then silanized (Monobond Plus, Ivoclar Vivadent) and after 60 seconds were heat dried at 100°C (DI500; Coltene, Altstätten, Switzerland) for 5 minutes, then adhesive resin was applied (Optibond FL Adhesive; Kerr Dental). The teeth (IDS layer and composite build-up) were conditioned with 2 to 3 seconds of silica coating (3M ESPE, St. Paul, MN, USA), following silanization (Bis-Silane, Bisco, Schaumburg, IL, USA) and dried for 5 minutes. Adhesive (Optibond FL; Kerr Dental) was applied to the teeth and heated (55°C, ENA heat; Micerium) composite (Enamel HFO UD2; Micerium) was placed to the preparation. All lithium disilicate restorations were luted by finger pressure until they were completely seated. Excess composite was removed using a probe, and afterwards each side was photopolymerized for 40 seconds (>1000 mW/cm<sup>2</sup>, Bluephase 20i; Ivoclar Vivadent). Glycerine gel (K-Y; Johnson & Johnson, Sezanne, France) was applied and again photopolymerized for 40 seconds on the 4 sides. The surplus photopolymerized composite was removed using a scaler (H6/H7; Hu-Friedy, Chicago, IL, USA) and the margins were polished using Ceragloss green (Edenta AG, Austria, Switzerland).

Aging was performed using thermocycling (Willytec, Munich, Germany): 10,000 times in baths of 5°C and 55°C, with a dwell time of 30 seconds. Fracture load was performed in a universal testing machine (MTS 810; MTS, Eden Prairie, MN, USA) using a stainless steel bar at 135° to imitate the oral situation, as shown in Figure 3. The samples were stored in water until fracture but were tested in a dry environment. The load was applied on the incisal edge with a crosshead speed of 1 mm per minute. The teeth were loaded until fracture, and the maximum fracture strength was used for the analysis. Failure modes were analyzed for and

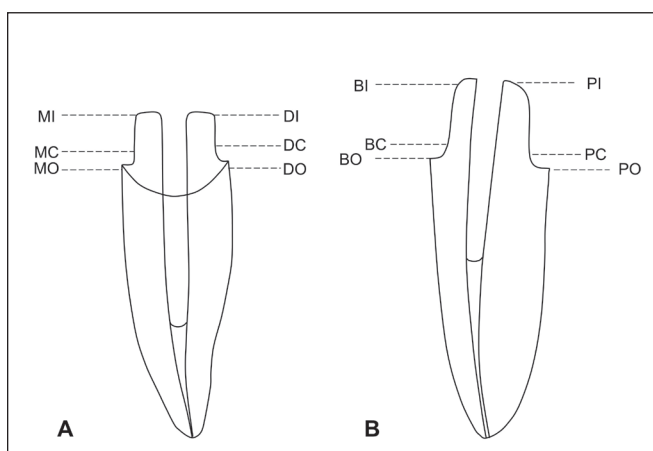


Figure 2. Locations for 12 measurements on a mesio-distal (A) and buccal-palatal (B) cross section. Abbreviations: B, buccal; C, cervical; D, distal; I, incisal; M, mesial; O, outline P, palatal.

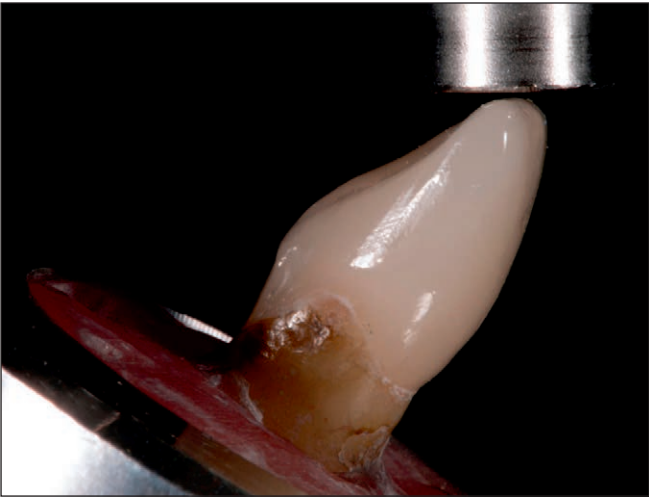


Figure 3. Sample in universal testing machine at 135° for fracture load test. Force applied until fracture by stainless steel rod (1 mm/minute).

categorized by: (1) cohesive failure in the material of the indirect restoration; (2) adhesive failure between the indirect restoration material and the dentin; (3) adhesive failure between the build-up and the crown; (4) loosening of the post and core and the crown; (5) cohesive failure in dentin; and (6) the fracture extending to the root. After this, all failures were classified as repairable or irreparable. Fractures more than 1 mm under the CEJ and extending into the root dentin were classified as irreparable.

The number of samples were calculated with a power analysis using G\*Power 3.1 (effect size = 0.5, power = 0.8, significance level = 0.05).<sup>28,29</sup> The surface area (mm<sup>2</sup>) of the preparations was determined using Geomagic (Control TM 2014, 64 bit). Data was analyzed using a statistical software program (SPSS 24.0; SPSS Inc., Chicago, IL, USA). One-way analysis of variance (ANOVA) was used to analyze the fracture strength results. A  $\chi^2$ -test was performed to analyze the differences in the mode of fracture and repairability between the different groups. A linear regression analysis was executed to analyze the influence of adhesive surface and remaining DWT on fracture strength.

RESULTS

The results of the fracture load test are presented in Table 3. One-way ANOVA was calculated on the fracture strengths. The analysis was not significant:  $F(3, 44)=1.20, p=0.319$ . There was no statistically significant difference in fracture strength between the groups, independent of the outlier (Figure 4). Analyzing the mode of failure (Figure 5) with a  $\chi^2$ -test did not result in statistical significance:  $\chi^2(9, n=48)=11.54, p=0.240$ .

Table 3: Results of Load to Fracture Test (N)				
	Mean	Standard Deviation	Minimum	Maximum
CP	319.9	139.9	101.4	517.3
LP	267.8	115.1	132.1	474.7
CE	258.3	102.9	108.5	524.7
LE	240.9	50.5	170.0	318.2

Abbreviations: CE, central endocrown; CP, central post and core; LE, lateral endocrown; LP, lateral post and core; N, newton.

There was no statistically significant difference found in failure mode. Most of the samples (>90%) had fractures extending into the root.

If reparability is considered, the LP, CP, and core groups all had irreparable fractures. In the CE group, 5/12 were repairable and in the LE group 1/12 was repairable. The  $\chi^2$  analysis was significant:  $\chi^2(3, n=48)=12.99, p=0.005$ . Only the analysis of CE (42% repairable) and CP (0% repairable) was statistically significant. Post and core crowns on central incisors had more irreparable fractures ( $\chi^2[1, n=24]=8.263, p=0.004$ ), which made extraction necessary. The LE group (8% repairable) was not significantly different from the LP group (0% repairable). Figure 6 shows a repairable endocrown sample (Figure 6A), an irreparable endocrown sample (Figure 6B), and an irreparable post and core sample (Figure 6C).

To determine the possible relation between adhesive surface/DWT and fracture strength, a linear regression analysis was calculated. In the endocrown groups (CE and LE), a nonsignificant regression equation was found ( $F(4, 19)=1.130, p=0.372$ ), with an  $R^2$  of 0.192. There was no statistically significant correlation between DWT and fracture strength, nor between the adhesive surface

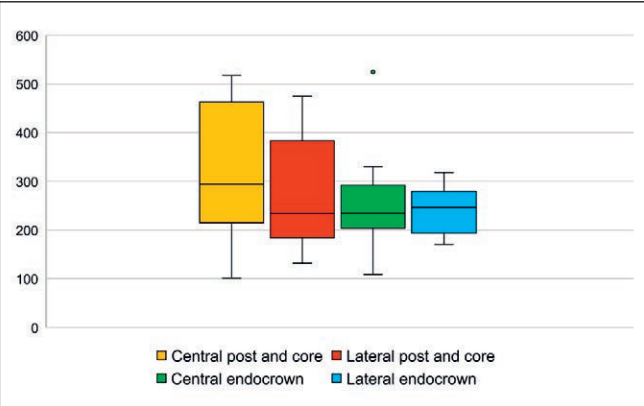


Figure 4. Fracture strength. Mean  $\pm$  1 standard deviation. Means are not significantly different ( $p=0.319$ ). °=outlier.



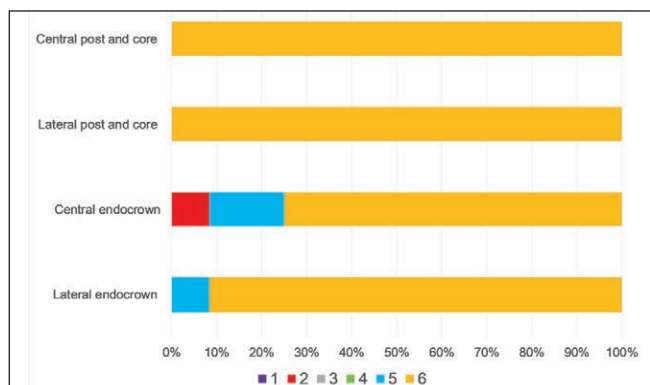


Figure 5. Frequency of failure mode: cohesive failure in the material of the indirect restoration (1); adhesive failure between the indirect restoration material and the dentin (2); adhesive failure between the build-up and the crown (3); loosening of the post and core crown (4); cohesive failure in dentin (5); and fracture extending to the root (6).

and fracture strength. In the post and core groups (CP and LP), a significant regression equation was found  $F(1,22)=19.086$ ,  $p<0.000$ , with an  $R^2$  of 0.465. There is a

statistically significant correlation between DWT and fracture strength, just as there is between adhesive surface and fracture strength. There were positive equations (Table 4), as, for example, the equation between cervical DWT and fracture strength: fracture strength =  $-118.302 + 244 \times \text{cervical DWT}$ . For each mm of cervical DWT, the fracture strength increased with 244 N. In Figure 7, the trendline for cervical DWT and fracture strength is shown. Trendlines for surface, cervical, and incisal DWT and fracture strength were comparable.

## DISCUSSION

Restoration of severely damaged anterior teeth depends highly on the amount of remaining ferrule.<sup>4</sup> In such situations the use of a post with a high elastic modulus is advised.<sup>30</sup> Improvements in adhesive technologies have led to the suggestion of endocrown restoration.<sup>10</sup> A recent systematic review indicates that the performance of endcrowns may be equal to that of conventional post and core treatments; however, most of the studies

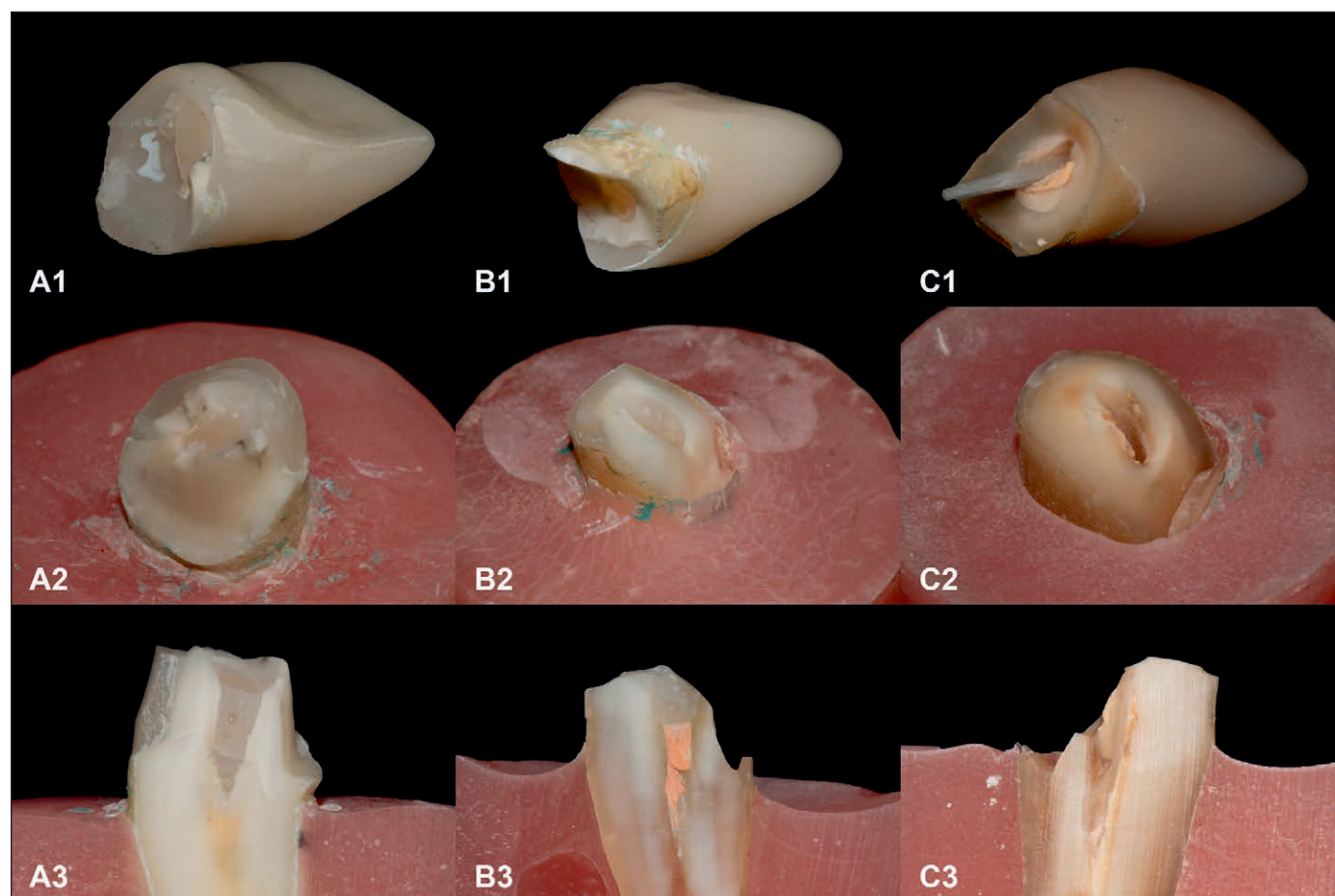


Figure 6. Examples and cross sections of fractured samples. **A1-A3:** Repairable fractured endocrown sample. **B1-B3:** Irreparable fracture endocrown sample. **C1-C3:** Irreparable fractured post and core sample.

Table 4: Test Outcome for Linear Regression and Regression Equation Between Independent Variables (Surface and Incisal, Cervical and Radix DWT) and the Dependent Variable (Fracture Strength)		
Correlation	Test Outcome	Regression Equation
Incisal DWT/fracture strength	F (1,22) = 14.51, $p=0.001$ ; $R^2$ of 0.397	Fracture strength (N) = 208.88 x incisal DWT (mm) + 25.71
Cervical DWT/fracture strength	F (1,22) = 19.086, $p<0.000$ ; $R^2$ of 0.465	Fracture strength (N) = 244 x cervical DWT (mm) - 118.30
Radix DWT/fracture strength	F (1,22) = 18.81, $p<0.000$ ; $R^2$ of 0.439	Fracture strength (N) = 229.00 x radix DWT (mm) -281.44
Surface/fracture strength	F (1,22) = 18.471, $p<0.000$ ; $R^2$ of 0.466	Fracture strength (N) = 3.38 x surface (mm <sup>2</sup> ) -43.76
All correlations are significant ( $p<0.05$ ). Abbreviations: DWT, dentin wall thickness; mm, millimeter; N= newton.		

on endocrown restorations have been done on posterior teeth.<sup>11</sup> If anterior teeth are studied, central incisors are chosen,<sup>23,25,31</sup> or bovine teeth are used;<sup>2,3</sup> there are no studies that include human maxillary lateral incisors. Therefore, the objectives of this study were to compare fracture strength and mode of failure, and determine the possible correlation of DWT/adhesive surface and fracture strength in endocrowns and post and core crowns on central and lateral incisors.

According to the results of this study, the first hypothesis, which states that there is no significant difference in fracture strength could be accepted as the fracture strength in all groups were not statistically different; however, the fracture strength is comparable with other studies on ETT, which were done on central incisors.<sup>3,31</sup> More importantly, all mean fracture strength results obtained in this study (>240 N) exceeded the

clinical bite forces of 93 and 150 N.<sup>17</sup> Considering fracture strength, both endocrown and post and core crown restorations should be applicable in a clinical situation.

Besides fracture strength, failure mode and repairability are also important. Considering failure mode, the second hypothesis could be accepted: there is no significant difference in failure mode. The mode of failure and being not significantly different between the groups is in accordance with the majority of studies done on this topic.<sup>2,3,23</sup> One study recorded more root fractures in endocrowns than in post and core crowns.<sup>31</sup> In comparison with studies on posterior teeth, the same nonsignificant results were found.<sup>32,33</sup>

There was a significant difference ( $p<0.05$ ) in clinical repairability between endocrowns and post and core crowns on central incisors in this study. Despite the similar failure modes, the endocrowns obtained more repairable failures. Therefore, the third hypothesis, stating that there is no difference in repairability, could be partially rejected, as there is a statistically significant difference in repairability between both restorations. Fractures observed in the endocrown groups were more horizontally oriented (Figure 6A), whereas the post and core crowns had more vertical root fractures (Figure 6C). Most of the endocrown samples broke in the upper part of the tooth together with the intrapulpal extension so the root remained intact (Figure 6A2). The small dimensions of this extension, which could be a disadvantage for its retention, here shows to be an advantage. Other research on repairability is inconsistent: There is a study that states that central incisors restored with-posts causes less fractures, but they compared a fiber post against no extension into the root canal.<sup>34</sup> Von Stein and others<sup>35</sup> reported no significant difference

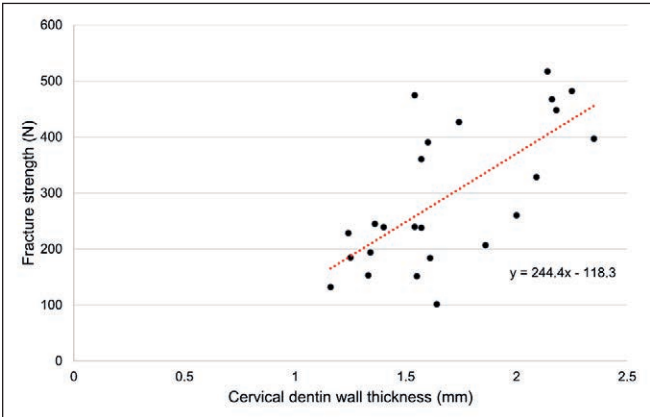


Figure 7. Statistically significant correlation equation between cervical DWT and fracture strength for post and core samples.  $y$ =fracture strength (N),  $x$ =cervical DWT. Abbreviation: DWT: dentin wall thickness.

between post and no-post, and both were repairable in 60% to 90% of specimens. Magne and others<sup>3</sup> studied the application of endocrowns on bovine incisors and found better repairability of endocrowns. They found 100% irreparable fractures in post and core crowns and 47% repairable fractures in endocrowns. The inconsistency in the literature is probably explained by the inconsistencies in the methodologies and study designs. The depth of posts, the design of no-post groups, and the materials used could have had an impact on fracture behavior. Posts are longer than the intrapulpal extension used in this study. Post and core crown samples caused fractures that extended further into the root than the endocrown samples caused. The shorter the extension, the smaller the fracture. The extension length should be balanced between macromechanical retention and the prevention of root fractures. In posterior teeth, additional studies found no difference in repairability between conventional (post-core) and endocrowns.<sup>32,33,36</sup>

There was a statistically positive correlation between the variables, DWT and surface, and fracture strength in the post and core crown samples. The fourth hypothesis, concerning the correlation between surface/DWT and fracture strength, can therefore be partially rejected. There was a significant positive correlation between both adhesive surface/DWT and fracture strength in the post and core crown restorations. When the walls were thicker, or the adhesive surface larger, higher fracture strengths were obtained. This correlation could also explain the difference in standard deviation for fracture strength between the post and core crown groups and the endocrown groups. Varying dimensions of the post and core crown samples determine the fracture strength and contribute to a higher standard deviation. The increase in fracture strength correlated to DWT was also found in other studies.<sup>27,37</sup> A significant difference was found between the 2 mm and 1 mm groups and the 0.5 mm remaining dentin wall groups.<sup>27</sup> The same findings were found on long posts (12 mm).<sup>37</sup> One study found no significant difference between 1- and 2-mm thick roots.<sup>38</sup> However, the failure mode is always significantly different and more destructive in samples with less DWT.<sup>37,38</sup> In all these studies, bovine teeth<sup>37</sup> or human canines<sup>27,38</sup> are used, and there are no studies considering DWT on human upper incisors. Due to the correlation found, it could be stated that the smaller the teeth, the less favorable a post placement becomes: The fracture strength decreases, and the failure mode seems to become more destructive. This tendency was not found in the endocrown groups.

A possible limitation of this study is the deficiency of the initial dimension measurement and the high variance in fracture strength, and, therefore, the low power of one-way ANOVA considering the fracture strength. Due to the variety in tooth dimensions there is a higher standard deviation in fracture strength. The estimated power of the one-way ANOVA is 0.3. This low power is a consequence of the study design in which the DWT is of interest and another test (regression analysis) was performed. If a stricter inclusion protocol on the size of teeth was used, the influence of DWT would be difficult to study. Because of the variance in tooth dimension, there was a variance in DWT after preparation, which led to variation in fracture strength (shown as significant regressions). There was no Weibull analysis performed based on the disadvantages of the restricted sample size and the diversity of materials noted by Quinn & Quinn.<sup>39</sup>

Based on a study by Marchionatti and others,<sup>40</sup> no simulation of periodontal ligament (PDL) was used in this study. They studied the influence of PDL on teeth restored with fiber posts, comparable with the current study and found no significant difference in fracture strength. Therefore, the use of a PDL was not applied, comparable with many articles on this subject.<sup>2,23,31,36,41</sup> If there was an impact on fracture strength: it was standardized for all groups.

The computer-aided design/computer-aided manufacturing (CAD/CAM) fabrication of the restorations was another limitation in this study, and probably also in a clinical situation. The goal of an endocrown is to create more macromechanical retention due to the intrapulpal extension, thereby obtaining higher fracture strength. In this study, the scanner wasn't able to properly detect the intrapulpal preparation for the endocrown samples; the software and milling unit could not design and mill the intrapulpal extension for a perfect fit with the cavity and increasing macromechanical retention. This led to a loose fit of the crowns on the preparations. These disadvantages of CAD/CAM are not found in earlier studies. One study used CAD/CAM, but on bovine teeth, which are larger and easier to scan.<sup>2</sup> Another study used a different preparation design and scanning method.<sup>25</sup> Taking the preparation, intraoral scanner, and milling unit used in this study in consideration, a better method to fabricate the restorations would be conventional impressions and using pressed ceramics. Next to that, multiple studies on the internal fit of ceramic crowns state that the fit of heat-pressed crowns is better than CAD/CAM milled crowns.<sup>42-44</sup> The tools of conventional impressions should be researched in future studies.



## CONCLUSIONS

1. Endocrowns and post and core crowns on central and lateral incisors had no statistically different fracture strengths.
2. The endocrown restorations had significantly more repairable failures than the post and core crowns in the central incisor groups.

There was a positive correlation between DWT and the fracture strength in the post and core crowns. When the walls were thicker or the adhesive surface larger, higher fracture strengths were obtained. For the endocrown groups these correlations were not found.

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## Conflict of Interest

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

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# The Evaluation of Different Treatments of Incipient Caries Lesions: An *in Situ* Study of Progression Using Fluorescence-based Methods

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## Clinical Relevance

Effective methods to control incipient caries lesions are needed. In this investigation, several methods provide encouraging results.

## SUMMARY

This study aimed to evaluate *in situ* the inhibition of incipient caries lesion progression using different treatment protocols and to evaluate the effectiveness of fluorescence-based methods (DIAGNOdent, DIAGNOdent pen, and VistaProof fluorescence camera [FC]) in monitoring this process. The research was conducted in four phases: (1) at

baseline, (2) after a first cariogenic challenge, (3) after treatment modalities, and (4) after a second cariogenic challenge. Sixteen volunteers used intraoral acrylic palatal appliances, each containing six enamel blocks (n=96). The cariogenic challenge was performed using a 20% sucrose solution over a 14-day period. The appliances were removed eight times a day and, upon removal, two drops

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of the solution were placed onto each enamel block. The enamel blocks were randomly assigned to three treatment groups: fluoride varnish ([FV] Duraphat;  $n=32$ ), resin infiltrant ([RI] Icon;  $n=32$ ), and adhesive system ([AS] Scotchbond;  $n=32$ ). At the end of each phase, the surface microhardness (SMH) was measured, and two trained examiners evaluated the specimens using fluorescence-based methods. In addition, integrated mineral loss ( $\Delta\Delta Z$ ;  $\text{vol\%} \cdot \text{mm} \times \mu\text{m}$ ) and lesion depth ( $\Delta LD$ ;  $\mu\text{m}$ ) were evaluated using transverse microradiography. A two-way analysis of variance and a Tukey *post hoc* test were calculated ( $\alpha=5\%$ ). Significant differences in SMH were observed according to the treatment, phases, and interaction of factors ( $p<0.001$ ). Treatment with FV resulted in significantly higher SMH values in phases 3 and 4 compared to RI and AS, with the last two treatments resulting in similar values ( $p>0.05$ ). The  $\Delta\Delta Z$  value was similar for FV and AS but significantly higher for RI ( $p=0.016$ ).  $\Delta LD$  was not significantly different among the groups ( $p=0.126$ ). Significant differences in the measurement of fluorescence for each fluorescence-based method were observed between each phase of the study ( $p<0.05$ ). It can be concluded that all treatments were effective in inhibiting the *in situ* progression of incipient lesions, although to different degrees, with minor mineral loss changes observed for the AS and FV. Besides, all fluorescence-based methods tested, except for that using the FC device, were effective in monitoring caries lesion progression.

## INTRODUCTION

The assessment of incipient caries lesion development and progression plays an important role in promoting adequate oral health care. It allows for the use of nonoperative preventive measures and minimally invasive treatments based on the control or arrest of caries lesion progression,<sup>1</sup> which is the current recommendation according to the Minimal Intervention Dentistry concept.<sup>2</sup>

In this context, it is possible to find several therapies, with different mechanisms of action, for controlling incipient caries lesions, such as fluoridated agents, adhesive sealants, and resin infiltration.<sup>2</sup> Fluoridated agents act by inhibiting enamel demineralization, increasing remineralization, and inhibiting acidic and aciduric bacterial enzyme production.<sup>3,4</sup> Adhesive sealants are used to arrest dental caries progression by preventing contact with the oral fluids through

the use of a mechanical support to the tissue, thereby inhibiting further cariogenic challenge.<sup>5-7</sup> Similarly, the use of a resin infiltrant (RI) is an alternative treatment for arresting and inhibiting the progression of noncavitated caries lesions through the penetration of low-viscosity photopolymerizable resins into the enamel pores present in the body of the lesion, thereby preventing the diffusion of cariogenic acids and mineral dissolution.<sup>8</sup> The question remains unanswered as to whether fluoride varnish (FV), resin infiltration, or conventional adhesive might be able to inhibit dental caries progression in an oral cavity with constant cariogenic challenges.

The slow progression of caries lesions allows for early detection and monitoring, providing the opportunity for the correct management of the patient.<sup>9</sup> Thus, fluorescence-based methods have been suggested for the detection and quantification of caries lesions.<sup>10</sup> DIAGNOdent 2095 ([LF] KaVo, Biberach, Germany) and DIAGNOdent 2190 pen ([LFpen] KaVo) are fluorescence-based devices that emit a diode laser at a wavelength of 655 nm (within the red range of the visible spectrum) and capture the fluorescence emitted by the endogenous porphyrins (fluorophores) produced by the cariogenic bacteria. The detected fluorescence is transformed into numerical values ranging from 0 to 99.<sup>11,12</sup> The VistaProof intraoral fluorescence camera ([FC] Dürer Dental, Bietigheim-Bissingen, Germany) is another fluorescence-based device that illuminates the dental surface with a 405 nm wavelength light emitted by six LEDs (within the blue range of the visible spectrum). This system captures the image of the dental surface at the time of fluorescence emission and transforms the ratio of green (wavelength of approximately 510 nm) and red (wavelength of approximately 680 nm) fluorescence emitted by the dental tissues into numerical values. According to the manufacturer, these values are correlated with the extent of the caries lesion.<sup>13</sup>

These fluorescence-based methods (LF, LFpen, and FC) appear to aid the process of detecting caries lesions. However, their performance is still questionable when used on smooth surfaces. Studies have evaluated the performance of these devices for detecting and monitoring caries lesions on smooth surfaces<sup>14-18</sup> and for monitoring the remineralization process.<sup>19-22</sup> Recent studies have evaluated the use of fluorescence-based devices as complementary methods for monitoring incipient caries lesions treated with RI or dental sealants on smooth and occlusal surfaces, with controversial results.<sup>23-26</sup>

No previous study has been performed to evaluate the effectiveness of LF, LFpen, and FC devices in monitoring the inhibition of the progression of



noncavitated caries lesions on smooth surfaces *in situ* after different treatments based on a minimally invasive dentistry (MID) philosophy. Thus, the aims of this *in situ* study were to evaluate the effectiveness of (1) different treatments in the inhibition of incipient caries lesion progression, and (2) fluorescence-based methods in monitoring this process. The null hypotheses were that (1) there is no difference among treatments based on an MID approach for inhibiting the progression of incipient caries lesions, and (2) fluorescence-based methods are not able to monitor enamel lesion progression.

## METHODS AND MATERIALS

### Experimental Design

This prospective, Institutional Review Board–approved, double-blind *in situ* study involved four phases: baseline (phase 1); a first cariogenic challenge, involving demineralization induction for 14 days (phase 2); treatment modalities of specimens (phase 3); and a second cariogenic challenge, involving demineralization induction for an additional 14 days (phase 4).

### Sample and Specimen Preparation (Phase 1)

The sample size was calculated based on the surface microhardness (SMH) remineralization data (primary outcome) from a previous study<sup>22</sup> and was calculated with a website power calculator ([www.sealedenvelope.com](http://www.sealedenvelope.com)). Considering a continuous outcome, a superiority trial was performed ( $\alpha$  at 5% and  $\beta$  at 80%); the SMH of the control group after the remineralization phase was  $43.9 \pm 25.2$  (mean  $\pm$  standard deviation) and the limit of equivalence was at 18%, thus a total of 31 samples per group were required to detect possible differences.

A total of 200 enamel blocks (4×4×2 mm) were obtained from freshly extracted sound bovine incisors. Teeth were disinfected in a 2% formaldehyde solution for one month. The enamel blocks were obtained after two double sectionings (Isomet 1000; Buehler, Lake Bluff, Illinois, USA) of the widest portion of dental crowns.<sup>27</sup> The enamel specimens were then stored in a 0.1% thymol solution.

Each enamel block was prepared and successively polished with carbide paper of different grits (Ecomet 250; Buehler, Lake Bluff, IL, USA) and diamond abrasive paste (Teclago; Vargem Grande Paulista, São Paulo, Brazil).<sup>22</sup>

A microhardness tester with a Knoop diamond (HNV-2; Shimadzu Corporation, Tokyo, Japan) was used for SMH analysis, as described in previous studies.<sup>20,22</sup> Of the 200 enamel specimens, only the 96 that exhibited hardness of  $368.3 \pm 80.0$  KHN were selected.

### Measurements Using Fluorescence-based Methods

Each enamel block was fixed in an acrylic resin disk and analyzed by two experienced examiners (MBD and PHC) using LF, LFpen, and FC devices.<sup>22</sup> Each examiner individually assessed the enamel blocks using the fluorescence-based devices three times, and the mean values were recorded.<sup>22</sup>

The LF and LFpen fluorescence analyses were performed following the manufacturer's instructions.<sup>22</sup> Before measurement, each device with its specific tip was calibrated using a standard reference and swept across each enamel block.<sup>21</sup> The maximum fluorescence value was recorded. The FC analysis was conducted in a dark room. The images of the enamel blocks were analyzed using DBSWIN software (Dürr Dental, Bietigheim-Bissingen, Germany), which translates fluorescence into numbers.<sup>13</sup>

### Participant Selection

A total of 16 healthy adult volunteers (3 males and 13 females, aged 20 to 40 years) who lived in a community with fluoridated water (0.7 ppm F) were selected and signed informed consent forms. They were in good general health (ie, no systemic illness, no drug use that affects salivary parameters, nonsmoking, not pregnant or breastfeeding, and no use of orthodontic appliances) and good oral health (ie, no active caries lesions or significant gingivitis/periodontitis).<sup>22</sup> All ethical and methodological aspects related to this *in situ* investigation were explained to the participants.

### First Cariogenic Challenge (Phase 2)

The enamel blocks were immersed in a 70% alcohol solution for 30 minutes.<sup>27</sup> Then, the outer one-third of the enamel surface of each block was covered with nail varnish (sound control area), leaving two-thirds of the enamel for induction of artificial demineralization<sup>27</sup> under the *in situ* protocol. The enamel blocks (n=96) were randomly allocated according to the different treatment modalities (n=32 per group). Then, two enamel blocks per group were randomly assigned to each participant (n=6; [www.sealedenvelope.com](http://www.sealedenvelope.com)).

Each participant wore an intraoral palatal appliance containing six spaces. One enamel block was fixed with wax in each space, leaving a 1-mm gap for biofilm formation, and protected by a plastic mesh.<sup>22</sup>

The cariogenic challenge was performed through exposure to a 20% sucrose solution over 14 days. Participants were instructed to remove the intraoral appliance eight times per day, and 2 drops of the solution were placed onto each enamel block.<sup>21</sup> Then,

the participant was instructed to put the appliance back into the mouth 5 minutes after sucrose exposure. Participants were also instructed to wear the appliances except during meals, drinking, and oral care.<sup>28</sup> Volunteers were instructed to brush their natural teeth with the provided nonfluoride dentifrice (Cocoricó; Bitufo, Itupeva, São Paulo, Brazil).<sup>21</sup> They were also instructed to not use any fluoridated or antibacterial products.

During the experimental phases, the participants were questioned about the use and stability of the intraoral acrylic palatal appliances and any possible discomfort. The participants' understanding of and compliance with the clinical protocol was constantly monitored.

After the first cariogenic challenge (phase 2), all enamel blocks (n=96) were removed from each intraoral appliance and mildly brushed to remove biofilm; they were then immersed in an ultrasonic bath, in deionized water for 2 minutes. Then, the enamel blocks were fixed in acrylic disks and kept in a humid environment in a refrigerator until further analysis. Fluorescence-based SMH analyses were performed as previously described.<sup>20,22</sup> Afterwards, the other outer one-third of the enamel surface of each specimen was covered with nail varnish (demineralized control area), leaving a central area of the enamel that was previously demineralized<sup>27</sup> for the treatment of enamel blocks (phase 3) and the second *in situ* cariogenic challenge (phase 4).

A 14-day nontreatment period between the first and second cariogenic challenges allowed for examinations, and the samples were kept in a humid environment in a refrigerator during this period. A previous study has shown that fluorescence values decrease after this period of nontreatment due to the storage method.<sup>29</sup>

### Treatment Modalities of Specimens (Phase 3)

The enamel blocks were treated according to the experimental groups (n=32): FV (5% NaF, Duraphat; Colgate-Palmolive, São Paulo, Brazil), RI (Icon; DMG, Hamburg, Germany), and AS (Adper Scotchbond Multi-Purpose; 3M ESPE Dental Products, St Paul, MN, USA).

Fluoride varnish was applied to the enamel surface using a standardized microbrush, and the samples were stored in artificial saliva<sup>30</sup> at 25°C for 6 hours (pH 6.8, 30 ml per sample) in order to promote the specimens' remineralization,<sup>30</sup> although this remineralization process continues with the effect of saliva during the use of the palatal appliances *in situ*. After that, the FV was removed using a blade and cotton swabs soaked

in 50% acetone.<sup>27</sup> After this procedure, the nail varnish was applied to recover the control areas.<sup>27</sup>

Resin infiltrant was applied to the enamel blocks following the manufacturer's instructions. The demineralized enamel surfaces were etched for 2 minutes with 15% hydrochloric acid (HCl; Icon-Etch; DMG, Hamburg, Germany), water rinsed for 30 seconds, and air dried for 10 seconds. Then, ethanol (Icon-Dry; DMG, Hamburg, Germany) was applied for 30 seconds, followed by additional air drying for 10 seconds. The low-viscosity RI (Icon-Infiltrant; DMG, Hamburg, Germany) was applied on the surface for 3 minutes. After that, the resin was light cured for 40 seconds using a light-emitting diode device at 900 mW/cm<sup>2</sup> (Radii Cal; SDI Dental Products, Victoria, Australia). The infiltrant was additionally applied for 1 minute and light cured for 40 seconds. Then, the enamel blocks were polished using #4000 grit aluminum oxide abrasive papers for 10 seconds.

Adhesive was applied to the enamel surface after 37% phosphoric acid gel etching (step 1) (Super Etch; SDI Dental Products, Victoria, Australia) for 60 seconds before rinsing with water for 60 seconds. After gentle air drying, only the bond component (step 3—the hydrophobic component) of Adper Scotchbond Multi-Purpose was applied for 20 seconds using a microbrush, air dried for 2 seconds, and light-cured using a light-emitting diode device (Radii Cal; SDI Dental Products, Victoria, Australia) with output at 900 mW/cm<sup>2</sup> (measured with a radiometer) for 20 seconds. The enamel blocks were also polished using #4000 grit aluminum oxide abrasive papers for 10 seconds.

Then, the specimens were fixed in acrylic disks. Surface microhardness and fluorescence-based measurements were obtained as previously described. The time interval between this phase (phase 3) and phase 4 was one week,<sup>22</sup> as fluorescence values decrease only after one to two weeks of sample storage.<sup>29</sup>

### Second Cariogenic Challenge (Phase 4)

In the second *in situ* cariogenic challenge, the six enamel blocks were washed with deionized water and fixed in each intraoral acrylic palatal appliance, with a new plastic mesh for biofilm accumulation.

The participants wore the appliances again for an additional 14 days and were instructed to follow the same protocol as described in phase 2. Then, the enamel blocks were removed from the intraoral appliances, cleaned, fixed in acrylic disks, and kept in a humid environment in a refrigerator until further analysis. Fluorescence-based and SMH analyses were performed as previously described.

### Transverse Microradiography

The enamel blocks were sectioned perpendicularly to the central area, and one-half was analyzed using transverse microradiography (TMR). The preparation of the enamel blocks and methodology for acquiring microradiographs for each specimen obtained were performed as previously described by Cardoso and others.<sup>27</sup> The mineral content was calculated from one picture of each enamel specimen (at the initial and final lesion areas), and the step-wedge grey levels were obtained using the formula from Angmar and others.<sup>31</sup> Sound enamel mineral content was assumed to be 87 vol%. The lesion depth (LD) was obtained using a 95% threshold of the mineral content of sound enamel (82.7%). For the comparison between the initial and final lesion enamel areas ( $\Delta Z$ , integrated mineral loss), the differences were calculated as follows:  $\Delta\Delta Z = \Delta Z$  initial lesion –  $\Delta Z$  final lesion;  $\Delta LD = LD$  initial lesion –  $LD$  final lesion.

Through the TMR analysis, it is possible to compare the percentage of mineral loss and LD of the first and second cariogenic challenges, as the enamel was protected with nail varnish in phase 1 (healthy control area) and after phase 2 (demineralized control area), leaving a central band of the demineralized enamel (for the treatment of specimens [phase 3] and the second *in situ* cariogenic challenge [phase 4]).<sup>27</sup> So, it is possible to compare, through the TMR software, the differences between the areas of demineralization of each phase.

### Statistical Analysis

Data analysis was performed using MedCalc for Windows (version 12.3.0; MedCalc Software, Mariakerke, Belgium) and Statistica for Windows (version 8.0; Stat Soft, Inc., Tulsa, OK, USA). The significance level was set at 5%. Outcome variables were the mean values of LF, LFpen, FC, SMH,  $\Delta\Delta Z$ , and  $\Delta LD$ , and the phases (1, 2, 3, and 4) and experimental

groups (FV, RI, and AS) were the variation factors.

The Kolmogorov-Smirnov and Shapiro-Wilk tests were used to check the data for normal distribution. All requirements for the analysis of variance (ANOVA) were met. Two-way ANOVA and the Tukey *post hoc* test were performed for statistical comparisons.

Quantitative data were represented as means and standard deviations for all phases of the study (phase 1 [baseline], phase 2 [after the first cariogenic challenge], phase 3 [after treatment modalities], and phase 4 [after the second cariogenic challenge]).

Inter-examiner reproducibility for fluorescence-based methods (LF, LFpen, and FC devices) was assessed by calculating the intraclass correlation coefficient (ICC), which ranges from poor (<0.40) to excellent (>0.75).

A receiver operating characteristic (ROC) analysis was carried out to assess the performance of LF, LFpen and FC devices in monitoring the inhibition of incipient caries lesion progression following different treatment modalities. The area under the ROC curve ( $A_z$ ) was calculated to indicate the overall accuracy of each device.<sup>9</sup> Moreover, with the ROC analysis, the optimal cut-off points between sound and demineralized surfaces were calculated. With these cut offs, sensitivity and specificity values were also calculated for each method in phases 2 and 4.

## RESULTS

In total, 16 subjects were able to finish the experimental periods. No participants reported adverse events or side effects.

Table 1 represents the SMH analysis of the enamel blocks with different treatments in all phases of the study. The two-way repeated measures ANOVA indicated differences for treatments ( $p < 0.001$ ), phases ( $p < 0.001$ ), and interactions between the two factors (treatments and phases) ( $p < 0.001$ ). With respect to the phases of the

Table 1: Surface Microhardness in KHN (mean  $\pm$  standard deviation) of the Enamel Blocks with Different Treatments (Experimental Groups) in All Phases of the Study<sup>a</sup>

Experimental Groups	Phase 1	Phase 2	Phase 3	Phase 4
FV	376.3 $\pm$ 21.7 A,a	143.5 $\pm$ 47.9 B,a	110.4 $\pm$ 64.2 C,a	62.7 $\pm$ 54.5 D,a
RI	371.3 $\pm$ 21.5 A,a	143.9 $\pm$ 56.1 B,a	34.2 $\pm$ 17.8 C,b	36.8 $\pm$ 19.1 C,b
AS	366.2 $\pm$ 27.3 A,a	149.4 $\pm$ 60.4 B,a	36.8 $\pm$ 19.1 C,b	25.4 $\pm$ 15.5 C,b

Abbreviations: ANOVA, analysis of variance; AS, adhesive system; FV, fluoride varnish; KHN, Knoop hardness number; RI, resin infiltrant.

<sup>a</sup>Significant differences are represented by different uppercase letters within the same row and different lowercase letters within the same column (two-way repeated measures ANOVA and Tukey *post hoc* test;  $p < 0.05$ ).

study, no statistically significant difference was noted for SMH values among groups ( $p>0.05$ ) within phases 1 and 2, demonstrating homogeneity for all groups at baseline and after the first *in situ* cariogenic challenge. However, FV SMH values were greater than RI and AS in both phase 3 (after treatment) and phase 4 (after the second *in situ* cariogenic challenge) ( $p<0.05$ ). With respect to treatment modalities and their respective phases, SMH values reduced in each assessed phase, except for RI and AS, in which no statistically significant difference was noted for SMH values in phases 3 and 4 ( $p>0.05$ ).

Table 2 shows the results of the TMR analysis. Fluoride varnish and the AS were able to inhibit lesion progression in a similar pattern and differed significantly from RI, which led to statistically significant higher demineralization according to the integrated mineral loss ( $p=0.016$ ). However, when LD was considered, none of the treatments resulted in significant changes when submitted to the second demineralization challenge, and they did not differ significantly from each other ( $p=0.126$ ).

Table 3 represents the fluorescence values of the enamel blocks with different treatments in all phases of the study. For the LF device, the two-way repeated measures analysis of variance indicated statistically significant differences only for phases ( $p<0.001$ ); no statistically significant difference was noted for treatments ( $p=0.3918$ ) and interaction between the factors (treatments and phases) ( $p=0.5631$ ). For the LFpen device, significant differences were indicated for phases ( $p<0.001$ ) and the interaction between the factors ( $p=0.0418$ ); no statistically significant difference was noted for the treatment ( $p=0.1662$ ). For the FC device, significant differences were indicated for phases ( $p<0.001$ ); no statistically significant difference was noted for treatments ( $p=0.1598$ ) and interaction

between the factors ( $p=0.5788$ ). The Tukey *post hoc* test analysis showed that LF values were ranked from lower to greater values as phase 1 < phase 3 < phase 4 < phase 2; LFpen values were ranked as phase 1 < phase 3 = phase 4 < phase 2; and FC values were ranked as phase 1 = phase 3 = phase 4 < phase 2. LFpen fluorescence values were lower for RI when compared to FV in phase 3 (after treatment). It was interpreted that there was interaction between the factors the LFpen device because all treatment modalities were different in phase 3, which was not found for the other phases.

Fluorescence-based methods detected significant differences after the first *in situ* cariogenic challenge (phase 2). However, after treatments (phases 3 and 4), fluorescence values were significantly lower compared to those observed in phase 2. These results are confirmed in Table 4, which presents sensitivity, specificity, and area under the ROC curve values in phases 2 and 4 for all fluorescence-based methods (LF, LFpen, and FC devices).

Table 5 represents the inter-examiner reproducibility calculated using ICC for the fluorescence measurements for the experimental groups in all phases of the study. Intraclass correlation coefficient values varied from 0.3653 (FC device, group 1, phase 3) to 0.8857 (FC device, group 1, phase 4). The reproducibility values indicated fair to good agreement for the fluorescence-based methods in phases 1 and 2, and fair to excellent agreement in phases 3 and 4 for all experimental groups.

DISCUSSION

Different treatment modalities for the inhibition of incipient caries lesion progression on smooth surfaces have been discussed in the literature. Some studies have evaluated the synergistic effect of resin infiltration

Table 2: Mean and Standard Deviation of the Lesion-integrated Mineral Loss ( $\Delta\Delta Z$ , %vol.min x $\mu\text{m}$ ; $\Delta Z$ Initial Lesion – $\Delta Z$ Final Lesion) and Depth ( $\mu\text{m}$ ; $\Delta LD$ = LD Initial Lesion – LD Final Lesion) for Enamel Specimens Treated With Different Materials (Experimental Groups) After Initial Demineralization <sup>a</sup>		
Experimental Groups (n)	$\Delta\Delta Z$ (%vol.min x $\mu\text{m}$ ) ( $\Delta Z$ initial – $\Delta Z$ final lesion)	$\Delta LD$ ( $\mu\text{m}$ ) (LD initial – LD final lesion)
FV (n=25)	-373.2 $\pm$ 113.8 A	-6.7 $\pm$ 4.3 A
RI (n=28)	-485.7 $\pm$ 187.9 B	-8.9 $\pm$ 6.1 A
AS (n=22)	-387.7 $\pm$ 132.4 A	-9.5 $\pm$ 4.6 A
p-value	0.016 <sup>b</sup>	0.126
Abbreviations: AS, adhesive system; FV, fluoride varnish; LD, lesion depth; RI, resin infiltrant; $\Delta\Delta Z$ , delta integrated mineral loss; $\Delta Z$ , integrated mineral loss; $\Delta LD$ , delta lesion depth; $\mu\text{m}$ , micrometer; %vol.min, volume percentage of minerals.		
<sup>a</sup> Significant differences are represented by different uppercase letters within the same column (ANOVA and Tukey <i>post hoc</i> test; <sup>b</sup> $p<0.05$ ).		



Table 3: Fluorescence Values (Mean  $\pm$  Standard Deviation) of the Enamel Blocks with Different Treatment Modalities (Experimental Groups) in all Phases of the Study<sup>a</sup>

Fluorescence-based Method	Experimental Groups	Phase 1	Phase 2	Phase 3	Phase 4
LF	FV	2.8 $\pm$ 2.1 A,a	17.1 $\pm$ 4.8 B,a	13.9 $\pm$ 4.1 C,a	14.1 $\pm$ 5.6 D,a
	RI	2.7 $\pm$ 2.3 A,a	17.0 $\pm$ 5.2 B,a	13.1 $\pm$ 3.6 C,a	14.6 $\pm$ 4.3 D,a
	AS	2.8 $\pm$ 2.1 A,a	16.6 $\pm$ 5.1 B,a	12.1 $\pm$ 4.0 C,a	14.3 $\pm$ 5.1 D,a
LFpen	FV	3.1 $\pm$ 2.4 A,a	20.7 $\pm$ 5.9 B,a	15.8 $\pm$ 5.4 C,a	15.2 $\pm$ 5.9 C,a
	RI	3.3 $\pm$ 2.4 A,a	20.4 $\pm$ 6.1 B,a	12.7 $\pm$ 3.1 C,b	15.1 $\pm$ 5.5 C,a
	AS	3.1 $\pm$ 2.0 A,a	19.9 $\pm$ 6.1 B,a	13.5 $\pm$ 3.3 C,a	14.9 $\pm$ 5.4 C,a
FC	FV	0.9 $\pm$ 0.1 A,a	1.1 $\pm$ 0.1 B,a	0.8 $\pm$ 0.1 A,a	0.9 $\pm$ 0.1 A,a
	RI	0.9 $\pm$ 0.1 A,a	1.1 $\pm$ 0.1 B,a	0.8 $\pm$ 0.1 A,a	0.8 $\pm$ 0.1 A,a
	AS	0.9 $\pm$ 0.1 A,a	1.1 $\pm$ 0.1 B,a	0.8 $\pm$ 0.1 A,a	0.8 $\pm$ 0.1 A,a

Abbreviations: ANOVA, analysis of variance; AS, adhesive system; FC, intraoral fluorescence camera; FV, fluoride varnish; LF, DIAGNOdent; LFpen, DIAGNOdent pen; RI, resin infiltrant.

<sup>a</sup>Significant differences are represented by different lowercase letters within the same column and different uppercase letters with the same row (two-way repeated measures ANOVA and Tukey post hoc test;  $p < 0.05$ ).

and fluoride application.<sup>32,33</sup> However, there is lack of comparison among them as separate treatments, as they have different mechanisms of action.

It is important to highlight that this is the first study that has evaluated caries lesion progression after different treatment modalities in a highly cariogenic environment, without the effect of residual fluoride from the dentifrice using an *in situ* model. To the best of our knowledge, there is one *in situ* study in the literature that has evaluated the inhibition of caries progression through the use of resin infiltration and sealing. In this study, volunteers used the appliances for approximately 3 months, with two 30-minute exposures to 10% sucrose daily.<sup>34</sup> In the present investigation, the null hypothesis

that there was no difference among the treatments based on an MID approach to inhibit the progression of incipient caries lesions was partially accepted. Fluoride varnish, RI and the AS were effective in inhibiting caries lesion progression, as indicated by the TMR analysis when considering LD ( $\Delta$ LD). Considering the differences between the LD values at baseline and after treatment and the second cariogenic challenge, it can be assumed that they are not clinically relevant, as they represent a difference of less than 10  $\mu$ m (as indicated by  $\Delta$ LD). Concerning the integrated mineral loss ( $\Delta$  $\Delta$ Z) values, FV and the AS led to a statistically significant lower subsurface demineralization compared with the RI Icon. This fact might be explained by the differences

Table 4: Sensitivity, Specificity, Area Under the ROC Curve (Az) Values, and Cut-off Points for LF, LFpen, and FC Devices for Phases 2 and 4

Phase	Fluorescence-based Method	Sensitivity	Specificity	Az	Cut-off Points
2	LF	0.835	0.933	0.868	>8
	LFpen	0.990	1.000	1.000	>9
	FC	0.948	0.807	0.932	>0.9
4	LF	0.469	0.667	0.556	>14
	LFpen	0.401	0.745	0.545	>16
	FC	0.349	0.781	0.557	>0.8

Abbreviations: Az, area under the ROC (receiver operating characteristic) curve; FC, intraoral fluorescence camera; LF, DIAGNOdent; LFpen, DIAGNOdent pen.

Table 5: Inter-examiner Reproducibility Represented by the Intraclass Correlation Coefficient and 95% Confidence Interval for LF, LFpen, and FC in the Experimental Groups for All Phases of the Study

Fluorescence-based Method	Experimental Groups	Phase 1	Phase 2	Phase 3	Phase 4
LF	FV	0.6019 (0.3845-0.8057)	0.6575 (0.3983-0.8328)	0.4561 (0.3141-0.7345)	0.5400 (0.3570-0.6290)
	RI	0.6074 (0.4055-0.9060)	0.5136 (0.3123-0.6355)	0.6693 (0.3225-0.8386)	0.6268 (0.2354-0.8178)
	AS	0.5301 (0.3744-0.7706)	0.6244 (0.3305-0.8166)	0.6050 (0.3109-0.8072)	0.4529 (0.3207-0.7330)
LFpen	FV	0.6919 (0.4416-0.8982)	0.5106 (0.3075-0.7123)	0.7525 (0.4929-0.8792)	0.4913 (0.3214-0.7517)
	RI	0.6127 (0.3038-0.8128)	0.5504 (0.3260-0.7317)	0.5385 (0.4463-0.7747)	0.7981 (0.5865-0.9015)
	AS	0.6626 (0.4562-0.8226)	0.6629 (0.3094-0.8354)	0.7576 (0.5035-0.8817)	0.4996 (0.3502-0.7558)
FC	FV	0.6884 (0.5080-0.8405)	0.7210 (0.4285-0.8638)	0.3653 (0.3001-0.6902)	0.8857 (0.7659-0.9442)
	RI	0.6750 (0.5546-0.9193)	0.7446 (0.4767-0.8753)	0.7187 (0.4238-0.8627)	0.5003 (0.2285-0.7073)
	AS	0.6937 (0.3721-0.8078)	0.6000 (0.3806-0.8047)	0.4510 (0.3633-0.5898)	0.6832 (0.6832-0.8454)

Abbreviations: AS, adhesive system; FC, intraoral fluorescence camera; FV, fluoride varnish; LF, DIAGNOdent; LFpen, DIAGNOdent pen; RI, resin infiltrant.

in the mechanism of action and application protocol of the products (per the manufacturer's instructions). The fact that RI demonstrated higher mineral loss can be attributed to the rather harsh etching procedure (the use of 15% hydrochloric acid for 2 minutes) that may have led to the removal of the surface layer of the lesion, increasing the subsurface demineralization, as shown by Freitas and others<sup>35</sup> in an *in vitro* model. In contrast, the study by Paris and Meyer-Lueckel<sup>34</sup> replaced the 15% hydrochloric acid with 37% phosphoric acid for 5 seconds for both RI and sealing treatments, for better permeability and to diminish the surface layer removal. Moreover, our results corroborated the study by Gelani and others<sup>32</sup> (with respect to  $\Delta\Delta Z$  values), who used an *in vitro* model for evaluating lesion progression. In their study, fluoride gel was able to inhibit lesion progression in the same way as resin infiltration combined with fluoride gel application, which was significantly better than RI alone. According to Meyer-Lueckel and Paris<sup>36</sup>—and also employed in the present study—RI should be applied after 2 minutes of etching with HCl to penetrate more deeply and occlude the enamel pores generated by demineralization, to inhibit lesion progression. Thus, considering the mechanism of action

of resin infiltration, greater mineral loss detected in this group might not be an indicator of lack of effectiveness.

One of the topics of *in situ* and *in vitro* investigations is the degree of demineralization and LD ( $<50\ \mu\text{m}$ ) of artificial caries-like enamel lesions, which is not consistent with what is observed *in vivo*, as discussed previously.<sup>35,37,38</sup> In the present study, the mean LD was approximately  $20 \pm 6.5\ \mu\text{m}$ , which can influence the results of the different treatment modalities.

Regarding the SMH analysis, a similar pattern of inhibition of lesion progression was observed after the second cariogenic challenge. It should be noted that the surfaces treated with resin infiltration and an AS exhibited lower SMH values after phases 3 and 4. Previous studies have shown increased caries lesion microhardness after resin infiltration,<sup>35,39,40</sup> since the low viscosity resin fills the lesion and creates a barrier to the lesion and the lesion body. However, the studies by Torres and others<sup>41</sup> and Neres and others<sup>42</sup> demonstrated a significant reduction in SMH of the group treated with resin infiltration after a new cariogenic challenge, which can be attributed to the incomplete dissolution of the remaining mineral content of the lesion body

that was not fully embedded in the resin matrix or polymerization contraction. Neres and others<sup>42</sup> also demonstrated higher surface roughness (ie, grooves and cracks) after enamel conditioning with HCl.

Adhesive systems do not have high penetration power, and, when present, penetration occurs only on the surface of natural enamel caries lesions.<sup>8</sup> However, in this study, the AS acted as a barrier to the new cariogenic challenge in the same pattern as the RI,<sup>43</sup> which might be attributed to the shallow artificial lesion produced in this study model. This is consistent with the literature, which shows better effectiveness in protecting enamel dissolution in early enamel lesions.<sup>44</sup> Previous studies using ASs to penetrate into artificial caries lesions showed surface sealing instead of penetration and occlusion of pore space.<sup>37,45</sup> On the other hand, RI leads to a full, but partially inhomogeneous, penetration of artificial caries lesions.<sup>44</sup> In addition, it is a treatment option for active white spot lesions because it promotes an aesthetic masking of these lesions,<sup>39,46-48</sup> with some color change after a new acid challenge.<sup>46,49</sup> If phase 2 had been completely skipped, the results for both the resin infiltration and AS treatments would probably be different for microhardness analysis, since it would be performed on a sound surface with no incipient lesion. Moreover, it is important to mention that applying HCl also promotes a rough surface and could influence SMH values.

With respect to the group treated with FV, a reduction in surface microhardness was also observed after phases 3 and 4. However, these SMH values were significantly higher than the values observed for RI and ASs. Fluoride precipitates calcium from saliva and promotes the formation of calcium fluoride ( $\text{CaF}_2$ ) reserves when highly concentrated agents are used. In the present study, the higher cariogenic environment and the absence of daily fluoride dentifrice use by the participants may have led to an increase in the dissolution rate of  $\text{CaF}_2$ , thereby increasing the demineralization rate of the lesion. However, when using fluoride therapies, the lesion body does not remineralize to the same level of the previous surface zone.<sup>50</sup> The remineralization of the outer surface of the enamel does not improve the aesthetics and structural properties of the deeper lesion.<sup>51</sup> It should be emphasized that FV was applied only once, simulating a professional clinical condition.<sup>40</sup> Thus, the short contact time between the varnish and the surface may have influenced the results. It is already known that regular applications of FV may increase anticaries properties.<sup>40</sup> In this context, RI can have a better aesthetic resolution, as it can penetrate the deeper

layers of the lesion. However, it can also present color alteration over time after staining processes.<sup>49</sup>

The second hypothesis of this investigation was rejected, as the fluorescence-based methods were able to monitor the enamel lesion progression. In general, the fluorescence values showed substantial differences between phases 1, 2, and 3 for all treatment modalities, proving to be effective for monitoring the progression of *in situ* enamel caries lesions. These results corroborate the findings of Spiguel and others,<sup>21</sup> Moriyama and others,<sup>22</sup> Diniz and others,<sup>17</sup> and Rodrigues and others,<sup>18</sup> who also used artificial caries-like lesions on smooth surfaces. The fluorescence values of LF and LFpen were significantly higher after *in situ* cariogenic challenge when compared with baseline values. According to Mendes and Nicolau,<sup>14</sup> an increase in LF values after artificial demineralization could be explained by an increase in porosity and light scattering on the enamel surface. However, LF's effectiveness is uncertain in artificial lesions created with no oral bacterial metabolites, as the LF device identifies changes in the organic content of the tooth structure (fluorophores and other chromophores produced by cariogenic bacteria) rather than inorganic content.<sup>10,11</sup> Previous studies reported that an increase in fluorescence values can be related to the penetration of bacteria, which produce substantial amounts of endogenous porphyrins and organic compounds, into the enamel lesions.<sup>17,21</sup>

It should be noted that LF and LFpen measurements were different at all phases of this investigation. The LFpen fluorescence values were higher than LF values, as shown in previous studies.<sup>13,17,52,53</sup> These differences between LF and LFpen fluorescence values can be associated with the type of probe tips in both devices (diameters and materials), which may impact the amount of light excitation and the level of fluorescence emitted by the dental tissues.

A significant difference could be observed between phases 3 and 4 for the LF device, with higher fluorescence values in phase 4, but the difference was not clinically relevant. In contrast, LFpen and FC devices demonstrated similar fluorescence values between phases 3 and 4. This was expected, as the different treatment modalities were able to hamper the lesion progression *in situ* in the same way. In recent clinical studies,<sup>25,26</sup> LFpen was able to detect significantly lower fluorescence values immediately after resin infiltration application and after six months of follow-up on white spot lesions on buccal surfaces when compared with baseline. The main idea of using fluorescence-based methods to monitor the inhibition of incipient caries lesion progression would be to detect a decrease in

light scattering on the enamel surface with the use of resin infiltration and, consequently, the change in fluorescence values. In an *in vitro* study, Markowitz and Carey<sup>24</sup> evaluated the LF device in assessing the ability of resin infiltration to improve the optical properties of artificial white spot lesions. The authors found no differences in the fluorescence values when analyzing the effect of demineralization and RI treatment on the appearance of the tooth structure. The fluorescence values remained at the low end of the instrument's range (close to zero). This fact could be attributed to the caries induction with pH 4.5 lactic acid gel and the absence of bacterial biofilm.

When evaluating the performance of fluorescence-based methods for caries detection and monitoring, there is no scientific evidence on the optimal cut-off points that should be used to determine the extent of caries lesions on smooth surfaces. In the present study, the baseline (sound surface) fluorescence values are consistent with the scale recommendation proposed by the manufacturers and by Lussi and Hellwig.<sup>12</sup> However, the range of fluorescence values for LF and LFpen devices in phases 2, 3, and 4 indicates enamel caries lesions. For the FC device, the range of fluorescence values in phases 3 and 4 are in the range of a sound surface, according to the manufacturer's interpretation (0.0-1.0), which could be a limitation of the device in detecting the progression of treated initial lesions.

The optimal cut-off points obtained by the ROC analysis for the detection of enamel lesions were >8 (LF), >9 (LFpen), and >0.9 (FC). The cut-offs for the detection of enamel lesion progression after treatment modalities were >14 (LF), >16 (LFpen), and >0.8 (FC). In phase 2, higher sensitivity, specificity, and Az values were observed for the fluorescence-based methods, showing their ability to identify initial caries lesion development, similar to the findings of Diniz and others.<sup>17</sup> However, in phase 4, the sensitivity and Az values were lower for all fluorescence methods, showing a moderate capacity to monitor the caries progression after different treatments. Another important aspect is related to the cut-off point for the FC device in this phase, which is not consistent with fluorescence values of enamel lesion progression and instead indicates a sound enamel surface. Thus, care must be taken when interpreting the FC values in monitoring incipient lesions.

A reliable caries detection method should present consistent data between diverse examiners and

evaluations in order to be useful for monitoring carious development.<sup>12</sup> In general, the inter-examiner reproducibility varied from fair to excellent values for all fluorescence devices and phases of the study, with different results according to the treatment modality. For the FC device, the wide range of agreement between the examiners was observed for the group treated with FV in phases 3 and 4. Previous studies have demonstrated good to excellent results for fluorescence-based devices in detecting and monitoring caries progression.<sup>13,16,17,22</sup>

Some limitations of the present investigation should be indicated, such as the depth of the enamel lesion that was expected to be deeper, the *in situ* study design that did not include a crossover model, and the high cariogenic challenge model simulating a patient with high caries risk. It should be mentioned that SMH analysis was performed after each phase of this *in situ* study once the surface test was not destructive and consider further surface assessments.<sup>17,20-22,27</sup> To confirm the results, TMR was done to provide a quantitative measure of the mineral content and LD, since it is an invasive technique that allows for the comparison of differences between the areas of demineralization of each phase.<sup>27</sup>

Despite the promising results of this *in situ* study, which is a close representation of the oral environment, new investigations should be performed in clinical conditions to confirm the capacity of different treatment modalities in order to inhibit enamel lesion progression and to confirm the performance of fluorescence-based methods in monitoring this process. It is important to highlight that fluorescence-based methods are adjunct methods and must be used in combination with visual examination in clinical practice.

## CONCLUSION

It can be concluded that FV, RI and AS were effective in inhibiting the *in situ* progression of incipient caries lesions, although at different levels, with minor mineral loss changes for AS and FV. In addition, the fluorescence-based methods were effective in monitoring caries lesion progression for all treatment modalities, except for the FC device.

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## Regulatory Statement

This study was approved by the local institutional review board of Cruzeiro do Sul University (protocol #013/2015, Brazil) and by the local animal research committee of Cruzeiro do Sul University (protocol #001/2015; Brazil) for the use of bovine teeth.

## Conflict of Interest

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

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# Debonding of Leucite-reinforced Glass-ceramic Veneers Using Er,Cr:YSGG Laser Device: Optimizing Speed with Thermal Safety

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## Clinical Relevance

Removing laminate veneers on anterior teeth by using an Er,Cr:YSGG dental laser can be completed faster than previously reported while maintaining thermal safety.

## SUMMARY

**Objective:** When laminate veneer restorations require removal, the process is tedious, time-consuming, and potentially damaging to the underlying tooth structure. The purpose of this study was to evaluate the removal of Empress CAD milled laminate veneers on extracted human central incisors by using an Er,Cr:YSGG dental laser while optimizing speed and maintaining thermal safety.

**Methods and Materials:** A total of 22 extracted human incisors were mounted in acrylic blocks. Conservative veneer preparations were made on all

samples with a high-speed dental handpiece with a diamond bur and air/water spray. The 22 blocks of IPS Empress CAD were designed and milled into laminate veneers with a CAD/CAM System and luted to the prepared teeth. An Er,Cr:YSGG dental laser was fitted with a handpiece and laser fiber (600- $\mu$ m diameter cylindrical fiber, 6 mm in length). Laser parameters were 333 mJ/pulse, 30 Hz, 80% air, 50% water, 600- $\mu$ m diameter fiber tip, at a fluence of 885.96 J/cm<sup>2</sup>. The laser fiber tip was held directly on the surface of each veneer in contact, perpendicular to the surface, and moved slowly, covering the labial surface while firing.

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**Results:** At the laser parameters tested (333 mJ/pulse, 30 Hz, 80% air, 50% water, 600- $\mu$ m diameter fiber tip), the average duration of exposure to completely remove each laminate veneer was  $14.16 \pm 0.60$  seconds, with a range of 10.75 to 21.25 seconds. The average thickness of each veneer measured at the midfacial was  $0.75 \pm 0.03$  mm. The mean intrapulpal temperature increase for this period was  $0.71^\circ\text{C} \pm 0.15^\circ\text{C}$ .

**Conclusions:** A regression model between time and thickness ( $p < 0.0001$ ) proved to be significant. However, the same cannot be said when the same modeling was tested between temperature and thickness. It can therefore be concluded that as the thickness of a veneer increases, more time is necessary to remove a veneer using Er,Cr:YSGG laser energy; however, increasing thickness does not necessarily result in an increase in pulpal temperature. Within the limitations of this study (single restorative material and single luting agent), it can be concluded that removing CAD Empress laminate veneer restorations using an Er,Cr:YSGG laser is reliable and thermally safe, even at an average of 10 W of power at 30 Hz. Additionally, thermal safety is maximized with adequate aerosolized water spray.

## INTRODUCTION

Porcelain and ceramic veneer restorations have evolved into a reliable aesthetic solution for many dental situations, including darkening, fractures, failing restorations, or misalignment. For over 30 years, these restorations have proven to be especially appropriate to improve the appearance of malpositioned teeth, and they can be a significantly more minimally invasive option (in lieu of full coverage) where there are extensive existing restorations or decay.<sup>1,2</sup> When a laminate veneer fails, it is most likely a result of decay or microleakage.<sup>3,4,5,6,7,8</sup>

Due to the ever-increasing bond strengths of current luting agents, the task of removing porcelain or ceramic indirect veneer and crown restorations can be a frustrating and time-consuming process.<sup>9</sup> The most common method of removing failed laminate veneer restorations is by using a high-speed handpiece fitted with a coarse diamond bur.<sup>10</sup> Albeit common, this method can be lengthy, uncomfortable, and lead to tooth or pulpal damage because of friction, heat, and vibration.

An additional challenge includes the issue that the highly aesthetic qualities of contemporary restorative materials can make it difficult to distinguish the margin between veneer and tooth during removal.<sup>9</sup> As a result, attempts have been made to cement laminate veneers using luting agents modified with a fluorescing dye. Should the need for veneer removal arise, the fluorescing agent results in less damage to the underlying tooth structure because of improved contrast.<sup>11</sup> Laminate veneer removal using an erbium laser eliminates the need for these specialized materials and procedures because the ablative process does not rely on visual inspection or visual acuity.

Existing research and clinical reports have demonstrated success when using an erbium laser for the purpose of removing failed porcelain or ceramic restorations.<sup>12</sup> It has been suggested that the primary effect of the laser energy occurs not on the veneer or the tooth surface, but instead, in the resin luting agent, which is caused at least partially by thermal softening of the material.<sup>13</sup> However, if the luting cement is ablated rapidly, thermal softening and heat conduction is avoidable.<sup>14,15,16</sup>

There are also reports of using other laser wavelengths to etch porcelain surfaces in order to improve bond strength.<sup>17,18,19,20,21</sup> Likewise, there have been studies demonstrating the effectiveness of using an erbium laser to reduce the shear bond strength of porcelain to tooth by laser irradiation.<sup>22</sup> However, scientific papers on the true effects of laser energy on these restorations and to the underlying teeth are limited.

The actual method of ablation of the resin luting cement by laser energy is multifaceted. Erbium lasers demonstrate the highest absorption in water. The pulsing laser energy is first absorbed by the water and organic components within the resin cement, causing expansion as a result of an increase in temperature and a subsequent increase in volume. These microexplosions can be seen as flashes of light and are visible both macro- and microscopically. The increase in internal pressure results in an explosive force that includes the inorganic substances, which separates the veneer from the tooth surface by hydrodynamic ejection.<sup>23,24,25</sup>

The aim of the present study was to test multiple laser parameters while using an Er,Cr:YSGG laser to remove milled leucite-reinforced glass-ceramic veneer restorations from extracted human central incisors. The IPS Empress CAD ingots used exhibit a homogeneous distribution of leucite crystals. The leucite crystals are evenly and densely distributed. The diameter of the crystals is 1–5  $\mu$ m, and the crystal phase volume is 35%–45% by volume.<sup>26</sup>

While previous recommendations regarding the parameters to remove laminate veneers were generally much lower than those used in this study, the authors intended to determine the most efficient laser parameters to successfully remove veneer restorations as quickly as possible without overheating the tooth and dental pulp. To this end, preliminary trials of various combinations of laser pulse, power, and water spray were completed prior to the initiation of this study. What is reported in this paper are the observations of the laser parameters at maximum power output on the device being tested. Saving time is of little help if the pulpal temperature increase becomes significant. Rechmann and others have demonstrated both conservative and “worst case” removal of crowns using an erbium laser.<sup>27</sup> In those trials, the goal was to remove the veneers intact in the rare case they were misaligned during cementation.

## METHODS AND MATERIALS

A total of 22 recently extracted human maxillary central incisors were obtained from a tooth bank and mounted in acrylic blocks, leaving the clinical crown and 2 mm of root surface exposed. Conservative veneer preparations (restricted to enamel, nonincisal wrap) were made on all samples by a single operator (DC), with a high-speed dental handpiece with a medium grit, round-ended diamond bur and air/water spray. Preparations were made by first using a depth cutting bur to 0.6 mm (MADC-006; Axis Dental, Coppel, Texas, USA) and finished with diamond burs to a feather-edge gingival margin (Peter Brasseler Holdings, LLC, Savannah, Georgia, USA). The root apices were opened with a Gates-Glidden bur to allow access for a 1.5-mm diameter Type-J sheathed and grounded thermocouple (IC-SS-116-G-6; Omega Engineering Inc, Stamford, Connecticut, USA). Prepared samples were stored in 0.1% thymol solution until use.

The 22 blocks of IPS Empress CAD (Ivoclar Vivadent, Inc, Amherst, New York, USA) were designed and milled by another operator (DR), with a Cerec Omnicam and Cerec MC XL CAD/CAM System (Dentsply Sirona, Inc, York, Pennsylvania, USA). The thickness of the completed veneers was recorded in the midfacial area by operator JG, using a 500-302 caliper (Kerr Corporation, Orange, California, USA). Before cementation, each veneer was placed on its respective tooth preparation and the fit was confirmed visually and with a sharp dental explorer, using 6.0x magnification loupes (EF Loupes; Designs for Vision, Bohemia, New York, USA). Veneer preparations were etched with 35% phosphoric acid solution (Ultra-Etch; Ultradent Products, Inc, South Jordan, Utah, USA) for

20 seconds, rinsed with water spray for 10 seconds, and air dried. Bonding agent was applied (Peak Universal Bond; Ultradent Products, Inc) for 10 seconds, air dried with 50% pressure for 10 seconds, and light cured for 10 seconds (DemiUltra; Kerr Co, Orange, California, USA). The veneers were then luted to the prepared teeth using Variolink Esthetic LC (Ivoclar Vivadent, Inc, Amherst, New York, USA), according to the manufacturer's specifications. Specimens were stored in distilled water for at least 48 hours before laser irradiation.

Each sample (tooth embedded in acrylic block) was secured to a ring stand using a 3-prong vinyl coated support clamp. The thermocouple probe was positioned vertically, directly below the tooth sample using a similar support clamp, with the tip of the probe extending into the amputated root to the top of the pulp chamber. A dental latex dam was used to protect the probe housing and another was placed to protect the root of each tooth from inadvertent water contact. In order to confirm accuracy and sensitivity of the thermocouple setup, a curing light with an irradiance of 1135 mW/cm<sup>2</sup> (8-mm diameter tip with a 60° angle) was used after first stabilizing the temperature for 30 seconds before each treatment, and again for 30 seconds following the complete removal of each veneer (DemiUltra). In all cases, intrapulpal temperature normalized after laser irradiation stopped, demonstrating that there was no lag in thermal transfer, which could potentially cause a latent rise in temperature following treatment. The curing light control was confirmed, as the pulpal temperature increased 2°C after 30 seconds of light activation. (See Figure 1)

An Er,Cr:YSGG dental laser (Waterlase iPlus; Biolase, Inc, Irvine, California, USA) was fitted with a handpiece and laser fiber tip (600-μm diameter cylindrical fiber, 6 mm in length). The laser parameters were 333 mJ/pulse, 30 Hz (10.0 Watts), 80% air, 50% water, 600-μm diameter fiber, at a fluence of 885.96 J/cm<sup>2</sup>. Quantitatively, laser energy is described in terms of the actual optical energy delivered per unit area (J/cm<sup>2</sup>), which is called the laser fluence. The pulp chamber was filled with a conductive silicone paste (Omegatherm 201; Omega Engineering, Inc, Stamford, Connecticut, USA). A single experienced operator (CW) performed all trials, while another operator (JG) set up and monitored the thermocouple and recorded time measurements. The laser fiber tip was held directly on the surface of each veneer in contact, perpendicular to the surface, and activated when instructed by the timekeeper. The fiber tip was slowly moved across the surface approximately 2

mm/second, in contact, until each veneer was either dislodged whole or in fragments. Veneer fragments were retained for future evaluation.

RESULTS

All of the veneer samples fractured into at least three pieces and dislodged during laser irradiation. Light microscopy confirmed that the debonding occurred at the cement to veneer interface. This is an important fact since ablation along the tooth surface would be undesirable and could lead to potential thermal effects. Additionally, the composite that remained on the prepared surface was often darkly discolored. In all cases, the remaining composite resin was left in a weakened, “powdery” state, which could be easily removed with a hand instrument and gauze.

At the laser parameters tested (333 mJ/pulse, 30 Hz, 80% air, 50% water, 600-μm diameter fiber), the average duration of laser exposure to completely remove each laminate veneer was  $14.71 \pm 3.05$  seconds, with a range of 11.5–21.25 seconds. The mean intrapulpal temperature increase for the irradiation period was  $0.85 \pm 0.88^\circ\text{C}$  increase (Figure 1).

A Pearson correlation analysis was used to measure the strength of the linear relationship between the time, thickness, and temperature variables. The correlation

coefficient between time and thickness was 0.67 and between time and temperature was 0.30 (Figures 2 and 3). Therefore, time and thickness were correlated moderately and positively; however, there was a weak positive correlation between time and temperature.

A simple linear regression analysis of the data between time in seconds, and thickness in mm was run. The regression line can be interpreted as follows: for every one-unit increase in the thickness of the veneer (1 mm), the value of time increased on average by 17.5 seconds ( $p \leq 0.00058$ ). Although the average midfacial thickness for all samples was greater than the manufacturer’s recommended 0.7 mm, 10 of 22 veneers were slightly less than 0.7 mm. It should be noted that this measurement was made at the true midfacial point of each veneer, which is positioned in a more gingival direction compared with the images on the manufacturer’s product brochure, which is closer to the incisal edge. Since the preparations gradually increased from 0.6 mm at the gingival margin to 0.7 mm or more at the incisal edge, it would make sense that the facial reduction as measured at the midfacial of each preparation would vary and often be between 0.6 mm and 0.7 mm. In general, the time it took to remove each veneer increased in direct proportion to the thickness.

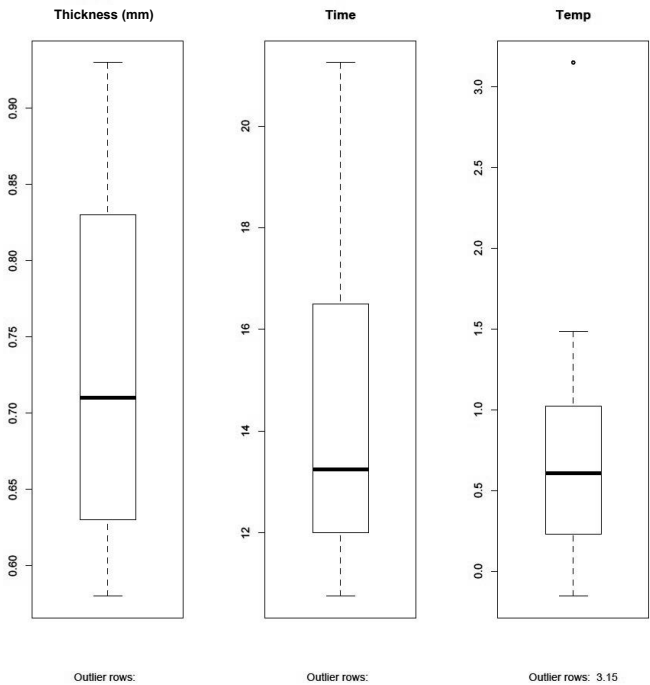


Figure 1. Plots representing thickness of veneer (mm), time for removal (sec), and pulpal temperature change ( $^\circ\text{C}$ ).

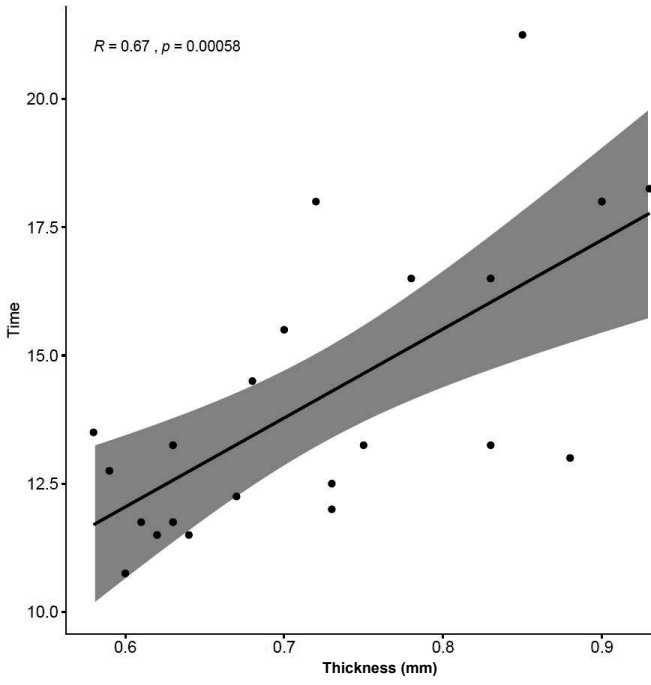


Figure 2. The Pearson correlation coefficient between time and thickness is 0.67 ( $p=0.00058$ ).

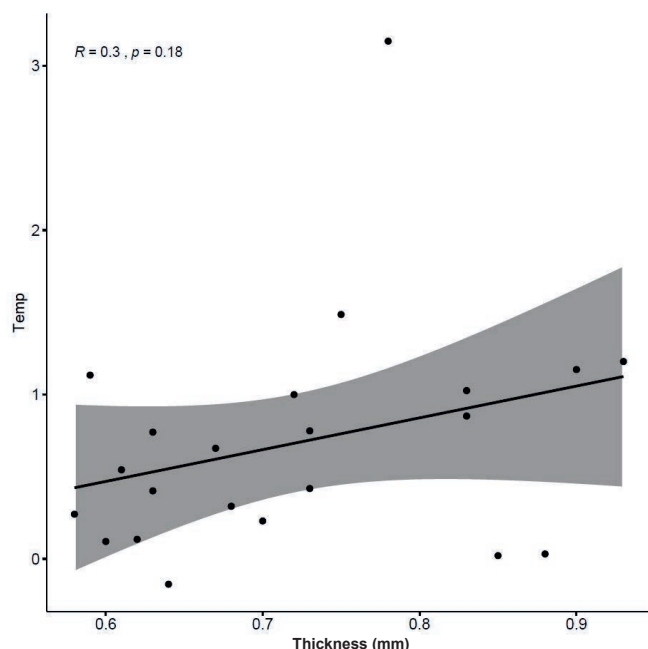


Figure 3. The Pearson correlation coefficient between temperature and thickness is 0.30 ( $p=0.18$ ).

## DISCUSSION

Van As and others have previously suggested lower laser fluences to debond laminate veneers.<sup>27</sup> In the Van As cases, the total laser treatment time was estimated to be as much as 60 seconds at 5–6 W average power; however, fiber size and fluence were not disclosed. Morford and others reported delivering varying average laser power values between 1.33 and 5.03 W delivered by way of a 1.1-mm diameter sapphire optical tip, in contact with the veneer surface.<sup>28</sup> The average treatment time was  $113 \pm 76$  seconds, with a range of 31–290 seconds. Because of a concern for potentially unsafe intrapulpal temperatures, during preliminary trials, the authors used similar laser parameters, which resulted in outcomes similar to those reported previously. In another study, Rechmann and others tested all-ceramic IPS E.max CAD crowns using an erbium laser at 560 mJ/pulse and 10 Hz (5.6 W). Fluence was  $45 \text{ J/cm}^2$  at the ceramic surface, which was approximately 5 mm from the tip.<sup>29</sup>

When comparing pulpal temperature rise in extracted human molars, Penn and others demonstrated that none of the tested devices (erbium laser,  $\text{CO}_2$  laser, and the traditional high speed handpiece) caused an increase of more than  $3.56^\circ\text{C}$ , which was well under the generally accepted threshold of  $5.5^\circ\text{C}$ .<sup>30,31,32,33,34,35</sup>

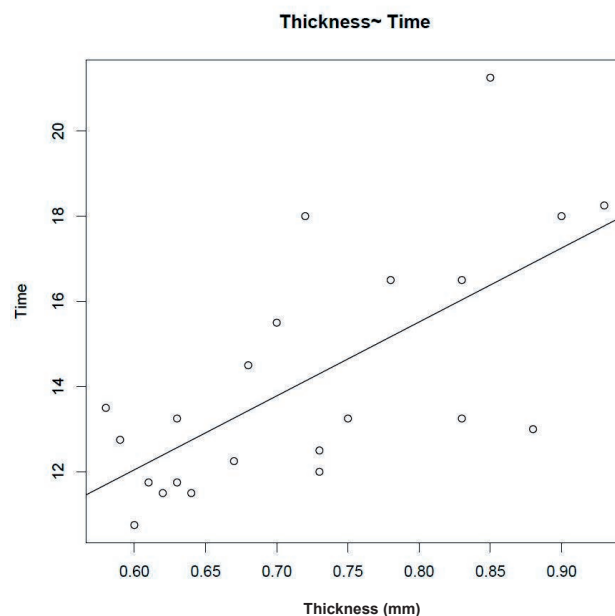


Figure 4. The regression line is  $\text{Time} = 1.637 + 17.348 \times \text{Thickness}$ . This regression line can be interpreted as follows: For every one-unit increase (1.0 mm) in Thickness of the veneer, the value of Time will increase on average by 17.348 seconds. The  $p$ -value of the model is reported as  $0.00058 \leq 0.05$ , which is significant.

A study presented by Rizoïu and others, and a more recent presentation by this author has shown a decrease in intrapulpal temperature during dental cavity preparation using an Er,Cr:YSGG laser device.<sup>36</sup> More recently, Zach and Cohen's work<sup>37</sup> has come under question, as others have suggested that the "probably tolerable" thermal limit may actually be significantly higher than  $5.5^\circ\text{C}$ .

The results presented in this report, including the thermal data, suggest that laser debonding of laminate veneers can be successful at higher power densities, as the increase in pulpal temperature is minimal with sufficient water spray. In fact, it was shown that, despite what might otherwise be expected, higher laser energy did not cause a significant rise in pulpal temperature because of the short duration of laser exposure. Of course, thickness of laminate veneer, material type or even luting agent may result in different irradiation times. The importance of copious amounts of water spray during laser ablation cannot be overstated. As far back as 2007, Kang and others demonstrated that charring and cracks were the result of dry laser ablation.<sup>38</sup> Craters created in human enamel with the addition of water spray were relatively clean and without thermal damage. For this reason, the maximum amount of aerosolized water spray was deemed necessary (100% = 36 ml/minute).<sup>39</sup>



The inert nature of the ceramic material used in this study suggests that fracturing is likely due to the lower flexural strength as compared with zirconia, for instance (200-220 MPa vs 1,000 MPa).<sup>28,29,30</sup> In the case of porcelain, it has been shown that there can be a minute amount of water absorption, intraorally. If veneers are made of porcelain, erbium laser energy does not pass through freely. Instead, the light energy is absorbed by the water contained within the porcelain, causing fracture of the material.<sup>27,31,32</sup>

## CONCLUSION

A regression model between time and thickness ( $p < 0.0001$ ) proved to be significant. However, the same cannot be said when the same modeling was tested between temperature and thickness. It can therefore be concluded that as the thickness of a veneer increases, more time is necessary to remove a veneer using Er,Cr:YSGG laser energy; however, increasing thickness does not necessarily result in an increase in pulpal temperature.

Within the limitations of this study, it can be concluded that removing CAD Empress laminate veneer restorations using an Er,Cr:YSGG laser is reliable and thermally safe, even at an average of 10 W at 30 Hz. Thermal safety is maximized so long as there is adequate aerosolized water spray.<sup>38</sup> Limitations of this study at the present time are that only one veneer material and one resin luting cement were tested. Further studies are necessary to compare results for different materials and various luting agents.

## Conflict of Interest

The corresponding author is a consultant for Biolase, Inc, the manufacturer of the laser device used in this study. No monetary or support of any kind was received for this study.

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# Lack of Neutralization of 10-MDP Primers by Zirconia May Affect the Degree of Conversion of Dual-cure Resin Cement

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## Clinical Relevance

Use of zirconia primers with a low pH and a high acidic monomer concentration should be employed in combination with dual-cure resin cements that are less sensitive to an acidic environment. Primers with lower 10-MDP concentrations attain better outcomes.

## SUMMARY

**Objective:** To assess the effects of different concentrations of 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) included in experimental ceramic primers on the degree of conversion (DC) and microshear bond strength ( $\mu$ SBS) of a dual-cure resin cement, and on the

acidity neutralization potential of zirconia ( $ZrO_2$ ) in comparison to hydroxyapatite (HAp).

**Methods:** Experimental ceramic primers were formulated using 5 wt%, 10 wt%, 20 wt%, or 40 wt% 10-MDP as an acidic functional monomer and camphorquinone (CQ)/amine or 1-phenyl-1,2-propanedione (PPD) as a photoinitiator system.

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Clearfil Ceramic Primer (Kuraray Dental, Tokyo, Japan) was used as the commercial control. Micro-Raman spectroscopy was used to assess the DC of uncured and light-cured resin cements applied onto primer-treated ZrO<sub>2</sub> surfaces. The  $\mu$ SBS and pH of primers were assayed in a universal testing machine and by a digital pH meter (Tec-3MP; Tecnal, Piracicaba, Brazil), respectively. Statistical analysis was performed by one-way analysis of variance (ANOVA) and Tukey's test ( $p < 0.05$ ).

**Results:** DC was not affected until a concentration of 10% 10-MDP in CQ primer and 5% 10-MDP in PPD primer was reached, when compared with the positive control ( $p > 0.05$ ). Groups 10-MDP 5% in CQ and PPD primers showed the highest  $\mu$ SBS compared with the positive control ( $p > 0.05$ ); however, higher concentrations of 10-MDP induced significant DC and  $\mu$ SBS reduction ( $p < 0.05$ ). HAP neutralized 10-MDP primers, but ZrO<sub>2</sub> provided higher acidity to the primers' pH.

**Conclusion:** 10-MDP monomer should be used in low concentrations in ZrO<sub>2</sub> primers to avoid reduction of the polymerization and bond strength of resin cement.

## INTRODUCTION

Seeking aesthetic dental treatments has increased the demand for metal-free prosthetic restorations. By replacing the metal framework for reinforced dental ceramics, several benefits are acquired beyond aesthetics, such as higher biocompatibility for nonprecious metal frameworks, lower thermal conductivity, higher hardness, and chemical stability.<sup>1,2</sup> Yttria-stabilized tetragonal ZrO<sub>2</sub> polycrystals (Y-TZP) ceramics may be applied as an alternative for the traditional metal framework.<sup>3</sup> Nevertheless, due to its high chemical stability and monolithic crystalline structure, conventional hydrofluoric acid conditioning is less effective on glass ceramics, thereby reducing the bonding ability when used along with dual-cure resin cements.<sup>4</sup>

Different chemical and mechanical surface pretreatments were, thus, recommended in order to improve the bonding of resin cements to ZrO<sub>2</sub> ceramics.<sup>3,5</sup> The use of chemical agents for resin cement luting Y-TZP structures has been shown to improve bonding to ZrO<sub>2</sub>.<sup>6</sup> Therefore, techniques promoting less damage to Y-TZP ceramics,<sup>7</sup> along with producing functionalized surfaces, are desirable. Among these techniques, tribochemical silica coating and subsequent

silanization have already demonstrated efficacy in enhancing the long-term durability of a resin–ZrO<sub>2</sub> bond<sup>8</sup>—even though such a procedure requires further laboratory steps and special equipment. Recently, there has been an increase in investigations and clinical applications of ZrO<sub>2</sub> primers and self-adhesive resin cements based on acidic functional monomers,<sup>5</sup> which improved the shear bond strength to Y-TZP ceramics.<sup>6,9</sup>

The most commonly used acidic functional monomer for ZrO<sub>2</sub> is 10-MDP,<sup>9</sup> and although most studies show that a higher bond strength results when a primer containing 10-MDP is used, this is not the consensus in the literature regarding reactions with a dual-cure resin cement.<sup>9</sup> This may be related to the fact that it is not exactly known what concentration of 10-MDP would provide best results.

Another problem related to the use of acidic functional monomers is that the presence of such monomers may interfere with the polymerization of dental adhesives and cements based on type II photoinitiator systems (such as camphoroquinone) with tertiary amine (CQ/amine), which might jeopardize the DC.<sup>10</sup> In the case of self-etch adhesives, dissolution of HAP from enamel/dentin and the reaction with the tertiary amine coiniciators in the primer may reduce this phenomenon due to the buffering of the acidic media<sup>11</sup> and binding of the functional monomer with calcium,<sup>12</sup> thereby avoiding the decrease on the DC during polymerization.<sup>10</sup> Such neutralization has been demonstrated with self-etch adhesives<sup>10</sup> as well as with self-adhesive resin cements.<sup>13</sup> Nevertheless, to the extent of our knowledge, no reports are available concerning the role of the reaction between the acidic functional monomer and Y-TZP ceramic on the neutralization trend of acidic pH from ceramic primers, which could potentially interfere with resin cement polymerization and bonding, depending also on the type of photoinitiator and presence of tertiary amine in ZrO<sub>2</sub> primer.

The aim of this study was to assess the effects of different concentrations of 10-MDP included in experimental ceramic primers on the DC and  $\mu$ SBS of a conventional dual-cure resin cement in comparison with a commercial ceramic primer. Additionally, the acidity neutralization potential of ZrO<sub>2</sub> and HAP were surveyed. The hypotheses tested are as follows: 1) the concentration of the acidic functional monomer does not interfere with the DC and adhesion of the resin composite cement applied to the Y-TZP-ceramic, 2) there is no effect of two photoinitiator systems in primers (with or without tertiary amine) on the DC and bond strength of resin cement to ZrO<sub>2</sub>, and 3) ZrO<sub>2</sub> is not able to neutralize the pH of experimental ceramic primers.



## METHODS AND MATERIALS

### Reagents

10-MDP was donated by FGM Company (Joinville, Brazil) and used without further purification. CQ (photoinitiator) and ethyl 4-(dimethylamino) benzoate (EDAB, coinitiator) were donated by Esstech Inc (Essington, Pennsylvania, USA), while type 1 photoinitiator PPD (photoinitiator) was purchased from Sigma Aldrich Chemicals (St. Louis, Missouri, USA).

### Experimental Primers

To formulate experimental ceramic primers, 10-MDP was employed as an acidic functional monomer and included in 5 wt%, 10 wt%, 20 wt%, or 40%, and diluted in 50 vol% ethanol/distilled water. In order to evaluate the influence of functional monomer acidity and photoinitiators, the primers were made light-curable by means of inclusion of CQ/EDAB or PPD. Clearfil Ceramic Primer (Kuraray Dental) was used for the commercial comparison primer (Table 1) and also it was added a negative control when no primer was applied.

### Degree of Conversion

Y-TZP ceramic blocks (Zirconcad, Angelus, Londrina, Brazil) with dimensions of 13.2 x 13.2 x 3.2 mm were obtained and sintered according to the manufacturer's instructions. After that, all blocks were polished for 30 seconds with 600-, 800-, and 1200-grit silicon carbide papers under water irrigation, ultrasonicated for 10 minutes to obtain standardized flat surfaces, air-dried,

and were then randomly assigned in one of the ten groups (n=3; three different ZrO<sub>2</sub> specimens tested in each group). Primer compositions are detailed in Table 1, and no application of ceramic primer was the negative control group. Primers were applied actively for 20 seconds using a microbrush and air-dried for 30 seconds with a strong blast of air. Thereafter, the dual-cure resin cement RelyX ARC (3M ESPE, St. Paul, Minnesota, USA) was used according to the manufacturer's instructions: a thin layer (1- ± 0.2-mm thick) was applied onto each ZrO<sub>2</sub> slab, covered with a Mylar strip, and then directly light-cured for 40 seconds using a Light Emitting Diode (LED) unit DB 685 (1100 mW/cm<sup>2</sup>; Dabi Atlante, Ribeirão Preto, Brazil). Each resin cement specimen was mixed only after the complete Micro-Raman spectroscopy analysis of the previous specimen.

Micro-Raman spectroscopy analysis was used to assess the DC of the resin cement 10 minutes after it was light cured. The Micro-Raman spectrophotometer (Xplora, Horiba Jobin Yvon Inc, Paris, France) was, firstly, calibrated using a standard silicon sample supplied by the manufacturer. A helium-neon laser with 3.2 W of power and a 532 nm wavelength was used, with a 1.5-μm spatial resolution and a 2.5 cm<sup>-1</sup> spectral resolution associated with a 10x magnification lens (Olympus, London, UK), to attain an approximately 60- x 70-μm field area with three accumulations of 10 seconds of acquisition time each. The DC was calculated based on a previous study<sup>14</sup> using the following formula:

$$DC = \left( 1 - \frac{R_{\text{cured}}}{R_{\text{uncured}}} \right) \times 100,$$

Table 1. *Compositions of Zirconia Primers Tested*

Groups	Composition
CQ5	5% 10-MDP, 46.5% ethanol, 46.5% distilled water, 0.5% CQ, 1.5% EDAB
CQ10	10% 10-MDP, 44% ethanol, 44% distilled water, 0.5% CQ, 1.5% EDAB
CQ20	20% 10-MDP, 39% ethanol, 39% distilled water, 0.5% CQ, 1.5% EDAB
CQ40	40% 10-MDP, 29% ethanol, 29% distilled water, 0.5% CQ, 1.5% EDAB
PPD5	5% 10-MDP, 46.5% ethanol, 46.5% distilled water, 2% PPD
PPD10	10% 10-MDP, 44% ethanol, 44% distilled water, 2% PPD
PPD20	20% 10-MDP, 39% ethanol, 39% distilled water, 2% PPD
PPD40	40% 10-MDP, 29% ethanol, 29% distilled water, 2% PPD
Commercial comparison (Clearfil Ceramic Primer)	1-5% 10-MDP, 3-TMSPMA (silane), ethanol
Negative control	No primer

Abbreviations: 3-TMSPMA, 3-(Trimethoxysilyl)propyl methacrylate; 10-MDP, 10-methacryloyloxy-decyl-dihydrogen-phosphate, CQ, camphorquinone; EDAB, ethyl 4-(dimethylamino) benzoate; PPD, phenyl-propanedione.

where R is the ratio between the heights of the 1638  $\text{cm}^{-1}$  and 1609  $\text{cm}^{-1}$  peaks, after baseline correction, of uncured and light-cured material. Three readings were taken from the top surface of each specimen according to a previous study.<sup>14</sup> These readings were averaged to obtain one statistical unit ( $n=3$ ). As three  $\text{ZrO}_2$  specimens were assessed per group, nine spectra total were surveyed in each group.

### Microshear Bond Strength

Y-TZP ceramic blocks (Zirconcad) with dimensions of 13.2 x 13.2 x 3.2 mm were obtained and sintered according to the manufacturer's instructions. They were embedded and fixed in PVC pipes by means of acrylic resin (JET; Artigos Odontologicos Classico Ltda, Campo Limpo Paulista, Brazil). The exposed flat  $\text{ZrO}_2$  surfaces were polished as described above.

The  $\mu\text{SBS}$  specimens were bonded to the  $\text{ZrO}_2$  surfaces using cylindrical translucent moulds (Tygon tubing, TYG-030; Saint-Gobain Performance Plastic, Clearwater, Florida, USA) as previously reported.<sup>6</sup> Six cylinders (0.75 mm diameter x 1 mm height) were bonded in each  $\text{ZrO}_2$  block using the dual-cure resin cement RelyX ARC (3M ESPE) in a similar set-up of DC analysis (10 groups), resulting in 36 cement cylinders per group. The six results from cylinders tested from the same  $\text{ZrO}_2$  block were averaged and used as a statistical unit ( $n=6$ , referring to the six  $\text{ZrO}_2$  blocks per group). Previous to cement application, either experimental primers or the commercial primer (Table 1) were actively applied for 20 seconds followed by a strong 30 second blast of air to evaporate all the solvent and ensure the monomer was left on the surface. In the negative control group, no primer was used before resin cement bonding. Resin cement was applied in the cylinders and light-cured for 40 seconds according to the manufacturer's instructions with an LED unit (DB 685, output wavelength peak at 470 nm, with 1100  $\text{mW}/\text{cm}^2$  irradiance periodically checked by a radiometer). All cylinders were group-cured to avoid overlapping exposure to the curing light. Specimens were analyzed and those with defects were discarded and replaced. Before the bond strength survey, all specimens were stored immersed in distilled water at 37°C for 48 hours in the dark to ensure the chemical cure of the dual-cure cements.

Bonded specimens were mounted in a device for a  $\mu\text{SBS}$  test (Odeme Dental Research, Joaçaba, Brazil) adapted in a universal testing machine (EMIC DL 2000; São José dos Pinhais, Brazil). An orthodontic wire (0.4-mm diameter) was positioned surrounding and in contact with half of the cylinder, and it was connected to the load cell (500 N) of the machine to exert shear

force in an upward direction. Each cylinder was tested individually with a 1 mm/minute crosshead speed up to the point of fracture. Maximum  $\mu\text{SBS}$  was recorded in N and transformed to MPa, with the analysis of each cylinder diameter to obtain the bonded area ( $\text{mm}^2$ ).

After debonding, all  $\text{ZrO}_2$  surfaces were examined with a stereomicroscope (SMZ800; Nikon, Tokyo, Japan) to determine the mode of failure, and which were classified in three types: A – adhesive fracture between ceramic and cement without signs of residual cement of  $\text{ZrO}_2$  surface; C – cohesive failure of the cement, with the full area presenting cement remnants; and M – mixed fracture, with areas depicting adhesive debonding and some residual cement indicating partial cohesive failure.

### Buffering of Primer Solutions

Initially, 1 mL of each primer solution was surveyed ( $n=3$ ) for its pH by using a digital pH meter (Tec-3MP). After the initial acquisition, 10 aliquots of 0.1 g of HAp<sup>15</sup> (Sigma Aldrich) or  $\text{ZrO}_2$  (Dinâmica Química Contemporânea, Diadema, São Paulo, Brazil) powders were added to each solution in order to track the variations in pH. At each aliquot, the primer was mixed for 60 seconds and the pH was re-assessed. Data was used to build a graph of the pH change for each powder. After the tenth aliquot, the final pH of each solution was used as the result for statistical analysis.

### Statistical Analysis

The results of the DC and  $\mu\text{SBS}$  were statistically analyzed by Shapiro-Wilk normality test ( $p>0.05$ ) and, after proving normal data, the data were analyzed by one-way ANOVA and Tukey's test ( $p<0.05$ ), with 93.2% power. The initial and final pH of each primer were statistically analyzed separately by *t*-test (to compare HAp vs  $\text{ZrO}_2$ ), with a 5% significance level.

## RESULTS

The degree of conversion outcomes are depicted in Figure 1. The positive control Clearfil Ceramic Primer (mean 89.0% DC), negative control (mean 89.0% DC), CQ5 (mean 94.8% DC), CQ10 (mean 93.5% DC), CQ20 (mean 81.5% DC), and PPD5 (mean 92.1% DC) treatments induced statistically similar conversions and the highest conversions ( $p>0.05$ ). The  $\text{ZrO}_2$  treatment using PPD40 (44.6% mean DC) presented significantly lower DC values when compared with all groups ( $p<0.05$ ). CQ40 and PPD20 presented intermediate results.

The results of  $\mu\text{SBS}$  (MPa) are shown in Figure 2. Groups with lower 10-MDP concentrations presented

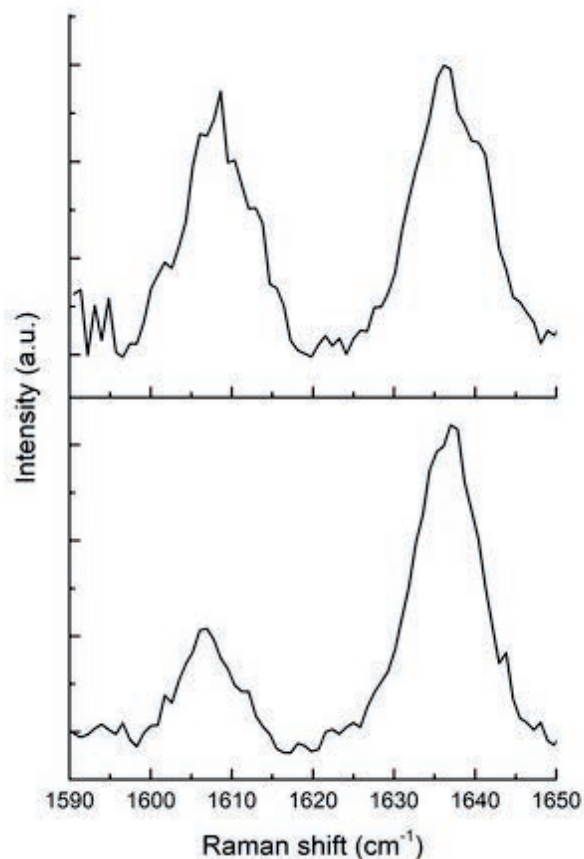


Figure 1. Representative Raman spectra of uncured (bottom) and cured (top) resin cement onto zirconia specimen.

similar and significantly higher  $\mu$ SBS, such as positive control Clearfil Ceramic Primer ( $14.0 \pm 1.2$ ), CQ5 ( $13.4 \pm 2.1$ ), and PPD5 ( $12.8 \pm 1.8$ ) when compared with other groups ( $p < 0.05$ ). As the concentration of acidic functional monomer increased,  $\mu$ SBS dropped significantly, usually with no significant difference when the same concentration of photoinitiator was compared ( $p > 0.05$ ). With exception of PPD40 and CQ40, PPD40 ( $8.1 \pm 0.7$ ) achieved significantly lower  $\mu$ SBS values when compared with CQ40 ( $p < 0.05$ ). The lowest  $\mu$ SBS value was found for the negative control no-primer group ( $0.5 \pm 0.2$ ), which was significantly different in comparison to all the groups ( $p < 0.05$ ); also, in this group, premature failures were observed in most of the specimens.

The results of the pH values are shown in Figure 3. Overall, regardless of the photoinitiator that was employed, the pH of all primers increased with the addition of HAp, while the pH slightly reduced with the addition of  $\text{ZrO}_2$  powder. The final pH was statistically different ( $p < 0.05$ ) between HAp and  $\text{ZrO}_2$  powders for all the primers tested.

## DISCUSSION

The efficiency and durability of adhesion between resin cement and  $\text{ZrO}_2$  ceramics depends upon several factors, such as wettability, microretentions, and the chemical interaction of functional monomers. Indeed,

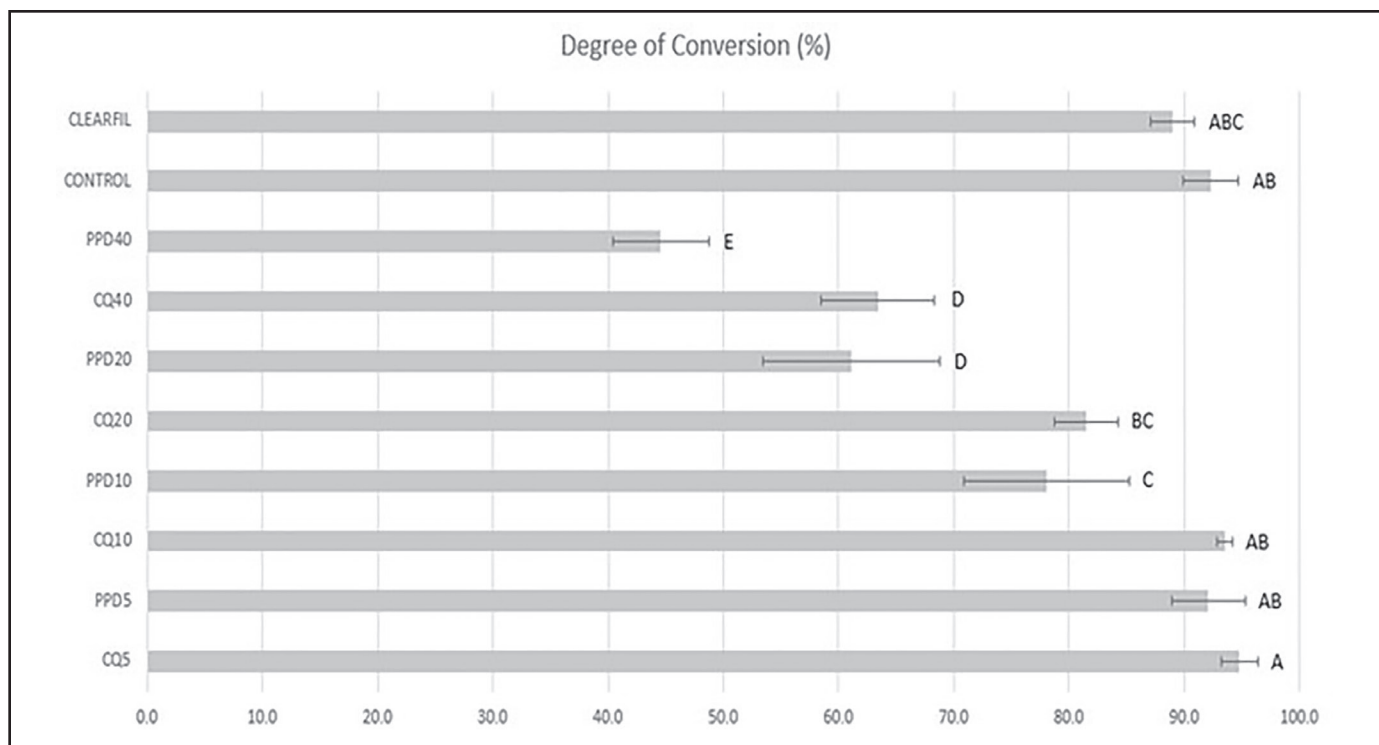


Figure 2. Means and standard deviations of degree of conversion (%) results from resin cement applied onto treated zirconia specimens. Different letters indicate statistical difference ( $p < 0.05$ ).

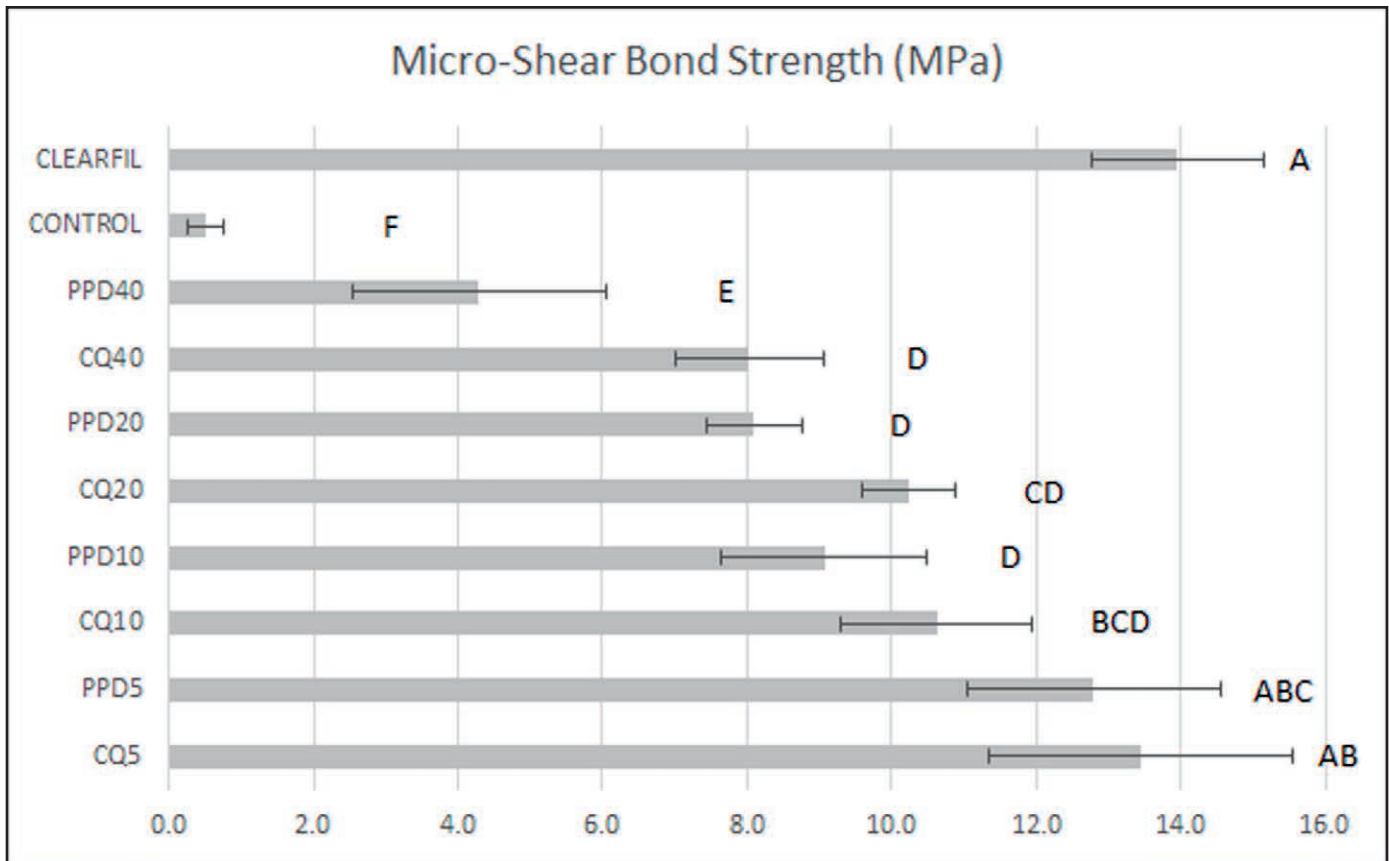


Figure 3. Means and standard deviations of micro-shear bond strength (MPa) outcomes. Different letters indicate statistical difference ( $p < 0.05$ ).

the interaction of an acidic monomer with a Y-TZP surface has proven to be a challenge;<sup>16</sup> besides, the optimal polymerization of resin cement is crucial to obtain high mechanical strength and stability of the ceramic-cement-dentin interface. In the present study, the DC of a commercial dual-cure resin cement applied onto a Y-TZP surface was investigated after the use of different ceramic primers containing a 10-MDP functional monomer and after they were combined with two photoinitiators. The first and second hypotheses tested were rejected because the concentration of the acidic monomer, as well as the two different photoinitiators tested, significantly altered the DC and  $\mu$ SBS of resin cement onto a Y-TZP ceramic, and the presence of high concentrations of acidic monomer together with PPD resulted in lower levels of these properties. However, the third hypothesis is accepted, as  $\text{ZrO}_2$  powder was not able to neutralize the pH of the primers tested.

The conditioning of feldspathic, leucite-reinforced, and lithium disilicate ceramics by hydrofluoric acid and subsequent silanization is a well-established method for luting glass ceramic prosthesis with a resin cement.<sup>3</sup> Silane increases the surface energy of

these ceramics, thereby providing chemical bonding between the siloxane functionality of the molecule and the silica-rich inorganic phases.<sup>6</sup> As Y-TZP is a monolithic ceramic without the presence of glass or silica in composition, conditioning with hydrofluoric acid does not increase surface roughness, and silane does not ameliorate the bond strength.<sup>7</sup> A recent review from Özcan and others<sup>5</sup> concluded that among all these strategies, the optimal durability of resin cement– $\text{ZrO}_2$  bonds is attained by using a 10-MDP monomer containing primers and cements, even after thermocycling.

The DC of resin-based materials containing a CQ/amine photoinitiator system may be affected by the presence of acidic functional monomers.<sup>17</sup> Excited CQ after light exposure turns into a single state that reacts with hydrogen donors, such as tertiary amines, thereby transferring electrons and protons, generating free radicals, and starting polymerization.<sup>18</sup> However, tertiary amines in resin-based materials may also react as Lewis bases are being neutralized by acidic functional monomers, which can impair polymerization.<sup>17</sup> Hanabusa and others<sup>10</sup> demonstrated this reaction with 10-MDP and 4-META functional monomers,



and depicted the negative effects on methacrylate-based polymerization initiated by the CQ/amine photoinitiator system. Nevertheless, in the presence of HAp (from enamel/dentin), its dissolution before light curing buffers acidic monomers and reduces the inhibitory influence on polymerization.<sup>10</sup>

Oguri and others<sup>17</sup> also tested the polymerization of CQ/amine and borate (type I photoinitiator) resins in the presence of an MAC-10 (11-methacryloxy-1,1-undecane dicarboxylic acid) acidic functional monomer. The DC in the CQ/amine system significantly dropped, while borate was not affected by the acidic monomer. In the present investigation, higher concentrations of an acidic functional monomer in experimental primers containing CQ/amine and PPD induced a lower DC to the resin cement; however, lower concentrations of 10-MDP had no influence. PPD's inclusion was proposed to investigate the influence of an amine-free photoinitiator system and its correlation with 10-MDP concentration. As for this photoinitiator, only  $\text{ZrO}_2$  could neutralize acidic monomers. In the PPD groups, more pronounced negative effects were observed (Figures 1 and 2), as the lowest bond strength and conversion among primers was achieved with a PPD40 primer. This suggests that the presence of an amine coinitiator in 10-MDP-containing ceramic primers could result in the additional neutralization of the primers' acidity, resulting in a less negative influence on resin cement polymerization and bonding.

Another factor that significantly decreases the DC of dual-cure resin cements when they are used to lute  $\text{ZrO}_2$  ceramics is the high opacity of  $\text{ZrO}_2$ . This reduces the light transmission and proper irradiance that reaches the underlying cement, thereby diminishing the polymerization, mechanical properties, and durability of resin cement.<sup>19</sup> In this study, light curing was performed directly over the resin cement without  $\text{ZrO}_2$  between the cement and the light unit's tip, disregarding the interference of ceramic opacity. Therefore, the polymerization tested was predominantly light-initiated. The chemical curing of dual-cure resin cements could likely compensate for the negative effect of acidic functional monomers from  $\text{ZrO}_2$  primers, but it was not investigated once the DC was attained 10 minutes after light curing. Future studies need to be done to test this hypothesis.

The reduction of polymerization conversion was concentration-dependent in 10-MDP-containing primers (Figure 1). Regarding Clearfil Ceramic Primer, the low concentration of 10-MDP (according to the manufacturer's MSDS) induces a higher pH and may leave free amine to react with CQ during light curing. Conversely, with experimental primers, the high

concentrations of 10-MDP (40 wt%) decreased the pH (Figure 3) and possibly most functional monomers chemically bonded to  $\text{ZrO}_2$ , but a significant amount was free to react with amine, thereby reducing resin cement's degree of conversion. From a clinical perspective, the viability of using self-etch or universal adhesives with higher concentrations of 10-MDP<sup>11</sup> than ceramic primers should be reconsidered and checked for potential negative effects of dual-cure resin cement polymerization. Concerning 10-MDP-containing adhesives, the study of Llerena-Icochea and others<sup>20</sup> showed no differences among acidic monomer concentrations of 3–15 wt%. In fact, these results corroborate the present outcomes, once even the lowest concentrations of 10-MDP achieved adequate  $\text{ZrO}_2$  bonding for the resin cement.

Indeed, some investigations demonstrated that a more alkaline pH favors the chemical interaction of 10-MDP with  $\text{ZrO}_2$ ,<sup>21</sup> which contributes to the present findings and which suggest a reduction of 10-MDP concentration for optimal adhesion to  $\text{ZrO}_2$ . However, a further alternative could be the use of other acidic monomers in ceramic primers and universal adhesives. For instance, Chen and others<sup>7</sup> studied PENTA (dipentaerythritol penta-acrylate phosphate) as a replacement for 10-MDP in ceramic primers. They concluded that the bond strength of resin cement to Y-TZP may be improved by using this alternative monomer, and that the increase in monomer concentration only enhances bond affinity, but not necessarily the efficacy. Therefore, PENTA may be applied in a very low concentration, reducing its potential inhibitory effects on resin cement's polymerization, which was not truly investigated in that study.

The attempt to improve bond strength by increasing the concentration of the acidic monomer in ceramic primers seems to be inefficient. A recent investigation<sup>22</sup> also showed, by nuclear magnetic resonance experiments, that high concentrations of 10-MDP in  $\text{ZrO}_2$  primers increased intermolecular hydrogen bonding between monomers, thereby jeopardizing the chemical interaction formation of hydrogen and ionic bonds between 10-MDP and  $\text{ZrO}_2$  ceramic.<sup>21</sup> As demonstrated herein, a high concentration of 10-MDP impairs the bond strength of resin cement to Y-TZP. The reduction in bond strength was positively correlated with an increase in 10-MDP concentration and the reduction of the ceramic primers' pH. This occurs due to the lack of neutralization of the primers' pH by the  $\text{ZrO}_2$  surface. Rather, in the pH tracking experiment,  $\text{ZrO}_2$  powder actually maintained or increased the acidity of primers (Figure 4). HAp, on the other hand,

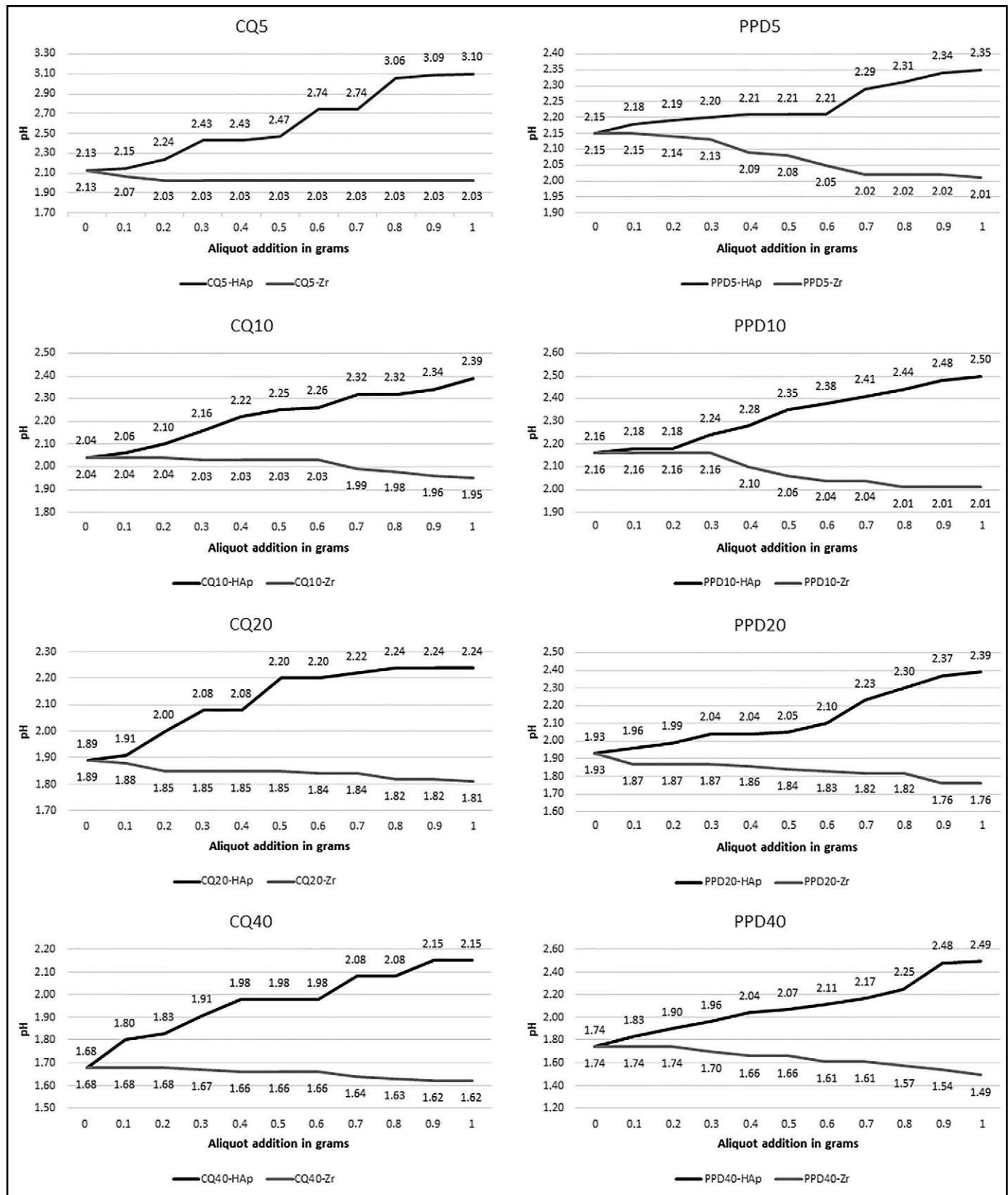


Figure 4. Spreading of pH variation of 10-MDP-containing primers after the addition of 0.1 g aliquots of hydroxyapatite (HAp; black lines) or zirconia (gray lines). Experiments were performed in triplicate and the graphs were formed by the mean values. Final pH levels were significantly different between HAp and zirconia ( $p < 0.05$ ) for all primers.

was able to effectively promote neutralization trend in primers. Indeed, the present outcomes suggest designing of  $\text{ZrO}_2$  primers towards less concentrated solutions with acidic functional monomers possessing optimal chemical interaction with  $\text{ZrO}_2$ , to allow for a reduction of their concentration.

## CONCLUSIONS

The ideal concentration of functional monomer 10-MDP to be used in ceramic primers should be up to 5 wt%, independently if combined with CQ/EDBA or PPD, to avoid the reduction of polymerization and bond strength of resin cement to  $\text{ZrO}_2$ .  $\text{ZrO}_2$  ceramic does not possess the neutralization capacity for very acidic ceramic primers generated by functional monomers, especially in high concentrations.

## Acknowledgments

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## Conflict of Interest

The authors have no financial interest in any of the companies or products mentioned in this article.

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## Departments

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### Errata:

Operative Dentistry apologizes for the layout and clarity errors in the manuscripts, “Time-dependent Microhardness Gradients of Self-adhesive Resin Cements Under Dual- and Self-curing Modes”, and “Effectiveness of Whitening Strips Use Compared With Supervised Dental Bleaching: A Systematic Review and Meta-analysis” published as online only articles attached to volume 45 issue 61.

Both articles were published without the final proof corrections being made. In both cases, the corrections to be made were only for style and readability and do not impact the science represented in the article.

The articles have been corrected and reposted to the website.

Our apologies to the authors and our readers for publishing content that was formatted below our standards.

The two articles affected are:

GRV da Rosa, BM Maran, VL Schmitt, AD Loguercio, A Reis, FS Naufel; Effectiveness of Whitening Strips Use Compared With Supervised Dental Bleaching: A Systematic Review and Meta-analysis. *Oper Dent* 1 November 2020; 45 (6): E289–E307. doi: <https://doi.org/10.2341/19-160-L>

T Geng, Y Pan, Z Liu, C Yuan, P Wang, X Meng; Time-dependent Microhardness Gradients of Self-adhesive Resin Cements Under Dual- and Self-curing Modes. *Oper Dent* 1 November 2020; 45 (6): E280–E288. doi: <https://doi.org/10.2341/19-006-L>

On occasion we receive manuscripts that we would like to publish, but do not have the page room to include in the print journal. For the full article, please go to <https://meridian.allenpress.com/operative-dentistry> or enter the provided address into your address bar.

### **Performance of a Universal Bonding System Associated With 2% Digluconate Chlorhexidine in Carious and Eroded Dentin**

JC Jacomine • M Giacomini • MA Agulhari • G Zabeu • H Honório • L Wang

Carious and eroded dentin represent clinical challenges. The use of a universal bonding system, in a self-etching mode, associated with chlorhexidine (CHX) seems to not improve its longevity. This may be attributed to the competition for calcium between the bonding agent functional monomer and CHX.

<http://doi.org/10.2341/19-123-L>

### **The Potential of a Bioactive, Pre-reacted, Glass-Ionomer Filler Resin Composite to Inhibit the Demineralization of Enamel in Vitro**

IF Leão • N Araújo • CK Scotti • RFL Mondelli • MM de Amoêdo Campos Velo • JFS Bombonatti

A prereacted, glass-ionomer filler fluoride-containing resin composite had lower remineralization potential than glass-ionomer cements but was able to inhibit enamel demineralization; thus, it may be an option for restoring dental surfaces for patients at high risk of caries.

<http://doi.org/10.2341/19-123-L>

### **Airborne-particle Abrasion and Dentin Bonding: Systematic Review and Meta-analysis**

VP Lima • KDA Soares • VS Caldeira • AL Faria-e-Silva • BAC Loomans • RR Moraes

The literature reviewed suggests that airborne particle abrasion has no negative effects on the bond strength of resin-based materials to dentin and that a positive influence on dentin bond strength was only achieved in specific air-abrasion conditions.

<https://doi.org/10.2341/19-216-L>

### **Clinical Performance of Filled/Nanofilled Versus Nonfilled Adhesive Systems in Noncarious Cervical Lesions: A Systematic Review and Meta-analysis**

JL de Geus • BM Maran • KA Cabral • A Davila-Sanchez • C Tardem  
MO Barceleiro • SD Heintze • A Reis • AD Loguercio

The use of filled adhesive systems does not influence the clinical performance of the adhesive restoration in noncarious cervical lesions.

<https://doi.org/10.2341/19-252-L>

### **Three-year Clinical Performance of Two Giomer Restorative Materials in Class-I Restorations**

F Ozer • O Irmak • O Yakymiv • A Mohammed • R Pande • N Saleh • M Blatz

The clinical performance of both conventional and flowable giomer restorative materials was particularly good in Class I restorations after three years of service.

<http://doi.org/10.2341/17-353-C>

# Performance of a Universal Bonding System Associated With 2% Digluconate Chlorhexidine in Carious and Eroded Dentin

JC Jacomine • M Giacomini • MA Agulhari • G Zabeu • H Honório • L Wang

## Clinical Relevance

Carious and eroded dentin represent clinical challenges. The use of a universal bonding system, in a self-etching mode, associated with chlorhexidine (CHX) seems to not improve its longevity. This may be attributed to the competition for calcium between the bonding agent functional monomer and CHX.

## SUMMARY

**Objectives:** The purpose of this study was to explore the interaction between two calcium-dependent agents: 10-methacryloyloxydecyl-dihydrogen phosphate (MDP) and 2% chlorhexidine (CHX) digluconate in association with a self-etching universal bonding system.

**Methods and Materials:** Flat dentin surfaces were obtained from 120 specimens (n=20/

group) prepared from extracted sound human third molars and randomly divided into three groups according to the dentin substrate: sound ([S] control), artificial carious ([C] 6 h/demineralization + 18 h/remineralization for 5 days + 48 h/remineralization), and artificial eroded ([E] 3 cycles for 5 min/day for 5 days using orange juice). Before bonding procedures, one-half of the specimens from each group were pretreated with deionized water (W) and the other half with

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2% chlorhexidine (CHX), forming six groups: S-W, S-CHX, C-W, C-CHX, E-W, and E-CHX. All specimens were restored with Adper Single Bond Universal (self-etching mode) and two increments of composite resin (Filtek Z-250), following the manufacturer's instructions. Slices (0.8 mm) were obtained from the specimens and subjected to scanning electron microscopy (SEM) analysis and sticks (0.64 mm<sup>2</sup>) were obtained and subjected to a microtensile bond strength test ( $\mu$ TBS) in a universal testing machine (0.5 mm/min) after 24 hours and 6 months of storage. Failure modes were classified using optical microscopy (40 $\times$ ). Data was statistically analyzed by three-way ANOVA and Tukey tests ( $p < 0.05$ ).

**Results:** Substrate type was a statistically significant factor ( $p < 0.0001$ ), whereas neither the pretreatment ( $p = 0.189$ ) nor time ( $p = 0.337$ ) were significant. No interaction considering all the factors was significant ( $p = 0.453$ ). **Conclusions:** Carious and eroded dentin substrates negatively interfered with the bonding potential of an MDP-based universal bonding system, regardless of the use of CHX. Likely, the reduction of available calcium impaired the effectiveness of the bonding system.

## INTRODUCTION

The complexity of the dentin substrate has resulted in a large number of adhesive related studies.<sup>1-4</sup> This is due to the characteristics of a dynamic biological substrate that results in limitations to creating a long-term stable interaction between resin monomers and dentin to achieve an ideal adhesive interface.<sup>5</sup> In clinical practice, dentin substrates are frequently altered by caries and erosion, which creates a vulnerable substrate for adhesion.<sup>1,3,4,6</sup>

Dental caries is still the most common and challenging clinical dental disease.<sup>7</sup> Robust scientific studies and technological advances have pushed for more conservative interventional procedures such as the selective removal of the carious tissue.<sup>8,9</sup> Simultaneously, lifestyle changes have caused an aged, clinical appearance of teeth due to premature enamel loss. This scenario corresponds to an increase in the prevalence of noncarious lesions, such as erosion.<sup>10-12</sup> In this case, the dental surface is demineralized by acids (extrinsic or intrinsic), without bacterial involvement, which provokes the softening of the enamel, followed by its mechanical removal with progressive tissue loss, especially by toothbrushing.<sup>10-12</sup> The erosion process

involves two phases. The first phase involves softening of the surface with mineral loss but without structural loss. The second phase involves the removal of this softened tissue, which occurs due to continuing erosion and/or mechanical processes.

Due to changes in these substrates, such as in mineral composition and the biological dynamic involving the organic matrix, the residual demineralized tissue can cause difficulties with bonding.<sup>4,6,13</sup> In this scenario, the bonding substrate can impair the long-term success of adhesion, regardless of the type of adhesive system.<sup>6,13</sup>

To restore these altered substrates, different types of dentin bonding systems are available and can be indicated specifically for each clinical situation. The most recently marketed category of bonding system was classified as universal adhesives.<sup>14,15</sup> Universal systems allow professionals to choose between two bonding methodologies: etch-and-rinse or self-etch. The ease of use of universal bonding systems combined with a promising bonding performance is clinically attractive.<sup>15,16</sup> Since one of the main ingredients in universal bonding systems is an acidic functional monomer, 10-methacryloyloxydecyl-dihydrogen phosphate (MDP), these systems enable the advantages of a chemically stable interaction with dentin and a reduction of sensitivity.<sup>14,15</sup> The deposition of stable MDP-calcium salts with the remaining mineral content seems to be responsible for this stability, due to the formation of a nanolayer that is less susceptible to hydrolysis.<sup>17</sup> Studies have shown increased bond strengths to dentin, attributed to this stable chemical salt formation.<sup>14,16</sup> Oliveira and others<sup>3</sup> demonstrated this optimized bonding performance using sclerotic dentin, which is particularly calcium-enriched.

Enzymatic activity also plays a relevant role in bond degradation and longevity.<sup>18-23</sup> The host matrix metalloproteinases (MMPs) can be activated with a low pH, such as found in carious or erosion processes. In the bonding procedures, MMPs are activated with mineral loss and the consequent exposure and nonprotection of the collagen fibrils due to incomplete monomer infiltration.<sup>24-28</sup> This fact may explain the progressive degradation of the hybrid layer over time.<sup>25,26</sup> As the action of MMPs depends on zinc and calcium, strategies that deprive these ions are being evaluated, such as the application of chlorhexidine (CHX) which presents a satisfactory antiproteolytic potential even at low concentrations.<sup>27,29-31</sup> Often, CHX has been used as a cleaning antimicrobial agent.<sup>27</sup> Its mechanism of action occurs by calcium chelation through the addition of sodium chloride that reverses or prevents the action of MMPs, especially MMP-2 and -9, both of which are present in the dentin substrate.<sup>31</sup> Among the types of

CHX, an aqueous solution of 2% CHX digluconate is the most accessible due to its low cost and can be used in the adhesive process after etching but before the adhesive application.<sup>1,24,28,30,32-34</sup> This methodology presents a high substantivity of the CHX in the organic matrix.<sup>26</sup> Some studies have shown that CHX provides a temporary effect, with 18-month substantivity.<sup>35,36</sup>

The combination of MDP and CHX could improve adhesion and increase the longevity of restorative treatments by minimizing the effects of degradation. However, current studies have demonstrated a possible interaction between these two components by observing precipitate formation near the adhesive interface, which may result negatively impact the interaction between monomers and dentin.<sup>2,28,34,37</sup> Therefore, considering the clinical challenges and possible interaction between these two beneficial strategies (MDP and CHX), more studies are needed to clarify their performance when used in combination on an altered dentin substrate.

The aim of this study was to investigate the microtensile bond strength of an MDP-containing universal bonding system used in self-etching mode combined with CHX in different clinical situations: sound, carious, and eroded dentin substrates. The null hypotheses tested were (1) there is no difference in bond strength to normal, carious, and eroded dentin substrate; (2) there is no difference in bond strength between pretreatment with deionized water or CHX; and (3) there is no difference in bond strength over 6 months, regardless of the substrate and pretreatment.

## METHODS AND MATERIALS

### Experimental Design

This laboratory study involved the analysis of three factors: substrate condition (three levels – sound [S, control], artificial carious [C], and artificial eroded [E] dentin), dentin pretreatment (two levels – deionized water [W] and CHX digluconate) and storage time (two levels – 24 hours and 6 months). The main response variable was the bond strength measured using a microtensile bond strength test. The failure mode was assessed using optical microscopy (40×), while SEM was used for additional qualitative analyses.

The sample size (120 specimens;  $n=20/\text{group}$ ) was determined based on a pilot study. The current authors considered six groups, a power of 80%, and an  $\alpha = 5\%$ . Based on the effect size of 10 and an estimated standard deviation of 8, the sample size was calculated as 18. Also, 10% more was added, as losses could occur, resulting in  $n=20$ .

### Specimen Preparation

The specimens with a flat dentin surface were randomized using Excel software. Extracted sound (caries and restoration-free) human third molars, obtained under the approval of the Local Institutional Ethics Committee (protocol CAAE 79124217.0.0000), were stored in a 0.1% salt solution of thymol at nearly 8°C until use. The occlusal enamel and roots were removed (perpendicular to the long axis of the tooth) using a water-cooled diamond disc (Isomet, Buehler Ltd. Lake Bluff, IL, USA). A 600-grit SiC abrasive paper was used under running water for 30 seconds (Politriz APL-4 AROTEC, Cotia, São Paulo, Brazil) to standardize the smear layer. The specimens were divided according to dentin substrate (S, C or E) and pretreatment (W or CHX) to constitute the groups: S-W, S-CHX, C-W, C-CHX, E-W, and E-CHX.

The control group (S) was maintained in artificial saliva (1.5 mM  $\text{Ca}[\text{NO}_3]_2 \cdot 4\text{H}_2\text{O}$ , 0.9 mM  $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ , 150 mM KCL, 0.1 mol/L Tris, 0.05 ppm F, pH 7.0). The C lesions were produced by 6-hour cycles in demineralization solution (1.5 mM  $\text{CaCl}_2$ , 0.9 mM  $\text{KH}_2\text{PO}_4$ , 50.0 mM lactic acid buffer, pH 5.0),<sup>38</sup> followed by 18 hours in remineralization solution (1.5 mM  $\text{CaCl}_2$ , 0.9 mM  $\text{KH}_2\text{PO}_4$ , 130.0 mM KCl, 20 mM HEPES buffer, 5.0 mM  $\text{NaN}_3$ , pH 7.0), which simulated the clinical situation of the carious process with an imbalance of demineralization and remineralization.<sup>39</sup> Each specimen was immersed in 30 mL of solution for each cycle. The solutions were renewed each day for 5 days, followed by 48 hours of incubation in a remineralizing solution, which was also renewed daily, for a total of 7 days of immersion. To create the E lesions, the specimens were immersed in industrialized orange juice at a pH of 4.0 (Suco Del Valle do Brasil, Leão Alimentos e Bebidas Ltda, Linhares, Espírito Santo, Brazil) that was composed of reconstituted orange juice, dietary fiber (acacia gum), vitamin C, and natural aroma. The specimens were immersed for 5 minutes, 3 times a day, for 5 days and stored in artificial saliva at all other times. Orange juice was selected for this step since it is a popular beverage in the population and is easy to use.<sup>2</sup> Both altered substrates were assessed by transverse microradiography after the challenge was completed to validate the formation of caries and erosion in dentin. In the C specimens (Figure 1), a thin demineralized subsurface was found with preservation of the outer surface, while eroded dentin revealed a superficial loss (Figure 2).

For the restorative treatment, as per the manufacturer's directions, enamel-selective acid-etching with 37% phosphoric acid gel (Dentscare LTDA, Joinville, Santa

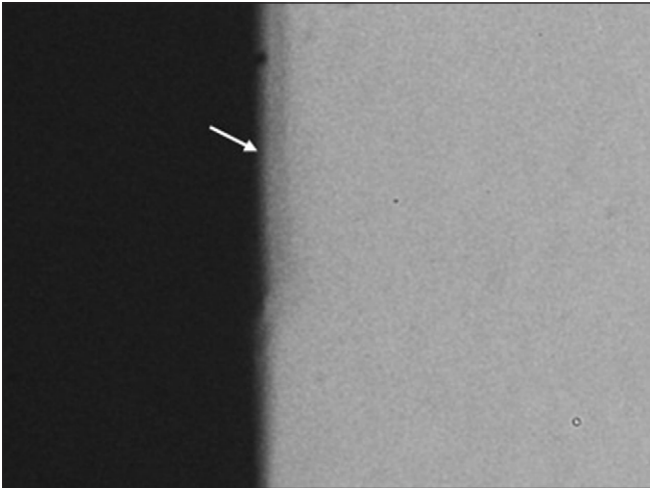


Figure 1. Transverse microradiography representative image of artificially carious dentin substrate. A subsurface lesion is observed.

Catarina, Brazil) was performed for all specimens for 30 seconds, followed by abundant washing with water for 30 seconds, and drying with absorbent paper (wet technique). Dentin was not etched, since the self-etching method was selected for this study. The specimens from the two dentin substrate pretreatment groups received an application of W or CHX aqueous solution at pH 5.8 (Sigma-Aldrich, Saint Louis, MN, USA). After 30 seconds of passive application, the excess was removed with absorbent paper. In sequence, the universal bonding system (Adper Single Bond Universal, 3M ESPE, St Paul, MN, USA; see Table 1) was applied following the self-etching protocol:

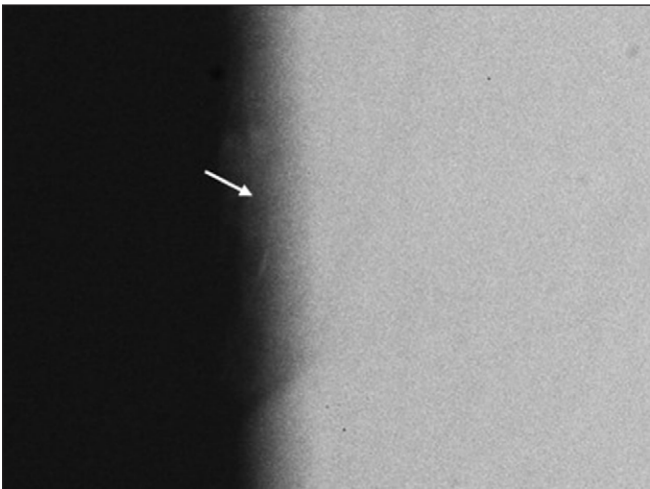


Figure 2. Transverse microradiography representative image of artificially eroded dentin substrate. Different from Figure 1, the external surface is not intact.

Table 1. Composition of Adhesive System – Adper Single Bond Universal	
Adhesive system	Composition
Adper Scotchbond Universal (3M ESPE, St. Paul, MN, EUA)	Methacryloyloxydecyl dihydrogen phosphate, dimethacrylates, 2-Hydroxyethylmethacrylate, methacrylate modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane.

rubbing for 20 seconds and solvent evaporation for 5 seconds. The bonding agent was light cured using a 1000 mW/cm<sup>2</sup> LED unit (Radii-Call, SDI, Bayswater, Victoria, Australia) for 10 seconds. Irradiance was checked after every 5 activations. The composite resin was placed in two increments of 2 mm layers (Filtek Z350 Universal Restorative, 3M ESPE, St Paul, MN, USA), which were both light cured for 20 seconds. The specimens were stored in artificial saliva for 24 hours at 37°C. All procedures were performed according to the manufacturer’s instructions and by the same operator.

Scanning Electron Microscopy

After 24 hours, the specimens were longitudinally sectioned, perpendicular to the bonding interface, using an Isomet 1000 digital saw (Buehler, Lake Bluff, IL, USA) to obtain slices of approximately 0.8 mm thickness. One slice from each subgroup was randomly selected for initial scanning electron microscopy (SEM) (24 hours) and 6 month analysis. The slices were stored in artificial saliva until time of observation. Then, they were immersed in an 18% hydrochloric acid solution for 30 seconds to remove the superficial smear layer, washed for 30 seconds in water, followed by immersion in a 5% sodium hypochlorite solution for 15 minutes to remove all noninfiltrating collagen by the dentin bonding system and subsequently washed for 30 seconds. The specimens were then dried for 12 hours at room temperature and then mounted on aluminum stubs to be sputter coated with palladium gold (DentronVaccum, Desk IV Moorestown, NJ, USA). The adhesive surface of all specimens was analyzed using SEM (JSM – T220A, JOEL LTDA, Tokyo, Japan) at a magnification of 1,500<sup>x</sup>.<sup>40,41</sup>

Microtensile Bond Strength Test

The test followed the current guidance.<sup>42,43</sup> The remaining slices of the restored specimens were again



longitudinally sectioned to prepare resin–dentin sticks of approximately 0.64 mm<sup>2</sup> area (0.8x0.8 mm) verified using a digital caliper (Mitutoyo Americana LTDA, Aurora, IL, USA). The sticks were then fixed to a device (JIG 1 – Plus, Odeme Dental Research, Luzerna, Santa Catarina, Brazil) using cyanoacrylate resin (Super Bonder Power Flex Gel – Loctite, Henckel LTDA, Itapevi, São Paulo, Brazil) and tested in tension with a universal testing machine (Instron 3342, Instron Co., Canton, MA, USA) at a 0.5 mm/minute crosshead speed and with a 500 N load cell. The  $\mu$ TBS was expressed in MPa by dividing the maximum load (kgf) by the specimen cross-sectional area (mm<sup>2</sup>). For this test, the operator was blinded regarding the stick group. During aging, the sticks were stored in weekly-renewed artificial saliva at 37°C.

Each fractured surface was analyzed with a handheld digital microscope (Dino-Liteplus digital microscope, AnMo Electronics Corp, Hsinchu, China) at approximately 40 $\times$  magnification and failure classified as adhesive, cohesive in dentin, cohesive in composite resin, or mixed.

The experimental unit considered was the tooth, so the sticks of each tooth were divided into two groups for the initial and six month evaluations. The average  $\mu$ TBS value for each tooth and time based on all the sticks was calculated and the premature failures were considered as zero for calculating the mean values. For the statistics analysis, the data satisfied the assumptions of a normal distribution and the equality of variance was tested for all the variables using Statistica software (Statsoft, Tulsa, OK, USA). Data was analyzed using three-way ANOVA and multiple comparison tests (Tukey test) with  $\alpha = 0.05$ .

## RESULTS

Bond strength mean values and standard deviations are shown in Table 2. The type of substrate was the only significant factor ( $p < 0.0001$ ). Pretreatment ( $p = 0.189$ ), time ( $p = 0.337$ ), and the interaction between all the factors ( $p = 0.453$ ) were not statistically significant.

Overall, the results suggest that the sound dentin substrate consistently provided the highest bond strength values, which were statistically different from the carious and eroded dentin substrates. The bond strength was compromised related to altered dentin substrates, presenting lower values in artificial carious and eroded conditions, regardless of pretreatment or storage time in this laboratory evaluation. Regarding the demineralized substrates, they presented similar performance, with no statistical differences between them.

Table 2. Mean (MPa) and standard deviation values of micro-tensile bond strength of a universal bonding system to dentin substrates treated or not with chlorhexidine.

Groups	Initial	6 months
S-W	39.27 (10.16) Aa*	39.23 (9.88) Aa*
S-CHX	40.55 (15.75) Aa*	33.39 (13.64) Aa*
C-W	27.67 (13.09) Ba*	26.17 (10.69) Ba*
C-CHX	24.09 (7.21) Ba*	24.44 (7.70) Ba*
E-W	25.73 (12.64) Ba*	26.63 (12.75) Ba*
E-CHX	25.83 (10.71) Ba*	24.87 (8.94) Ba*

N=20. Different capital letters mean statistical significance between substrates (S x C x E) ( $p < 0.05$ ). Equal lowercase letter means no statistical significance between pretreatments (W x CHX) ( $p < 0.05$ ). Asterisk symbol means no statistical significance between time (initial x 6 months) ( $p < 0.05$ ).

When considering time, no significant difference was noted for any condition. The same performance was attributed to CHX, which did not provide any differences with regards to substrate or time.

Figure 3 shows the distribution of the failure modes, revealing that adhesive failure was observed mostly in the initial groups and mixed failure was predominant in the six-month storage groups. Cohesive failures were not absent, although they were present at only a small percentage.

Representative SEM images (1500 $\times$ ) of sound, carious, and eroded dentin are presented, respectively, in Figure 4 and combined with their subgroups (W and CHX, initial and six months). The images of the sound group showed a homogeneous distribution of adhesive agent (self-etching mode) constituting a thin hybrid layer with the presence of some resinous tags, which are notable on the groups treated with CHX. When altered substrates were observed, a discontinuous structure was visible, even for the carious and eroded dentin.

## DISCUSSION

In clinical practice, the most common substrates are morphological and structurally modified, as with carious and eroded dentin, which frequently require restorative treatment.<sup>4,6,8</sup> As most studies often use sound dentin, this study evaluated the influence of a demineralized substrate in different interactions, resembling the actual clinical condition.<sup>14,15,16,25</sup>



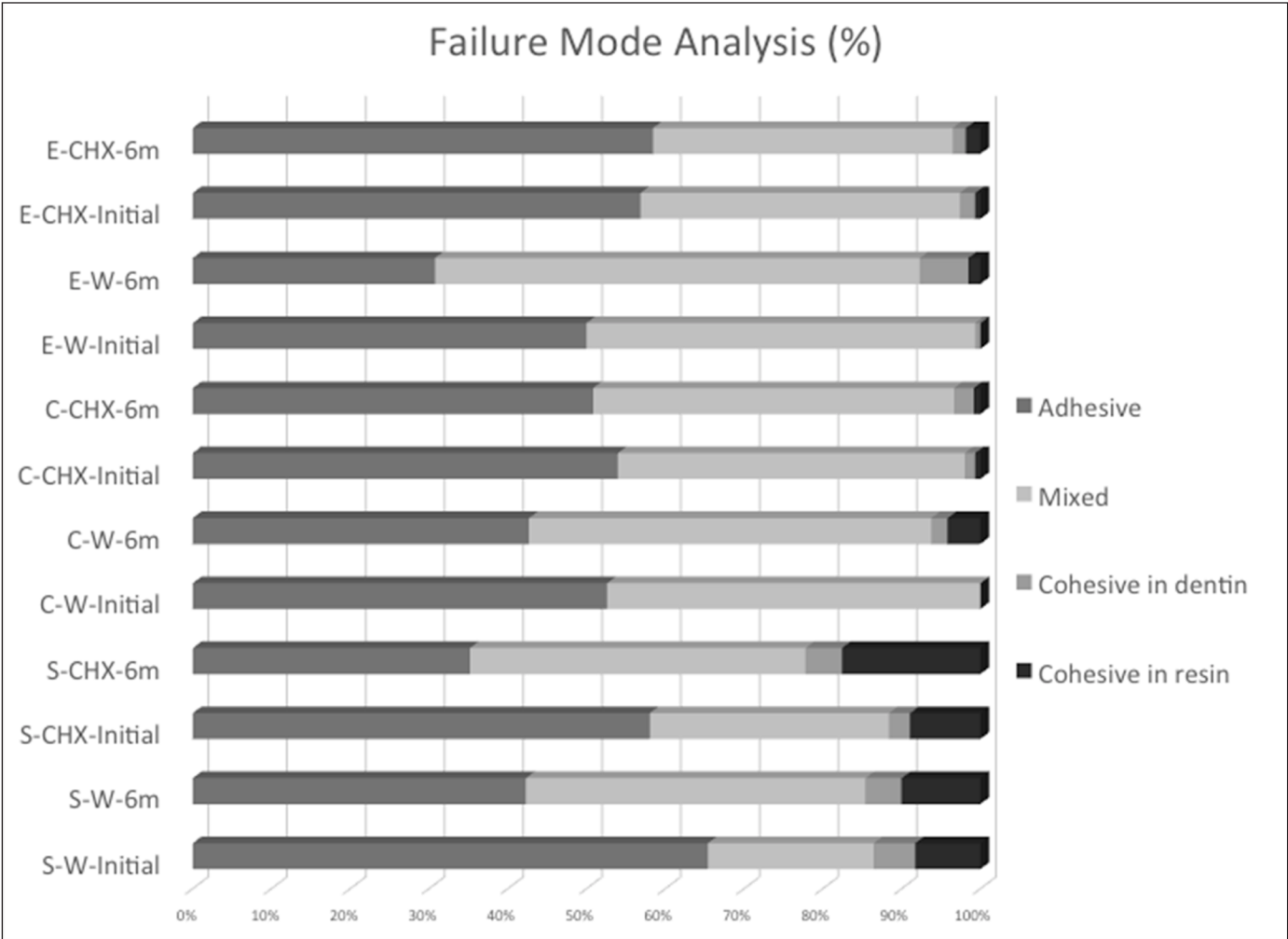


Figure 3. Classification of the failure mode distribution (%) for all substrates, pretreatment, and time evaluation (initial and 6 months). Predominance of adhesive and mixed failure pattern in all groups.

According to the present results, when sound dentin was used, bond strength reached the highest values, regardless of time and pretreatment. Both artificially carious and eroded dentin impaired the bond strength of the self-etching mode of a universal bonding system to dentin (Table 2), which supports rejecting the first null hypothesis tested. Likely, this performance suggests that a lower mineral content negatively affects the chemical interaction of these remaining minerals with MDP-based bonding agents, even immediately after bonding. Therefore, a reduced calcium content in dentin might reduce the formation of stable calcium-based salts and impair the quality of bonding.

This poor bond strength performance for carious lesions is supported by Isolan and others.<sup>6</sup> In this

systematic review, a significantly higher bond strength to sound dentin was found when compared to carious substrates, regardless of the cycling protocols. Also, the lower values found for eroded dentin are in accordance with the literature, which indicates a reduction of adhesion to these substrates.<sup>1,2,32,33</sup>

Controversially, Giacomini and others<sup>2</sup> did not present differences between sound and eroded substrates, with only artificially carious dentin diminishing bond strength to dentin. According to the authors, this difference may be attributed to structural and chemical changes of carious dentin,<sup>44,45</sup> as the denuded collagen fibrils of the organic matrix are degraded.<sup>45</sup> In eroded dentin, the main modification relies on mineral loss, without affecting the organic matrix. For the artificial

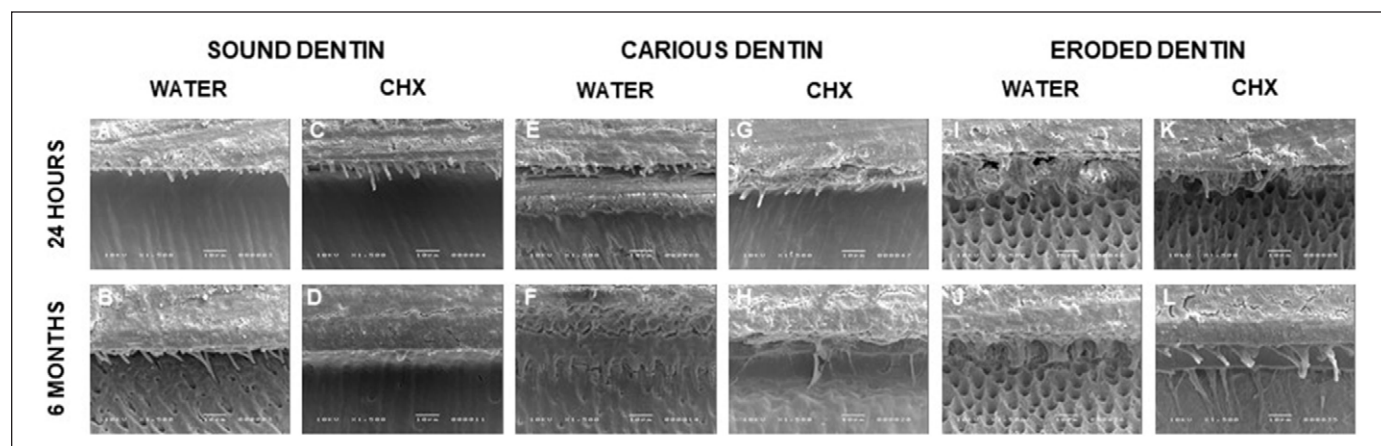


Figure 4. Representative scanning electron microscope images (1500 $\times$ ) of the sound groups (A–D), carious groups (E–H), and eroded groups (I–L), with water or chlorhexidine (CHX) at initial and 6-month evaluation. A specific and particular pattern of the self-etching adhesive system was observed in the sound groups, with eventual formation of short resin tags with a sparse and homogeneous distribution. A pattern similar to that found in the sound group, but with lower homogeneity, was observed in the carious and eroded groups due to the complexity of the demineralized substrates. Additionally, a standard feature of eroded dentin substrate was observed with the exposure of dentin tubules.

carious substrate, a greater effect to the adhesive quality is likely due to the degradation of collagen.<sup>45</sup> Muñoz and others<sup>16</sup> did not observe any difference between application modes (etch-and-rinse or self-etch) in the use of Adper Single Bond, although it should be noted that those authors used sound dentin. Based on the present study, this substrate overestimates the bonding performance and is probably not realistic enough to simulate clinical conditions.

In the Giacomini and others<sup>2</sup> study, the etch-and-rinse strategy used on artificial carious and eroded substrates exacerbated the lack of mineral content. This scenario may impair the ability of the acidic functional monomer MDP to bond to dentin, with a reduced formation of MDP-calcium salt.

The 10-MDP monomer is often present in the composition of universal bonding systems and allows for chemical bonding to the dental structure by a calcium-dependent mechanism, forming a stable nanolayer with various MDP-calcium salts in the adhesive interface.<sup>46,47</sup> Substrates with a lower calcium ion concentration associated with MDP-based bonding systems could result in greater adhesion, especially on modified substrates if they undergo further demineralization by etching during the adhesive process. Therefore, the self-etching mode could allow for more effective interaction of MDP with the calcium in the substrate due to its greater availability, which would be less harmful for adhesion. The same rationale could be applied to any other phosphate-based functional monomer.

In terms of pretreatment with CHX at the initial and six-month evaluations, no statistical difference was observed, regardless of substrate type and time. Therefore, the second null hypothesis was accepted. This association was anticipated, as CHX is robustly supported as an antiproteolytic agent.<sup>1,28</sup> However, as no influence was observed, it is supposed that the available calcium concentration, even in demineralized dentin, was sufficient to allow complete action for both agents (MDP and CHX) when the self-etching mode was employed, as no adverse additional demineralization was provoked by phosphoric acid.<sup>31,46,47</sup> In Giacomini and others<sup>2</sup> the difference between treatments (W and CHX) on all substrates (sound, carious and eroded) when using the etch-and-rinse mode would support this observation. Over time, this perspective may change, as substantivity of CHX is approximately 18 months, according to other studies.<sup>35,36</sup> When considering the time of evaluation, no statistical difference was observed among the initial and six-month groups; therefore, the third null hypothesis was not rejected.

Failure mode analysis (Figure 3) indicated a predominance of adhesive and mixed failure patterns, which validate the bond strength test and was compatible with the current literature. These analyses guarantee that the interface was being tested while avoiding a material cohesive test. The increase in the percentage of cohesive fractures in the sound groups confirmed the fact that the adhesive resistance in the sound substrate was higher than the observed bond

strength for the carious and eroded demineralized substrates. The representative SEM image analyses are shown in Figure 4, which supports the quantitative data and is in accordance with the literature.<sup>48</sup> The overall performance of the bonding agent shows a classical particular pattern of self-etching adhesive systems. The hybrid layer was not distinct in the SEM images and the eventual formation of short resin tags of sparsely and homogeneous distribution was detected.

The characteristics presented in the images also support the stable values of bond strength detected for the different groups in the time comparison, regardless of the pretreatment used. However, poorly homogeneous images are observed in the demineralized groups, which suggests a greater complexity of the substrates when affected by caries or erosion. For the eroded group, it is possible to note the characteristic pattern of eroded substrates with great exposure of dentinal tubules throughout the dentin surface.<sup>4</sup> Possible fractures can be seen in areas corresponding to the composite resin, especially in the six-month sound groups, demonstrating a probable degradation of the resin, which may correspond to the increase of cohesive fractures in resin over time.

Therefore, the supposed competition in this study between MDP and CHX for the calcium ions present in the substrate would not cause an interference in bonding effectiveness when testing initially and at six months in sound, carious, and eroded substrates with an MDP-based bonding system. Within the limitations of this study, this strategy is feasible and reliable for clinical use, even though demineralized substrates always impair the bonding potential.

## CONCLUSION

Cariou and eroded dentin substrates negatively interfered with the bond strength of an MDP-based universal bonding system when using the self-etch mode, regardless of its use with CHX.

## Conflict of Interest

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

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# The Potential of a Bioactive, Pre-reacted, Glass-Ionomer Filler Resin Composite to Inhibit the Demineralization of Enamel *in Vitro*

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## Clinical Relevance

A prereacted, glass-ionomer filler fluoride-containing resin composite had lower remineralization potential than glass-ionomer cements but was able to inhibit enamel demineralization; thus, it may be an option for restoring dental surfaces for patients at high risk of caries.

## SUMMARY

Evidence is lacking on the use of surface prereacted glass-ionomer filler resin composites to inhibit demineralization and that simulate real clinical conditions. The present laboratory study evaluated the potential of such composites to prevent demineralization and quantified fluoride (F) and other ions released from restorative materials after a dynamic pH-cycling regimen applied to the tooth

material interface *in vitro*. The pH-cycling regimen was assessed by measuring surface hardness (SH) along with energy dispersive X-ray spectroscopy (EDX).

**Methods and Materials:** Ninety blocks of bovine enamel were subjected to composition analysis with EDX, and were further categorized based on SH. The blocks were randomly divided into 6 treatment groups (n=15 each): F IX (Fuji IX

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Extra; GC Corporation); IZ (Ion Z, FGM); F II (Fuji II LC, GC Corporation); B II (Beautifil II, Shofu); F250 (Filtek Z250 XT, 3M ESPE); and NT (control, no treatment). The blocks were subjected to a dynamic pH-cycling regimen at 37°C for 7 days concurrently with daily alternations of immersion in demineralizing/remineralizing solutions. EDX was conducted and a final SH was determined at standard distances from the restorative materials (150, 300, and 400  $\mu\text{m}$ ).

**Results:** The EDX findings revealed a significant increase in F concentration and a decrease in  $\text{Ca}^{2+}$  in the enamel blocks of group B II after the pH-cycling regimen ( $p < 0.05$ ). SH values for groups F IX, IZ, and F II were greater than those for groups B II, F250, and NT at all distances from the materials.

**Conclusions:** The results suggest that each of 3 restorative materials, F IX, IZ, and F II, partially inhibited enamel demineralization under a dynamic pH-cycling regimen.

## INTRODUCTION

Owing to their bonding capacity and good physical properties, composite resins have become the most commonly used restorative materials in clinical practice.<sup>1,2</sup> Acid conditioning of enamel as proposed by Buonocore<sup>3</sup> and new adhesive systems have significantly improved the long-term stability of composite resin restorations, mainly of those restricted to the enamel or with enamel-located margins. Although the adhesion of restorative materials to the enamel results in a stable, disease-resistant, and long-lived restoration, the lack of marginal integrity and sealing increases the risk of caries at the margin of the restoration. The main challenges to achieving a long-lived marginal seal include the existence of extensive cavities, the clinician's knowledge of adhesive-system techniques and composition, the type of enamel substrate (ie, sound or carious), the presence of fluorosis or anomalies, and whether the tooth is primary or permanent.<sup>4</sup> Although these variables are known to not affect marginal sealing, it is essential to develop new restorative materials that can boost the long-term performance of restorations.

Composite resins are used for restoration, but several clinical studies have shown higher failure rates for resin composites than for amalgam restorations,<sup>5,6</sup> mainly attributed to secondary caries that develop adjacent to the filling.<sup>7</sup> Secondary caries are responsible for 60% of all replacement restorations in dental practice;<sup>8</sup> for this reason, a Cochrane systematic review has reinforced

the benefit of amalgam restorations to restore posterior teeth because the incidence of secondary caries is higher with composites than with amalgam or other restorative materials.<sup>9,10</sup> However, dental amalgam contains mercury, and reducing the environmental burden of metals through improved environmental practices is a concern, as highlighted by the Treaty of Minamata.<sup>11</sup> Therefore, the use of amalgam is being gradually reduced in clinical use, focusing rather on alternative materials that also are based on minimally invasive dentistry.<sup>12</sup>

Owing to the limitations of composite materials, the principle of restorative dentistry in recent decades has prompted new technologies that improve restorative materials while aligning aesthetics as well as the function/integrity of the dental structure with the challenges inherent in the oral environment. Conventional glass-ionomer cements have interesting properties such as biocompatibility, fluoride (F) release, modulus of elasticity similar to tooth structure, and the ability to chemically bond to the tooth structure,<sup>13</sup> but poor mechanical properties. To overcome these poor properties, resin-modified glass-ionomer cements were developed. These are considered to be a significant advancement, as they improved the physical properties of the cements and also enabled an ion-exchange–based adhesive surface to form and concomitantly release F, which can inhibit dental caries from forming adjacent to the restoration.<sup>14–16</sup> However, the hydrophilic nature of polyhydroxyethylmethacrylate hydrogels results in increased water uptake and solubility, which negatively influences the mechanical properties and clinical performance of these materials in areas that bear stress, such as the posterior teeth.<sup>17,18</sup>

Based on the F-releasing mechanism of glass-ionomer cements during the acid-base reaction phase, in 1999, Roberts and others synthesized a prereacted glass-ionomer (PRG) filler that could be incorporated with polyalkenoic acids into resinous materials from the complete or partial reaction of ion-leachable glasses.<sup>19</sup> PRG fillers consist of fluoroaluminosilicate glass that forms a water-rich siliceous hydrogel in the presence of water. The result is a stable PRG filler with a trilaminar structure that allows the release and recharge of F via a ligand-exchange mechanism within the prereacted hydrogel.<sup>20,21</sup>

The giomer (glass-ionomer + polymer), a novel group of hybrid composite restorative materials based on surface-PRG (S-PRG) fillers, is a technology of interest because it provides biofunctions to restorative materials. Such hybrids have been used in various dental materials such as composite resins, bonding agents, cements, and resin sealants.<sup>22,23</sup> Previous studies have reported

that materials based on S-PRG fillers have the potential to prevent the demineralization of enamel and dentin, as shown by the use of coatings, solutions, or sealing agents;<sup>23,24</sup> therefore, this new generation of F-releasing materials deserves further investigation. Evidence is lacking on restorations carried out with S-PRG fillers to prevent the development of caries and that simulate real clinical conditions, and a dynamic pH-cycling regimen could help clarify this aspect. Moreover, it is important to evaluate the ions released from this class of restorative material that could act as a mineral reservoir to combat caries developing around restorations; these ions can be detected by quantifying the inorganic components in caries-like lesions adjacent to materials.

We aimed to evaluate the potential of S-PRG fillers and F-releasing restorative materials to inhibit the demineralization of enamel compared with conventional and resin-modified glass-ionomer cements. We quantified the ions released from these materials in areas adjacent to the tooth–restoration interface using the combined analyses of surface hardness (SH) and energy dispersive X-ray spectroscopy (EDX). The null hypotheses tested were: (1) restorations using S-PRG

fillers would not prevent enamel demineralization inhibition; and (2) that there would be no difference in the quantification of chemical elements in the enamel mineral elements around restorative materials submitted for compositional analysis via EDX.

## METHODS AND MATERIALS

### Experimental Design

The current laboratory study involved 1 factor restorative material in 6 levels: NT (no treatment [control group]); conventional glass-ionomer cement F IX (Fuji IX Extra; GC Corporation, Tokyo, Japan); conventional glass-ionomer cement IZ (Ion Z; FGM, Pembroke Pines, FL, USA); resin-modified glass-ionomer cement F II (Fuji II LC; GC Corporation); S-PRG fillers, F-releasing restorative material B II (Beautifil II; Shofu, San Marcos, CA, USA); and composite resin F250 (Filtek Z250 XT; 3M ESPE, St. Paul, MN, USA). The experimental units were enamel blocks obtained from bovine incisors and selected by SH. The response variables were based on the SH and EDX analyses. Table 1 lists the specifications of all materials used.

Table 1: *Materials, Classification, and Composition of Materials Evaluated in this Study*

Materials	Manufacturer	Classification	Composition
Beautifil II (B II)	Shofu (Kyoto, Japan)	Composite resin: fluoride-containing resin composite (bioactive prereacted glass-ionomer filler; giomer system)	Glass particle S-PRG, glass fluoride, aluminum, borosilicate particles, TEGDMA, Bis-GMA, particle size 20–40 nm
Filtek Z250 (F250)	3M ESPE (St. Paul, USA)	Composite resin (negative control)	Bis-GMA, UDMA, Bis-EMA (zirconia/silica), particle size 0.01–3.5µm
Fuji IX Extra (F IX)	GC Corporation (Tokyo, Japan)	Conventional glass ionomer cement (positive control)	Fluoroaluminosilicate glass, potassium persulphate, ascorbic acid
Fuji II LC (F II)	GC Corporation (Tokyo, Japan)	Modified glass ionomer cement (positive control)	Fluoroaluminosilicate glass particles, composite monomers, photo initiators
Ion Z (IZ)	FGM (Joinville, Brazil)	Conventional glass ionomer cement (positive control)	Glass of calcium, aluminum, zinc, fluoride, silicate, polycarboxilic acid, deionized water, titanium dioxide, iron oxide

Abbreviations: Bis-EMA, bisphenol A diglycidyl methacrylate ethoxylated; Bis-GMA, bisphenol A-glycidyl methacrylate; S-PRG, surface prereacted glass; TEGDMA, triethyleneglycoldimethacrylate; UDMA, urethanedimethacrylate.



## Preparation and Selection of Samples from Enamel Blocks

Enamel specimens (4×4×2 mm) were obtained from bovine incisors, which were cut using an ISOMET low-speed saw (Buehler Ltd, Lake Bluff, IL, USA). The blocks were polished sequentially using #600 and #1200 grit sandpaper discs (CarbiMet paper discs; Buehler Ltd). For the final polishing a felt disc with a 1- $\mu$ m diamond suspension (Buehler Ltd) was used at high speed under a weight of 172 g. During each change of grit, as well as at the end of the polishing process, the specimens were ultrasonicated in deionized water for 2 minutes using an ultrasonic device (USC 750; Unique Group, Indaiatuba, São Paulo, Brazil). Baseline Knoop SH (KHN) was determined by making 5 indentations (spaced 100- $\mu$ m apart) using a microhardness tester (Model HMV-2000 OR HMV-2; Shimadzu Corporation, Kiyamachi-Nijo, Kyoto, Japan) under a 25-g load for 10 seconds. Assessments were made under a 25-g load for 10 seconds. To establish the homogeneity of the samples, specimens with an average SH >20% or <350 KHN were excluded.

## Treatment of the Enamel Blocks

The selected enamel blocks were randomized according to baseline SH and randomly divided into 6 groups (n=15 each): NT (no treatment [control]), F IX, F II (positive control), B II (evaluated material), and F250 (negative control). The 4 × 4 mm surface of each of the 90 randomized blocks was divided into 2 regions, delimiting the area where standardized cavities were prepared (3×1.5 mm), with diamond tip N° 1093/1093F (Figure 1A) for posterior restorative treatment.

For restorative procedures, resin composites were inserted incrementally, covered with a polyester strip, pressed with a glass slide to delimit the thickness of the material by digital pressure, and then light-cured with a light-emitting diode—curing device (Dabi Atlante, São Paulo, Brazil) operating at 961 mW/cm<sup>2</sup> for 20 seconds.

Before initiating the pH-cycling regimen, half of each specimen was secured with tape to treat the enamel with the various resin composites, limiting the experimental area. After restorative treatment, the protective tape was removed, and the specimens were stored for 24 hours in a chamber with relative humidity of 100% at 37°C for 24 hours before testing. Specimens were protected using a base, which was then removed by acetone.<sup>25</sup> No cavities were prepared in the blocks used for the control group, and half of each control block was coated with an acid-resistant varnish to protect that surface for subsequent pH cycling (Figure 1B).

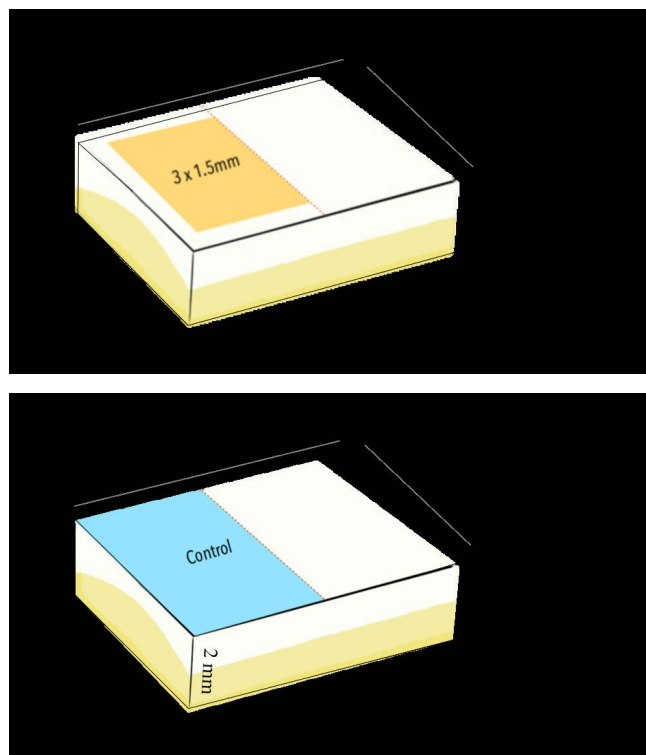


Figure 1. **A:** Delimited area that was used to prepare a standardized cavity (3×1.5 mm). **B:** Control area of the block coated with varnish protecting this region for posterior pH cycling.

## The pH-cycling Regimen

After the treatments, the specimens were subjected to a dynamic pH-cycling regimen for 7 days at 37°C.<sup>26</sup> Each day, the specimens were subjected to alternating immersion in 30 mL of demineralizing solution (2.0 mM Ca(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O, 2.0 mM NaH<sub>2</sub>PO<sub>4</sub>·2H<sub>2</sub>O, 0.077 mM acetate buffer, 0.02 ppm F, pH 4.7) for 6 hours and in a remineralizing solution (1.5 mM Ca(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O, 0.9 mM NaH<sub>2</sub>PO<sub>4</sub>·2H<sub>2</sub>O, 150 mM KCl, 0.1 mM sodium acetate, 0.03 ppm F, pH 7.0) for 18 hours for 5 days.<sup>26</sup> In the last 2 days, the blocks were immersed in the remineralization solution according to Vieira and others.<sup>26</sup> Each specimen was stored in a plastic container to avoid any possible effects of the F released by the materials. When each solution was exchanged, the blocks were washed under deionized water and then dried with blotting paper before being transferred to the next solution.

## Surface Hardness Analysis

The SH of the blocks (n=15) was again determined at the end of the pH-cycling regimen. Five indentations were made at 3 standard distances (150, 300, and 450  $\mu$ m) from the treatment; the indentations were separated by 100  $\mu$ m (Figure 2). For each block, the mean value of

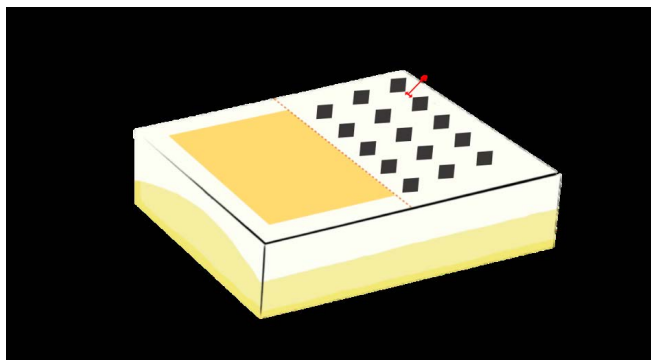


Figure 2. Representative figure of the cavities at three standard distances from the treatment area (150, 300, and 450  $\mu\text{m}$ , with a separation of 100  $\mu\text{m}$  between indentations).

the 5 indentations were calculated and compared with the baseline mean.

### Energy Dispersive X-ray Spectroscopy Analysis

The amount of each enamel component was assessed by EDX, as described by Velo and others.<sup>27</sup> The blocks were mounted onto aluminum stubs with acrylic resin (Palavit M, Heraeus, Germany) without contaminating the treated enamel surfaces. Only specimens restored with glass-ionomer cement were protected in the restoration area by a base acid during analysis. All specimens were examined by scanning electron microscopy (Personal SEM EeXpress; Aspek Corporation, Delmont, PA, USA) at an accelerating voltage of 15–20 kV before and after the pH-cycling regimen *in vacuo*. Elemental analysis by EDX was conducted over the surface area of each block around restorations to determine the relative amounts of calcium (Ca), phosphorus (P), and F by the weight percentage. Parameters for sound enamel were the chemical formula of hydroxyapatite  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , with a density of 3.021 g/cm<sup>3</sup>.<sup>28</sup>

### Statistical Analyses

Data were subjected to statistical analysis using the statistics program SPSS-17 (SPSS; IBM, Chicago, IL, USA). Normal distribution and equality of variances were checked for all the variables using the Kolmogorov–Smirnov test. SH was analyzed using two-way repeated-measures analysis of variance followed by Tukey's test. For EDX data, the amounts of Ca, P, and F were compared between samples using paired *t*-tests. The level of statistical significance was set at  $p < 0.05$ .

## RESULTS

### Surface Hardness Measurements

Table 2 presents all the mean values and corresponding standard deviation values for SH (KHN). All groups had essentially the same initial SH ( $p > 0.05$ ), implying all the blocks had a uniform hardness to conduct the treatments. For all treatment groups, the pH-cycling regimen caused demineralization of the enamel proximal to the restorative material. The glass-ionomer cements F IX, IZ, and F II had the highest postcycling values for hardness at all 3 distances evaluated (150, 300, and 450  $\mu\text{m}$ ), and these values were significantly different from those of the other groups ( $p < 0.001$ ). The Z250 composite resin had the lowest SH value, which did not differ significantly from that of the NT group at all distances evaluated ( $p > 0.05$ ).

The S-PRG filler F-releasing restorative material B II group (giomer system) yielded a postcycle SH value that was intermediary among the other glass-ionomer cements we evaluated, and it was significantly higher than the SH values for the Z250 resin and NT groups at the 3 distances evaluated ( $p < 0.05$ ). For all groups, the SH value decreased as the distance from the restoration increased (Table 2).

Table 2: Mean  $\pm$  Standard Deviation of the Initial and Final Surface Hardness (SH, KHN) at Three Distances from the Treatment Area (150, 300, and 450  $\mu\text{m}$ ) That Were Evaluated for All Groups<sup>a</sup>

Groups	Initial SH (Kg/mm <sup>2</sup> )	SH (Kg/mm <sup>2</sup> ) (final 150 $\mu\text{m}$ )	SH (Kg/mm <sup>2</sup> ) (final 300 $\mu\text{m}$ )	SH (Kg/mm <sup>2</sup> ) (final 450 $\mu\text{m}$ )
F IX	345.53+36.74 Aa	287.40+57.78 Ba	267.07+58.29B Ca	242.93+63.32 Ca
I Z	346.00+35.59 Aa	284.00+39.08 Ba	261.53+47.93B Ca	244.80+46.22 Ca
F II	352.93+31.04 Aa	309.13+29.67 Ba	267.80+23.02 Ca	231.73+35.85 Da
B II	345.93+33.70 Aa	216.87+45.63 Bb	175.67+31.41 Cb	151.73+32.32 Cb
F 250	379.60+24.55 Aa	122.67+22.21 Bc	89.67+11.69 Cc	86.67+20.28 Cc
NT	374.80+32.23 Aa	88.13+6.48 Bc	81.80+6.28 Bc	77.07+9.31 Bc

Abbreviation: SH, surface hardness.

<sup>a</sup> Different capital letters in the same row indicate statistically significant differences ( $p < 0.05$ ; repeated measures analysis of variance and Tukey's tests); different lower case letters in same column indicate statistically significant differences ( $p < 0.05$ ; analysis of variance and Tukey's tests).

Energy Dispersive X-ray Spectroscopy

Table 3 presents the atomic percentages of Ca, P, F, and the Ca/P weight ratio on the enamel substrates that were determined by EDX. The postcycling P content did not differ significantly among the groups ( $p>0.05$ ). After pH cycling, the content of Ca, P, and F did not differ among the groups (including NT), with the exception of the B II group, which had a significantly lower amount of Ca ( $p=0.003$ ) but a significantly higher amount of F ( $p=0.003$ ) in the enamel immediately adjacent to the restoration.

DISCUSSION

Hybrid composite restorative materials based on S-PRG fillers have been touted in the dental materials field to provide biofunctionality to restorative materials. In the current study, we evaluated the potential of restorations with an S-PRG filler F-released composite to inhibit enamel demineralization against glass-ionomer cements and conventional composite resins to prevent caries developing adjacent to restorations, simulating real clinical conditions. In addition, we evaluated the elemental inorganic content of enamel around restorations before and after treatments. The null hypothesis that the S-PRG restorative materials would not have the potential to prevent demineralization was rejected, as demonstrated by the SH and EDX data.

In this study, the pH-cycling regimen was used according to Vieira and others<sup>26</sup> to simulate the demineralizing and remineralizing episodes that occur

in the oral cavity and create caries-like lesions similar to those occurring *in vivo*.<sup>24</sup> Dental caries is a biofilm-sugar dependent disease<sup>29</sup> and, therefore, caries lesions will develop on intact or restored dental surfaces on which a biofilm forms. The acidic pH environment produced from the fermentation of dietary sugars promotes the dissolution of the underlying dental minerals.<sup>30</sup> This is the first step in the demineralization process, when acid reaches the site on a crystal surface and Ca/P are dissolved into the surrounding aqueous phase between the crystals.<sup>31</sup> Thus, pH is a driving force that regulates the loss or gain of Ca and P from the mineral structure of teeth.<sup>32</sup> If F ions are present at the crystal surface during demineralization, these ions can adsorb onto the surface of the crystals and inhibit demineralization by acids.<sup>31</sup>

The difference between caries progression on tooth substrates adjacent to restorations and the sound tooth surface is the possibility of a biofilm accumulating at the interface. This problem associated with the shrinkage stress of restorative materials could boost secondary caries development.<sup>33,34</sup> Therefore, for restorative materials that release F, besides restoring function and esthetics, they can also control the development of caries adjacent to the filling,<sup>32</sup> as F can reduce demineralization and promote remineralization of dental hard tissues. Based on our EDX results, the S-PRG material (group B II) retained the largest amount of F ( $p=0.003$ ) levels in the enamel adjacent to the restoration (Table 3). Naoum and others demonstrated that Beautifil II released more F than other resin-based

Table 3: Element Content in Atomic Percentage (At%; mean ± SD) at the Initial Condition and After Undergoing the pH-cycling Regimen According to Different Groups								
Groups	Initial Ca	Final Ca	Initial P	Final P	Initial F	Final F	Ca/P Ratio	Ca/P Ratio
F IX	54.05 ±0.95	53.60 ±0.83	37.09 ±0.21	37.16 ±0.15	1.55 ±0.15	1.60 ±0.15	1.45 ±0.03	1.44 ±0.02
I Z	53.80 ±0.95	53.89 ±0.93	37.12 ±0.13	37.20 ±0.26	1.58 ±0.17	1.55 ±0.14	1.44 ±0.02	1.44 ±0.03
F II	54.01 ±0.77	53.29 ±1.04	37.11 ±0.15	37.2 ±0.32	1.52 ±0.13	1.62 ±0.14	1.45 ±0.02	1.43 ±0.03
B II	54.05 ±0.69 <sup>a</sup>	53.13 ±0.75 <sup>a</sup>	37.16 ±0.19	37.11 ±0.27	1.51 ±0.10 <sup>*</sup>	1.67 ±0.12 <sup>a</sup>	1.45 ±0.02 <sup>a</sup>	1.43 ±0.02 <sup>a</sup>
F 250	53.65 ±0.92	52.89 ±1.52	37.35 ±0.17	37.39 ±0.34	1.52 ±0.15	1.54 ±0.16	1.43 ±0.02	1.41 ±0.05
NT	53.69 ±1.02	53.82 ±1.51	37.37 ±0.25	37.41 ±0.26	1.55 ±0.15	1.52 ±0.24	1.43 ±0.03	1.43 ±0.04
Abbreviations: Ca, calcium; F, fluoride; P, phosphorous. <sup>a</sup> Statistically significant difference ( $p<0.05$ )								

materials.<sup>35</sup> In this study, which simulates real clinical challenges, the higher F levels presented are explained as follows: S-PRG fillers promote a rapid F release via ligand exchange between F and cations within the prereacted hydrogel.<sup>36</sup> This ability to release F implies that B II is the most capable of providing F to the surrounding tooth structure at times when the adjacent enamel is most susceptible to demineralization.<sup>35</sup>

On the other hand, the pH-cycling regimen significantly decreased the Ca level for B II, which confirms the dissolution of hydroxyapatite. Such results were unexpected because when F is present, the amount of mineral dissolved is reduced because a certain proportion of Ca and P ions are incorporated into enamel as fluorapatite, thus reducing demineralization.<sup>32</sup> Therefore, based on these results, we can state that although the S-PRG material is able to release F in the enamel adjacent to the restoration, the bioavailable F is not enough to develop a calcium fluoride (CaF<sub>2</sub>)-like particles reservoir and increase mineral resistance to acid through the formation of fluorapatite (ie, remineralization process).<sup>37</sup>

Despite F-enhanced remineralization incorporated Ca and P ions into the surface, in low concentrations it only partially inhibited the net dissolution of enamel, while remineralization requires the presence of Ca and P and an F reservoir preventing the oral environment from becoming unsaturated.<sup>38</sup> In addition, F release in B II is accompanied by other ions, such as aluminum, which present a strong affinity to fluoride-forming Al-F complex and reduces the levels of bioavailable F ions.<sup>39</sup> This fact can interfere with the dynamic caries process since the presence of free ions is important to ensure F bioavailability. These results were confirmed by the SH analysis, because the B II group had a lower mean value for hardness than the glass-ionomer cements, for which the Ca concentration remained unchanged after pH cycling.

A previous study has shown that an S-PRG filler-containing tooth-coating material inhibited demineralization around the coating.<sup>40</sup> However, it used a static model (not a dynamic pH-cycling model) to induce demineralization, which may have resulted in overestimating the effect of the coating.<sup>40</sup> In the present study, we speculate that although the F release was not able to form a CaF<sub>2</sub>-like particles reservoir, F ions might be bioavailable in the environment, but more studies are necessary to confirm this. In this study, the higher F release values occur by exposure to an acid pH (4.7), which enhances hydrolysis of the F component in the material.<sup>41</sup> However, although EDX analysis evaluated the atomic percentage composition of the blocks, it is recommended to ensure the bioavailable ions to

determine the potential protective benefits of this class of material to tooth structure.

At the same time, the B II group presented lower values of hardness than glass-ionomer cements, which were able to maintain a constant Ca concentration after the pH-cycling regimen. The Ca/P weight ratio and Ca/P molar ratio determine the rate of hydroxyapatite mineralization, and it is important to evaluate them as the mechanical properties of the tooth substrate, as its rate of biodegradation strongly depends on it. This ratio was calculated for stoichiometric hydroxyapatite (HA; Ca/P weight ratio = 253/2.151) and varies accompanying tissue mineralization.<sup>42</sup> The lower values of Ca and the Ca/P ratio presented by B II confirm that despite F release being able to prevent demineralization, it is not enough to improve the remineralization process because of the uptake of lower levels of Ca ions. Therefore, F released from a material should not be the only factor that determines the potential protective benefits of different bioactive materials to the tooth structure.

Besides that, B II presents less controlled F-release than glass-ionomer cement, as the F glass within B II presents little or no glass-ionomer matrix phase due to the lack of a significant acid-base reaction. Glass-ionomer cements are also more porous, which may influence the amounts of F released. In addition, when compared with glass-ionomers, giomer composites have more resin contents added and the barrier through which water and F diffuse also increases.<sup>43,44</sup>

Glass-ionomer cements release F into the oral environment via 2 processes: (1) a short-term reaction involving a fast transfer of F to the oral environment, and (2) a gradual diffusion of F through the developing matrix, which undergoes a gradual increase in crosslink.<sup>35,36</sup> The amount of F released in this second process depends on the nature of the matrix formed.<sup>45</sup> In the present study, the release of F shown in the F IX, IZ, and F II groups (conventional and resin-modified glass-ionomer cements) probably occurred by the second process, and for this reason we did not observe an initial increase in the F level for these materials when evaluated by EDX (Table 3). In this same context F diffused through the cement, because the enamel around the F IX, IZ, and F II glass-ionomer cements had the highest values for hardness at the 3 distances evaluated (150, 300, and 450  $\mu$ m), and these values differed significantly from those of the B II and Z250 groups ( $p < 0.001$ ; Table 2). The SH results for our glass ionomers agree with the results of Okada and others: we observed an uptake of Ca and P ions with a consequent increase in hardness.<sup>46</sup> The delayed release of F enhanced their potential to inhibit the recurrence of



caries. The outcomes presented here reflect the ability of certain resins to reduce demineralization based on F availability as a consequence of the dissemination of F, since the continuous availability of F in the oral environment can slow demineralization.<sup>32</sup> This slow release of F presented by glass-ionomer cements has clinical implications, as F-released from an ionomer follows a continuous uptake process and increases F concentration in the oral environment.

Among the F-releasing materials we evaluated, the results showed that hardness values decrease with distance from the restorative material (300 and 450 µm), although the F IX, IZ, and F II groups maintained consistently high hardness values (Table 2) in comparison with the other groups. The loss of hardness in the NT group was important for validating the pH cycling used in our study;<sup>24</sup> it demonstrated that the bovine enamel had demineralized, providing the proposed demineralizing challenge. Our results show that EDX is an effective method for detecting minor alterations in Ca, P, and F mineral content. The glass-ionomer cements of the F IX, IZ, and F II groups yielded similar results, without significant differences in the percentages of Ca, P, and F between the initial and final conditions after the pH-cycling regimen. These results agree with previous findings.<sup>37,47</sup> For the Z250 and NT (negative control groups), there was no significant shift in Ca, P, or F content values after the pH-cycling experiment, as expected.

A limitation of the current study must be highlighted. Although the percentages of F did not differ between the initial and final conditions for the glass-ionomer cements, they are known to have better capability to act as an F reservoir than composite resin-based materials,<sup>48</sup> implying that over long-term continuous episodes of demineralization/remineralization, the amount of F released by the glass-ionomer cements differed from that of the other cements, as shown by the EDX results. Therefore, further studies are necessary to evaluate the effect of F release into the adjacent enamel in real time over a long period.

Within the limitations of this laboratory study, based in the current findings, the glass-ionomer-based materials we evaluated were able to release F at sufficient doses to slow the rate of demineralization. The bioactive PRG filler F-containing resin composite can be considered an effective option for restorations in patients at high risk of dental caries, especially in stress-bearing areas such as in posterior tooth restorations or when the aesthetic factor is essential, as this class of material has the potential to prevent new carious lesions

developing around restorations. However, further long-term analysis and *in vivo* studies are required to determine the efficacy of these materials for controlling caries lesions.

### Conflict of Interest

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

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# Airborne-particle Abrasion and Dentin Bonding: Systematic Review and Meta-analysis

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## Clinical Relevance

The literature reviewed suggests that airborne particle abrasion has no negative effects on the bond strength of resin-based materials to dentin and that a positive influence on dentin bond strength was only achieved in specific air-abrasion conditions.

## SUMMARY

In this systematic review the authors investigated how airborne-particle abrasion (APA) using aluminum oxide affects the bond strength of resin-based materials to dentin. The search was performed in three databases. *In vitro* studies (Type of study) comparing the bond strength of resin-based materials (Outcome) to air-abraded (Intervention) compared with non-air-abraded (Comparison) human dentin (Population) were included (the PICOT elements are given parenthetically). From 5437 unique articles, 65 were read in full, 33 were included in the qualitative synthesis, and 32 were included in the meta-analysis. Methodologic quality and risk of bias were assessed. Comparisons were performed between air-abraded and control dentin groups by adopt-

ing a random-effects model ( $\alpha=0.05$ ). Additional analyses were carried out for the different parameters used in APA: type of surface treatment in the control group, particle size, air pressure, and APA duration. The bond strength to air-abraded dentin was favored only when the control surface was treated with a hand excavator. For particle size, APA was favored when the particle size was  $>30\ \mu\text{m}$  and the controls were no treatment or hand excavator or when the particle size was  $\leq 30\ \mu\text{m}$  and the control was bur. In addition, the results favored air-abraded groups only when the pressure was  $> 5$  bar and bur was used in the control group. No significant differences were observed for duration of APA. No comparison on bond strength considering the presence of aging conditions was possible in the included

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**studies due to the low number of studies that aged the specimens. In conclusion, APA had no negative effects on the bond strength of resin-based materials to dentin and was able to improve the dentin bond strength only when the particle size was  $> 30 \mu\text{m}$  and air pressure was  $> 5$  bar. PROSPERO registration protocol: CRD42018096128**

## INTRODUCTION

Airborne-particle abrasion (APA) is a procedure used for several applications in dentistry, with the first report dating back to the 1940s.<sup>1</sup> Different air-abrasion devices have been introduced to the market for applications including cavity preparation,<sup>1-3</sup> prophylaxis and removal of surface stains,<sup>1</sup> selective caries removal,<sup>4</sup> tribochemical coating,<sup>5</sup> and surface polishing or roughening.<sup>6</sup> APA involves propelling a well-defined, sharply focused stream of particles expelled from a small nozzle under high pressure against a surface. The fluid used is usually compressed air, and many particle types, such as sodium bicarbonate, glycine, and aluminum oxide, can be used depending on the intended goal of APA.<sup>7,8</sup> The particle size, pressure, and duration of APA may also vary depending on the clinical application<sup>9-12</sup> and affect the result of the abrasion process.

Depending on the abrasive particle, the kinetic energy of the accelerated hard particles may result in rapid substance removal on impact.<sup>13</sup> Whereas sodium bicarbonate is usually used for polishing procedures, APA with aluminum oxide is commonly used to prepare surfaces to enhance micromechanical retention of restorative materials, such as glass ceramics,<sup>14,15</sup> oxide ceramics,<sup>16,17</sup> and resin composites.<sup>18,19</sup> The objective is usually to increase the area for micromechanical interlocking of adhesive materials.<sup>20-22</sup> Despite a limited number of clinical studies, clinical applications of APA of dental substrates using aluminum oxide particles have been reported as a cleaning method, a pretreatment technique before adhesive luting of indirect restorations, and surface treatment before resin composite restorations.<sup>23-25</sup>

Despite the potential benefits for bonding restorative materials, APA has also been shown to produce surface flaws and microcracks that can compromise the strength of ceramic restorations.<sup>26,27</sup> Thus, evaluation of the effects of APA on human dentin is warranted. In several *in vitro* studies, investigators have examined the effect of APA on dentin<sup>10,21,22,28-32</sup> and have usually focused on applying air-abrasion to improve the bond strength of adhesive materi-

als.<sup>10,17,24,33-38</sup> Large variability exists among the size of the abrasive particles used, as well as the time duration and pressure used in APA. Pooled *in vitro* data could help determine whether APA has a positive effect on dentin and ascertain whether the technique can be applied clinically to dentin surfaces without major concerns. The aim of this systematic review of *in vitro* studies, therefore, was to investigate how APA using aluminum oxide particles affects the bond strength of resin-based materials to human dentin. The null hypothesis was that APA does not have a negative effect on dentin bond strength.

## METHODS AND MATERIALS

This systematic review adheres to the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) statement.<sup>39</sup> The review protocol was registered with the international database for systematic reviews – PROSPERO (protocol CRD42018096128). The PICOT elements were as follows: Population, human dentin; Intervention, air abrasion considering a procedure in which dentin surfaces were subjected to abrasive blasting by a stream of aluminum oxide particles propelled under high pressure with compressed air as the fluid; Comparison, non-air-abraded dentin; Outcome, bond strength of resin-based materials to dentin; and Type of study, *in vitro* tests.

## Systematic Literature Search

The literature search aimed to identify all studies that evaluated the effect of APA using aluminum oxide particles on dentin. The search was systematically performed by two independent reviewers (VPL and VSC) using three online international scientific databases: The National Library of Medicine (MEDLINE/PubMed), ISI Web of Science, and Scopus. The search strategy used in PubMed is shown in Table 1. The strategy was adapted to the other databases accordingly. The final search was performed in October 2018. After the articles were searched, all were imported into Endnote X7 software (Thompson Reuters, Philadelphia, PA, USA) to remove duplicates.

Titles and abstracts were read to verify the inclusion criteria: *in vitro* studies that reported comparison between air-abraded and non-air-abraded dentin bond strengths. When the study did not clearly define the control group, the non-air-abraded group was considered the control. The following terms were considered in the inclusion criteria: “air abrasion,” “airborne particle abrasion,” “air polishing,” or “sandblasting.” Aluminum oxide particles

Table 1: Search Strategy Used in PubMed and Adapted to the Other Databases

Search Terms	
#3	Search #1 AND #2
#2	Dentin* OR Dental
#1	Air-Abrasion OR Air Abrasion OR Airborne Abrasion OR Airborne-Particle Abrasion OR Particle Abrasion OR Air Abrasion, Dental OR Abrasion, Dental Air OR Abrasions, Dental Air OR Air Abrasions, Dental OR Dental Air Abrasion OR Dental Air Abrasion OR Prophylaxis OR Sandblast* OR Air Polishing

were the only abrasive eligible for this review. Only studies that evaluated the bond strength of resin-based materials to sound dentin from human teeth exposed to APA were included. Studies that evaluated bovine dentin and abrasive particles other than aluminum oxide were excluded. Only articles published in English were considered, with no restrictions on year of publication. Any disagreement regarding the eligibility of the included studies was resolved through discussion and consensus, or a third reviewer (RRM) was consulted. Only studies that fulfilled all eligibility criteria were included. Whenever information relevant to eligibility was unavailable in the abstract or the abstract itself was unavailable, the article was selected for full-text reading. The reviewers manually searched the reference lists of the included articles for additional relevant studies.

### Data Recorded From the Selected Studies

For each included study, the following data and information were recorded using a standard form in spreadsheet format (Excel for Mac version 16.31, Microsoft Corporation, Redmond, WA, USA): control group and its surface treatment, particle size, air-abrasion distance, angle with the surface during air-abrasion, air-abrasion pressure, air-abrasion duration, cleaning method or surface treatment after APA, type of bond strength test, bond strength mean values in MPa, standard deviations, and number of specimens tested.

### Data Analysis

Pooled effect estimates were obtained by comparing the standardized mean difference between the air-abraded and control groups within each study with estimated 95% confidence intervals. The standardized mean difference was used to minimize differences in bond strength values measured by different methods, for example, shear or tensile tests. The analyses were performed by adopting a random-effects model using Review Manager version 5.1 (The Nordic Cochrane Centre, The Cochrane Collaboration, Copenhagen, Denmark). As the studies

adopted different surface treatments in the control groups, a meta-analysis was carried out considering the same intervention-control subgroups, that is, the control groups were separated according to their surface treatments: no treatment, bur, SiC abrasive paper, hand excavator, or acid etching. Additional analyses were carried considering the different air-abrasion parameters adopted, that is, the abrasive particle size ( $\leq 30 \mu\text{m}$  or  $> 30 \mu\text{m}$ ), air pressure ( $\leq 5$  bar or  $> 5$  bar), APA duration ( $\leq 10$  seconds or  $\geq 15$  seconds) and presence of aging conditions (yes/no). All meta-analyses considered the same combinations of intervention-control groups. Statistical heterogeneity of the treatment effect among studies was appraised using the Cochran Q test, in which values  $> 50\%$  were considered to suggest substantial heterogeneity.<sup>40</sup> Multiple groups from the same study were analyzed according to the Cochrane guidelines formula for combining groups.<sup>40</sup>

### Quality Assessment and Risk of Bias

The methodologic quality and risk of bias of the included studies was assessed according to Cochrane guidelines<sup>40</sup> and criteria adapted from previous studies<sup>41,42</sup> as follows: selection bias (random sequence generation), sample-size calculation, presence of a clearly defined control group, and performance and detection bias (blinding of operator/examiner). Each criterion was judged to have high, low, or unclear risk of bias, which was also used for quality assessment. The assessment of risk of bias was performed using Review Manager version 5.1.

## RESULTS

The search resulted in the retrieval of 7340 articles, as shown in the study flowchart presented in Figure 1. After removing duplicates, 5437 unique publications were screened, of which 5372 were excluded because they did not meet the eligibility criteria. A total of 65 articles were assessed in full, including two found in the manual search. From these 65 publications, 32 were excluded for reasons detailed in Figure 1. The list of articles excluded after the eligibility screening is provided as supplementary

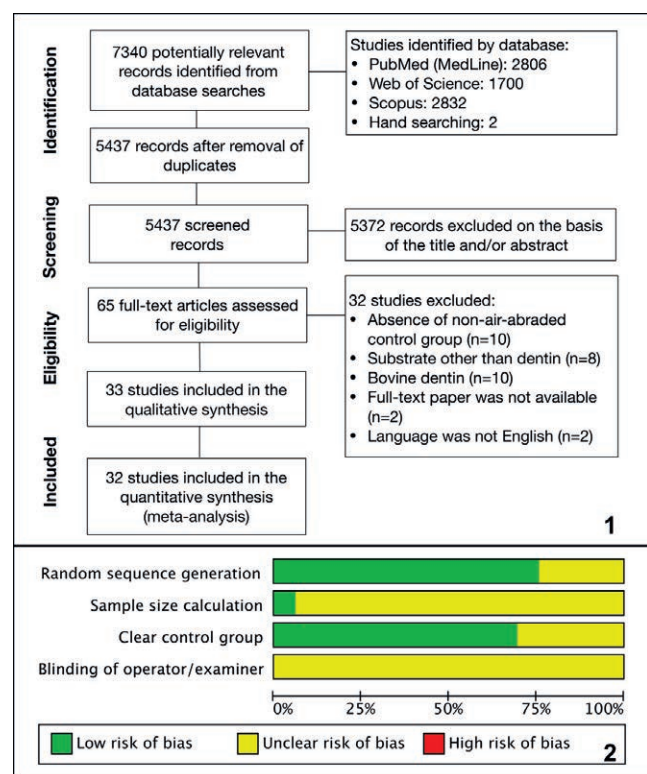


Figure 1. Flowchart of the systematic review.

Figure 2. Risk of bias graph: proportion of studies with low, unclear, or high risk of bias for each item.

material in the Appendix. A total of 33 studies were included in the qualitative synthesis, and 32 were included in the meta-analysis. One study that evaluated bond strength<sup>43</sup> was not included in the quantitative analysis because the standard deviation value was missing; nevertheless, this study reported similar bond strengths between air-abraded and control dentin.

Varied APA parameters were used across the studies: 70% used aluminum oxide particles with size  $>30\ \mu\text{m}$ , the air pressure most commonly used was up to 5 bar (51.5%), and air-abrasion duration was usually up to 10 seconds (54.5%). All studies included in this review and the details of the air-abrasive procedures are provided in the Appendix. Only data that were within the scope of this study are reported. Regarding quality assessment (Figure 2), most included studies presented low risk of bias relative to random sequence generation, and the majority of studies presented a clearly defined control group. Sample-size calculation was reported in two studies, and blinding of operator/examiner was not reported in any of the included studies. The risk of bias for each item judged in each included study is shown in Figure 3.

The meta-analysis on dentin bond strength values considering the different combinations of intervention-control comparisons is presented in Figure 4. A significant difference was found between the groups favoring dentin subjected to APA compared with non-air-abraded dentin only when the control surface was treated with a hand excavator ( $p=0.02$ ,  $I^2=67\%$ ). When the other control dentin surface treatments were considered (no treatment, bur, SiC paper, or acid etching), no significant differences between air-abraded and non-air-abraded dentin were detected.

Considering the APA parameter particle size (Figure 5), the results favored APA when the particle size was  $>30\ \mu\text{m}$  and the controls were no treatment ( $p=0.02$ ,  $I^2=64\%$ ) and hand excavator ( $p<0.00001$ ,  $I^2=0\%$ ). APA was also favored when the particle size was  $\leq 30\ \mu\text{m}$  and the control was bur ( $p=0.0004$ ,  $I^2=0\%$ ). For the other control surfaces no significant differences between the experimental and control groups were observed. When the parameter air-abrasion pressure was considered in the meta-analysis (Figure 6), the results favored air-abraded groups only when the air pressure was  $>5$  bar and bur was used to treat the control surfaces ( $p=0.01$ ,  $I^2=0\%$ ), with no other significant differences. Two studies<sup>44,45</sup> did not report air pressure, thus were not considered in the subgroup analysis. Regarding the parameter APA duration (Figure 7), no significant differences were observed in bond strength between control and air-abraded dentin. Three studies did not report the duration of air abrasion.<sup>46-48</sup> and, in two studies, air-abrasion duration was not standardized since it depended on the removal of cement from the surface<sup>20,49</sup> or tooth preparation.<sup>8,50</sup>

It was not possible to perform a meta-analysis considering the presence of aging conditions in the studies as only four articles reported dentin bond strengths for immediate and aged groups.<sup>45,49,51,52</sup> Each study had a different type of control, hindering comparisons within the same combinations of intervention-control groups.

## DISCUSSION

The null hypothesis tested was accepted since application of APA with aluminum oxide particles had no detrimental effect on the bond strength of resin-based materials to dentin in any of the meta-analyses. Previous studies have reported possible negative effects of APA on dentin characteristics. These studies showed that APA may result in more irregular,<sup>11,12,26,53-55</sup> or rougher dentin surfaces compared with non-air-abraded dentin.<sup>56-59</sup> How-

	Random sequence generation	Sample size calculation	Clear control group	Blinding of operator/examiner
Abo-Hamar <sup>49</sup>	+	?	+	?
Ahid <sup>44</sup>	+	?	?	?
Anja <sup>33</sup>	+	?	+	?
Burnett <sup>29</sup>	?	?	?	?
Chaiyabutr <sup>20</sup>	+	?	?	?
Chaves <sup>30</sup>	+	?	?	?
Chimello <sup>9</sup>	+	?	?	?
Coli <sup>10</sup>	?	?	+	?
D'Amario <sup>21</sup>	+	?	+	?
de Oliveira <sup>62</sup>	+	?	+	?
Dilber <sup>11</sup>	+	+	+	?
Flury <sup>36</sup>	?	?	?	?
França <sup>61</sup>	+	?	?	?
Freeman <sup>62</sup>	?	?	+	?
Geitel <sup>37</sup>	?	?	?	?
Ilday <sup>38</sup>	+	?	+	?
Leite <sup>43</sup>	+	?	?	?
Los <sup>46</sup>	?	?	+	?
Manhart <sup>61</sup>	+	?	+	?
Manhart <sup>13</sup>	+	?	+	?
Moritz <sup>47</sup>	?	?	+	?
Motisuki <sup>12</sup>	+	?	+	?
Pahlavan <sup>63</sup>	+	?	?	?
Pilo <sup>31</sup>	+	?	+	?
Roeder <sup>48</sup>	+	?	+	?
Santos <sup>64</sup>	+	?	+	?
Santos <sup>65</sup>	+	?	+	?
Soares <sup>66</sup>	+	?	+	?
Souza-Zaroni <sup>60</sup>	+	?	+	?
Sutil <sup>6</sup>	+	+	+	?
Van Meerbeek <sup>67</sup>	+	?	+	?
Yazici <sup>68</sup>	+	?	+	?
Zimmerli <sup>45</sup>	?	?	+	?

Figure 3. Risk of bias for each item judged as low, unclear or high in each included study.

ever, findings of the present investigation suggest that those irregular surface aspects may not negatively interfere with the bonding of resin-based materials to dentin. In fact, increasing dentin surface roughness and producing a more irregular surface texture are the goals of APA in many clinical cases. Propelling aluminum oxide particles to dentin may result in substance removal from the surface because of the kinetic energy of the accelerated particles and differences in hardness between the abrasive and the dentin tissue. Aluminum oxide particles have a Vickers hardness of approximately 1200 kg/mm<sup>2</sup>, whereas the Vickers hardness of dentin is approximately 57-60 kg/mm<sup>2</sup>.<sup>60</sup> The rougher dentin surfaces may improve micromechanical interlocking between adhesive agents and restorative materials or improve the wettability of dentin surfaces. This may explain the findings showing that APA was able to improve the dentin bond strength, although only in a few cases and only in the short term.

The improved bond strength to air-abraded dentin was dependent on particle size and pressure of the air stream used.<sup>20,47,61</sup> Aluminum oxide particles >30 µm in size generally yielded better bond strength, although the same effect was observed for particles ≤30 µm when the control was bur. Application of any particle size could potentially increase surface roughness and, thus, the interaction of adhesive agents with dentin. The differences observed for the distinct particle sizes could be explained by their distinct ability in generating morphologic changes for micromechanical keying on dentin surfaces. In addition, air pressures >5 bar, which are in the range of air pressures produced by dental turbines, also led to improved dentin bond strength in a few cases, whereas lower pressures did not yield the same result. In contrast, APA duration was not particularly important for the bond strength to dentin. Therefore, it appears reasonable to suggest that in cases of use of APA in dentin as surface pretreatment seeking for improved bonding,<sup>23-25</sup> aluminum oxide particle sizes >30 µm and air pressure >5 bar should be preferred, although further analyses in this regard are warranted. In case the dentist intends to apply APA to the dentin for other clinical purposes, such as surface cleaning, any particle size or air pressure could be used. It should be highlighted, however, that the actual ability of APA in cleaning the dentin was not investigated here.

Variability in methods used among studies for air-abrading the dentin surfaces was observed,



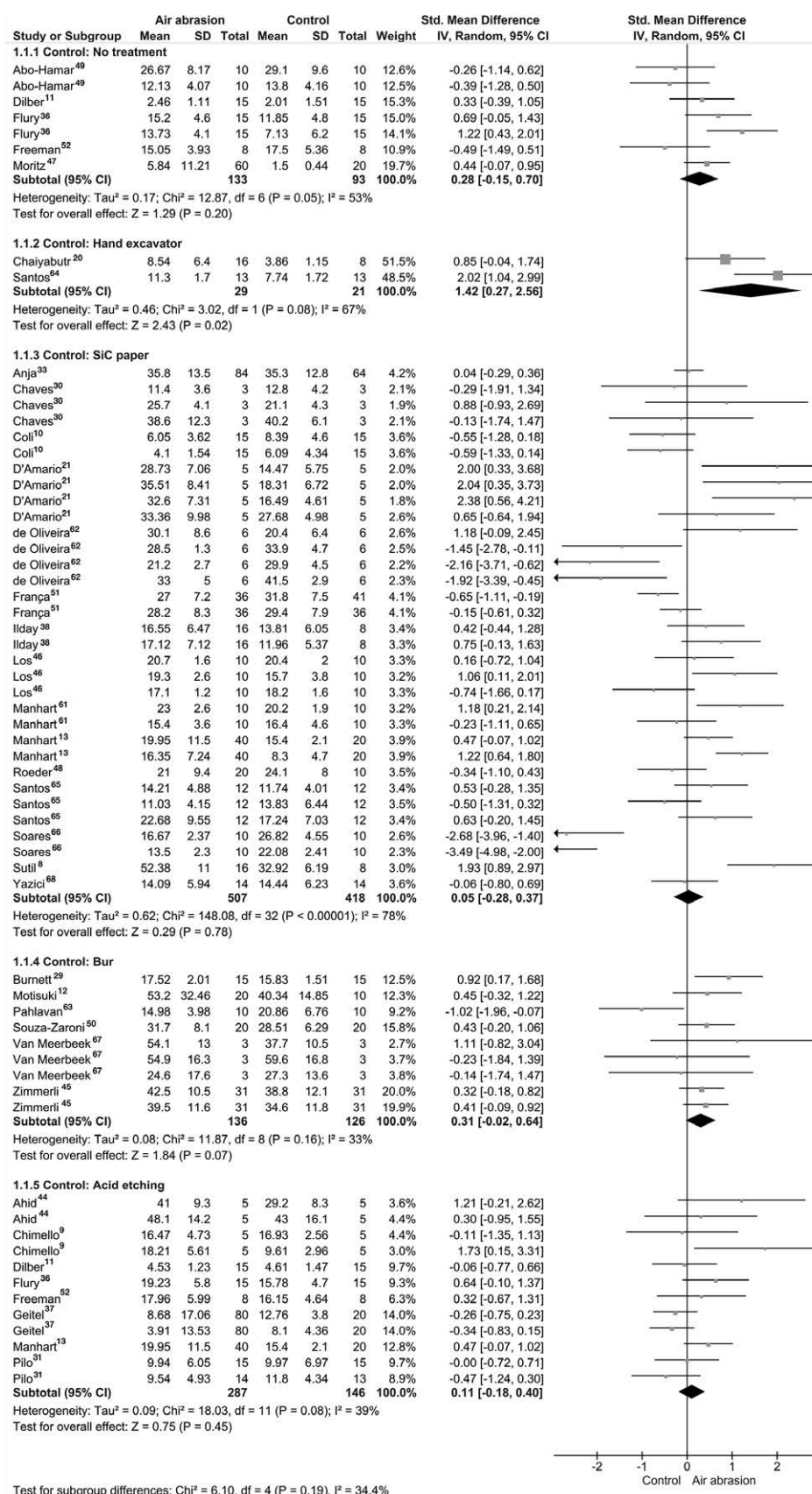


Figure 4. Meta-analysis on dentin bond strength values considering the different combinations of intervention-control comparisons. Statistically significant difference was observed when the control surface was treated with a hand excavator ( $p=0.02$ ).

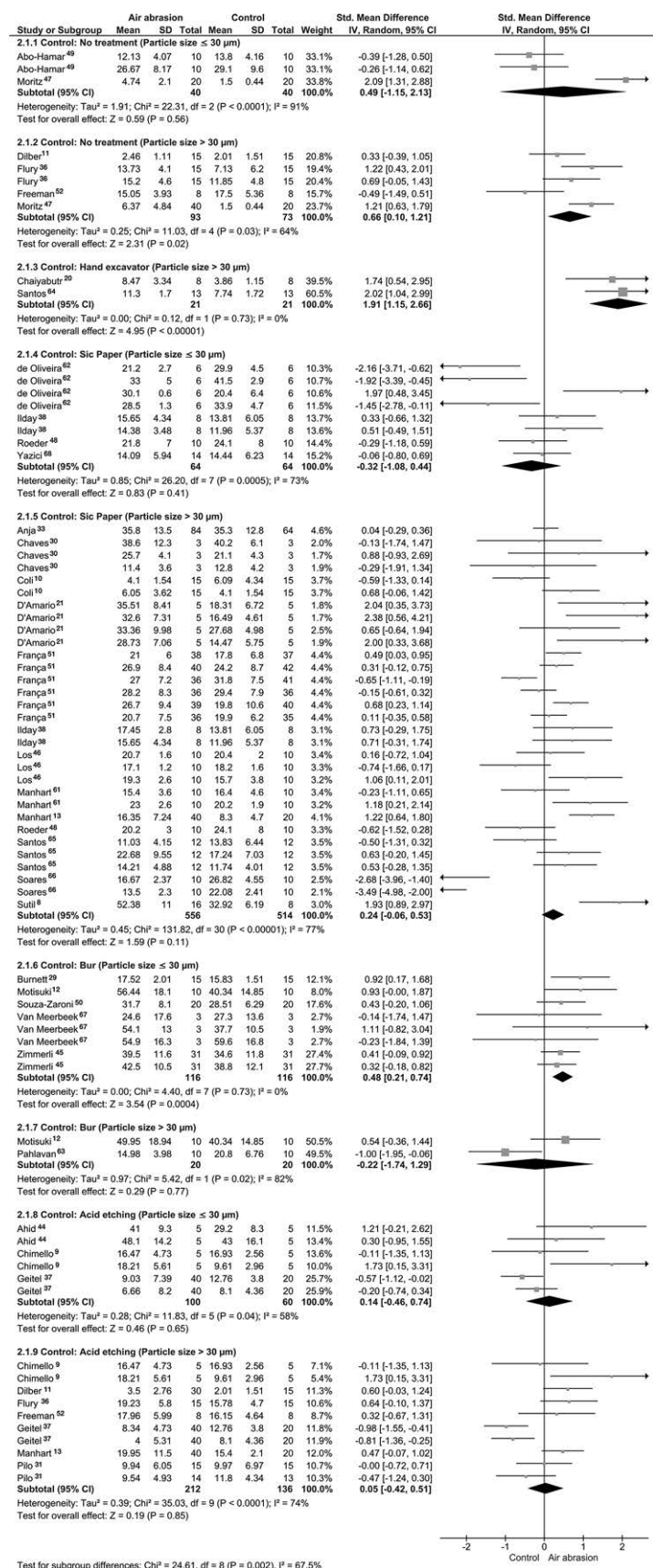


Figure 5. Meta-analysis for particle size ( $\leq 30 \mu\text{m}$  or  $> 30 \mu\text{m}$ ) with same intervention-control groups. Statistically significant differences were observed when the particle size was  $> 30 \mu\text{m}$  and the controls were no treatment ( $p=0.02$ ) or hand excavator ( $p<0.00001$ ). Considering particle size  $\leq 30 \mu\text{m}$ , a statistically significant difference was observed when the control was bur ( $p=0.0004$ ).

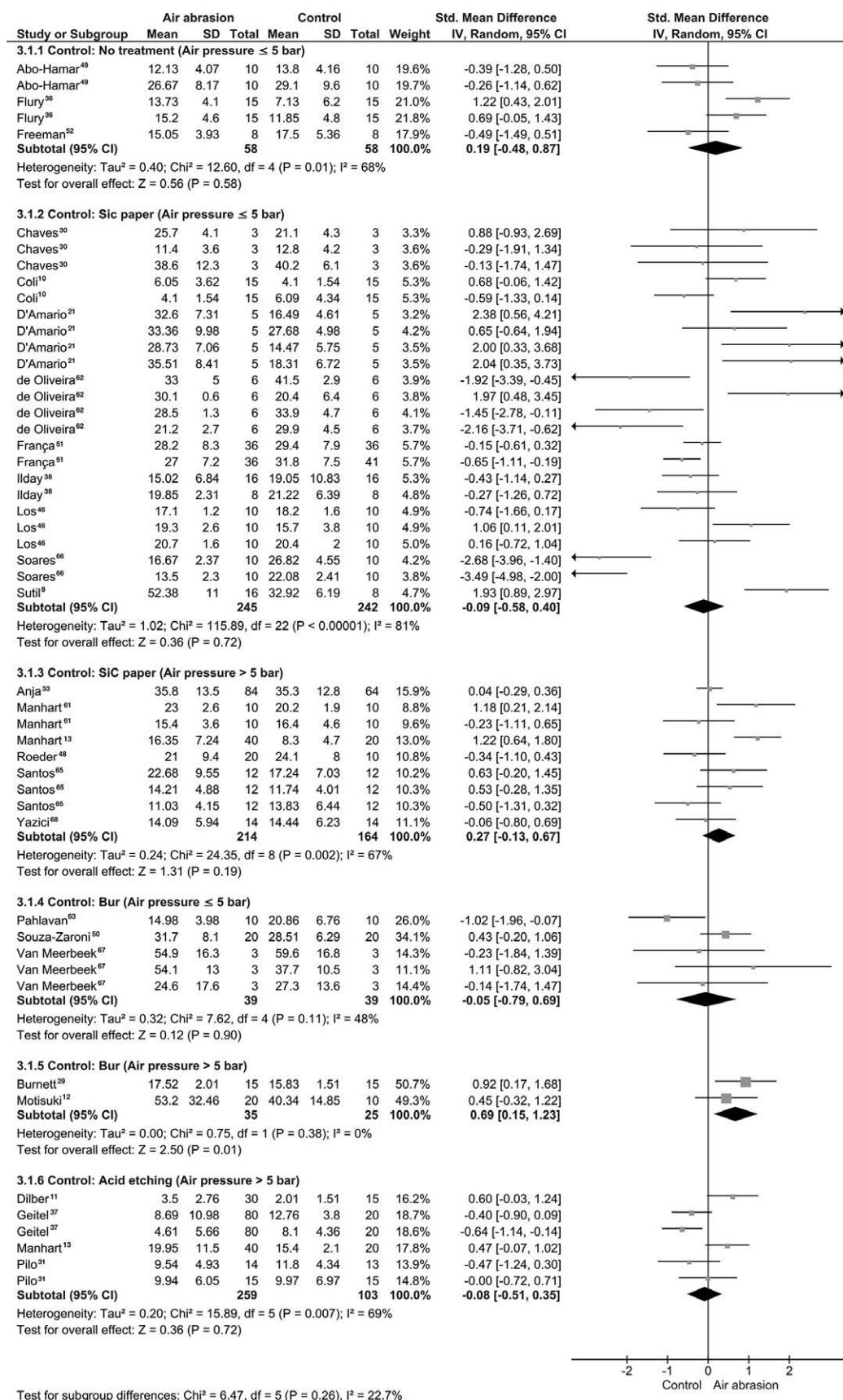


Figure 6. Meta-analysis for air pressure ( $\leq 5$  bar or  $> 5$  bar) with same intervention-control groups. Statistically significant difference was observed when the air pressure was  $> 5$  bar and bur was used to treat the control surfaces ( $p = 0.01$ ).

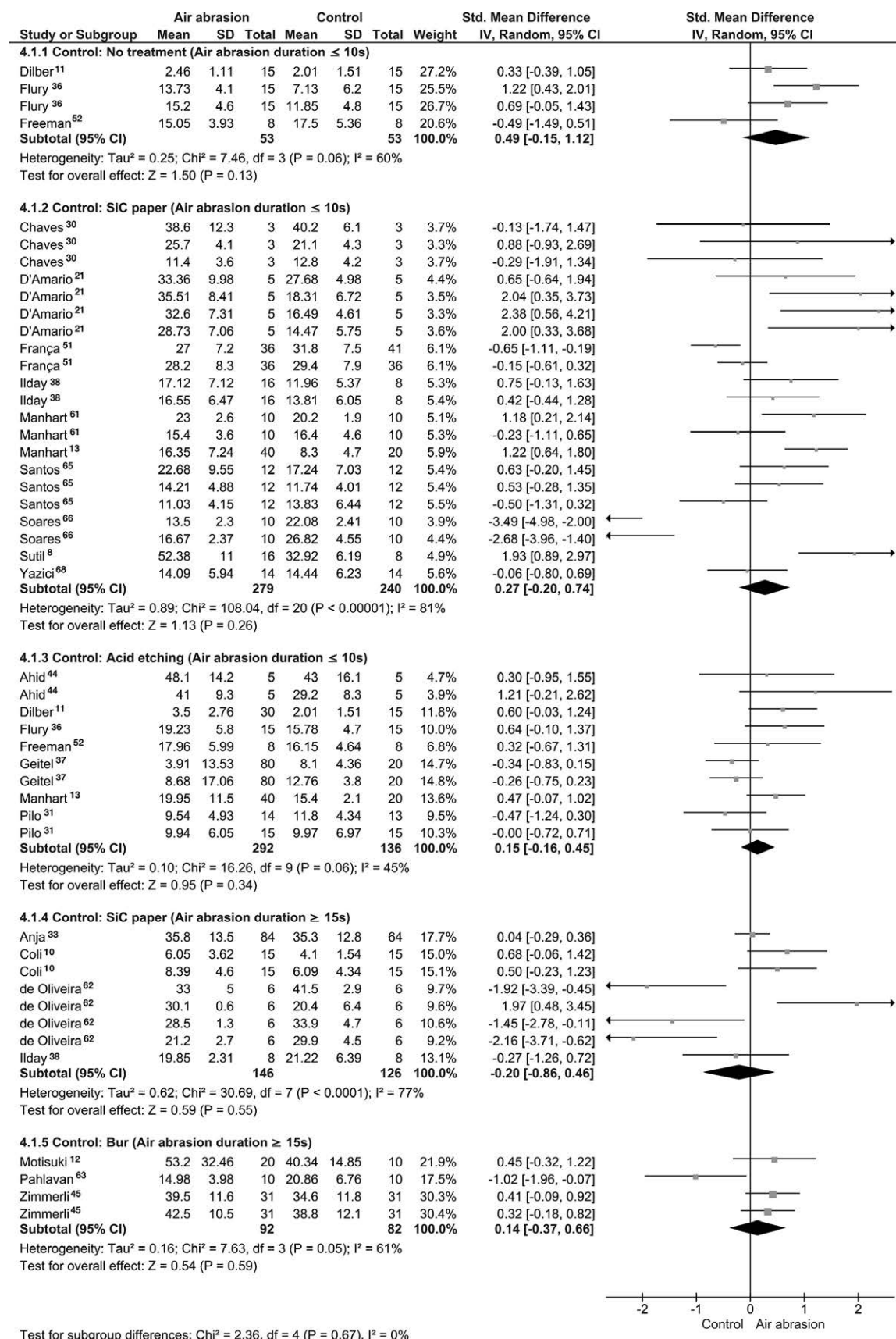


Figure 7. Meta-analysis for airborne-particle abrasion duration ( $\leq 10$  seconds or  $\geq 15$  seconds) with same intervention-control groups. No significant differences were observed in bond strength between control and air-abraded dentin.



including particle sizes, distances, angulations, cleaning methods after abrasion, and air pressures. Analyses according to the parameters employed were used to aid in minimizing those heterogeneities. The surface treatments applied to the dentin specimens before the bond strength tests were not homogeneous either. Therefore, to minimize the clinical heterogeneity regarding surface conditions, the extracted data were separated according to same intervention-control subgroups, that is, the dentin surface treatments: no treatment, bur, SiC abrasive paper, hand excavator, or acid etching. Comparisons were restricted to same intervention-control conditions. The *in vitro* literature is known for having problems regarding good reporting practices, especially because no guidelines are available for reporting the results of the numerous types of *in vitro* tests used in dentistry. In addition, the extracted data were limited to sound dentin to reduce structural and morphologic variability regarding the dentin substrate; thus, the conclusions should not be extrapolated to caries-affected or sclerotic dentin.

Most studies included in this review tested only immediate bond strengths to dentin; that is, there was no evaluation of the effects of water degradation or other aging method on the dentin bonds. *In vitro* studies are urged to always include a storage group when testing adhesive bond strengths. Previous studies showed that differences reported between treatments in the short term were not observed when the bonded specimens were stored in water for some time before testing.<sup>45,49</sup> This finding is of particular importance for APA enthusiasts: the positive effects of the treatment may not persist in the long term. Therefore, it seems that the decision to apply APA to dentin should be made by the dentists, taking into account their own clinical experience; the literature cannot give a definitive answer on whether APA may effectively generate dentin bonds that last longer than non-air-abraded dentin. However, no negative effects for APA applied to dentin were observed either; thus, the treatment seems to be safe with respect to bonding to dentin.

### CONCLUSION

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

- APA with aluminum oxide particles had no negative effects on the bond strength of resin-based materials to dentin.
- In a few subgroup analyses, air abrasion was able to improve the immediate bond strength to dentin

when the particle size was  $>30\ \mu\text{m}$  and air pressure was  $>5\ \text{bar}$ .

- APA duration had no significant effect on immediate dentin bond strengths.

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### Conflict of Interest

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

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# Clinical Performance of Filled/ Nanofilled Versus Nonfilled Adhesive Systems in Noncarious Cervical Lesions: A Systematic Review and Meta-analysis

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## Clinical Relevance

The use of filled adhesive systems does not influence the clinical performance of the adhesive restoration in noncarious cervical lesions.

## SUMMARY

**Objective:** The aim of this meta-analysis was to investigate the clinical performance of filled vs unfilled adhesive systems when applied in noncarious cervical lesions.

**Methods and Materials:** A systematic search was performed in PubMed, Scopus, Web of Science, LILACS, BBO, Cochrane Library, and SIGLE. Gray literature was also screened. Only

randomized controlled clinical trials were included. The risk of bias of the studies was evaluated using the Cochrane Collaboration's tool. A random-effects meta-analysis was conducted to compare the retention rate, marginal discoloration, and secondary caries of noncarious cervical lesions restored with filled adhesives vs unfilled adhesives. The quality of the body of evidence was assessed using the GRADE approach.

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**Results:** A total of 3662 studies were identified after removal of duplicates. Twenty-nine studies remained for qualitative analyses and 28 studies for the meta-analysis. Only one study was judged to have a low risk of bias, and the other 28 were considered to have unclear risk of bias. There was no statistically significant difference between filled adhesives compared with unfilled adhesives in relation to loss of retention, marginal discoloration, or secondary caries at any of the follow-up periods (12-18 months, 24-30 months, 3 years, and 5 years or longer). The quality of evidence was graded as moderate for most outcomes at the respective follow-ups, except when there was an explained heterogeneity, which occurred mainly for loss of retention at the 12-month to 3-year follow-up. The results did not depend on whether microfilled or nanofilled adhesives had been investigated.

**Conclusions:** The addition of fillers into the composition of adhesive systems did not increase the clinical performance (retention rates, marginal discoloration, or secondary caries) of composite restorations placed in noncarious cervical lesions when compared with unfilled adhesives.

## INTRODUCTION

In recent decades, because of an increasing demand for esthetic restorations, composite resins have gained a prominent role in modern restorative dentistry. Nowadays, composite resins are the most widely used dental material, representing 65% of the restorations currently placed in the United States.<sup>1,2</sup> However, it is worth mentioning that 50% to 70% of newly placed restorations are the result of failure of preexisting restorations, which results in millions of dental care dollars spent annually on replacement of these restorations.<sup>2-4</sup> Many of those replacements, however, are unnecessary as either the defects that led to the replacement of the restoration could be repaired adhesively with composite resins or the restorations are replaced due to economic reasons or false diagnosis by the dentist (eg, confusion of discolored margin with caries at the margins).<sup>5-7</sup>

Among several clinical problems of esthetic restorations, the bonding interface between the dentin and the direct restorative material is considered one of the Achilles' heels of esthetic restorations. Recently published reviews have reported that although an improvement in the clinical performance of adhesive restorations has been observed, the

retention rates of composite restorations placed in noncarious cervical lesions are still a clinical problem.<sup>8,9</sup>

Although the exact mechanism responsible for bond degradation is not completely understood,<sup>2</sup> one contributing factor for debonding may arise from the low mechanical properties of the adhesive layer that bonds the composite resin material to the dental substrate. Indeed, among the substrates of this bonded interface, the adhesive layer has the lowest elastic modulus.<sup>10,11</sup> When submitted to masticatory stresses, the adhesive layer suffers the greatest level of strain among the components. Stress that exceeds the inherent strength of the adhesive layer results in defects, cracks, or abrupt catastrophic failure of the resin-dentin bond.<sup>12,13</sup>

Adhesive systems traditionally do not contain filler particles.<sup>14</sup> However, from a theoretical perspective and by analogy with resin composites, the addition of fillers increases the mechanical properties of the adhesive layer.<sup>15,16</sup> This concept was called the elastic cavity wall concept.<sup>17,18</sup> In the past, manufacturers added varying proportions of glass filler particles (microfiller 1-5  $\mu\text{m}$ ) in the hydrophobic bonding bottle of three-step etch-and-rinse adhesives.<sup>16,19,20</sup> These filled adhesives were loaded up to 40-50 wt%,<sup>19</sup> for example, of Optibond FL (Kerr Co, Orange, CA, USA) and PermaQuick (Ultradent, South Jordan, UT, USA). Because of the very good clinical performance in long-term clinical trials of these highly filled adhesives,<sup>21-24</sup> the same strategy was used in simplified versions of two-step etch-and-rinse adhesives and in the self-etch adhesives.<sup>8</sup>

In simplified adhesives, hydrophobic resins are combined with priming and/or acidic monomers, which do not allow the addition of a large filler amount. For example, two-step etch-and-rinse adhesives contain about 8.5-15 wt% of fillers in their composition (OptiBond Solo, Kerr Co.; One-Step Plus, Bisco Inc, Schaumburg, IL, USA),<sup>16,25-27</sup> which is less than half of the amount that is added in three-step etch-and-rinse systems. By adding large filler amounts, adhesives become more viscous, and this jeopardizes the wettability of the dental substrates.<sup>15,16</sup>

Instead of microfillers, nanofillers have been added into the adhesive systems.<sup>28</sup> Apart from improving the strength of the adhesive layers, nanofillers can penetrate into dentin tubules and into the collagen network.<sup>16,27</sup> Nanometer-sized silica (pure silicon dioxide) smaller than 20 nm are usually added.<sup>26,29</sup> Some two-step etch-and-rinse

systems (Prime & Bond NT and XP Bond, Dentsply Sirona and Adper Scotchbond 2 XT, 3M OralCare) and one-step self-etch adhesive systems (Clearfil S3 Bond, Kuraray and G-Bond, GC Corp) that contain nanofillers are available on the market; the amount usually ranges between 5 wt% and 10 wt%.<sup>16</sup>

Studies have shown that simplified adhesives with nanofillers may have better mechanical properties compared with unfilled adhesive systems; however, the improvement is material dependent.<sup>30,31</sup> In addition, studies have also proven that the addition of nanofillers does not increase the bond strength to dentin.<sup>32-35</sup> A closer view showed inconclusive results when clinical studies evaluating filled vs unfilled adhesives were evaluated.<sup>36-45</sup> Therefore, the aim of this systematic review and meta-analysis was to answer the following focused PICO question- (P, participant; I, intervention; C, comparator; O, outcome): “Are the retention rates, marginal discoloration, and secondary caries of composite resin restorations placed in noncarious cervical lesions of patients superior when bonded with filled/nanofilled adhesives compared with unfilled adhesives?”

## METHODS AND MATERIALS

The methodology described in the present study follows the PRISMA requirements (Preferred Reporting Items for Systematic Reviews and Meta-Analyses statement).<sup>46</sup>

### Protocol and Registration

The study was registered in the PROSPERO database (CRD42018093198) and performed from May to August 2018 at the State University of Ponta Grossa, Paraná, Brazil.

### Information Sources and Search Strategy

The search strategy used in the PubMed database was developed based on the concepts of patient and intervention from the focused PICO question described at the end of the Introduction section. Within each concept, the controlled vocabulary (Medical Subject Headings terms) and free keywords were combined with the Boolean operator “OR.” Then, the concepts were combined with the Boolean operator “AND” to restrict the search. A filter for randomized clinical trials was also used for the PubMed database (Table 1). Table 1 also lists other electronic databases that were searched (Web of Science, Scopus, Cochrane Library, Latin American and Caribbean Health Sciences Literature database [LILACS] and Brazilian Library in Dentistry [BBO]). The reference

lists of all primary studies were hand searched for additional relevant publications as well as links to related articles of each primary study in the PubMed database. No restrictions on publication date or languages were made.

The gray literature was also inspected by looking up abstracts of the International Association for Dental Research and their regional divisions (1990-2016), the System for Information on Grey literature in Europe (SIGLE), dissertations and theses using the ProQuest Dissertations and Theses full-text database, as well as the *Periodicos Capes* Theses database. Ongoing trials were searched in the following clinical trials registries: Current Controlled Trials ([www.controlled-trials.com](http://www.controlled-trials.com)), International Clinical trials registry platform (<http://apps.who.int/trialsearch/>), ClinicalTrials.gov ([www.clinicaltrials.gov](http://www.clinicaltrials.gov)), Rebec ([www.rebec.gov.br](http://www.rebec.gov.br)), and EU Clinical Trials Register (<https://www.clinicaltrialsregister.eu>).

### Eligibility Criteria

We included randomized clinical trials (RCTs) with parallel and split-mouth designs that compared the retention rates or other secondary outcomes (caries at restorative margins and marginal discoloration) of filled/nanofilled adhesives vs unfilled adhesives for bonding composite resin restorations in noncarious cervical lesions. RCTs were excluded if they 1) compared the same type of adhesive, 2) compared the association among different adhesives in the same restorations, or 3) compared filled vs nanofilled adhesives.

### Study Selection and Data Collection Process

After database screening, duplicates were removed and possible eligible articles were selected according to title and abstracts. Full-text articles were obtained by two authors (JLG and BMM), and they were classified according to the inclusion criteria. Pilot-tested, customized extraction forms were used to register details about the studies, such as study design, participants, interventions, and outcomes. Each study received an identification number (study ID), combining the first author name and the publication year. Authors were not contacted for further information to avoid recall bias.

### Data Items

When there were multiple reports of the same study (ie, reports with different follow-ups), data from all reports were extracted directly into a single data

Table 1: *Electronic Database and Search Strategy*

PubMed, March 22, 2018: 2980			
<p>#1 (tooth erosion[MeSH Terms] OR tooth abrasion[MeSH Terms] OR tooth cervix[MeSH Terms] OR “cervical lesion”[Title/Abstract]) OR “cervical lesions”[Title/Abstract]) OR “class V”[Title/Abstract] OR “class 5”[Title/Abstract] OR abfraction[Title/Abstract] OR “tooth cervix”[Title/Abstract])</p>	<p>#2 (dentin-bonding agents[mh:noexp]) OR “adhesive system”[Title/Abstract] OR “adhesive systems”[Title/Abstract] OR “bonding agent”[Title/Abstract] OR “bonding agents”[Title/Abstract] OR “dental adhesive”[Title/Abstract] OR “dental adhesives”[Title/Abstract] OR “dentin bonding agent”[Title/Abstract] OR “dentin bonding agents”[Title/Abstract] OR “adhesive material”[Title/Abstract] OR “adhesive materials”[Title/Abstract] OR “etch-and-rinse adhesive”[Title/Abstract] OR “etch-and-rinse adhesives”[Title/Abstract] OR “total-etch adhesive”[Title/Abstract] OR “total-etch adhesives”[Title/Abstract] OR “self-etch adhesive”[Title/Abstract] OR “self-etch adhesives”[Title/Abstract] OR “self-etching adhesive”[Title/Abstract] OR “self-etching adhesives”[Title/Abstract] OR “all-in-one adhesive”[Title/Abstract] OR “all-in-one adhesives”[Title/Abstract] OR “one-bottle adhesive”[Title/Abstract] OR “one-bottle adhesives”[Title/Abstract] OR “filled adhesive” [Title/Abstract] OR “unfilled adhesive” [Title/Abstract])</p>	<p>#3 (dental restoration, permanent[MeSH Terms] OR composite resins[MeSH Terms] OR “resin composite”[Title/Abstract] OR “resin composites”[Title/Abstract] OR “composite resin”[Title/Abstract] OR “composite resins”[Title/Abstract] OR “resin restoration”[Title/Abstract] OR “resin restorations”[Title/Abstract] OR “composite restoration”[Title/Abstract] OR “composite restorations”[Title/Abstract])</p>	<p>#4 (randomized controlled trial[pt] OR controlled clinical trial[pt] OR randomized controlled trials[mh] OR random allocation[mh] OR double-blind method[mh] OR single-blind method[mh] OR clinical trial[pt] OR clinical trials[mh] OR (“clinical trial”[tw] OR ((singl*[tw] OR doubl*[tw] OR trebl*[tw] OR tripl*[tw]) AND (mask*[tw] OR blind*[tw]))) OR (placebos[mh] OR placebo*[tw] OR random*[tw] OR research design[mh:noexp] OR comparative study[pt] OR evaluation studies as topic[mh] OR follow-up studies[mh] OR prospective studies[mh] OR control*[tw] OR prospective*[tw] OR volunteer*[tw]) NOT (animals[mh] NOT humans[mh]))</p>
<b>Scopus: March 22, 2018: 742</b>			
<p>#1 ( TITLE-ABS-KEY ( “tooth erosion” ) OR TITLE-ABS-KEY ( “tooth abrasion” ) OR TITLE-ABS-KEY ( “tooth cervix” ) OR TITLE-ABS-KEY ( “cervical lesion” ) OR TITLE-ABS-KEY ( “class V” ) OR TITLE-ABS-KEY ( “class 5” ) OR TITLE-ABS-KEY ( abfraction ) )</p>	<p>#2 TITLE-ABS-KEY(“adhesive system”) OR TITLE-ABS-KEY(“bonding agent”) OR TITLE-ABS-KEY(“dental adhesive”) OR TITLE-ABS-KEY(“adhesive material”) OR TITLE-ABS-KEY(“etch-and-rinse”) OR TITLE-ABS-KEY(“total-etch”) OR TITLE-ABS-KEY(“self-etch”) OR TITLE-ABS-KEY(“all-in-one”) OR TITLE-ABS-KEY(“one-bottle”) OR TITLE-ABS-KEY(“filled adhesive”) OR TITLE-ABS-KEY(“unfilled adhesive”)</p>	<p>#3 TITLE-ABS-KEY(“composite resin”) OR TITLE-ABS-KEY(“resin composite”) OR TITLE-ABS-KEY(“resin restoration”) OR TITLE-ABS-KEY(“composite restoration”)OR TITLE-ABS-KEY ( “dental restoration” ) AND ( LIMIT-TO ( SUBJAREA , “DENT” ) )</p>	
Web of Science search: March 22, 2018: 515			
<p><b>Tópico:</b> (“tooth erosion”)  <b>ORTópico:</b> (“tooth abrasion”)  <b>ORTópico:</b> (“tooth cervix”)  <b>ORTópico:</b> (“cervical lesion”)  <b>ORTópico:</b> (“class V”)  <b>ORTópico:</b> (“class 5”)  <b>ORTópico:</b> (abfraction)</p>	<p><b>#2Topic:</b> (“adhesive system”)  OR Topic: (“bonding agent”) OR  Topic: (“dental adhesive”) OR  Topic: (“dentin bonding”) OR  Topic: (“adhesive material”) OR  Topic: (“etch and rinse”) OR  Topic: (“total etch”) OR Topic:  (“self etch”) OR Topic: (“all in  one”) OR Topic: (“one bottle”)  OR Topic: (“filled adhesive”)</p>	<p><b>#3Topic:</b> (“resin composite”)  <b>ORTópico:</b> (“dental  restoration”)OR Topic:  (“composite resin”) OR Topic:  (“resin restoration”) OR Topic:  (“composite restoration”)</p>	



Table 1: *Electronic Database and Search Strategy (cont.)*

PubMed, March 22, 2018: 2980		
#1 AND #2 AND #3		
Lilacs and BBO: March 22, 2018: 358		
<p>(MH:"tooth erosion" OR MH:"tooth abrasion" OR MH:"tooth cervix" OR "cervical lesion" OR "lesão cervical" OR "lesión cervical" OR "cervical lesions" OR "lesões cervicais" OR "lesiones cervicales" OR "class V" OR "classe V" OR "clase V" OR "class 5" OR "classe 5" OR "clase 5" OR abfraction OR "abfração" OR "abfracción")</p>	<p>#2(MH:"dentin-bonding agents" OR "adhesive system" OR "adhesive systems" OR "sistema adesivo" OR "sistemas adesivos" OR "sistema adhesivo" OR "sistemas adhesivos" OR "bonding agent" OR "bonding agents" OR "agentes de união" OR "agentes de unión" OR "agentes de ligación" OR "agentes de enlace" OR "dental adhesive" OR "dental adhesives" OR "adesivo dental" OR "adhesivo dental" OR "adesivos dentaís" OR "adhesivos dentaies" OR "adhesive material" OR "material adesivo" OR "material adhesivo" OR "adhesive materials" OR "materiais adesivos" OR "materiales adhesivos" OR "adesivo dentinário" OR "adesivos dentinários" OR "adhesives dentinarios" OR "adhesive material" OR "adhesive materials" OR "dentin bonding agent" OR "dentin bonding agents" OR "etch-and- rinse adhesive" OR "etch-and- rinse adhesives" OR "adesivo convencional" OR "adesivos convencionais" OR "adhesive convencional" OR "adhesives convencionales" OR "total-etch adhesive" OR "total-etch adhesives" OR "condicionamento ácido total" OR "adesivo de grabado total" OR "adhesivos de grabado total" OR "self-etch adhesive" OR "self-etch adhesives" OR "adesivo autocondicionante" OR "adesivos autocondicionantes" OR "adhesive autograbado" OR "adhesives autograbados" OR "self-etching adhesive" OR "self- etching adhesives" OR "all-in-one adhesive" OR "all-in-one adhesives" OR "adesivo de passo único" OR "adesivos de passo único" OR "adesivo de passo unico" OR "adhesivos de passo unico" OR "one-bottle adhesive" OR "one-bottle adhesives" OR "adesivo de frasco único" OR "adesivos de frasco único" OR "filled adhesive" OR "unfilled adhesive" OR "filled adhesives" OR "unfilled adhesives")</p>	<p>#3 (MH: "composite resins" OR MH:"dental restoration, permanent" OR "resin composite" OR "resin composites" OR "resina composta" OR "resinas compostas" OR "resina compuesta" OR "resinas compuestas" OR "composite resin" OR "composite resins" OR "compósito" OR "compósitos" OR "resin restoration" OR "resin restorations" OR "restauração de resina" OR "restauração de resinas" OR "restauración de resina" OR "restauraciones de resina" OR "composite restoration" OR "composite restorations" OR "restauração de compósito" OR "restaurações de compósitos" OR "restauração de resina composta" OR "restaurações de resinas compostas")</p>

Table 1: *Electronic Database and Search Strategy (cont.)*

PubMed, March 22, 2018: 2980		
#1 AND #2 AND #3		
Cochrane Library: March 22, 2018: 286		
#1MeSH descriptor: [Tooth Erosion] explode all trees #2MeSH descriptor: [Tooth Abrasion] explode all trees #3MeSH descriptor: [Tooth Cervix] explode all trees #4cervical next lesion?:ti,ab,kw #5"class V":ti,ab,kw #5"class 5":ti,ab,kw #7abfraction:ti,ab,kw #8tooth next cervix:ti,ab,kw #9 tooth next erosion:ti,ab,kw # 10 tooth next abrasion:ti,ab,kw #11 #1 or #2 or #3 or #4 Or #5 or #6 or #7 or #8 or #9 or #10	#12MeSH descriptor: [Dentin-Bonding Agents] #13adhesive next system*:ti,ab,kw #14bonding next agent*:ti,ab,kw #15dental next adhesive*:ti,ab,kw #16dentin bonding agent*:ti,ab,kw #17adhesive next material*:ti,ab,kw #18"etch and rinse":ti,ab,kw #19total next etch*:ti,ab,kw #20"self etch*":ti,ab,kw #21"all in one":ti,ab,kw #22"one bottle":ti,ab,kw	#23*filled adhesive* #24 #12 or #13 or #14 or #15 or #16 or #17 or #18 or #19 or #20 or #21 or #22 or #23 #25MeSH descriptor: [Composite Resins] #26MeSH descriptor: [Dental Restoration, Permanent] #27resin near composite*:ti,ab,kw #28composite next resin* #29resin near restoration* #30composite next restoration*:ti,ab,kw #31#25 or #26 or #27 or #28 or #29 or #30 #32#11 and #24 and #31

collection form to avoid overlapping data. We collected data about retention rates, marginal discoloration, and secondary caries. Usually, clinical studies on restorative materials use USPHS criteria, which are classified as Alpha, Bravo, and Charlie. We dichotomized the ordinal data into Alpha+Bravo/Charlie. For clinical studies using World Dental Federation criteria, the ordinal data were dichotomized as clinically acceptable or clinically unacceptable. The data were collected into different follow-up evaluations: 12 to 18 months, 24 to 30 months, 3 years, and 5 years or longer. When more than one adhesive of each type was included in the study, their values were combined to make a single entry. In the case of data inconsistencies between reports of different follow-up evaluations of the same study, data were collected from the most recent article. Subgroup analysis based on the type of filler (regular or nanofillers) was performed whenever data were available.

**Risk of Bias in Individual Studies**

Two authors (JLG and BMM) independently assessed the risk of bias of the studies selected using the Cochrane Collaboration’s tool for assessing risk of bias in randomized trials.<sup>47</sup> The risk of bias tool contains six domains: sequence generation, allocation concealment, blinding of the outcome assessors, incomplete outcome data, selective outcome reporting, and other possible sources of bias. Each domain was judged to be at low, unclear, or high risk of bias according to the Cochrane Handbook for Systematic Reviews of Interventions 5.1.0 (<http://handbook.cochrane.org>).

The key domains of this study were sequence generation, allocation concealment, and examiner blinding. At the study level, the study was at low risk of bias if all key domains were at low risk of bias. If one key domain was judged as having high risk of bias, the study was considered as having a high risk of bias. If at least one key domain was judged as at unclear risk among other low-risk of bias domains, the study was considered as having unclear risk of bias. During data selection and quality assessment, any disagreements between the reviewers were solved through discussion and if needed by consulting a third reviewer (ADL).

**Summary Measures and Synthesis of the Results**

Dichotomous data (loss of retention, marginal discoloration, and secondary caries) were meta-analyzed to obtain a combined estimate of the overall risk difference (RD) with a 95% confidence interval. This procedure was done in different follow-ups: 12 to 18 months, 24 to 30 months, 3 years, and 5 years or longer. Subgroup analysis based on the type of filler (microfillers or nanofillers) was performed whenever data were available in each follow-up. Random effect models were used for all meta-analyses, and we assessed heterogeneity (which represents any kind of variability among studies) by using the Cochran Q test and I<sup>2</sup> statistics. We carried out the analyses by using the software RevMan 5.3 (Review Manager version 5, The Cochrane Collaboration, Copenhagen, Denmark).

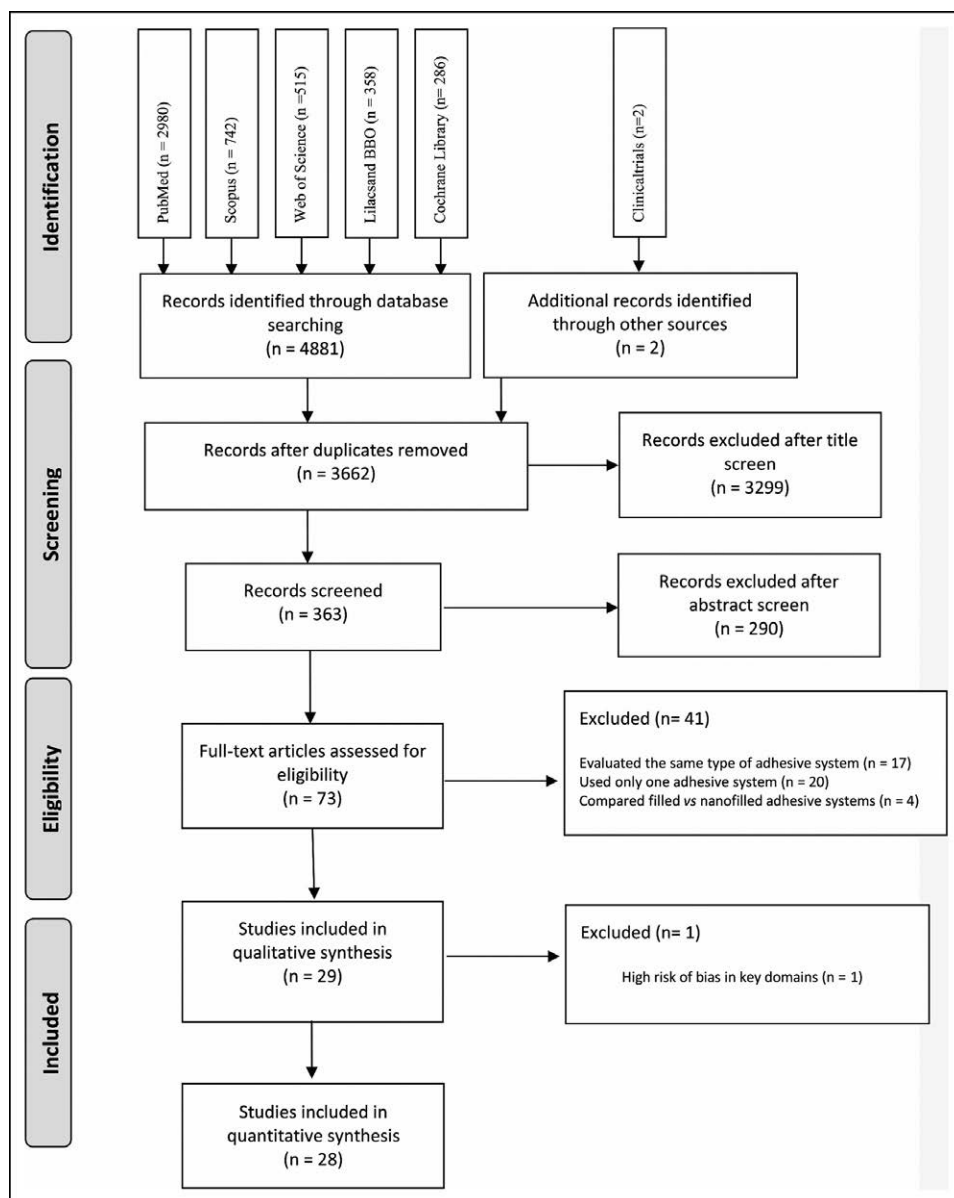


Figure 1. Flowchart diagram showing the number of articles obtained in the different phases of the study.

In case of heterogeneity, a sensitivity analysis was performed.

### Assessment of the Quality of Evidence Using GRADE

The quality of the evidence was graded for each outcome variable across studies (body of evidence) using the Grading of Recommendations: Assessment, Development and Evaluation (GRADE; <http://www.gradeworkinggroup.org/>) to determine the overall strength of evidence. The GRADE approach is used to contextualize or justify

intervention recommendations with four levels of evidence quality, ranging from high to very low.

The GRADE approach begins with the study design (RCTs or observational studies) and then addresses five reasons (risk of bias, imprecision, inconsistency, indirectness of evidence, and publication bias) to possibly rate down the quality of evidence (one or two levels) and three to possibly rate up the quality (large effect, management of confounding factors, dose-response gradient).<sup>48</sup> Each one of these topics was assessed as “no limitation,” “serious limitations,” or “very serious limitations” to allow categorization of the quality of the evidence for

Table 2: Summary of the Studies Included in the Systematic Review

Study ID	Follow-up, mo	Study Design	Subjects' Age, Mean $\pm$ SD [range], y	Total Number of Subjects [Male]	Number of Restorations per Group at Baseline
Abdalla and Garcia-Godoy <sup>105</sup>	12 and 24	Multiple restorations	n.r. $\pm$ n.r. [35-52]	48 [n.r.]	AB – 65 CSE – 65 HB – 65
Aw and others <sup>94</sup>	6, 12, 24, and 36	Multiple restorations	51 $\pm$ n.r. [29-75]	57 [n.r.]	SB – 47 SM – 51 OCB – 48
Boushell and others <sup>95</sup>	6, 18, 36, and 72	Multiple restorations	55.4 $\pm$ 9.5 [30-75]	39 [13]	XIII – 39 XIV – 40 XP – 41
Burrow and Tyas <sup>96</sup>	6, 12, 24, and 36	Multiple restorations	61 $\pm$ n.r. [n.r.-n.r.]	20 [n.r.]	SB – 30 CSE – 31
Eliguzeloglu, Dalkilic, and Omurlu <sup>43</sup>	3, 12, and 24	Multiple restorations	n.r. $\pm$ n.r. [30-70]	29 [16]	SB – 60 CSE – 102 XIII – 90
De Araújo and others <sup>91</sup>	6 and 12	Multiple restorations	n.r. $\pm$ n.r. [23-54]	17 [n.r.]	SM – 31 EO – 31
Dutra-Correa and others <sup>97</sup>	6 and 18	Multiple restorations	48.7 $\pm$ n.r. [27-79]	37 [n.r.]	XV – 30 XP – 30
Hafer and others <sup>98</sup>	6, 12, 24, and 36	Multiple restorations	46.7 $\pm$ 14.1 [18-66]	40 [n.r.]	FM – 40 SoM – 40 SC – 30
Hansen and others <sup>37,38</sup>	36, 48, and 60	Multiple restorations	n.r. $\pm$ n.r. [n.r. $\pm$ n.r.]	n.r. [n.r.]	G – 75 SM – 30
Horsted-Bindslev and others <sup>36</sup>	6, 12, 18, and 24	Multiple restorations	n.r. $\pm$ n.r. [n.r. $\pm$ n.r.]	10 [n.r.]	G – 26 SM – 26
Jang and others <sup>45</sup>	6, 12, 18, and 24	Multiple restorations	55 $\pm$ n.r. [30-73]	35 [n.r.]	CSE – 83 XV – 81
Jordan and Suzuki <sup>92</sup>	6 and 12	Multiple restorations	n.r. $\pm$ n.r. [n.r. $\pm$ n.r.]	n.r. [n.r.]	G2000 – 95 T – 115 PUB 3 – 100 AB2 – 101
Kubo and others <sup>99</sup>	12, 24, 36, 48, and 60	Multiple restorations	61.3 $\pm$ n.r. [45-78]	8 [4]	CLB – 36 SB – 35
Kurokawa and others <sup>93</sup>	3, 6, and 12	Multiple restorations	46 $\pm$ n.r. [31-82]	46 [20]	APL – 21 AQ – 21 GB – 14 OBF – 18
Lawson and others <sup>106</sup>	6, 12, and 24	Multiple restorations	n.r. $\pm$ n.r. [n.r. $\pm$ n.r.]	37 [n.r.]	SM – 42 SU – 84
Matis and others <sup>100</sup>	6, 12, and 36	Multiple restorations	45 $\pm$ n.r. [30-75]	30 [12]	FL – 40 SM – 40
Neo and others <sup>42</sup>	18	Multiple restorations	47 $\pm$ n.r. [n.r.-n.r.]	10 [4]	PUB 3 – 21 IB – 20
Pena and others <sup>102</sup>	3, 6, 12, 18, and 24	Multiple restorations	n.r. $\pm$ n.r. [n.r. $\pm$ n.r.]	25 [13]	CSE – 56 XV – 56
Perdigão and others <sup>89</sup>	6 and 18	Multiple restorations	n.r. $\pm$ n.r. [24-63]	35 [16]	PBNT – 63 SB – 65
Perdigão and others <sup>44</sup>	6 and 18	Multiple restorations	47.6 $\pm$ n.r. [22-78]	39 [n.r.]	SM – 29 SSE – 30 SBP – 32 EB – 34



Table 2: Summary of the Studies Included in the Systematic Review (ext.)

Rubber Dam?	Mechanical Preparation?	Materials [Type of Particles]	Type of Adhesive
Yes	No	Admira Bond <sup>a</sup> – AB [FI] Clearfil SE Bond <sup>b</sup> – CSE [NA] Hybrid Bond <sup>c</sup> – HB [UN]	AB – two-step etch and rinse CSE – two-step self-etch HB – one-step self-etch
No	Bevel	Single Bond <sup>d</sup> – SB [UN] Scotchbond Multipurpose <sup>d</sup> – SM [UN] One Coat Bond <sup>e</sup> – OCB [FI]	SB – two-step etch and rinse SM: three-step etch and rinse OCB two-step etch and rinse
No	No	Xeno III <sup>f</sup> – XIII [UN] Xeno IV <sup>f</sup> – XIV [UN] XP Bond <sup>f</sup> – XP [NA]	XIII – one-step self-etch XIV – one-step self-etch XP – two-step etch and rinse
n.r.	n.r.	Single Bond <sup>d</sup> – SB [UN] Clearfil SE Bond <sup>b</sup> – CSE [NA]	SB – two-step etch and rinse CSE – two-step self-etch
No	No	Single Bond <sup>d</sup> – SB [UN] Clearfil SE Bond <sup>b</sup> – CSE [NA] Xeno III <sup>f</sup> – XIII [UN]	SB – two-step etch and rinse CSE – two-step self-etch XIII – one-step self-etch
No	No	Scotchbond Multipurpose <sup>d</sup> – SM [UN] Easy One <sup>d</sup> – EO [NA]	SM – three-step etch and rinse EO – one-step self-etch
No	No	Xeno V <sup>f</sup> – XV [UN] XP Bond <sup>f</sup> – XP [NA]	XV – one-step self-etch XP – two-step etch-and-rinse
Yes	No	Futurabond M <sup>a</sup> – FM [NA] Solobond M <sup>a</sup> – SoM [UN] Syntac Classic <sup>g</sup> – SC [UN]	FM – one-step self-etch SoM – two-step etch and rinse SC – four-step etch and rinse
No	Bevel	Gluma <sup>h</sup> – G [FI] Scotchbond Multipurpose <sup>d</sup> – SM [UN]	G – two-step self-etch SM – three-step etch and rinse
n.r.	n.r.	Gluma <sup>h</sup> – G [FI] Scotchbond Multipurpose <sup>d</sup> – SM [UN]	G – two-step self-etch SM – three-step etch and rinse
No	No	Clearfil SE Bond <sup>b</sup> – CSE [NA] Xeno V <sup>f</sup> – XV [UN]	CSE – two-step self-etch XV – one-step self-etch
Yes	n.r.	Gluma 2000 <sup>i</sup> – G2000 [FI] Tenure <sup>j</sup> – T [FI] Prisma Universal Bond 3 <sup>f</sup> – PUB3 [UN] AllBond 2 <sup>k</sup> – AB2 [UN]	G2000 – two-step etch and rinse T – two-step self-etch PUB 3 – two-step etch and rinse AB2 – three-step etch and rinse
No	Bevel	Clearfil Liner Bond II <sup>b</sup> – CLB [FI] Single Bond <sup>d</sup> – SB [UN]	CLB – two-step self-etch SB – two-step etch and rinse
No	No	Adper Prompt L-Pop <sup>d</sup> – APL [UN] AQ bond plus <sup>c</sup> – AQ [UN] G Bond <sup>l</sup> – GB [NA] One-up Bond F Plus <sup>m</sup> – OBF [FI]	APL – one-step self-etch AQ – one-step self-etch GB – one-step self-etch OBF – one-step self-etch
Yes	n.r.	Scotchbond Multipurpose <sup>d</sup> – SM [UN] Scotchbond Universal <sup>d</sup> – SM [NA]	SM – three-step etch and rinse SU – one-step self-etch or two-step etch and rinse
Yes	No	FL Bond <sup>n</sup> – FL [FI] Scotchbond Multipurpose <sup>d</sup> – SM [UN]	FL – two-step self-etch SM – three-step etch and rinse
No	No	Prisma Universal Bond 3 <sup>f</sup> – PUB3 [UN] Imperva Bond <sup>n</sup> – IB [FI]	PUB 3 – two-step self-etch IB – three-step etch and rinse
No	Bevel	Clearfil SE Bond <sup>b</sup> – CSE [NA] Xeno V <sup>f</sup> – XV [UN]	CSE – two-step self-etch XV – one-step self-etch
Yes or No	No	Prime & Bond NT <sup>f</sup> – PBNT [NA] Single Bond <sup>d</sup> – SB [UN]	PBNT – two-step etch and rinse SB – two-step etch and rinse
No	No	Scotchbond Multipurpose <sup>d</sup> – SM [UN] Scotchbond SE <sup>d</sup> – SSE [NA] Single Bond Plus <sup>d</sup> – SBP [NA] Easy-Bond <sup>d</sup> – EB [NA]	SM – three-step each and rinse SSE – two-step self-etch SBP – two-step etch and rinse EB – one-step self-etch

Table 2: Summary of the Studies Included in the Systematic Review (cont.)

Study ID	Follow-up, mo	Study Design	Subjects' Age, Mean $\pm$ SD [range], y	Total Number of Subjects [Male]	Number of Restorations per Group at Baseline
Ritter and others <sup>90</sup> / Swift and others <sup>114</sup>	6, 18, 36, and 96	Multiple restorations	53 $\pm$ 12.4 [27-77]	33 [19]	OS – 48 PB – 51
Sartori and others <sup>103</sup>	6, 18, and 30	Multiple restorations	n.r. $\pm$ n.r. [n.r. $\pm$ n.r.]	27 [n.r.]	FNR – 30 SoM – 33
Stojanac and others <sup>104</sup>	12 and 24	Multiple restorations	n.r. $\pm$ n.r. [18-50]	30 [n.r.]	PBNT – 30 A – 30 XIII – 30
Turkun <sup>101</sup>	3, 6, 9, and 12	Multiple restorations	44 $\pm$ n.r. [26-59]	35 [16]	CPB – 85 XIII – 78
Tyas <sup>109</sup>	3, 6, 12, 24, and 36	Multiple restorations	n.r. $\pm$ n.r. [n.r. $\pm$ n.r.]	36 [n.r.]	G – 20 SM – 20 PUB – 20
Van Dijken <sup>115</sup>	6, 12, 18, 24, 30, 36, 42, and 48	Multiple restorations	56.4 $\pm$ n.r. [26-82]	81 [44]	T – 47 Tri – 53 S2 – 53
Van Dijken <sup>41</sup>	6, 12, 18, and 24	Multiple restorations	58 $\pm$ n.r. [46-72]	90 [51]	CLB – 46 OCB – 46 APL – 52
Van Meerbeek and others <sup>107</sup>	6, 12, and 24	Multiple restorations	n.r. $\pm$ n.r. [n.r. $\pm$ n.r.]	35 [n.r.]	T – 32 Tri – 40
Van Meerbeek and others <sup>108</sup>	6, 12, 24, and 36	Multiple restorations	n.r. $\pm$ n.r. [20-79]	125 [n.r.]	G2000 – 103 CLB – 110 SM – 107

Abbreviations: FI, filled adhesive system; ID, identification; NA, nanofilled adhesive system; n.a., not applicable; n.r., not reported in the study; SD, standard deviation; UN, unfilled adhesive system.

<sup>a</sup> Voco, Cuxhaven, Germany.

<sup>b</sup> Kuraray Medical, Tokyo, Japan.

<sup>c</sup> Sun Medical, Moriyama City, Chiga, Japan.

<sup>d</sup> 3M Oral Care, St Paul, MN, USA.

<sup>e</sup> Coltène Whaledent, Cuyahoga Falls, OH, USA.

<sup>f</sup> Dentsply Sirona, York, PA, USA.

<sup>g</sup> Ivoclar Vivadent, Schaan, Liechtenstein.

<sup>h</sup> Bayer, Leverkusen, Germany.

<sup>i</sup> Columbus Dental, St Louis, MO, USA.

<sup>j</sup> DenMat Corp., Santa Maria, CA, USA.

<sup>k</sup> Bisco Inc. Schaumburg, IL, USA.

<sup>l</sup> GC Corp., Tokyo, Japan.

<sup>m</sup> Tokuyama Dental, Tokyo, Japan.

<sup>n</sup> Shofu Inc., Kyoto, Japan.

<sup>o</sup> Kerr, Orange, CA, USA.

<sup>p</sup> ICI Dental, Macclesfield, UK.

each outcome into high, moderate, low, and very low. The “high-quality” level suggests that we are very confident that the true effect lies close to the estimate of the effect. On the other extreme, a study of “very low quality” suggests that we have very little confidence in the effect estimate and the estimate reported can be substantially different from what was measured.

## RESULTS

### Characteristics of Included Studies

After the database screening and removal of duplicates, 3662 articles were identified (Figure 1). After title screening, 363 articles remained, and this number was reduced to 75 articles after careful examination of the abstracts (Figure 1). Among these articles, 41 were excluded for the following reasons:

Table 2: Summary of the Studies Included in the Systematic Review (ext.)

Rubber Dam?	Mechanical Preparation?	Materials [Type of Particles]	Type of Adhesive
No	No	Optibond Solo <sup>o</sup> – OS [FI] Prime & Bond <sup>f</sup> – PB [UN]	OS – two-step etch and rinse PB – two-step etch and rinse
No	n.r.	Futurabond NR <sup>a</sup> – FNR [NA] Solobond M <sup>a</sup> – SoM [UN]	FNR – one-step self-etch SoM – two-step etch and rinse
No	No	Prime & Bond NT <sup>f</sup> – PBNT [NA] AdheSE <sup>g</sup> – A [NA] Xeno III <sup>f</sup> – XIII [UN]	PBNT – two-step etch and rinse A – two-step self-etch XIII – one-step self-etch
No	No	Clearfil Protect Bond <sup>b</sup> – CPB [NA] Xeno III <sup>f</sup> – XIII [UN]	CPB – two-step self-etch XIII – one-step self-etch
n.r.	n.r.	Gluma <sup>h</sup> – G [FI] Scotchbond Multipurpose <sup>d</sup> – SM [UN] Prisma Universal Bond <sup>f</sup> – PUB [UN]	G – two-step etch and rinse SM – three-step etch and rinse PUB – two-step etch and rinse
No	No	Tenure <sup>j</sup> – T [FI] Tripton <sup>o</sup> – Tri [UN] Scotchbond 2 <sup>d</sup> – S2 [UN]	T – two-step self-etch Tri – two-step self-etch S2 – two-step self-etch
n.r.	No	Clearfil Liner Bond II <sup>b</sup> – CLB [FI] One Coat Bond <sup>e</sup> – OCB [FI] Adper Prompt L-Pop <sup>d</sup> – APL [UN]	CLB – two-step self-etch OCB – two-step etch-and-rinse APL – one-step self-etch
Yes	Bevel	Tenure <sup>j</sup> – T [FI] Tripton <sup>o</sup> – Tri [UN]	T – two-step self-etch Tri – two-step self-etch
Yes	With or without bevel	Gluma 2000 <sup>i</sup> – G2000 [FI] Clearfil Liner Bond II <sup>b</sup> – CLB [FI] Scotchbond Multipurpose <sup>d</sup> – SM [UN]	G2000 – two-step self-etch CLB – two-step self-etch SM – three-step etch and rinse

1) the studies compared the same type of adhesive system (n=19),<sup>40,49-66</sup> 2) the studies used the same type of adhesive system in both study groups (n=18),<sup>67-84</sup> and 3) the studies compared filled vs nanofilled adhesive systems (n=4).<sup>85-88</sup>

A total of 32 articles remained for qualitative evaluation. From these 32 articles, 3 articles<sup>38,89,90</sup> reported longer follow-ups of earlier studies. Therefore, there were 29 studies among 32 publications. Tables 2, 3, and 4 characterize the 29 included studies. The follow-up time of the studies varied from 12 months<sup>91-93</sup> to 8 years.<sup>90</sup> All studies placed multiple restorations per patient. In this design, any patient could receive as many restorations as possible, depending on the number of available noncarious cervical lesions.

The mean age of the participants was approximately 50 ( $\pm 7$ ) years.<sup>a</sup> Most of the studies (n=17,

<sup>a</sup> References 39, 41, 42, 44, 45, 90, 93-101.

59%) used cotton rolls and a saliva ejector to prevent contamination during the restorative protocol,<sup>b</sup> while 8 studies used a rubber dam.<sup>89,92,98,100,105-108</sup>

In few studies (n=6), the enamel was beveled.<sup>38,94,99,102,107,108</sup> Different types of adhesive systems were used in the studies, varying from three-step etch-and-rinse adhesives (n=11)<sup>c</sup> to one-step self-etch adhesives (n=14).<sup>d</sup> The number of restorations per adhesive system used in the studies evaluated varied from 14 restorations<sup>93</sup> to 189 restorations.<sup>107</sup>

<sup>b</sup> References 38, 39, 42-45, 90, 91, 93-95, 97, 99, 101-104.

<sup>c</sup> References 36, 38, 42, 44, 91, 92, 94, 100, 106, 108, 109.

<sup>d</sup> References 41, 43-45, 91, 93, 95, 97, 98, 101-105.

Table 3: Summary of the Studies Included in the Systematic Review: Part 2

Study ID	Conditioner	Wet-Bonding Adhesion Technique?	Application Under Agitation?
Abdalla and Garcia-Godoy <sup>105</sup>	AB – 36% phosphoric acid <sup>n.s.</sup> CSE – Cleafil SE <sup>primera</sup> HB – n.a.	AB – yes CSE – n.r. HB – n.r.	AB – n.r. CSE – n.r. HB – n.r.
Aw and others <sup>94</sup>	SB – 35% phosphoric acid <sup>n.s.</sup> SM – 35% phosphoric acid <sup>n.s.</sup> OCB – 15% phosphoric acid <sup>n.s.</sup>	SB – yes SM – yes OCB – yes	SB – n.r. SM – n.r. OCB – yes
Boushell and others <sup>95</sup>	XIII – n.a. XIV – n.a. XP – Caulk 34% Conditioner Gel <sup>e</sup>	XIII – yes XIV – yes XP – yes	XIII – n.r. XIV – n.r. XP – n.r.
Burrow and Tyas <sup>96</sup>	SB – n.r. CSE – Cleafil SE primer <sup>a</sup>	SB – yes CSE – yes	SB – n.r. CSE – n.r.
Eliguzeloglu, Dalkilic, and Omurlu <sup>43</sup>	SB – 35% phosphoric acid <sup>f</sup> CSE – Cleafil SE primer <sup>a</sup> or 37% phosphoric acid <sup>n.s.</sup> XIII – n.a. or 37% phosphoric acid <sup>n.s.</sup>	SB – yes CSE – yes XIII – yes	SB – n.r. CSE – n.r. XIII – n.r.
De Araújo and others <sup>91</sup>	SM – 35% phosphoric acid <sup>n.s.</sup> EO – n.a.	SM – yes EO – yes	SM – n.r. EO – n.r.
Dutra-Correa and others <sup>97</sup>	XV – n.a. XP – 36% phosphoric acid <sup>n.s.</sup>	XV – n.r. XP – yes	XV – yes XP – n.r.
Hafer and others <sup>98</sup>	FM – n.a. SoM – 35% phosphoric acid <sup>f</sup> SC – 37% phosphoric acid <sup>g</sup>	FM – yes SoM – yes SC – yes	FM – n.r. SoM – n.r. SC – n.r.
Hansen and others <sup>37, 38</sup>	G – n.r. SM – n.r.	G – n.r. SM – n.r.	G – n.r. SM – n.r.
Horsted-Bindslev and others <sup>36</sup>	G – 35% phosphoric acid <sup>h</sup> SM – 35% phosphoric acid <sup>h</sup>	G – yes SM – yes	G – n.r. SM – n.r.
Jang and others <sup>45</sup>	CSE – Cleafil SE primer <sup>a</sup> XV – n.a.	CSE – n.r. XV – n.r.	CSE – n.r. XV – yes
Jordan and Suzuki <sup>92</sup>	G2000 – n.r. T – n.r. PUB 3 – n.r. AB2 – n.r.	G2000 – n.r. T – n.r. PUB 3 – n.r. AB2 – n.r.	G2000 – n.r. T – n.r. PUB 3 – n.r. AB2 – n.r.
Kubo and others <sup>99</sup>	CLB – 37% phosphoric acid <sup>a</sup> SB – 37% phosphoric acid <sup>a</sup>	CLB – yes SB – n.r.	CLB – n.r. SB – n.r.
Kurokawa and others <sup>93</sup>	APL – n.a. AQ – n.a. GB – n.a. OBF – n.a.	APL – n.r. AQ – n.r. GB – n.r. OBF – n.r.	APL – yes AQ – n.r. GB – n.r. OBF – yes
Lawson and others <sup>106</sup>	SM – 37% phosphoric acid <sup>c</sup> SU – n.a. or 37% phosphoric acid <sup>c</sup>	SM – yes SU – yes	SM – yes SU – yes
Matis and others <sup>100</sup>	FL – n.a. SM – 37% phosphoric acid <sup>n.s.</sup>	FL – yes SM – yes	FL – n.r. SM – n.r.
Neo and others <sup>42</sup>	PUB 3 – n.r. IB – n.r.	PUB 3 – n.r. IB – n.r.	PUB 3 – n.r. IB – n.r.
Pena and others <sup>102</sup>	CSE – Cleafil SE primer <sup>a</sup> XV – n.a.	CSE – n.r. XV – n.r.	CSE – n.r. XV – n.r.
Perdigão and others <sup>89</sup>	PBNT – 34% phosphoric acid <sup>e</sup> SB – 37% phosphoric acid <sup>c</sup>	PBNT – yes or not SB – yes or not	PBNT – n.r. SB – n.r.
Perdigão and others <sup>44</sup>	SM – 35% phosphoric acid <sup>c</sup> SSE – n.a. SBP – 35% phosphoric acid <sup>c</sup> EB – n.a.	SM – n.r. SSE – yes SBP – n.r. EB – yes	SM – n.r. SSE – yes SBP – n.r. EB – n.r.
Ritter and others <sup>90</sup> /Swift and others <sup>114</sup>	OS – 37% phosphoric acid <sup>n.s.</sup> PB – 34% phosphoric acid <sup>n.s.</sup>	OS – yes PB – yes	OS – yes PB – n.r.
Sartori and others <sup>103</sup>	FNR – n.a. SoM – 35% phosphoric acid <sup>f</sup>	FNR – yes SoM – yes	FNR – n.r. SoM – n.r.



Table 3: Summary of the Studies Included in the Systematic Review: Part 2 (ext.)

Time of Evaporation of the Solvent (s)	Type of Solvent	Composite Resin Used	Operator(s) Experience (Graduate, Dentist, or Postgraduate)
AB – 2 - 3 CSE – n.r. HB – 5	AB – acetone CSE – water HB – acetone/water	Clearfil APX <sup>a</sup>	Dentist
SB – 5 SM – 5 OCB – 2	SB – ethanol SM – water OCB – water	SB – Silux Plus <sup>b</sup> SM – Silux Plus <sup>b</sup> OCB – Synergy <sup>c</sup>	n.r.
XIII – 5 XIV – 2 XP – 5	XIII – ethanol XIV – ethanol XP – tert-butanol	TPH <sup>d</sup>	Dentist
SB – n.r. CSE – n.r.	SB – ethanol CSE – water	SB – Filtek A110 <sup>b</sup> CSE – Clearfil ST <sup>a</sup>	n.r.
SB – n.r. CSE – n.r. XIII – n.r.	SB – ethanol CSE – ethanol XIII – water	Filtek Supreme <sup>b</sup>	n.r.
SM – 5 EO – 5	SM – water EO – ethanol/water	Z350 <sup>c</sup>	n.r.
XV – 5 XP – 5	XV – ethanol XP – tert-butanol	Exthet X <sup>d</sup>	n.r.
FM – 5 SoM – n.r. SC – n.r.	FM – water SoM – water/acetone SC – water/acetone	FM – Amaris <sup>h</sup> SoM – Amaris <sup>h</sup> SC – Tetric EvoCeram <sup>f</sup>	n.r.
G – n.r. SM – n.r.	G – ethanol SM – water	Silux Enamel Bond <sup>b</sup>	n.r.
G – n.r. SM – n.r.	G – ethanol SM – water	P-30 <sup>b</sup>	n.r.
CSE – n.r. XV – 5	CSE – water XV – ethanol	Z250 <sup>b</sup>	n.r.
G2000 – n.r. T – n.r. PUB 3 – n.r. AB2 – n.r.	G2000 – ethanol T – acetone PUB 3 – ethanol AB2 – acetone	G2000 – Pekafile <sup>i</sup> T – Marathon <sup>i</sup> PUB 3 – Prisma APH <sup>d</sup> AB2 – Bisfil M <sup>k</sup>	n.r.
CLB – n.r. SB – n.r.	CLB – water SB – ethanol	Clearfil APX <sup>a</sup>	Dentist
APL – n.r. AQ – n.r. GB – n.r. OBF – n.r.	APL – water AQ – water/acetone GB – water OBF – water	APL – Filtek Supreme <sup>b</sup> AQ – Metafil C <sup>n</sup> GB – Gradia Direct <sup>l</sup> OBF – Palfique Estelite <sup>o</sup>	n.r.
SM – 5 SU – 5	SM – water SU – water/ethanol	Filtek Supreme Ultra <sup>b</sup>	Dentist
FL – 10 SM – 5	FL – water SM – water	FL – Beautifil <sup>p</sup> SM – Silux Plus <sup>b</sup>	n.r.
PUB 3 – n.r. IB – n.r.	PUB 3 – ethanol IB – water/ethanol	PUB 3 – APH <sup>d</sup> IB – Lite Fil II <sup>p</sup>	n.r.
CSE – n.r. XV – n.r.	CSE – water XV – ethanol	Esthet X <sup>d</sup>	Dentist
PBNT – 5 SB – n.r.	PBNT – acetone SB – ethanol	Filtek A110 <sup>b</sup>	Dentist
SM – 5 SSE – 5 SBP – 10 EB – 5	SM – water SSE – ethanol SBP – water EB – water/ethanol	Filtek Supreme Plus <sup>b</sup>	n.r.
OS – n.r. PB – 5	OS – ethanol PB – acetone	OS – Prodigy <sup>m</sup> PB – TPH Spectrum <sup>d</sup>	Dentist
FNR – 5 SoM – 5	FNR – water SoM – water/acetone	Polofil M <sup>h</sup>	Graduate

Table 3: Summary of the Studies Included in the Systematic Review: Part 2 (Cont.)

Study ID	Conditioner	Wet-Bonding Adhesion Technique?	Application Under Agitation?
Stojanac and others <sup>104</sup>	PBNT – 36% orthophosphoric acid <sup>e</sup> A – AdheSE primer <sup>d</sup> XIII – n.a.	PBNT – yes A – yes XIII – yes	PBNT – n.r. A – n.r. XIII – n.r.
Turkun <sup>101</sup>	CPB – CPB primer <sup>a</sup> XIII – n.a.	CPB – n.r. XIII – n.r.	CPB – n.r. XIII – n.r.
Tyas <sup>109</sup>	G – n.r. SM – n.r. PUB – n.r.	G – n.r. SM – n.r. PUB – n.r.	G – n.r. SM – n.r. PUB – n.r.
Van Dijken <sup>115</sup>	T – n.r. Tri – n.r. S2 – n.r.	T – n.r. Tri – n.r. S2 – n.r.	T – n.r. Tri – n.r. S2 – n.r.
Van Dijken <sup>41</sup>	CLB – CLB primer <sup>a</sup> OCB – 15% phosphoric acid gel <sup>n.s.</sup> APL – n.a.	CLB – n.r. OCB – n.r. APL – n.r.	CLB – n.r. OCB – n.r. APL – yes
Van Meerbeek and others <sup>107</sup>	T – 37% phosphoric acid <sup>c</sup> Tri – 37% phosphoric acid <sup>c</sup>	T – yes Tri – n.r.	T – n.r. Tri – n.r.
Van Meerbeek and others <sup>108</sup>	G2000 – n.r. CLB – n.r. SM – n.r.	G2000 – n.r. CLB – n.r. SM – n.r.	G2000 – n.r. CLB – n.r. SM – n.r.

Abbreviations: ID, identification; n.a., not applicable; n.r., not reported in the study; n.s., not specified.

<sup>a</sup> Kuraray Medical, Tokyo, Japan.  
<sup>b</sup> 3M Oral Care, St Paul, MN, USA.  
<sup>c</sup> Coltène Whaledent, Cuyahoga Falls, OH, USA.  
<sup>d</sup> Dentsply Sirona, York, PA, USA.  
<sup>e</sup> Benlioglu Dental Inc., Ankara, Turkey.  
<sup>f</sup> Ivoclar Vivadent, Schaan, Liechtenstein.  
<sup>g</sup> DMC, Joinville, SC, Brazil.  
<sup>h</sup> Voco, Cuxhaven, Germany.  
<sup>i</sup> Columbus Dental, St Louis, MO, USA.  
<sup>j</sup> DenMat Corp, Santa Maria, CA, USA.  
<sup>k</sup> Bisco Inc, Schaumburg, IL, USA.  
<sup>l</sup> GC Corp, Tokyo, Japan.  
<sup>m</sup> Kerr, Danbury, CT, USA.  
<sup>n</sup> Sun Medical, Moriyama City, Chiga, Japan.  
<sup>o</sup> Tokuyama Dental, Tokyo, Japan.  
<sup>p</sup> Shofu Inc, Kyoto, Japan.  
<sup>q</sup> SDI, Bayswater, Australia.  
<sup>r</sup> Degussa, Düsseldorf, Germany.  
<sup>s</sup> ICI Dental, Macclesfield, UK.

The wet bonding technique was applied in 18 studies.<sup>e</sup> Some studies (n=8) mentioned that the application of the adhesive system was done while the adhesive was actively moved on the surface (agitation).<sup>f</sup> The time to evaporate the solvent was 5 seconds in most studies (n=14).<sup>g</sup> Adhesives were composed of different solvents such as water, ethanol, acetone, and tert-butanol. Most of the studies did not report on the operator experience (graduate, postgraduate, academic dentist, general practitioner), but for those for which this informa-

tion was reported, most of the operators were academic dentists.<sup>89,90,95,99,102,105-108</sup> Only one study reported that the operator was a graduate student.<sup>103</sup> No study was conducted with general practitioners.

### Meta-analysis

A meta-analysis was performed that included all studies with exception of one,<sup>98</sup> which was considered at high risk of bias in the key domain examiner blinding. The risk of bias assessment is provided in Figure 2. Some follow-ups could not be integrated into the meta-analysis because of lack of information. If data were not available or could not be extracted, the study was not considered for the meta-analysis. No difference was observed between the

<sup>e</sup> References 36, 43, 44, 89-91, 94-100, 103-107.

<sup>f</sup> References 41, 44, 45, 90, 93, 94, 97, 106.

<sup>g</sup> References 41, 44, 45, 90, 91, 94, 95, 97, 98, 100, 101, 103, 105, 106.

Table 3: Summary of the Studies Included in the Systematic Review: Part 2 (Cont.)

Time of Evaporation of the Solvent (s)	Type of Solvent	Composite Resin Used	Operator(s) Experience (Graduate, Dentist, or Postgraduate)
PBNT – n.r. A – n.r. XIII – n.r.	PBNT – acetone A – water XIII – ethanol	PBNT – Esthet X <sup>d</sup> A – Tetric EvoCeram <sup>f</sup> XIII – Dyract Extra <sup>d</sup>	n.r.
CPB – 5 XIII – n.r.	CPB – water XIII – ethanol	Esthet X <sup>d</sup>	n.r.
G – n.r. SM – n.r. PUB – n.r.	G – ethanol SM – water PUB – ethanol	G – Lumifor <sup>b</sup> SM – Silux <sup>b</sup> PUB – Prismafine <sup>d</sup>	n.r.
T – n.r. Tri – n.r. S2 – n.r.	T – acetone Tri – water S2 – water	T – Opalux <sup>s</sup> Tri – Opalux <sup>s</sup> S2 – Silux <sup>b</sup>	n.r.
CLB – 3 - 5 OCB – 3 APL – 5	CLB – water OCB – water APL – water	CLB – Clearfil APX <sup>a</sup> OCB – Synergy <sup>c</sup> APL – Pertac Hybrid <sup>b</sup>	n.r.
T – n.r. Tri – n.r.	T – acetone Tri – water	T – Herculite XR <sup>m</sup> Tri – Opalux <sup>l</sup>	Dentist
G2000 – n.r. CLB – n.r. SM – n.r.	G2000 – ethanol CLB – water SM – water	G2000 – Pekafill <sup>b</sup> CLB – Clearfil Photo Anterior <sup>a</sup> SM – Silux Plus <sup>b</sup>	Dentist

subgroup analysis in any of the meta-analyses that had been conducted.

**Loss of Retention**—This analysis was based on 27 studies.<sup>h</sup> In the overall analysis, which took into consideration both subgroups (filled vs unfilled and nanofilled vs unfilled), no significant difference between the two groups was detected in the follow-ups of 12 to 18 months (RD=−0.01; 95% confidence interval [CI], −0.03 to 0.02;  $p=0.60$ ; Figure 3), 24 to 30 months (RD=0.00; 95% CI, −0.03 to 0.03;  $p=0.95$ ; Figure 3), 3 years (RD=−0.04; 95% CI, −0.10 to 0.03;  $p=0.26$ ; Figure 4), and 5 or more years (RD=−0.01; 95% CI, −0.10 to 0.07;  $p=0.77$ ; Figure 4). Analysis of heterogeneity revealed that data were heterogeneous at 12 to 18 months, 24 to 30 months, and 3-year follow-ups ( $p<0.03$ ;  $I^2>45\%$ ; Figures 3 and 4) but not at the 5-year recall ( $p=0.28$ ;  $I^2=21\%$ ; Figure 4).

**Marginal Discoloration**—This analysis was based on 22 studies.<sup>i</sup> In the overall analysis, which took into consideration both subgroups, no significant difference between the two groups was detected in the follow-ups of 12 to 18 months (RD=−0.02; 95% CI, −0.04 to 0.00;  $p=0.07$ ; Figure 5), 24 to 30 months (RD=−0.04; 95% CI, −0.10 to 0.02;  $p=0.18$ ; Figure 5), or 3 years (RD=0.01; 95% CI, −0.06 to 0.09;  $p=0.75$ ; Figure 5). Analysis of heterogeneity revealed that

data were heterogeneous at 12 to 18 months ( $p=0.16$ ;  $I^2=22\%$ ; Figure 5), and 3-year follow-up ( $p=0.84$ ;  $I^2=0\%$ ; Figure 6) but not at the 24 to 30 months recall ( $p<0.0002$ ;  $I^2=69\%$ ; Figure 5).

**Secondary Caries**—This analysis was based on 17 studies.<sup>j</sup> In the overall analysis, which took into consideration both subgroups, no significant difference between groups was detected in the follow-ups of 12 to 18 months (RD=−0.00; 95% CI, −0.01 to 0.01;  $p=0.88$ ; Figure 7), 24 to 30 months (RD=−0.00; 95% CI, −0.02 to 0.01;  $p=0.59$ ; Figure 7), or 3 years (RD=−0.02; 95% CI, −0.06 to 0.01;  $p=0.16$ ; Figure 8). Analysis of heterogeneity revealed that data were not heterogeneous at any given recall time ( $p>0.32$ ;  $I^2<13\%$ ; Figures 7 and 8).

**Assessment of the Quality of Evidence**—In the summary of findings in Table 4, we can observe that for the outcome variable loss of retention, most of the follow-ups were graded as having a low quality of evidence, except for 5 or more year recalls, which were graded as moderate. Unclear risk of bias and unexplained heterogeneity were the reasons for downgrading the level of evidence. For the outcome variable marginal discoloration, the 12- to 18-month recall and the 3-year recall were graded as moderate (unclear risk of bias of the eligible studies) and the 24- to 30-month recall was graded as having a low quality of evidence (unclear risk of bias and

<sup>h</sup> References 36, 38, 39, 41-45, 89-94, 96, 97, 99-109.

<sup>i</sup> References 41-45, 89-94, 96, 97, 100-108.

<sup>j</sup> References 36, 41, 43-45, 89, 90, 93, 97, 100-106, 108.

Outcome	Anticipated Absolute Effects <sup>b</sup> (95% CI)		Relative Effect (95% CI)	No. of Restorations (studies)	Quality of the Evidence (GRADE) <sup>c</sup>
	Filled/Nanofilled Adhesives	Unfilled Adhesives			
Loss of retention (1 year): dichotomous scale (yes/no)	54 per 1000 (–54 to 107)	65 per 1000	RR –0.01 (–0.03 to 0.02)	2801 (25 RCTs)	⊕⊕○○ LOW <sup>d,e</sup>
Loss of retention (2 years): dichotomous scale (yes/no)	78 per 1000 (–234 to 234)	88 per 1000	RD –0.00 (–0.03 to 0.03)	1601 (15 RCTs)	⊕⊕○○ LOW <sup>d,e</sup>
Loss of retention (3 years): dichotomous scale (yes/no)	88 per 1000 (–66 to 220)	166 per 1000	RD –0.04 (–0.10 to 0.03)	759 (7 RCTs)	⊕⊕○○ LOW <sup>d,e</sup>
Loss of retention (5 or more years): dichotomous scale (yes/no)	169 per 1000 (–241 to 1690)	241 per 1000	RD –0.01 (–0.10 to 0.07)	215 (3 RCTs)	⊕⊕⊕○ MODERATE <sup>d</sup>
Marginal discoloration (1 year): dichotomous scale (yes/no)	68 per 1000 (–68 to 136)	104 per 1000	RD –0.02 (–0.04 to 0.00)	2273 (21 RCTs)	⊕⊕⊕○ MODERATE <sup>d</sup>
Marginal discoloration (2 years): dichotomous scale (yes/no)	172 per 1000 (–86 to 430)	223 per 1000	RD –0.04 (–0.10 to 0.02)	1327 (12 RCTs)	⊕⊕○○ LOW <sup>d,e</sup>
Marginal discoloration (3 years): dichotomous scale (yes/no)	306 per 1000 (–1836 to 2754)	302 per 1000	RD 0.01 (–0.06 to 0.09)	516 (4 RCTs)	⊕⊕⊕○ MODERATE <sup>d</sup>
Secondary caries (1 year): dichotomous scale (yes/no)	5 per 1000 (–5 to 10)	7 per 1000	RD –0.00 (–0.01 to 0.01)	1857 (16 RCTs)	⊕⊕⊕○ MODERATE <sup>d</sup>
Secondary caries (2 years): dichotomous scale (yes/no)	2 per 1000 (–2 to 4)	6 per 1000	RD –0.00 (–0.02 to 0.01)	1137 (10 RCTs)	⊕⊕⊕○ MODERATE <sup>d</sup>
Secondary caries (3 years): dichotomous scale (yes/no)	0 per 1000	30 per 1000	RD –0.02 (–0.06 to 0.01)	390 (3 RCTs)	⊕⊕⊕○ MODERATE <sup>d</sup>
<sup>a</sup> Patient or population: noncarious cervical lesions; intervention: filled/nanofilled adhesives; comparison: unfilled adhesives. <sup>b</sup> The risk in the intervention group (and its 95% confidence interval) is based on the assumed risk in the comparison group and the relative effect of the intervention (and its 95% CI). <sup>c</sup> GRADE Workgroup grades of evidence: High quality: We are very confident that the true effect lies close to that of the estimate of the effect. Moderate quality: We are moderately confident in the effect estimate: the true effect is likely to be close to the estimate of the effect, but there is a possibility that it is substantially different. Low quality: Our confidence in the effect estimate is limited: the true effect may be substantially different from the estimate of the effect. Very low quality: We have very little confidence in the effect estimate: the true effect is likely to be substantially different from the estimate of effect. <sup>d</sup> Unclear risk of bias. <sup>e</sup> Unexplained statistical heterogeneity.					

unexplained heterogeneity). All meta-analyses of the outcome variable secondary caries were graded as moderate because of the unclear risk of bias of the studies.

## DISCUSSION

Systematic reviews and meta-analyses are important for resolving controversies between clinical trials and to provide clinical input for guidelines

that address adequate clinical care delivered by oral health personnel, especially general practitioners.<sup>110</sup> According to the results of the present study, the addition of fillers or nanofillers in adhesive systems does not significantly improve the clinical performance of the retention rate, marginal discoloration, or secondary caries.

When the first filled adhesive systems emerged in the market, the theoretical concept was that



	Adequate sequence generation?	Allocation concealment?	Examiner blinding?	Incomplete outcome data addressed?	Free of selective reporting?
Abdalla & Garcia-Godoy <sup>105</sup>	?	?	+	+	+
Aw and others <sup>94</sup>	?	?	+	?	+
Boushell and others <sup>95</sup>	?	?	?	+	+
Burrow & Tyas <sup>96</sup>	?	?	+	+	+
Eliguzeloglu Dalkilic & Omurlu <sup>43</sup>	?	?	+	-	+
De Araújo and others <sup>91</sup>	?	?	+	+	+
Dutra-Correa and others <sup>97</sup>	?	?	+	+	+
Hafer and others <sup>98</sup>	?	?	-	+	+
Hansen and others <sup>37,38</sup>	?	?	?	+	+
Horsted-Blindslev and others <sup>36</sup>	?	?	?	+	+
Jang and others <sup>45</sup>	?	?	+	+	+
Jordan & Suzuki <sup>92</sup>	?	?	?	+	+
Kubo and others <sup>99</sup>	?	?	+	+	+
Kurokawa and others <sup>93</sup>	?	?	+	+	+
Lawson and others <sup>106</sup>	+	?	+	+	+
Matis and others <sup>100</sup>	?	?	?	+	+
Neo and others <sup>42</sup>	?	?	?	+	+
Pena and others <sup>102</sup>	?	?	+	+	+
Perdigão and others <sup>99</sup>	?	?	+	+	+
Perdigão and others <sup>44</sup>	?	?	+	+	+
Ritter and others /Swift and others <sup>90</sup> <sup>114</sup>	+	?	?	+	+
Sartori and others <sup>103</sup>	?	?	+	+	+
Stojanac and others <sup>104</sup>	?	?	+	+	+
Turkun <sup>101</sup>	?	?	+	+	+
Tyas <sup>109</sup>	?	?	?	+	+
Van Dijken <sup>115</sup>	?	?	?	+	+
Van Dijken <sup>41</sup>	?	?	?	+	+
Van Meerbeek and others <sup>107</sup>	?	?	?	+	+
Van Meerbeek and others <sup>108</sup>	?	?	+	+	+

Figure 2. Summary of the risk of bias assessment for the 27 studies included in the meta-analysis according to the Cochrane Collaboration tool. The risk of bias tool contains six domains: sequence generation, allocation concealment, blinding of the outcome assessors, incomplete outcome data, selective outcome reporting, and other possible sources of bias. Each domain was judged to be at low, unclear, or high risk of bias according to the Cochrane Handbook for Systematic Reviews of Interventions 5.1.0

filled adhesive systems act as thickening agents within the adhesive layer. The formation of a thick layer of adhesive interface will improve the mechanical properties, and<sup>15,16</sup> according to the so-called elastic bonding concept,<sup>17,18</sup> the adhesive layer should absorb the compression produced by the tooth-flexure stress, thus reducing interfacial stresses and preserving the marginal integrity,<sup>17,18,111</sup> which eventually should result in better retention rate of the adhesively bonded restorations.<sup>12,13</sup>

There are, however, options to increase the thickness of the adhesive layer: first, to apply two layers of adhesive, and second, to use a separate hydrophobic layer such as the three-step etch-and-rinse or two-step self-etch adhesive systems.<sup>8,112</sup> Some systematic reviews came to the conclusion that simplified adhesive systems such as the one-step self-etch systems reduce the retention rates and increase marginal discoloration of Class V composite resin restorations.<sup>8,113</sup> However, a closer view of the RCTs of the present study showed that although 16 studies evaluated filled vs unfilled adhesives, only a few compared a filled or unfilled adhesive within the same adhesive system group.<sup>k</sup> This prevented us from investigating this variable by a subgroup analysis or meta-regression.

It is worthwhile to mention that flowable composites are also used with the goal of absorbing occlusal stress (“elastic bonding concept”).<sup>17,18</sup> However, several systematic reviews have shown that the use of flowable resin composite compared with high-viscous resin composites did not affect the retention rate or marginal discoloration of Class V restorations.<sup>8,113,116</sup> Microfillers in adhesive systems (1-5 µm) do not penetrate into the interfibrillar spaces but are observed within the adhesive layer.<sup>117</sup> Therefore, there are adhesive systems with glass particles of 20-nm size or lower (pure silicon dioxide, from either colloidal or pyrogenic origin).<sup>26,29</sup> According to the manufacturers, the nanofillers are small enough to penetrate into dentin tubules and infiltrate the interfibrillar spaces of demineralized dentin. Furthermore, it was suggested that infiltration of the interfibrillar channels could provide a strengthening element for demineralized dentin.<sup>16,27</sup>

However, the nanofillers must be physically and chemically stabilized to prevent them from aggregating during storage and/or during the application of the adhesive, which makes these “filler clusters”

<sup>k</sup> References 89, 93, 94, 105, 107-109, 114, 115.

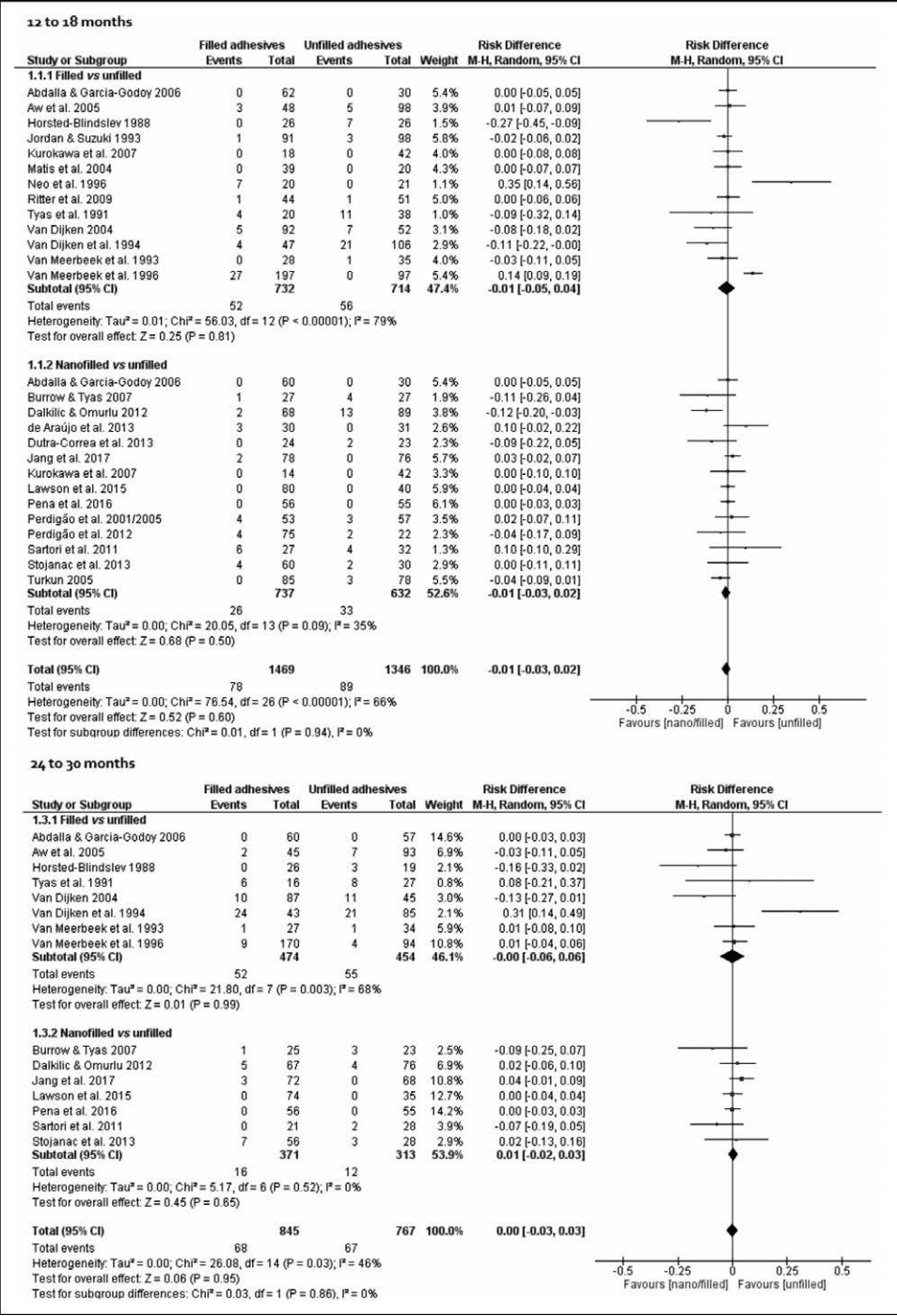


Figure 3. Forest plot of restorations that suffered retention loss comparing filled/nanofilled vs unfilled adhesives at 12 to 18 months and 24 to 30 months.

too large to infiltrate the interfibrillar spaces.<sup>27,118</sup> However, several studies showed that no nanofiller had been found inside the hybrid layer or the demineralized dentin.<sup>27,119,120</sup> Furthermore, it has been reported that exposed collagen may function as a filter<sup>121</sup> that does not allow the nanofillers to penetrate. The molecular weight of the nanofillers

and the resin monomers of the adhesives differ substantially. Therefore, the diffusion rate is very different, which inhibits the complete infiltration of the nanofillers into the interfibrillar space.<sup>27,122</sup> Some authors claim that in demineralizing dentin, there is a formation of a hydrogel of residual substance, proteoglycans, and noncollagenous pro-

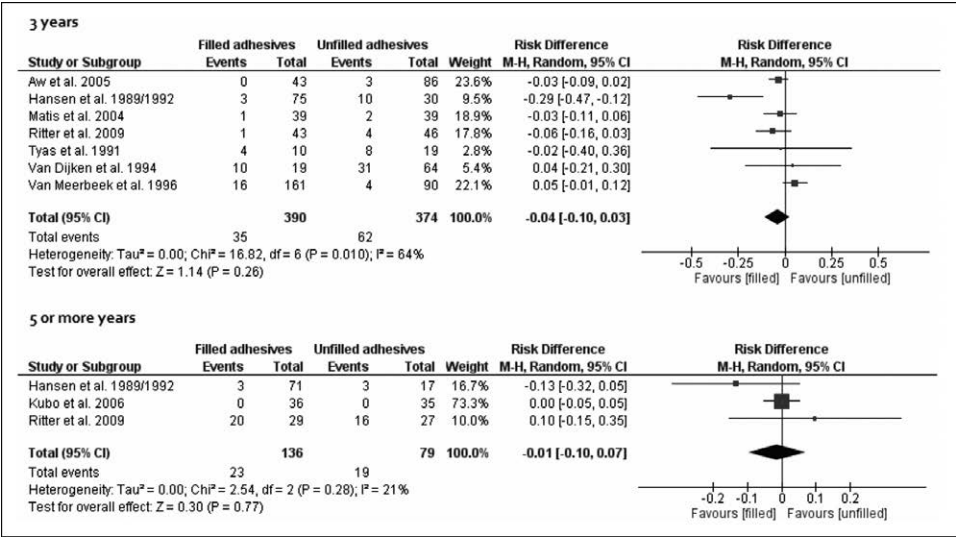


Figure 4. Forest plot of restorations that suffered retention loss comparing filled vs unfilled adhesives at 3 and 5 or more years.

teins that may physically impede the infiltration of nanofillers.<sup>123</sup>

Also due to the natural tendency to aggregate, micrometric electrodense filler clusters will form that are larger than the interfibrillar spaces.<sup>16,27</sup> Osorio and others<sup>119</sup> showed that in self-etch adhesive systems, large clusters were observed that were beyond the dimensions of the interfibrillar spaces of the collagen fibers. Some authors suggested that if the volume of the nanofillers within the adhesive was lower than 3.0 wt%, they did not aggregate that easily and would increase the bond strength to dentin.<sup>124</sup> However, in commercial simplified etch-and-rinse and self-etch adhesives, more than 5 wt% is found.<sup>16</sup> On the other hand, the lower amount of nanofillers did not significantly improve the mechanical properties of the adhesive layer.<sup>20,30,31</sup> Other researchers used specific techniques to produce nonaggregated nanoparticles with high antimicrobial potential.<sup>125,126</sup> These facts may also explain why no significant increase in the bond strength to dentin could be observed when nano-filled-containing simplified adhesives had been tested compared with unfilled simplified adhesives.<sup>32-35</sup>

The results of the present study should be interpreted with caution because they represent an overall comparison without taking into consideration specific variations in the products (monomer and solvent composition, application technique, evaporation solvent time, and moisture control). However, if one of these factors has an important role in the clinical performance of an adhesive, merging studies in a meta-analysis will increase the power to detect the role of such a variable. This

would not be possible in primary studies with low sample sizes.

Also, the inadequate randomization of some clinical studies may have led to the fact that the chances of a patient being allocated to the test or control group were not the same for all patients, and known and unknown prognostic factors had not been balanced out among the groups.<sup>47,110,127</sup> The random sequence should be protected until implementation<sup>127</sup> (allocation concealment). Most of the eligible studies that had been included in this systematic review were classified as having unclear risk of bias. This judgment was based on the lack of clear description of the randomization and allocation concealment process. This is in accordance with what was recently published by Reis and others in 2018,<sup>128</sup> who reported that more than 60% of RCTs about adhesive systems that had been tested in noncarious cervical lesions had a high or unclear risk of bias for randomization and allocation concealment.

Therefore, long-term and well-conducted RCTs that comply with the requirements of an RCT are needed to evaluate possible technological improvements of adhesive systems such as the addition of nanofillers to improve the longevity of the bonding interface to dentin.

CONCLUSIONS

The addition of micro or nanofillers to the composition of adhesive systems did not increase the clinical performance (retention rates, marginal discoloration, or secondary caries) in noncarious cervical lesions compared with unfilled adhesive systems.

Figure 5. Forest plot of restorations with marginal discoloration comparing filled/nanofilled vs unfilled adhesives at 12 to 18 months and 24 to 30 months.

Figure 6. Forest plot of restorations with marginal discoloration comparing filled vs unfilled adhesives at 3 years.



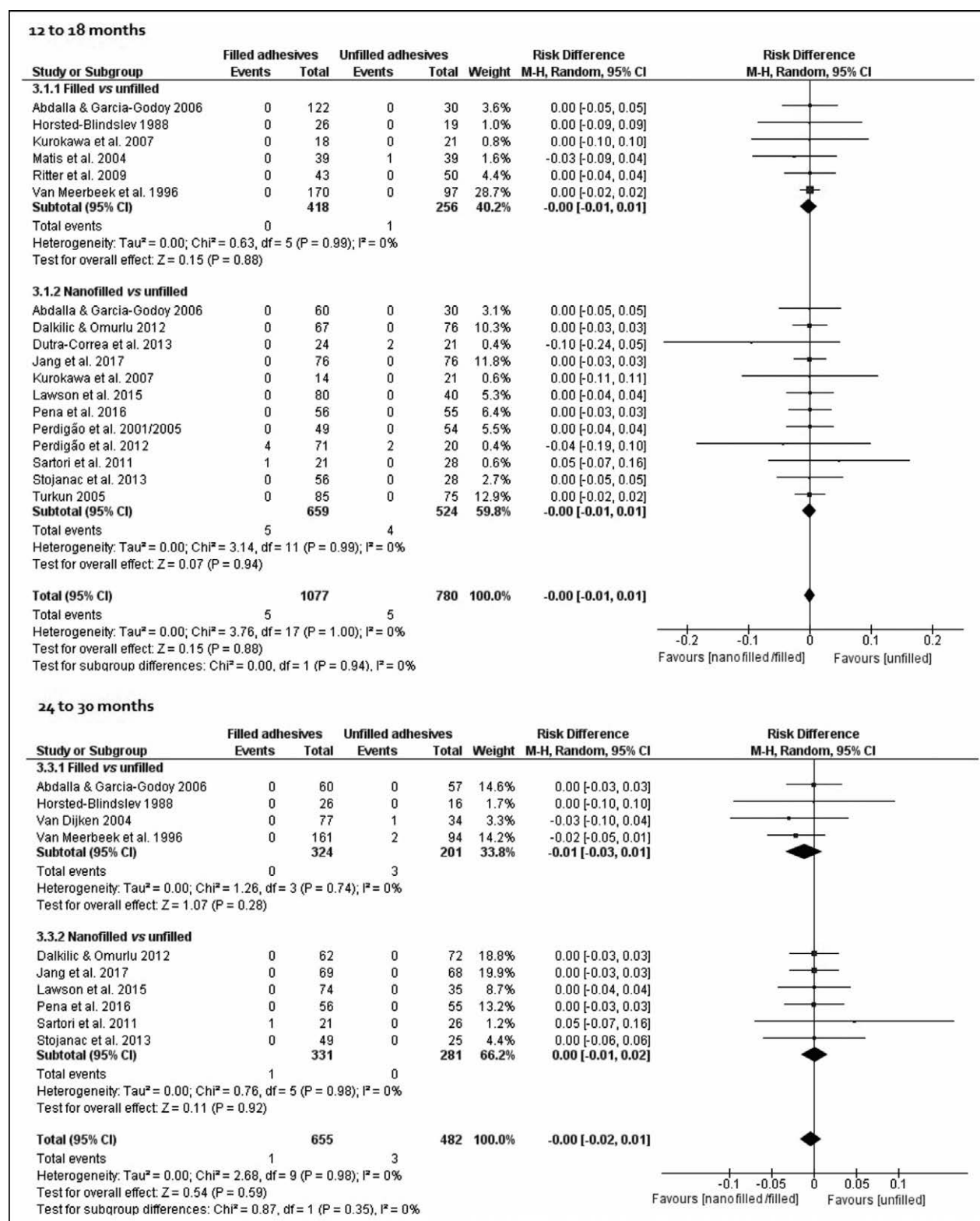


Figure 7. Forest plot of restorations with secondary caries comparing filled/nanofilled vs unfilled adhesives at 12 to 18 months and 24 to 30 months.

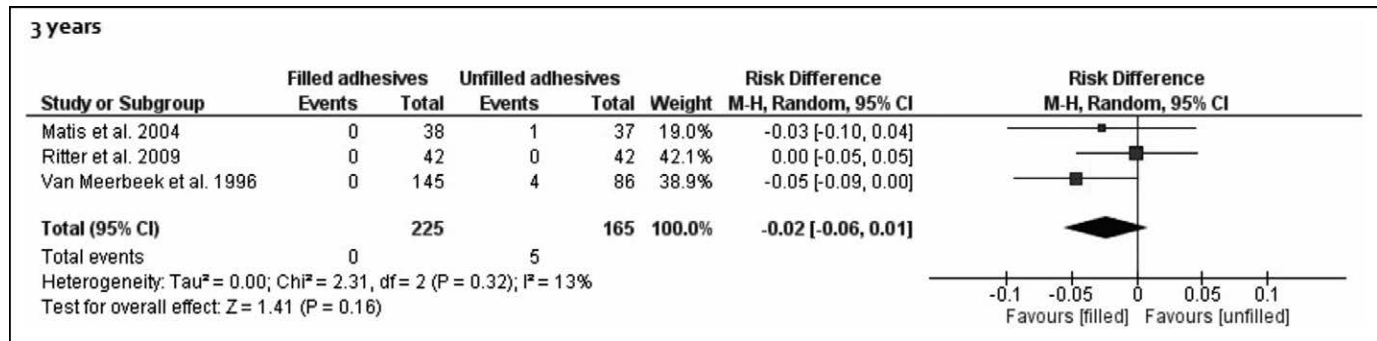


Figure 8. Forest plot of restorations with secondary caries comparing filled vs unfilled adhesives at 3 years.

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### Conflict of Interest

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

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# Three-year Clinical Performance of Two Giomer Restorative Materials in Restorations

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## Clinical Relevance

The clinical performance of both conventional and flowable giomer restorative materials was particularly good in Class I restorations after three years of service.

## SUMMARY

This study evaluated and compared the clinical performance of a flowable and a conventional giomer restorative material after three years. Forty-four pairs of restorations (total n=88) were placed in Class I cavities with either a flowable giomer (Beautifil Flow Plus F00; Shofu Inc, Kyoto, Japan) or a conventional giomer restorative material (Beautifil II; Shofu Inc) after the application of a dentin adhesive (FL-Bond II; Shofu Inc) and a flowable liner (Beautifil Flow Plus F03; Shofu Inc). After 3 years, 39 pairs of restorations were evaluated with the modified United States Public Health Service criteria, and digital color photographs of restorations were taken at each patient visit. The evaluation parameters were as follows: color

match, marginal integrity, marginal discoloration, retention, secondary caries formation, anatomic form, surface texture, and postoperative sensitivity. Evaluations were recorded as a clinically ideal situation (Alpha), a clinically acceptable situation (Bravo), or a clinically unacceptable situation (Charlie). Data were analyzed with Fisher's exact and McNemar tests ( $\alpha=0.05$ ).

None of the restorations showed retention loss, postoperative sensitivity, secondary caries, or color change. The performance of Beautifil II in terms of marginal integrity, marginal discoloration, and surface anatomic form was significantly lower at the 36-month follow-up than at baseline ( $p=0.007$ ). There were no significant differences between the baseline and 36-month follow-up scores for

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the other criteria for Beautifil II ( $p>0.05$ ). No differences were found between the baseline and the 36-month follow-up scores for any of the criteria for Beautifil Flow Plus F00 ( $p>0.05$ ). No statistically significant difference in overall clinical performance was found between the 2 materials after 36 months ( $p>0.05$ ).

The three-year clinical performance of both restorative materials (Beautifil Flow Plus F00 and Beautifil II) was very good and not significantly different for any of the parameters evaluated.

## INTRODUCTION

Resin composites have been used in dentistry for more than five decades. In recent years, their formulations were significantly improved to expand the range of clinical indications.<sup>1</sup> Different manufacturing techniques, compositions, filler types, and filler sizes affected the overall properties of these materials. Improvements in filler technology and formulations made composite resin materials suitable even for the stress-bearing areas of posterior teeth.<sup>2</sup> Nanocomposites are among the most recent developments, offering reduced polymerization shrinkage, increased mechanical properties such as tensile and compressive strength to fracture, improved optical characteristics, and better retention.<sup>2,3</sup>

Flowable resin composites are lower viscosity resins that typically have a lower filler content than universal composites. Their flow characteristics make them useful for restoring small cavities or as a cavity liner for improved adaptation to the cavity walls of larger cavities.<sup>4</sup> They effectively seal the microstructural irregularities of cavity preparations prior to conventional resin composite placement. Therefore, it has been confirmed that using flowable resin composites as a liner improves marginal integrity and reduces the microleakage of resin composite restoration.<sup>5</sup> Filler content and monomer composition vary among different brands of flowable resin composites, offering various properties.

Giomer (glass ionomer + polymer) restorative materials were introduced more than 15 years ago, and they contain prereacted glass ionomer (PRG) filler particles embedded in a resin matrix.<sup>6</sup> Gomers are manufactured by reacting acid-reactive fluoride-containing glass with polyacids in the presence of water.<sup>7</sup> PRG fillers are divided into two categories: full reaction type PRG (F-PRG) fillers and surface reaction type PRG (S-PRG) fillers. In F-PRG fillers, the entire filler particle reacts with polyacrylic acid and releases a large amount of fluoride as the core of the particle is completely reacted. Therefore, unlike S-PRG fillers,

F-PRG fillers degrade faster. In S-PRG fillers, only the surface of the filler reacts with polyacrylic acid, and the glass core remains intact.<sup>8</sup> Gomers offer improved clinical handling and physical characteristics compared with conventional and resin-modified glass ionomers while providing the esthetic properties of resin composites.<sup>8</sup> S-PRG fillers in giomer materials also allow for the release and recharge of fluoride that is comparable to glass ionomer materials but is more than that of fluoride-containing resin composites.<sup>9</sup>

Gomers have a successful short- to long-term clinical history in Class I, II, and V lesions.<sup>7,8,10-12</sup> Beautifil II (Shofu Inc) is one of the universal second-generation giomer restorative resin materials, which combines the characteristics of both composite resins and glass ionomers. This giomer-based resin actually represents a special class of composites that offers both protection against caries and provides functional and esthetic results. Based on S-PRG technology, it is comprised of aluminofluoro-borosilicate glass and multifunctional glass fillers, with particle sizes ranging from 0.01–4.0  $\mu\text{m}$ . Additionally, it contains discrete nanofillers (10–20 nm) and has a total filler content of 83.3 wt% (68.6 vol%),<sup>13</sup> which means that a glass-ionomer-like structure surrounds multifunctional glass fillers, with an external hard glass layer. Therefore, the fillers gain great physical strength and release fluoride (F) and 5 other ions (Na, sodium; B, borate; Al, aluminum; Si, silicate; and Sr, strontium) without causing deterioration of the properties of the material.<sup>14</sup> More recently, a flowable giomer restorative material, Beautifil Flow Plus F00 (Shofu Inc), was introduced. It is indicated as a flowable base, liner, and final restorative material. Similar to Beautifil II, Beautifil Flow Plus F00 is also based on S-PRG technology. It has a filler content of 67.3 wt% (47.0 vol%).<sup>15,16</sup> Both materials are indicated for Class I–V lesions.

The longevity of resin composite restorations has been previously reviewed;<sup>17</sup> they exhibit lower clinical success than other materials.<sup>18,19</sup> The main reasons for the failure of composite resin restorations are secondary caries and fracture.<sup>17</sup> The properties of resin restorative materials utilizing PRG technology include increased wear resistance and a high level of radiopacity, due to the presence of multifunctional glass fillers and shade conformity, owing to the improved light diffusion and fluorescence of the material.<sup>8</sup> One of the very specific advantages of giomer restoratives is their release of fluoride<sup>20</sup> and, therefore, their possible ability to prevent secondary caries.<sup>21</sup> Gomers with S-PRG fillers could release a greater amount of fluoride than that of other fluoride releasing resin composites.<sup>22,23</sup> Additionally, it was speculated that the amount of released fluoride

was possibly related to the flow level of the restorative material: the higher the flow, the greater the amount of fluoride release.<sup>22</sup>

This prospective study investigated and compared the three-year clinical performance of a flowable and a conventional fluoride-releasing giomer restorative material containing S-PRG fillers, bonded with a two-step, self-etch adhesive to restore posterior Class I lesions. The null hypothesis tested was that there would be no difference in the clinical performance of the two giomer materials.

## METHODS AND MATERIALS

In this prospective, open clinical study, 44 pairs of Class I cavities were restored with either a flowable giomer restorative material (Beautifil Flow Plus F00; Shofu Inc) or a conventional giomer restorative material (Beautifil II; Shofu Inc) after the application of a two-step, self-etch adhesive system (FL-Bond II; Shofu Inc) and a flowable giomer liner (Beautifil Flow Plus F03; Shofu Inc) (Table 1).

## Patient Selection

The study was approved by the Institutional Review Board of the University of Pennsylvania (Protocol #815836). Written, informed consent was obtained from all participants before the initiation of treatment. Participants of this study had molar-supported permanent dentition with normal occlusion. The patient inclusion criteria were as follows: primary shallow/moderate caries not reaching the inner one-third of dentin, with no risk of pulpal exposure in the occlusal surface; occlusal contact with the antagonist tooth; with at least two similar sized occlusal lesions; and in good state of general health. The exclusion criteria were as follows: intense bruxism or severe periodontal problems, molars with a carious lesion on a surface other than the occlusal surface and in continuity with the occlusal cavity, pulp exposure during caries removal, cavities with imminent risk of pulp exposure, and spontaneous pain or sensitivity to percussion. All patients received oral prophylaxis treatment and oral hygiene instructions 2 weeks before the placement of restorations.

Table 1. Restorative Materials and Adhesive System Used in the Study

Material Description	Material Name	Composition	Manufacturer
Giomer restorative	Beautifil II	Base resin: Bis-GMA (7.5 wt%)/TEGDMA (5 wt%); resin filler: multifunctional glass filler and S-PRG (surface prereacted glass-ionomer) filler based on aluminofluoro-borosilicate glass  Filler loading: 83.3 wt% (68.6 vol%); particle size range: 0.01–4.0 µm; mean particle size: 0.8 µm DL-Camphorquinone	Shofu, Kyoto, Japan
Flowable giomer restorative	Beautifil Flow Plus F00	Base resin: Bis-GMA (15 wt%)/TEGDMA (13wt%); resin filler: multifunctional glass filler and S-PRG filler based on aluminofluoro-borosilicate glass  Filler loading: 67.3 wt% (47.0 vol%); particle size range: 0.01–4.0 µm; mean particle size: 0.8 µm DL-Camphorquinone	Shofu, Kyoto, Japan
Two-step, self-etch adhesive	FL-Bond II	Primer: carboxylic acid monomer, phosphonic acid monomer, 6-MHPA, water, solvent, photoinitiator  Adhesive: HEMA, UDMA, TEGDMA, 40% fluoride releasing and recharging S-PRG filler, photoinitiator	Shofu, Kyoto, Japan
Abbreviations: 6-MHPA, 6-methacryloxyhexyl 3- phosphonoacetate; Bis-GMA, bisphenol-A-diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; S-PRG filler, surface prereacted glass-ionomer filler; TEGDMA, triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate.			



Thirty-four patients (14 men, 20 women; age range, 20–45 years) were included in this study. A total of 88 molar teeth (44 pairs) with Class I primary carious lesions were restored. Each patient received at least 1 pair of restorations with both materials placed in either tooth. A “pair” means that the 2 materials were used in the same patient in at least 2 molar teeth, based on the patient’s needs.

**Clinical Procedures**

The teeth were randomly assigned to the restorative materials. All lesions were restored by 2 calibrated operators using local anesthesia and rubber dam isolation. The initial access to the carious dentin was accomplished using a diamond bur attached to a high-speed handpiece under water cooling. Cavity preparations were limited to the removal of carious tissue. The average faciolingual width of the cavities was approximately one-third of the intercuspatal width. No bevel was prepared on the enamel margins. Only loose enamel prisms were removed with finishing diamond burs. Each preparation was performed with new burs.

The self-etch adhesive (FL-Bond II; Shofu Inc) was used according to the manufacturer’s instruction. The

primer was thoroughly applied to the cavity and left undisturbed for 10 seconds, after which it was air-dried for 5 seconds. Subsequently, the bonding agent was applied to the cavity and light-cured for 10 seconds. A thin layer of flowable material (Beautifil Flow Plus F03; Shofu Inc) was applied to the cavity base and light-cured for 10 seconds with a light-emitting diode (LED) curing unit (Elipar S 10; 3M Espe, St. Paul, MN, USA). The cavities were then restored incrementally with either Beautifil Flow Plus F00 or Beautifil II giomer resin restorative material. Each 2-mm increment was light-cured with the same curing unit. After polymerization, occlusal adjustment, contouring, and finishing were performed with diamond finishing burs (Brasseler, Savannah, GA, USA) and the restorations were polished thoroughly with composite polishing kits (Enhance and PoGo Polishing System; Dentsply Caulk, Milford, DE, USA).

**Clinical Evaluation of the Restorations**

The restorations were evaluated according to the modified United States Public Health Service criteria<sup>24</sup> at baseline, 6-month, 18-month, and 36-month follow-up visits (Table 2). Two calibrated examiners who were

Table 2. Evaluation Criteria of the Restorations According to Modified United States Public Health Service Criteria		
No.	Category	Rating and Characteristics
1	Retention	Alfa (A): no loss of restoration Bravo (B): any loss of restorative material
2	Marginal integrity	Alfa (A): explorer doesn’t catch or slight catch with no visible crevice Bravo (B): explorer catches and crevice is visible, but there is no exposure of dentin or base Charlie (C): explorer penetrates crevice and defect extended to dentin–enamel junction
3	Secondary caries	Alfa (A): no caries present Bravo (B): caries present associated with the restoration
4	Surface anatomic form conditions	Alfa (A): restoration is continuous with existing anatomic form Bravo (B): restoration isn’t continuous with existing anatomic form, but missing material is not sufficient to expose dentin or lining Charlie (C): sufficient material is lost to expose dentin
5	Postoperative sensitivity	Alfa (A): no sensitivity Bravo (B): sensitivity, but diminishing in intensity Charlie (C): constant sensitivity, not diminishing in intensity
6	Surface texture	Alfa (A): enamel-like surface Bravo (B): surface rougher than enamel, clinically acceptable Charlie (C): surface unacceptably rough
7	Color match	Alfa (A): restoration matches adjacent tooth structure in shade and/or translucency Bravo (B): mismatch in shade and/or translucency is within normal range of tooth shades Charlie (C): match in shade and/or translucency is outside normal range of tooth shade
8	Marginal discoloration	Alfa (A): no visible evidence of marginal discoloration Bravo (B): marginal discoloration present but has not penetrated in pulpal direction

not involved in the operative process evaluated the restorations with a mirror, explorer, and air stream. In case of a disagreement, examiners re-evaluated the restorations until a consensus was reached. Digital color photographs of the lesions and the restorations were taken at baseline and at each follow-up visit for documentation purposes.

Both tested materials were compared with Fisher's exact test. Each tested criterion for each material was analyzed separately (with respect to different follow-up periods) using Friedman's test.

## RESULTS

Of the 44 original pairs of restorations placed, 39 were available for evaluation at the 3-year follow-up visit. The number of patients, score percentages, and statistical significance values for the criterions, which revealed different outcomes at different follow-up periods, are presented in Tables 3, 4, 5, and 6. None of the restorations showed retention loss, postoperative sensitivity, secondary caries, or color change. For the marginal integrity, marginal discoloration, and surface anatomic form, Friedman's test revealed that the quality of Beautifil II restorations was significantly lower at 36 months than at baseline ( $p=0.007$ ) (Tables 3, 4, 5). However, there was no significant difference

between the baseline and 36-month follow-up scores for the surface texture criteria with Beautifil II ( $p>0.05$ ) (Table 6). For Beautifil Flow Plus F00, no differences were found between the baseline and the 36-month follow-up scores for any of the criteria ( $p>0.05$ ).

Fisher's exact test revealed no difference between the performance of Beautifil Flow Plus F00 and Beautifil II at 36 months for all the criteria evaluated ( $p>0.05$ ).

## DISCUSSION

Clinical studies can provide important information in respect to material performance and changes over time.<sup>25</sup> This study employed a 36-month observation period with 6- and 18-month intervals. The recall rate of this study was 100% at 6 months and 88.64% at 18 months and 36 months. A similar study reported a recall rate of 80% at 36 months,<sup>16</sup> while another study revealed a recall rate of only 59%,<sup>26</sup> which is far lower than that of this study.

In this study, all restorations of the presented patients (39 patients) remained intact, with no postoperative sensitivity or secondary caries at the 36-month follow-up. The absence of postoperative sensitivity may be attributed to the use of a two-step, self-etch adhesive, which does not entirely remove the smear layer.<sup>26</sup> Therefore, the outcomes in respect to this parameter

Table 3. Results of Clinical Evaluation of Marginal Integrity Criterion

		Baseline (n=44)	6 m (n=44)	18 m (n=44)	36 m (n=39)	Friedman Test p-value
Beautifil II	A	40 (90.9%)	40 (90.9%)	40 (90.9%)	35 (89.7%)	0.007
	B	4 (9.1%)	4 (9.1%)	4 (9.1%)	4 (10.3%)	
Beautifil Flow Plus F00	A	43 (97.7%)	42 (95.4%)	42 (95.4%)	37 (94.9%)	0.112
	B	1 (2.3%)	2 (4.6%)	2 (4.6%)	2 (5.1%)	
Fisher's exact p-value		1	0.676	0.676	0.675	–

Abbreviations: A, Alpha; B, Bravo; m, months.

Table 4. Results of Clinical Evaluation of Surface Anatomic Form Criterion

		Baseline (n=44)	6 m (n=44)	18 m (n=44)	36 m (n=39)	Friedman Test p-value
Beautifil II	A	41 (93.2%)	40 (90.9%)	40 (90.9%)	35 (89.7%)	0.007
	B	3 (6.8%)	4 (9.1%)	4 (9.1%)	4 (10.3%)	
Beautifil Flow Plus F00	A	44 (100%)	43 (97.7%)	43 (97.7%)	38 (97.4%)	0.392
	B	0 (0%)	1 (2.3%)	1 (2.3%)	1 (2.6%)	
Fisher's exact p-value		0.241	0.36	0.36	0.358	–

Abbreviations: A, Alpha; B, Bravo; m, months.

Table 5. Results of Clinical Evaluation of Marginal Discoloration Criterion						
		Baseline (n=44)	6 m (n=44)	18 m (n=44)	36 m (n=39)	Friedman Test <i>p</i> -value
Beautiful II	A	44 (100%)	43 (97.7%)	41 (93.2%)	35 (89.7%)	0.007
	B	0 (0%)	1 (2.3%)	3 (6.8%)	4 (10.3%)	
Beautiful Flow Plus F00	A	44 (100%)	44 (100%)	43 (97.7%)	38 (97.4%)	0.392
	B	0 (0%)	0 (0%)	1 (2.3%)	1 (2.6%)	
Fisher's exact <i>p</i> -value		1	1	0.66	0.358	–
Abbreviations: A, Alpha; B, Bravo; m, months.						

Table 6. Results of Clinical Evaluation of Surface Texture Criterion						
		Baseline (n=44)	6 m (n=44)	18 m (n=44)	36 m (n=39)	Friedman Test <i>p</i> -value
Beautiful II	A	43 (97.7%)	42 (95.4%)	42 (95.4%)	37 (94.9%)	0.112
	B	1 (2.3%)	2 (4.6%)	2 (4.6%)	2 (5.1%)	
Beautiful Flow Plus F00	A	44 (100%)	43 (97.7%)	43 (97.7%)	38 (97.4%)	0.392
	B	0 (0%)	1 (2.3%)	1 (2.3%)	1 (2.6%)	
Fisher's exact <i>p</i> -value		0.241	1	1	0.308	–
Abbreviations: A, Alpha; B, Bravo; m, months.						

may not be solely related to the type of the giomer resin material used. The only deterioration observed was minimal loss of marginal integration and slight marginal discoloration in the teeth restored with Beautiful II after 36 months. Regarding marginal integrity and marginal discoloration, 10.3% of the restorations received Bravo ratings for both of these criteria. Another study found higher Bravo ratings—41.9% for marginal integrity and 16.1% for marginal discoloration—for Beautiful II.<sup>24</sup> The difference between the results could be due to lower recall rates in the previous study. Additionally, in this study, a flowable material, Beautiful Flow Plus F03, was used as a liner underneath both restorative materials. Such a low-modulus material could act as an elastic layer and dissipate the stresses generated by occlusal loads,<sup>27</sup> which may explain the different findings. Additionally, the use of a flowable material as a liner could improve the adaptation of the initial increment of the covering restorative material and may, therefore, influence the outcomes.

Teeth restored with the flowable giomer restorative material, Beautiful Flow Plus F00, did not exhibit any significant changes at the 36-month recall compared with baseline. Its flowable behavior has the ability to provide better adaptation to the cavity walls,<sup>28,29</sup> which may be a contributing factor for the sustained marginal integrity over time.

Previous clinical studies reported an acceptable clinical performance of first-generation giomer restorative materials.<sup>7,8</sup> In this study, a second-generation giomer restorative material, Beautiful II, also showed clinically acceptable results, confirming the results of previous studies with Beautiful II.<sup>16,26,30</sup> A recent observation showed that surface roughness, marginal adaptation, and marginal discoloration were the most frequent changes observed for Beautiful II after 36 months.<sup>26</sup> Forty percent of the restorations showed signs of slight crevices along the margin at occlusal surfaces.<sup>26</sup> This study revealed similar results for Beautiful II.

In a clinical trial,<sup>16</sup> which compared Beautiful II and Beautiful Flow Plus F00 for 3 years, Beautiful Flow Plus F00 showed better performance regarding marginal integrity, marginal discoloration, surface roughness, and surface morphology (anatomic form) at the 36-month recall. Those materials performed similarly in this study.

Among the most prevalent factors for gap formation during long-term clinical service are polymerization shrinkage and differences between the thermal expansion coefficients of the restorative material and the tooth structure.<sup>31</sup> This gap along the restoration—tooth interface potentially leads to leakage, marginal discoloration, secondary caries, and postoperative sensitivity. Cavities with high configuration factors

may produce contraction stresses along the cavity walls,<sup>32</sup> affecting the long-term marginal integrity of the restoration. In this study, Class I cavities, which have the highest configuration factor, were restored with giomer restoratives with different viscosities. The high cavity configuration factor must be viewed as a reason for the Bravo ratings observed for marginal integrity and marginal discoloration with Beautifil II at 36 months. Lower viscosity and higher elasticity may be contributing factors for the finding that Beautifil Flow Plus F00 performed better at the 36-month follow-up in respect to marginal integrity and discoloration. Besides material-dependent properties, finishing/polishing procedures, and chipping of restorative material at the cavosurface margins also influence marginal deterioration.<sup>8</sup>

Flowable restorative materials typically have a lower filler load than conventional composite resins.<sup>33</sup> The flowable giomer restorative material Beautifil Flow Plus F00 has a filler load of 67.3 wt % (47.0 vol %), which is higher than that of conventional flowable composites.<sup>33</sup> The high filler content with S-PRG fillers has shown to provide the flowable giomer material with superior physical properties and increased fluoride release over time.<sup>15</sup> Therefore, it can be assumed that these superior properties can expand the range of indications for this material to the posterior regions. However, further clinical studies would be necessary to test this hypothesis. Although the hardness value of Beautifil Flow Plus F00 was reported to be lower than that of Beautifil II,<sup>15</sup> 36-months follow-up evaluation did not yield any difference between them in respect to anatomic form. It was reported that fluorosilicate glass fillers in giomers are susceptible to degradation by weak acids<sup>34</sup> and that both Beautifil II and Beautifil Flow Plus F00 can be degraded by citric acid,<sup>15</sup> which might influence their anatomic form during clinical service. However, longer-term follow-up trials would be needed to verify this. In addition, the size and the location of the restorations influence occlusal wear of the restorative materials.<sup>35</sup> Wear increases with a larger surface area and length of cavosurface margins. Since it was not possible to standardize cavities in this study, the location and the size of the restorations have possibly impacted the outcomes.

In general, it has been confirmed that giomer restorative materials present promising clinical outcomes.<sup>7,8,10-12,16,26</sup> In this study, second-generation giomer restorative materials, both Beautifil II conventional giomer restorative and Beautifil Flow Plus F00 flowable giomer materials, clinically showed very good results without any significant difference from each other for all parameters evaluated in the study.

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## Conflict of Interest

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

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