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We thank all our reviewers for their time and dedication to Operative Dentistry.

On The Cover

Mount Arenal in the morning, an active stratovolcano in La Fortuna, Costa Rica. Photo provided by Dan Henry of Pensacola, FL USA. Photo taken with a Sony A350 20mm f5.6 – 1/50 sec. © Operative Dentistry, Inc.

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563–674

Editorial

Literature Review

Clinical Research

Laboratory Research

Departments

Online Only Articles

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Editorial

PEER REVIEW - 2015

JA Platt, Editor

The dental scientific literature is being challenged as a rapidly increasing number of "peer-reviewed" journals strains the ability of the qualified reviewer pool to meet the demand. (In 2014, Thomas Reuters Journal Citation Reports assigned an Impact Factor for 88 dental journals. Five years earlier, they rated only 64.) In addition to this expanding group of traditional publications is a large number of recently introduced online journals. Just as with print journals, online publications demonstrate a wide range of scientific rigor and review. Some of them deserve and are receiving support from reviewers.

Significant changes are also occurring within the reviewer pool. In some places in the world, such as the United States, the number of tenured and tenure-track faculty has greatly decreased. It is this group which has provided significant peer-review support over the past decades. Many of the recently hired younger faculty come on board as clinical track faculty with little incentive to be involved with research and the peer review process. Oftentimes, the new faculty have minimal previous research experience and no desire to participate as reviewers.

These factors (increasing journals and decreasing reviewers) result in fewer people being asked to carry a greater load in providing peer review oversight of our literature. When I gather with other editors, a common topic of conversation is the shortage of qualified and willing reviewers. Clearly, the future of peer review, and how it is accomplished, is being challenged. Thankfully, *Operative Dentistry* continues to benefit from the dedicated service of a large number of volunteer reviewers who provide important feedback for the authors of manuscripts to improve the content and presentation of information. Reviewers also provide critical information for the editor as ultimate publication decisions are made for each article. Of the 504 articles submitted to this journal over the past year, a total of 120 articles were accepted. Each article had at least two reviewers who worked with the authors and the editorial team to improve, and then approve the submission. To be done well, this task requires significant effort and a commitment to excellence.

I remain extremely grateful to the group of scientists and clinicians who provide this critical service. What follows is a list of people who have provided reviews for us over the past year. I thank each one of them. And you, as a reader of *Operative Dentistry*, if you know any of these people, give them your thanks as well. They are a committed group of volunteers working diligently to maintain the integrity of peer review!

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Reviewers

Operative Dentistry, Inc. would like to thank our conscientious team of Reviewers for their hard work, tenacity, and dedication in the furthering of operative dentistry around the word. These individuals dedicate innumerable hours in reading, re-reading, and critiquing manuscripts. Submitted articles, accepted for publication or not, all benefit from these reviewers who help authors present their hard work, as well as verifying that the work we publish is scientifically accurate, clinically relevant, and professionally uplifting. We cannot thank these individuals enough for their contributions, but we do want to publically recognize them.

Be it known here, and throughout the world, that the following named individuals have contributed real and invaluable service to the profession of Dentistry by volunteering their time and talents to the cause of peer-review for Operative Dentistry, Inc. The time of service listed below is for the 2015 publication year.

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Literature Review

Can Silanization Increase the Retention of Glass-fiber posts? A Systematic Review and Meta-analysis of *In Vitro* Studies

AP Moraes • R Sarkis-Onofre • RR Moraes
MS Cenci • CJ Soares • T Pereira-Cenci

Clinical Relevance

The improved retention of glass-fiber posts (GFPs) with a combination of post pretreatment and silanization is of particular interest because it could impact the clinical survival of GFP-retained restorations.

SUMMARY

The role of silanes in the bonding of resin luting agents to glass-fiber posts (GFPs) is a controversial topic, and the question still remains whether post silanization is able to improve the retention of GFPs luted into root canals. Thus, this study was designed to determine whether evidence exists to justify silanization of GFPs before cementation to increase their retention into root canals. *In vitro* studies that evaluated the retention of GFPs cemented into root canals or artificial substrates and that used silane coupling agents for pretreatment of the post were selected. Searches were carried out in PubMed and Scopus databases with no publication year or language limits. The last search was carried out in August 2014. Two distinct data analyses were carried out: 1) control group (no post pretreatment) vs silane only and 2) post pretreatment + silane vs silane only. Pooled-effect estimates were obtained by comparing the difference between each bond strength mean value and were expressed as the weighted mean difference between groups ($p \leq 0.05$). A total of 178 articles were found, and 23 were included in

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the review. The results were affected by the substrate into which the GFPs were luted (teeth or artificial devices). The analysis between control group and silane only for studies that used artificial devices favored the use of silane ($p<0.0001$), but considering studies that used teeth as substrate, no significant difference was observed ($p=0.35$). The analysis between silane only and pretreatment + silane did not show a significant difference between groups when artificial devices were used ($p=0.71$), whereas the analysis favored the use of post pretreatment + silane over silane ($p<0.00001$) only when the GFPs were luted into teeth. In conclusion, this review indicates that silanization improves the retention of GFPs luted into root canals provided that selective surface pretreatments are applied to the post before silanization.

INTRODUCTION

Glass-fiber posts (GFPs) have been developed to improve the optical effects of esthetic restorations[1,2] and are widely used for restoring endodontically treated teeth with insufficient coronal structure to serve as a core for the restoration.[3,4] The use of GFPs in cases in which the coronal tooth structure has been destroyed as a result of caries, trauma, or overaggressive endodontic procedures is gaining widespread acceptance among dental clinicians.[5,6] Together with the increased use of prefabricated posts, particularly GFPs, an increase has also been observed in the number of studies on this subject available in the literature. These studies evaluate different cementation protocols, adhesive systems, and surface treatments for improving the bond between resin cements and GFPs. Yet the main reason for failure of GFPs is still debonding, which occurs mainly as a result of the difficulties clinicians face in achieving proper adhesion to the intraradicular dentin.[7]

Various surface pretreatments of GFPs have been tested in the literature. These pretreatments can be divided into 1) physical/chemical means intended to create surface irregularities and expose the inorganic glass fibers and 2) chemical treatments applied to improve micromechanical and/or chemical attachment to the post.[8-12] Silanization is the most frequently used chemical pretreatment. Organosilane coupling agents are bifunctional molecules in which one end of the molecule is capable of reacting with inorganic glass fiber and the other end with the resin cement.[13] The role of silanes in the bonding of resin luting agents to GFPs is, however, a controversial topic.[2] Some studies[2,12,14] reported that silanization does not have a significant effect on the bond strength between resin cements and GFPs, whereas other studies[15-17] reported improved bonding by silanization. It is also a possibility that increased exposure of the glass fibers to physical/chemical pretreatments could have a synergic effect with silanization, thereby improving the retention of GFPs.

Despite the large number of *in vitro* studies in the literature, the question still remains whether post silanization is able to improve the retention of GFPs luted into root canals. This question cannot be easily answered because of the large variability in methods and results among primary studies. Therefore, the aim of this study was to systematically review the literature to determine whether there is *in vitro* evidence to justify the use of silanes to improve the bond strength of GFPs to intraradicular dentin. The hypothesis tested was that application of silane does not improve the retention of GFPs.

METHODS

Search Strategy

This systematic review was based on the guidelines of the *Cochrane Handbook for Systematic Reviews of Interventions*[18] and followed the four-phase flow diagram based on the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) Statement.[19] Two electronic databases (PubMed and Scopus) were searched to identify manuscripts that could meet the following inclusion criteria: *in vitro* studies that evaluated the retention (bond strength) of GFPs luted into root canals (human or bovine teeth) or into artificial devices that used silane coupling agents for pretreatment of the post. The following search strategies were used: (glass fib* post*) AND (silane*); (endodontically-treated teeth) AND (silane*).

Screening and Selection

No publication year or language limits were set. The last search was carried out in August 2014. Reference lists of included studies were hand searched for additional articles. Excluded from the study were investigations reporting *in situ* studies, literature reviews, types of posts other than GFPs, and studies that did not use silane coupling agents for post pretreatment. Two reviewers (APM and RSO) independently screened the titles identified in the searches. If the title indicated possible inclusion,

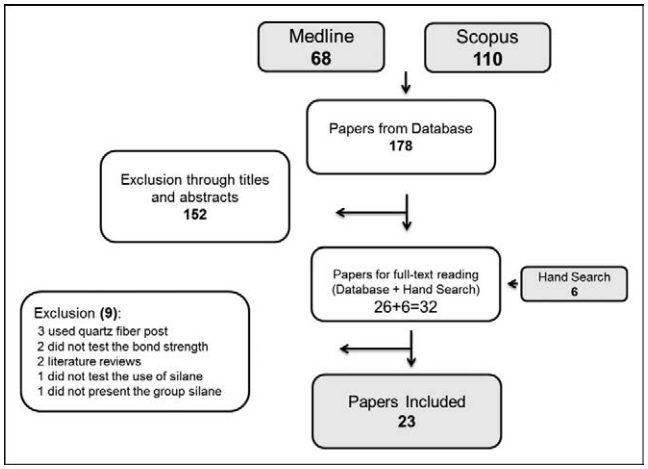

Figure 1. *Flow diagram of the systematic review according to the PRISMA Statement.*

the abstract was evaluated. After the abstracts were carefully appraised, the manuscripts considered eligible for the review and those with which there was some doubt were selected for full-text reading. In case of disagreement, a third reviewer (TPC) decided if the article should be included or not.

Data Collection

The two reviewers extracted all data simultaneously using a standardized outline. To make identification of variables found in the articles easier, the authors categorized similar information into groups (eg, post pretreatment used, bond strength mean reported in the articles). In case of measurement of bond strength values for different root thirds (push-out test, for instance), the arithmetic average of the values of the thirds was used. For studies that did not report bond strength means in tables, the authors were contacted via e-mail if data were missing or when more information was needed.

Statistical Analysis

Two distinct data analyses were carried out: 1) control group (untreated posts) vs silane only and 2) post pretreatment + silane vs silane only. Every possible comparison of bond strength between groups within the articles was simulated. Pooled-effect estimates were obtained by comparing the difference between each bond set of strength mean values and were expressed as the weighted mean difference between groups. A p-value < 0.05 was considered statistically significant (Z-test).

Statistical heterogeneity of the treatment effect among studies was assessed using the Cochran Q test, with a threshold p-value of 0.1, and the inconsistency I^2 test, in which values greater than 50% were considered indicative of high heterogeneity.[18] The analyses were carried out using a random-effects model. Taking into account that the analyses of substrate used in the test could present high heterogeneity, subgroup analyses considering artificial devices or teeth as distinct substrates were carried out to explore that influence on the results. All analyses were conducted using Review Manager Software, version 5.1 (The Nordic Cochrane Centre, The Cochrane Collaboration, Copenhagen, Denmark).

Assessment of Risk of Bias

Risk of bias of each included study was evaluated according to the description in the articles of the following parameters for the study quality assessment:[20] randomization of teeth, use of teeth free of caries or restoration, materials used according to the manufacturers' instructions, use of teeth with similar dimensions, endodontic treatment performed by the same operator, description of sample size calculation, and blinding of the operator of the testing machine. If the authors reported the parameter, the article had a "Y" (yes) on that specific parameter; if it was not possible to find the information, the article received an "N" (no). Articles that reported one to three items were classified as having high risk of bias, those that reported four or five items were classified as having medium risk of bias, and those reporting six or seven items were classified as having low risk of bias. Only articles that used teeth as substrate for luting the GFPs had the risk of bias classified; the other studies had other parameters evaluated except those related to the teeth.

RESULTS

Figure 1 shows the flow diagram of the systematic review. A total of 178 articles were found, and 26 were eligible for full-text analysis. The hand searches revealed six more articles for full-text reading. From the 32 studies, 23 articles were included in the review.[2,6,9,16,21-39] Nine studies were excluded for the following reasons: two did not test the bond strength;[40,41] two were literature reviews;[42,43] one study did not test the use of silane;[44] three studies used quartz-fiber posts;[45-47] and one did not present the group silane.[48] In the included studies, the main outcomes evaluated were type of pretreatment, substrate used for luting the GFPs, bond strength test, and resin cement (Tables 1 through 3).

Table 1: *Characteristics of Studies that Used Artificial Devices as Substrate*

Author, Year	Substrate	Comparison	Bond Strength Test	Conclusion
Aksornmuang and others, 2004[6]	Resin blocks	Control[a], dual-cure bonding agent, dual-cure bonding agent followed by light-curing for 20 s, silane coupling bonding agent, silane coupling bonding agent followed by light-curing for 20 s	Microtensile	Application of a silane coupling agent improved the bond strength of dual-cure resin core material to glass fiber posts.
Bitter and others, 2007[22]	Composite disk and plastic mold	Four different silane solutions	Push-out	The effects of silanization appeared to be clinically negligible.
Bitter and others, 2008[21]	Plastic mold	Silane and control[a]	Push-out	The silanization had negligible effects.
Cekic-Nagas and others, 2011[23]	Cylindrical plastic tube	Sandblasting was followed by the application of a silane; immersion in 9.6% hydrofluoric acid gel; silanization and control[a]	Micro–push-out	Different surface treatments of fiber posts might affect the bonding capacity of resin-core systems to these posts.
Costa Dantas and others, 2012[24]	Metal matrix	Silane, hydrofluoric acid, hydrofluoric acid + silane, plasma polymerization with argon, ethylenediamine plasma (EDA), control[a]	Push-out	Adhesion improvement was only observed after EDA treatment.
Debnath and others, 2003[25]	Fixed bottom grip	Two different silanes using various concentrations (1%, 5%, and 10%)	Pull-out	Five percent of samples had the highest strength.
Goracci and others, 2005[26]	Plastic matrix	Silane and control[a]	Microtensile	The application of a silane onto the post surface prior to building up the core significantly increased the post–core bond strength.
Magni and others, 2007[28]	Plastic matrix	Sandblasting, sandblasting + silanization, silanization, control[a]	Microtensile	Silanization was confirmed to be a reliabe method for improving the bond strength of resin luting agents to fiber posts.
Mosharraf and others 2012[29]	Cylindrical Plexiglas matrix	Silanization, sandblasting, treatment with 24% H_2O_2, and control[a]	Tensile	Although silanization and sandblasting can improve the bond strength, there was not any significant difference between surface treatments used.
Novais and others, 2011[32]	Plastic matrix	Three prehydrolyzed silanes and one two-component silane followed by air-drying temperatures, 23°C and 60°C	Push-out	The use of warm air-drying after silane application produced no increase in the bond strength between the fiber-reinforced composite post and the composite core. The two-component silane produced higher bond strength than all prehydrolyzed silanes when it was used with air-drying at room temperature.
Oliveira and others, 2011[16]	Elastomer mold	Silane and control[a]	Shear	Silanization of glass fiber posts is not necessary when self-adhesive resin cements are used.
Radovic and others, 2007[9]	Plastic matrix	Sandblasting or no pretreatment in each of the two groups; posts received three types of additional "chair-side" treatments: silane; adhesive; control[a]	Microtensile	Sandblasting may give an increase in microtensile strength to methacrylate-based glass fiber posts, eliminating the need to apply additional "chair-side" treatments.
Soares and others, 2008[36]	Metal stubs	Silane, silane and adhesive, airborne-particle abrasion with 50-μm Al_2O_3 and silane, airborne-particle abrasion, silane, and adhesive	Microtensile	Treatment with silane only was sufficient as a surface treatment for adhesive bonding.
Zicari and others, 2012[38]	Artificial root canals	Control[a]; silane, or coated with silica-coated alumina particles	Push-out	Laboratory testing revealed that different variables, such as type of post, composite, cement, and post-surface pretreatment, may influence the cement-post interface.

[a] *Control stands for no treatment*

Table 2: *Characteristics of Studies that Used Teeth as Substrate*

Author, Year	Substrate	Comparison	Bond Strength Test	Conclusion
Leme and others, 2013[27]	Human roots	Control[a]; silane; silane and Solobond; silane and Scotchbond Adhesive; silane and Excite	Push-out	Silane application may be necessary to improve the adhesion of fiber posts.
Liu and others, 2014[39]	Human maxillary central incisors and canines	Control[a], sandblasting, silanization, sandblasting followed by silanization	Push-out	Silanization of the post surface has no significant effect on the interfacial bond strength between the post and the resin cement.
Mosharraf and others, 2013[30]	Human maxillary incisors	Control[a];Silanization after etching with 20% H_2O_2; silanization after airborne-particle abrasion; silanization	Tensile	Application of hydrogen peroxide before silanization increased the bond strength between resin cements and fiber posts.
Narene and others, 2011[31]	Human root dentin	Silane, Cojet and Silane, 10% sodium ethoxide and silane and and 10% H_2O_2	Push-out	Cojet/silane showed the highest bond strength.
Perdigão and others, 2006[2]	Human maxillary central incisors and canines	Silane and control[a]	Push-out	The use of a silane coupling agent did not increase the push-out bond strengths of the fiber posts used in this study.
Rathke and others, 2009[33]	Human teeth	Silane and control[a]	Push-out	Silanization seems to be less relevant for intra–root canal bonding, but may have beneficial effects on post-to-core strengths.
Rödig and others, 2010[34]	Human teeth	Control[a], silanization, sandblasting + silanization and tribochemical coating	Push-out	Silanization of the posts seems to have no significant effect on bond strength.
Sahafi and others, 2003[35]	Human maxillary incisors	Roughening (sandblasting, hydrofluoric acid etching), application of primer (Alloy Primer, Metalprimer II, silane), or roughening followed by application of primer (sandblasting or etching followed by primer, Cojet treatment)	Shear bond strength	The bond strength of resin cement could be improved by surface treatment, Cojet treatment and sandblasting were the most effective pretreatments, and etching the posts used with hydrofluoric acid cannot be recommended.
Tian and others, 2012[37]	Human roots	Silane and control[a]	Pullout	Silanization of fiber posts does not make a difference in terms of preventing dislocation of a post.

[a] *Control indicates no treatment.*

Results of the meta-analyses are presented in Figures 2 and 3. The analysis between control group (untreated posts) and silane only for studies that used artificial devices (Figure 2) favored the use of silane ($p<0.0001$), with $I^2 = 94\%$. Considering studies that used teeth as substrate, no significant difference was observed between groups ($p=0.35$; $I^2=87\%$). The analysis between silane only vs pretreatment + silane (Figure 3) did not show a significant difference between groups when artificial devices were used ($p=0.71$; $I^2=81\%$), whereas the analysis favored the use of post pretreatment + silane ($p<0.00001$; $I^2=94\%$) over silane only when the GFPs were luted into teeth. The articles by Bitter and others[21,22] were not included in the analyses because the data necessary for analysis

Table 3: *Resin Cements Used in the Included Studies*

Author, Year	Comparison	Resin Cement	Conclusion
Aksornmuang and others, 2004[6]	Control[a], dual-cure bonding agent, dual-cure bonding agent followed by light-curing for 20 s, silane coupling bonding agent followed by bonding Clearfil Photobond with Porcelain Bond Activator, Clearfil Photobond with Porcelain Bond Activator followed by light-curing for 20 s	Clearfil DC Core (conventional)	Application of a silane coupling agent improved the bond strength of dual-cure resin core material to glass fiber posts.
Bitter and others, 2007[22]	Four different silane solutions	Panavia F (self-etch); PermaFlo DC (conventional); VariolinK II (conventional); RelyX Unicem (self-adhesive)	Variolink II demonstrated significantly higher bond strengths than the other investigated materials.
Bitter and others, 2008[21]	Silane and control[a]	Clearfil Core (conventional); MultiCore Flow (conventional)	Bond strengths were significantly affected by thermocycling, post type, and pretreatment, but in general not by the core material.
Cekic-Nagas and others, 2011[23]	Sandblasting was followed by the application of a silane; immersion in 9.6% hydrofluoric acid gel and silanization and control[a]	Biscore (resin-core material); Admira (composite resin)	The highest mean micro–push-out bond strength value was achieved in DT-light post, HF-silane treatment with the Biscore core material.
Costa Dantas and others, 2012[24]	Silane, hydrofluoric acid, hydrofluoric acid + silane, plasma polymerization with argon, ethylenediamine plasma (EDA), and the control[a]	RelyX Unicem (self-adhesive)	The RelyX Unicem cement showed an affinity with fiber posts treated with EDA plasma, which was observed for the highest bond strength.
Debnath and others, 2003[25]	Two different silanes using various concentrations (1%, 5%, and 10%)	Experimental resin	Five percent of samples had the highest strength.
Goracci and others, 2005[26]	Silane and control[a]	UnifilFlow; Tetric Flow (flowable composites)	Any combination of post and core material, post silanization increased the interfacial bond strength.
Leme and others, 2013[27]	Control[a]; silane; silane and Solobond; silane and Scotchbond Adhesive; silane and Excite	RelyX Unicem (self-adhesive)	Silane application may be necessary to improve the adhesion of fiber posts luted with the self-adhesive resin cement evaluated here.
Liu and others, 2014[39]	Control[a], sandblasting, silanization, sandblasting followed by silanization	DMG LUXACORE Smartmix Dual, Multilink Automix, Panavia F2.0, RelyX Unicem	It can be concluded that especially when DMG LUXACORE Smartmix Dual is used, air abrasion of glass fiber posts has a significantly helpful effect on the micro–push-out bond strength.
Magni and others, 2007[28]	Sandblasting, sandblasting + silanization, silanization, control[a]	Multilink (conventional); Variolink II (conventional); MultiCore Flow (conventional)	The type of luting agent did not significantly influence bond strength.
Mosharraf and others, 2012[29]	Silanization, sandblasting, treatment with 24% H_2O_2, and control[a]	Clearfil Photo Core Composite (composite resin)	Both silanization and sandblasting improved the bonding strength of fiber posts to composite resin core, but there were not any significant differences between these groups and the control group.
Mosharraf and others, 2013[30]	Control[a]; silanization after etching with 20% H_2O_2; silanization after airborne-particle abrasion; silanization	Panavia F 2.0 (self-etch)	Application of hydrogen peroxide before silanization increased the bond strength between resin cements and fiber posts.
Narene and others, 2011[31]	Silane, Cojet and Silane, 10% sodium ethoxide and silane and and 10% H_2O_2	Variolink II (conventional)	The results showed no significant differences between the control group and the silane treatment. The use of Cojet/silane associated with Variolink II showed the highest bond strength.

Table 3: Resin Cements Used in the Included Studies (cont.)

Author, Year	Comparison	Resin Cement	Conclusion
Novais and others, 2011[32]	Three prehydrolyzed silanes and one two-component silane followed by air-drying temperatures, 23°C and 60°C	Filtek™ Z250 Universal Restorative (composite resin)	The use of warm air-drying after silane application produced no increase in the bond strength between the fiber-reinforced composite post and the composite core.
Oliveira and others, 2011[16]	Silane and control[a]	Maxcem Elite (MXE, self-adhesive); RelyX Unicem clicker (UNI, self-adhesive); seT capsule (SET, self-adhesive); SmartCem 2 (SC2, self-adhesive); RelyX ARC (conventional)	For ARC, MXE, and SET, the silanated groups had higher bond strengths.
Perdigão and others, 2006[2]	Silane and control[a]	Post Cement Hi-X Base/Catalyst (conventional), Variolink II (conventional), ParaPost Resin Cement (conventional)	The use of a silane coupling agent did not increase the push-out bond strengths of the fiber posts used in this study.
Radovic and others, 2007[9]	Sandblasting or no pretreatment in each of the two groups; posts received three types of additional "chair-side" treatments: silane; adhesive; control[a]	Unifil Core (composite resin)	Sandblasting may give an increase in microtensile strength to methacrylate-based glass fiber posts, eliminating the need to apply additional "chair-side" treatments.
Rathke and others, 2009[33]	Silane and control[a]	Dyract Cem Plus (self-adhesive); Variolink II (conventional); Panavia F 2.0 (self-etch); RelyX Unicem (self-adhesive)	The highest mean post-to-dentin strength was measured using the etch-and-rinse luting agent, Variolink II, and the lowest mean post-to-dentin strength was measured using the etch-and-rinse luting agent, Dyract Cem Plus.
Rödig and others, 2010[34]	Control[a], silanization, sandblasting + silanization and tribochemical coating	Variolink II (conventional); Calibra (conventional); Luxacore (composite core material)	The significantly highest bond strengths were measured with the core buildup material Luxacore.
Sahafi and others, 2003[35]	Roughening (sandblasting, hydrofluoric acid etching), application of primer (Alloy Primer, Metalprimer II, silane), or roughening followed by application of primer (sandblasting or etching followed by primer, Cojet treatment)	ParaPost Resin Cement (conventional); Panavia F (self-etch)	Panavia F had significantly higher bond strength to ground ParaPost XH, Cerapost, and dentin than did ParaPost Cement.
Soares and others, 2008[36]	Silane, silane and adhesive, airborne-particle abrasion with 50-μm Al_2O_3 and silane, airborne-particle abrasion, silane, and adhesive	RelyX ARC (conventional)	Treatment with silane only was sufficient as a surface treatment for adhesive bonding.
Tian and others, 2012[37]	Silane and control[a]	ParaCore (PAR, composite resin); Relyx Unicem (RXU, self-adhesive); Relyx ARC (RXA, conventional)	PAR was significantly different from RXU and RXA ($p<0.05$). There was no statistically significant difference between RXU and RXA and between the use of silanization or not.
Zicari and others, 2012[38]	Control[a]; silane, or coated with silica-coated alumina particles	Variolink II (conventional); Clearfil Esthetic Cement (conventional); RelyX Unicem (self-adhesive)	A significantly higher push-out bond strength was recorded for the self-adhesive cement Unicem (3M ESPE).

[a] *Control indicates no treatment.*

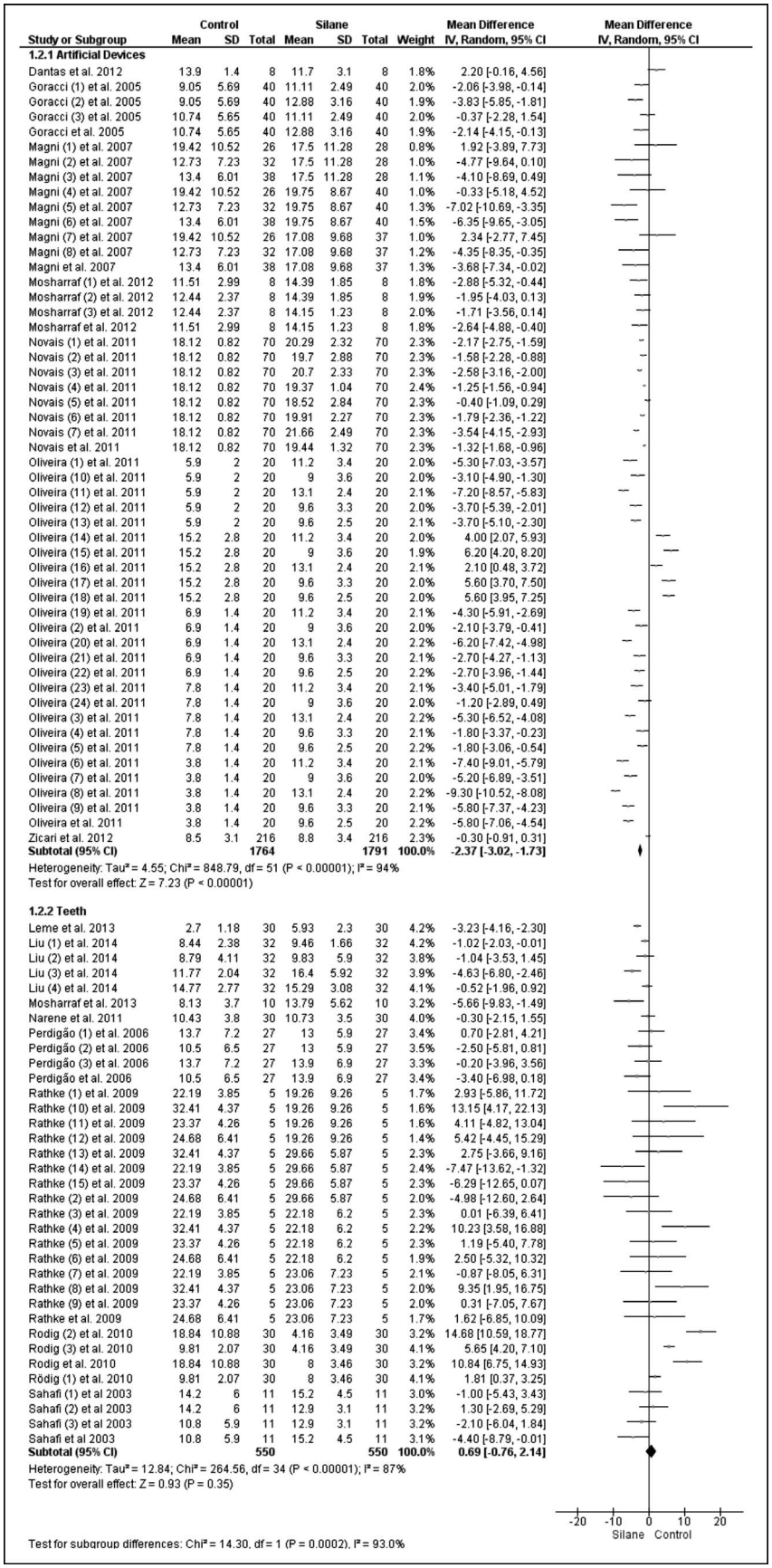

Figure 2. *Forest plot for the analysis between control group (untreated posts) and silane only. Studies that used artificial devices favored the use of silane, whereas studies that used teeth as a substrate for luting the posts reflected no significant difference.*

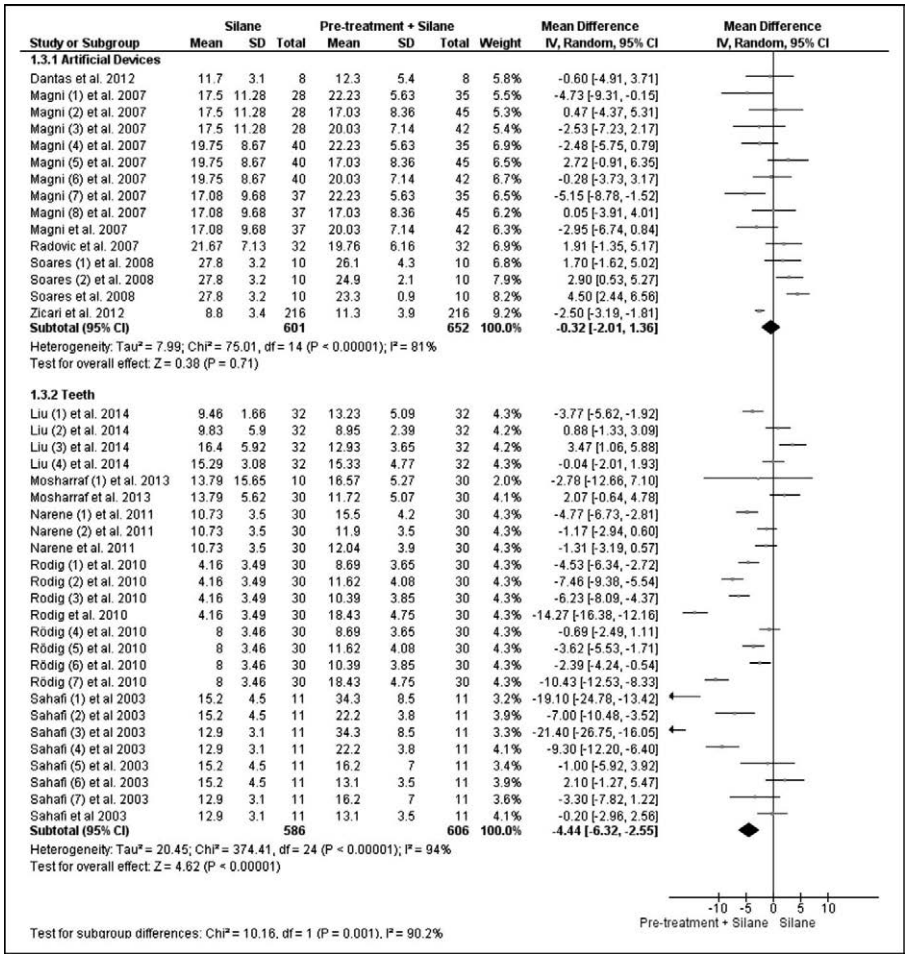

Figure 3. *Forest plot for the analysis between silane only vs pretreatment + silane. No significant difference between groups was observed when artificial devices were used, whereas the analysis favored the use of post pretreatment + silane over silane only when the posts were luted into teeth.*

were not obtained after an attempt at e-mail contact with the authors.

Table 3 shows that a wide variety of resin cements were used in the selected studies, with varied results reported. One study[27] reported that post silanization improved the adhesion of GFPs luted with self-adhesive resin cements, and 12 studies[6,22,23,26,28-32,34-36] showed a positive effect of silane on the bond strength of posts luted with regular resin cements. Yet other studies showed no significant improvement in the retention of GFPs by silanization using self-adhesive,[24] regular resin cements,[2,9,21] or both.[39] It was not possible to observe any interaction among resin cements, post silanization, or other post treatments.

Risk of Bias

The nine articles[2,27,30,31,33-35,37,39] that used teeth as substrate had the risk of bias classified as high. From the studies that used artificial devices as substrate, 11 articles[6,9,21-24,26,28,32,36,38] reported that the materials were used according to manufacturers' instructions, and none reported sample size calculation or whether blinding of the operator of the testing machine was used (Table 4).

DISCUSSION

This systematic review is the first to summarize the *in vitro* data on the influence of silanization on the retention of GFPs into root canals. Several materials, surface treatments, and cementation strategies have been tested in the literature in an endeavor to increase the retention of GFPs into root canals. Analysis of all available data together could clarify the role of silane with regard to the performance of luted GFPs and give support for the clinician in terms of evidence-based decision making. The hypothesis tested that application of silane does not improve the retention of GFPs was rejected.

Several surface pretreatments for posts have been tested to improve the bonding between GFPs and resin cements.[10-12,26,41,49-51] Pretreatment procedures aim to generally improve the adhesion to GFPs by facilitating chemical and/or mechanical

Table 4: *Risk of Bias Considering Aspects Reported in the "Materials and Methods" Section*

Author, Year of Article	Important Aspects Related to "Materials and Methods" Section—Yes (Y), No (N), Not Applied (NA)							
	Teeth Randomization	Teeth Free of Caries or Restoration	Materials Used According to Manufacturer's Instructions	Teeth with Similar Dimensions	Endodontic Treatment Performed by the Same Operator	Sample Size Calculation	Blinding of the Operator of the Test Machine	Risk of Bias
Aksornmuang and others, 2004[6]	NA	NA	Y	NA	NA	N	N	NA
Bitter and others, 2007[22]	NA	NA	Y	NA	NA	N	N	NA
Bitter and others, 2008[21]	NA	NA	Y	NA	NA	N	N	NA
Cekic-Nagas and others, 2011[23]	NA	NA	Y	NA	NA	N	N	NA
Costa Dantas and others, 2011[24]	NA	NA	Y	NA	NA	N	N	NA
Debnath and others, 2003[25]	NA	NA	N	NA	NA	N	N	NA
Goracci and others, 2005[26]	NA	NA	Y	NA	NA	N	N	NA
Leme and others, 2013[27]	Y	N	Y	N	Y	N	N	High
Liu and others, 2014[39]	Y	N	N	N	N	N	N	High
Magni and others, 2007[28]	NA	NA	Y	NA	NA	N	N	NA
Mosharraf and others, 2012[29]	NA	NA	N	NA	NA	N	N	NA
Mosharraf and others, 2013[30]	N	Y	N	N	Y	N	N	High
Narene and others, 2011[31]	Y	Y	N	N	N	N	N	High
Novais and others, 2011[32]	NA	NA	Y	NA	NA	N	N	NA
Oliveira and others, 2011[16]	NA	NA	N	NA	NA	N	N	NA
Perdigão and others, 2006[2]	Y	N	N	N	N	Y	N	High
Radovic and others, 2007[9]	NA	NA	Y	NA	NA	N	N	NA
Rathke and others, 2009[33]	N	Y	Y	Y	NA	N	N	High
Rödig and others, 2010[34]	N	N	Y	N	Y	N	N	High
Sahafi and others, 2003[35]	N	N	Y	N	NA	N	N	High
Soares and others, 2008[36]	NA	NA	Y	NA	NA	N	N	NA
Tian and others, 2012[37]	Y	N	Y	N	N	N	N	NA
Zicari and others, 2012[38]	NA	NA	Y	NA	NA	N	N	NA

interaction between the different substrates at the bonded interface. The results of the present study indicate that silanization improves the retention of GFPs only when appropriate surface pretreatment of the post is performed before application of silane. This finding is explained by the fact that the glass fibers in untreated posts are covered by the highly cross-linked, low-reactive epoxy resin. Application of surface pretreatments might expose the glass fibers, allowing more effective formation of siloxane bonds between silane and glass. The rough surface left by the surface pretreatments may also aid in improving micromechanical retention at the post-resin cement interface.[46,52]

Previous studies[16,17] have clearly indicated the positive effect that silanization might have on the bond strength between GFPs and methacrylate-based materials. However, the question that remained unanswered was whether post silanization would have a role in improving its retention into root canals. In this study, investigations that did not lute the GFPs into dental root canals or artificial root canals were excluded, since the retention analysis was the main focus here. It was noted that silanization alone is not sufficient to improve the retention of GFPs luted into root canals, whereas the combination of surface pretreatment + silanization was able to improve the retention into root canals.

Post debonding is the main reason for clinical failure of GFP-retained restorations.[7] This clinical failure type might result from poor interaction between resin cement and intraradicular dentin and/or poor interaction of resin cement and post. The findings of the present study indicate that when the posts were luted into natural root canals, the combination of post pretreatment + silanization significantly improved the post retention. This result is explained by a better interaction between resin cement and post surface leading to a situation in which the mechanical stresses during testing concentrate at the interface between the resin cement and root dentin only. In such a scenario, the better mechanical keying at the post-cement interface does not contribute significantly toward stress concentration and/or magnification during the test, leading to higher bond strength values.

In contrast to the findings from studies performed using extracted teeth, no significant improvement in the retention of GFPs was observed for the combination of post pretreatment + silane when the posts were luted into artificial devices. When artificial devices are used, there is no dentin-resin cement interface; in other words, the resin cements used to lute the posts do not interact with dental hard tissues but rather with synthetic materials such as methacrylate-based composites. In such a scenario, the interaction of the cement with the artificial devices is expected to be improved as compared with that associated with dentin, which is acknowledged to be the weakest link in adhesive bonding. In addition, the use of artificial devices usually does not have the same limitations that are imposed upon extracted teeth, such as great variability in root canal diameter and resin cement film thickness between specimens. Therefore, it is suggested that the use of artificial devices to lute GFPs should be restricted to situations in which the post-cement interface is the main focus of the investigation.

Among the surface pretreatments tested in the included studies, sandblasting stands out as the pretreatment most often used. A total of 80% of comparisons carried out here on the effect of surface pretreatments on the retention of GFPs into artificial devices, and ~62% of the comparisons on the retention of GFPs into root canals, used sandblasting as pretreatment. As an overall result, the present findings indicate a positive effect of surface pretreatments before silanization; however, this result should be mainly concentrated at the combination of sandblasting + silanization on the retention of GFPs, because most studies only tested that specific combination. That notwithstanding, surface pretreatments that only selectively expose the glass fiber by chemical means could be considered the ideal situation to enhance the silanization effect. Sandblasting is known not to be selective in exposing the glass and may cause structure damage to the post, although there is no evidence regarding whether this could affect the mechanical stability of post-and-core restored teeth.

Different mechanical tests to measure the bond strength and a wide variety of adhesives and resin cements are reported in the *in vitro* literature, resulting in a tough scenario for one seeking comparisons between the results of different studies. Authors sometimes do not follow the manufacturers' directions in applying materials, underscoring the problem of comparing studies in the literature. Systematic reviews have the advantage of analyzing the literature data together, but they also suffer from the limitation that the methods employed in distinct studies differ to extents that often are difficult to predict. With that in mind, we have used a tool to assess the risk of bias of each study.

The results indicate that all selected studies present a high risk of bias, demonstrating that

variables that could influence the results of the studies were not controlled by researchers, favoring the high heterogeneity of the findings in the present study. However, the risk of bias assessment can be subjective and should be interpreted as such. Heterogeneity among the studies was in fact expected, since it is known that laboratory analyses have intrinsic variability related to experimental setups, procedures for specimen preparation, and the mechanical tests themselves.

The results of the present review should be interpreted with caution considering that laboratory studies have intrinsic limitations in terms of simulating *in vivo* conditions. However, the improved retention of GFPs by a combination of post pretreatment and silanization is of particular interest, bearing in mind that it could affect the clinical survival of GFP-retained restorations. Additionally, clinicians should be aware of the beneficial effects that post silanization might have on the clinical performance of restoration, particularly because post silanization is a procedure that might be overlooked in the clinical practice if it is regarded as being of minor significance. Furthermore, it is important to know if the posts are commercially available in a presilanized or pretreatment form by the manufacturer. For this reason, following the manufacturers' recommendations when preparing the GFPs before luting is necessary. Regardless of the results presented here, well-designed randomized controlled clinical trials (RCTs) with long follow-up periods would provide the ultimate answer as to whether use of a silane coupling agent could result in improved clinical success rates for GFP-retained restorations. However, it is known that RCTs cannot be used indiscriminately to support all clinical decisions. Therefore, the overall results of the present study favor the combination of post surface pretreatment and silanization for the retention of GFPs.

CONCLUSIONS

Analysis of the *in vitro* literature indicates that silanization improves the retention of GFPs luted into root canals provided that selective surface pretreatments are applied to the post before silanization.

Regulatory Statement

This study was conducted at the Federal University of Pelotas, Graduate Program in Dentistry, in Brazil.

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 Research

Influence of Isolation Method of the Operative Field on Gingival Damage, Patients' Preference, and Restoration Retention in Noncarious Cervical Lesions

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Clinical Relevance

The use of cotton rolls/retraction cord is as effective as rubber dam isolation for restoration of noncarious cervical lesions. In addition, patient's preference, gingival damage, or chairside time was similar for both isolation techniques.

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SUMMARY

Objectives: To evaluate the retention rates, gingival damage, and patients' preferences for adhesive restorations in noncarious cervical lesions (NCCL) associated with the use of rubber dam vs cotton rolls/retraction cord isolation.

Methods: Thirty patients having one pair of similar NCCL on opposing sides of the same arch were enrolled in this study. A total of 60 restorations were placed. In each patient one restoration was placed under rubber dam isolation (RD) using dental retainers, and the other one was placed using cotton rolls/retraction cord (CR/RC) isolation. Dental residents with more than 10 years of clinical experience restored all NCCL using the same adhesive (GO!, SDI Limited, Bayswater, Australia) and composite resin (Ice, SDI). The patients' preferences were recorded. The gingival condition

(bleeding, gingival laceration, and gingival insertion level) was evaluated immediately after the restorative procedure and after one week. Gingival sensitivity was also assessed one week after the end of the restorative procedures. The clinical time required to perform each restoration was recorded. The performance of the restorations was assessed using the FDI criteria at baseline and six, 12, and 18 months after clinical service. All criteria evaluated were submitted to appropriate statistical analysis (α=0.05).

Results: The retention rates of the restorations at each recall time were not affected by the isolation method (p>0.05). No significant difference between isolation methods was found in regard to patients' preferences (p=0.86), gingival bleeding (p=0.57), laceration (p=0.64), insertion (p>0.52), gingival sensitivity (p=0.52), or chairside time (p=0.77).

Conclusions: The use of CR/RC was shown to be similar to the use of RD in terms of retention rates, patient's preference, gingival damage, and chairside time for adhesive restorations in NCCL.

INTRODUCTION

Nowadays, many patients demand that their dental restorations not only function well but also resemble natural teeth. It is well known that resin composite and adhesive systems are especially technique sensitive,[1] given that proper material handling and adequate field isolation are critical for the success and longevity of the restorations.

This means that the adhesive procedures need to be performed on clean tooth surfaces without the presence of any contaminants such as intraoral humidity, saliva, and gingival/sulcular fluid or blood.[2,3] Contaminants have been shown to jeopardize the bonding effectiveness of adhesive systems to the dental structure.[4,5]

For more than a century, a rubber dam has been considered the optimal method to isolate a dental operating field. Rubber dam isolation prevents moisture contamination during the placement of direct restorations and endodontic treatments,[2,3,6] and most dental schools regularly teach this method.[7] Many faculty members consider rubber dams to be an essential component of modern adhesive dentistry, [8,9] and many advantages have been listed elsewhere.[2,3]

However, the use of a rubber dam during operative dentistry procedures in a private practice is not common.[6,7,9,10] In one of the most relevant studies of rubber dam use, which involved a questionnaire completed by US general dentists, 53% of the dentists reported that they had never used a rubber dam for amalgam restorations, 45% had never used a rubber dam for anterior direct resin composites, and 39% had never used a rubber dam for posterior direct resin composites. More recently, Gilbert et al.,[10] in a practice-based study, collected data on 9890 consecutive restorations done in previously unrestored tooth surfaces from 5810 patients. Most dentists (63%) in this study did not use a rubber dam for any restoration. A rubber dam was used for only 12% of restorations. Reasons for not using a rubber dam in routine practice include patient discomfort, insufficient time, technical difficulty, insufficient training, and the cost and low fees for treatment.[6,7,9]

Ryan O'Connell[2] and Mala and others[3] reported that almost 50% of the clinicians evaluated in a survey considered rubber dams difficult to apply, and almost 50% felt that adult patients do not like it. There is not much evidence to support the following cited claims: patient acceptance/discomfort and insufficient time, technical difficulty, insufficient training/lack of skill, and costs and fees. In fact, there are studies that support and contradict each of these claims.[9]

The influence of a rubber dam vs cotton rolls/retraction cord isolation on the performance of adhesive restorations is also the subject of controversy. Although three meta-analyses revealed no influence of the type of isolation on the survival rate of posterior composite restorations[11,12] or that of anterior composite restoration,[13] most of the clinical trials were observational and not prospective.[10] It might be more appropriate to evaluate both methods of isolation in a prospective, split-mouth design to determine the patients' actual perceptions of both procedures.

Few clinical studies[14,15] have attempted to address these issues for noncarious cervical lesions (NCCL), where placement of dental retainers is considered more challenging. Thus, the primary outcome of this examiner-blind, randomized clinical trial was to evaluate whether the type of isolation technique (rubber dam vs cotton rolls/retraction cord) influenced the retention rates of NCCLs bonded with a one-step, self-etch adhesive over the course of six, 12, and 18 months. As secondary outcomes, we also compared the chairside time, the gingival damage, and the patients' preferences toward the use of

Table 1: *Characteristics of Research Subjects and Features of the NCCLs Included in This Study for Both Study Groups*

Characteristics of Research Subjects	Patients, n	
Gender distribution		
Male	12	
Female	18	
Age distribution, y		
20-29	0	
30-39	6	
39-49	24	
>49	30	
Characteristics of Class V lesions	**Lesions, n**	
Shape, ° of angle	RD	CR/RC
<45	6	8
45-90	14	16
90-135	10	6
>135		
Cervico-incisal height, mm		
<1.5	8	6
1.5-2.5	12	16
2.5-4.0	10	6
>4.0	0	2
Degree of sclerotic dentin		
1	0	0
2	14	12
3	16	18
4	0	0
Presence of antagonist		
Yes	30	30
No	0	0
Attrition facet		
Yes	0	0
No	30	30
Preoperative sensitivity (spontaneous)		
Yes	0	0
No	30	30
Preoperative sensitivity (air dry)		
Yes	18	16
No	12	14
Preoperative sensitivity (touch)		
Yes	16	18
No	14	12
Tooth distribution		
Anterior		
Incisor	6	2
Posterior		
Premolar	24	28
Molar	0	0
Arch distribution		
Maxillary	10	12
Mandibular	20	18

Abbreviations: CR/RC, cotton rolls/retraction cord; NCCLs, noncarious cervical lesions; RD, rubber dam.

rubber dams vs cotton rolls/retraction cord isolation. The null hypothesis to be tested was that the 18-month retention rates of adhesive restorations placed in NCCLs are similar for both isolation methods.

METHODS AND MATERIALS

Patient and Lesion Selection

The local Ethics Committee on Investigations Involving Human Subjects reviewed and approved the protocol and consent form for this study (protocol 6291/06). This study was reported in accordance with the Consolidated Standards of Reporting Trials statement.[16]

Study Design—This was an examiner-blind, split-mouth randomized clinical trial. The study was carried out in the Clinic of the School of Dentistry at the State University of Ponta Grossa (Paraná, Brazil) from April-November 2010. We informed all participants about the nature and the objectives of the study.

Inclusion Criteria—Two calibrated dental residents screened patients to determine whether they met the study entry criteria. The qualified patients were recruited in the order in which they reported for the screening session, thus forming a convenience sample.

The evaluations were performed using a mouth mirror, an explorer, and a periodontal probe. All participants were healthy and had at least 20 teeth. Each participant had at least one pair of similarly sized NCCLs, without undercuts. The lesions were located in the same arch but on opposing sides. All teeth selected for the study had occlusal contacts, with no more than 50% of the cavo-surface margin involving enamel.[17,18] Patients must also have been willing to sign the informed consent form before starting treatment. All baseline details relative to the research subjects and the NCCLs are displayed in Table 1.

Exclusion Criteria—Patients with extremely poor oral hygiene, criteria 2 and 3 of periodontitis,[19] or with heavy bruxism habits were excluded from the

study. Patients with NCCLs exhibiting self-reported spontaneous hypersensitivity were also excluded.[20] Of 51 patients, 21 were excluded from the study because they did not fulfill the inclusion criteria. Thus, a total of 30 subjects (12 men and 18 women), with a mean age of 45 years were enrolled in this study.

Characterization of Noncarious Cervical Lesions— The degree of sclerotic dentin was measured according to the criteria described by Swift and others.[20] The lateral visualization of the cavity allowed the determination of the angle of the cavity (<45°; 45°-90°; 90°-135°; >135°). The gingival-incisal height of the cavity was measured using a periodontal probe. Other features such as presence of antagonist and attrition facet were also observed and recorded to allow identification of comparability of the groups at baseline (Table 1).

Before evaluation, the examiners were trained in the criteria of Swift and others[20] for evaluation of the sclerotic dentin and cavity angle. They observed 15-20 photographs that were representative of each score in each criterion (n=4), and the criteria were discussed with an expert. After that and on two further occasions, they evaluated 10-20 teeth that were not included in the study sample. An intra-examiner and interexaminer agreement of at least 85% was necessary before we began the evaluation.[21]

Clinical photographs were taken prior to the beginning of the treatment, at an original magnification of 1.5× using a digital camera (D70; Nikon Inc, Melville, NY, USA) with a 120-mm Medical Nikkor lens (Nikon Inc) to record the periodontal conditions. Gingival conditions (gingival insertion level and bleeding) were evaluated using the Löe and Silness Gingival Index.[22] A blunt instrument, such as a periodontal probe, was used to assess the bleeding potential of the gingival tissues[22] and the gingival sensitivity (yes/no). One calibrated dental resident specializing in periodontics performed this exam. Before evaluation, the examiner was trained in the criteria for the Löe and Silness Gingival Index,[22] by one professor, a specialist in periodontics with more than 15 years of experience. They observed 10-20 photographs that were representative of each score. After that and on two occasions, they evaluated 10-20 teeth that were not included in the study sample. An intraexaminer and interexaminer agreement of at least 85% was necessary before we began the evaluation.[21]

Operative Procedure and Experimental Design— All patients were given oral hygiene instructions prior to the operative treatment. The same calibrated dental residents who participated in the patient screening restored all teeth under the supervision of an experienced clinician. The dental residents were clinical professors who were at the end of their doctoral courses at the School of Dentistry at the State University of Ponta Grossa (Paraná, Brazil). At the time the study was conducted, they each had clinical experience of more than 10 years.

For the calibration procedure step, the experienced clinician placed one restoration from each group to identify all restorative steps involved in the application technique. Then, each operator placed two restorations per group, also under the supervision of the experienced clinician. The restoration deficiencies were shown to the clinician prior to starting the study. Only after that were the operators considered calibrated to perform the restorative procedures.

A maximum of two restorations per patient were placed, one from each group. A total of 60 restorations were placed, with 30 for each group. In each subject, the choice of each isolation method was randomly determined by tossing a coin before the restorative intervention in order to guarantee concealment of the allocation. Both restorations were placed in the same day. The patient and the operator were not blinded to the procedure, but the examiner was.

In the RD group, the rubber dam was inserted (Madeitex, São José dos Campos, Brazil) along with the rubber dam retainer No. 212 (KG Sorensen, Barueri, Brazil). In the CR/RC group, a mouth retractor (Arc-Flex, FGM Dent Prod Ltda, Joinville, Brazil) was applied, and cotton rolls and saliva ejectors were used to keep the operative field dry. The gingival tissue of teeth from the CR/RC group was retracted with the retraction cord (Proretract, FGM Dent Prod Ltda).

After allocating the groups, the proper shade of the composite was determined using a shade selection guide (Ice Shade Guide, SDI Limited, Baywater, Australia). Both lesions were restored in the same clinical appointment, and the order of the procedure varied from patient to patient.

To determine the need for dental anesthesia, the dental retainer or retraction cord was placed and the operators asked the patient if he or she felt any discomfort. In the case of a positive answer, teeth were anesthetized (Citanest, Dentsply, Petrópolis, Brazil). In the case of a negative answer, the operative intervention continued on from that point. The lesions were cleaned with pumice and water in a rubber cup (Ref No. 8040RA and No. 8045RA, KG

Table 2: *Composition of the Materials Used in This Study*

Adhesive Systems	Composition	Application Mode
Go! (SDI Limited, Bayswater, Victoria, Australia)	Phosphoric acid ester monomer; dimethacrylate monomer; monomethacrylate monomer; silicon dioxide filler; water; acetone; photoinitiators; stabilizer and sodium fluoride	1. Apply first coat of adhesive to saturate all surfaces. 2. Leave in place for 20 s. 3. Blow with dry, high-pressure, oil-free air for 10 s to evaporate solvent. 4. Apply second coat of adhesive to saturate all surfaces. 5. Leave in place for 20 s. 6. Blow with dry, high-pressure, oil-free air for 10 s to evaporate solvent. Leave surface glossy. 7. Light-cure for 10 s with an LED light (Radii-Cal LED, SDI Limited).
Ice (SDI Limited, Bayswater, Victoria, Australia)	22.5% wt (39% vol) multifunctional methacrylic ester and 77.5% wt (61% vol) inorganic filler (40 nm-1.5 μm).	8. Place composite in increments of 2 mm or less. 9. Light-cure each increment for at least 20 s using an LED light (Radii-Cal LED).

Sorensen, Barueri, Brazil), followed by rinsing and drying procedures. The operators did not prepare any additional retention or bevel in the NCCL, according to the guidelines of the American Dental Association.[23]

The self-etch adhesive system GO! (SDI Limited) and the composite resin Ice (SDI Limited) was applied according to the manufacturer's directions (Table 2). The cavities were restored in three increments, and each one was light-cured for 20 seconds (Radii-cal, SDI Limited; 800 mW/cm^2).

The restorations were finished with fine-grit diamond burs and the polishing procedure was performed with abrasive discs (Sof-Lex Pop-On discs, 3M ESPE, St Paul, MN, USA) immediately after placing the restorations. The time required for the restorative intervention from the beginning of the isolation procedure until the final polishing was recorded for both groups.

Clinical Evaluation

Patients' Preferences—Immediately after removal of each isolation method, we asked the patients about which method of isolation they preferred.

Evaluation of the Gingival Tissue Damage—Clinical photographs were taken immediately after the restorative procedure and after one week using the same parameters as for the baseline picture. The presence of gingival laceration (yes/no) was evaluated immediately after the restorative procedure and again after one week. The gingival condition (gingival insertion level and bleeding) was evaluated as described earlier only after one week of the restorative procedure. Gingival sensitivity (yes/no) after one week following the procedure was also evaluated by asking the patient if he or she had any kind of sensitivity in the gingivae.

Performance of Adhesive Restorations—We used the FDI criteria[24,25] to evaluate the restorations at baseline and after six, 12, and 18 months of clinical service. Only the most relevant items for testing the adhesive performance were selected (Table 3): marginal staining, fractures/retention, marginal adaptation, postoperative sensitivity, and recurrent caries.

Two experienced examiners, blinded to the group assignment, performed the follow-up examinations using a mirror and a double probe. Both examiners evaluated all the restorations once and independently. When disagreements occurred during the evaluations, they had to reach consensus.

Calibration Step—Before evaluation, the examiners were trained in the FDI criteria.[24,25] They observed 10 photographs that were representative of each score for each criterion. After that and on two occasions, they evaluated 10-15 teeth that were not included in the study sample. An intraexaminer and interexaminer agreement of at least 85% was necessary before we began the evaluation.[21] The training was performed by one professor, a specialist in restorative dentistry with more than 15 years of clinical and research experience.

Statistical Analysis

The patients' preferences, the need for anesthesia, presence of laceration, self-report of gingival sensitivity, and the one-week gingival bleeding between the two groups were evaluated by the McNemar test.

Due to the non-normal distribution of the data from the gingival insertion level, the baseline and one-week levels for each isolation method were

Table 3: *World Dental Federation (FDI) Criteria Used for Clinical Evaluation*[24,25]

Classification	Esthetic Property	Functional Properties		Biological Properties	
	1. Staining margin	2. Fractures and retention	3. Marginal adaptation	4. Postoperative (hyper-) sensitivity	5. Recurrence of caries
1. Clinically very good	1.1 No marginal staining.	2.1 Restoration retained, no fractures/ cracks.	3.1 Harmonious outline, no gaps, no discoloration.	4.1 No hypersensitivity.	5.1 No secondary or primary caries
2. Clinically good (after correction very good)	1.2 Minor marginal staining, easily removable by polishing.	2.2 Small hairline crack.	3.2.1 Marginal gap (50 μm). 3.2.2 Small marginal fracture removable by polishing.	4.2 Low hypersensitivity for a limited period of time.	5.2 Very small and localized demineralization. No operative treatment required
3.Clinically sufficient/ satisfactory (minor shortcomings with no adverse effects but not adjustable without damage to the tooth)	1.3 Moderate marginal staining, not esthetically unacceptable.	2.3 Two or more or larger hairline cracks and/or chipping (not affecting the marginal integrity).	3.3.1 Gap $<$ 150 μm not removable. 3.3.2. Several small enamel or dentin fractures.	4.3.1 Premature/ slightly more intense. 4.3.2 Delayed/weak sensitivity; no subjective complaints, no treatment needed.	5.3 Larger areas of demineralization, but only preventive measures necessary (dentin not exposed).
4. Clinically unsatisfactory (repair for prophylactic reasons)	1.4 Pronounced marginal staining; major intervention necessary for improvement.	2.4 Chipping fractures which damage marginal quality; bulk fractures with or without partial loss (less than half of the restoration).	3.4.1 Gap $>$ 250 μm or dentin/base exposed. 3.4.2. Chip fracture damaging margins. 3.4.3 Notable enamel or dentin wall fracture.	4.4.1 Premature/ very intense. 4.4.2 Extremely delayed/weak with subjective complaints. 4.4.3 Negative sensitivity intervention necessary but not replacement.	5. 4 Caries with cavitation (localized and accessible and can be repaired.
5. Clinically poor (replacement necessary)	1.5 Deep marginal staining not accessible for intervention.	2.5 (Partial or complete) loss of restoration.	3.5 Filling is loose but *in situ*.	4.5 Very intense, acute pulpitis or nonvital. Endodontic treatment is necessary and restoration has to be replaced.	5.5 Deep secondary caries or exposed dentin that is not accessible for repair of restoration.

compared with each other by the Wilcoxon signed rank test. The gingival insertion level of the two groups, at each period, was also evaluated by the Wilcoxon signed rank test. The total time required for the restorative procedure was evaluated by Student *t*-test for dependent samples.

The differences in the ratings of the two groups in each recall time (six, 12, and 18 months) were tested with the Fisher exact test (α=0.05), and differences in the ratings of each recall time (six, 12, and 18 months) vs baseline findings were compared using the McNemar test (α=0.05).

RESULTS

Patients' Perceptions and Gingival Conditions

Two patients slept during the restorative procedure, and therefore they did not report any preference for the isolation methods. Fifteen and 13 patients preferred the rubber dam and cotton roll/retraction cord, respectively, and this difference was not statistically significant (p=0.86). The total time required to perform the restoration of NCCL lesions was 20.8 ± 5.2 minutes and 21.2 ± 5.2 minutes for the RD and CR/RC groups, respectively, and this difference was not significant (p=0.77).

Approximately 70% of the patients required anesthesia, and no significant difference was observed between groups (Table 4; p=1.00). Gingival bleeding and laceration were more common for the RD group in comparison with CR/RC group; however, this difference was not significant (Table 4; p=0.57 and p=0.64, respectively). No significant difference in gingival sensitivity was reported after one week between isolation methods (Table 4; p=0.52).

The median of gingival insertion levels (mm) at baseline and at one week were similar for both isolation methods, and no significant differences between them were observed when the baseline (p=0.82) and 1-week gingival insertion levels (p=0.56) were compared with each other (Table 5).

Table 4: *Comparison of the Patient's Preference, Need for Anesthesia, Gingival Laceration, Gingival Bleeding, and Sensitivity for the Study Group (%, 95% Confidence Interval [n])*

Characteristic	RD	CR/RC	p-Value*
Patient's preference	54 (36-71) [15]	46 (30-64) [13]	0.86
Needed anesthesia	67 (49-81) [20]	70 (52-83) [21]	1.00
Gingival laceration	63 (46-78) [19]	37 (22-55) [11]	0.64
1 wk gingival bleeding	60 (42-75) [18]	47 (30-64) [14]	0.57
1 wk gingival sensitivity	43 (27-61) [13]	27 (14-45) [8]	0.52

Abbreviations: CR/RC, cotton rolls/retraction cord; RD, rubber dam.
** McNemar test.*

Performance of the Adhesive Restorations

All research subjects attended the follow-up recalls. None of the patients reported postoperative sensitivity, and we did not detect caries at any of the follow-up periods.

In regard to retention, the six-month retention rates (with 95% confidence interval in parentheses) of the restorations were 93% (79%-98%) for both isolation methods, and after 12 months, 90% (74%-96%) and 86% (70%-95%), respectively, for the RD and CR/RC groups. At the 18-month recall, the retention rates were 73% (55%-86%) for the RD group and 73% (55%-86%) for the CR/RC group, with no statistical difference between any pair of groups at the six, 12, and 18-month recall ($p>0.05$). A significant difference was detected between the 12-month and 18-month data compared with the baseline for both groups (Table 6; $p<0.05$). An overall retention rate after 18 months of only 73% (55%-86%) was observed for the adhesive tested.

Only a few restorations were considered to have clinically relevant discrepancies in the item fracture (Table 6), but no significant difference was detected between any pair of groups at the six-, 12-, and 18-month recall and for each group when the six-, 12-, and 18-month times were compared with baseline ($p>0.05$).

In regard to marginal staining and marginal adaptation, no significant difference was found between groups at each recall time and for each group when the six-, 12-, and 18-month times were compared with baseline (Table 6; $p>0.05$). Some clinical cases can be found in Figures 1-3.

DISCUSSION

Although one of the aims of rubber dam isolation is to protect soft tissues against physical and chemical trauma resulting from the operative procedure,[3,4] the occurrence of gingival abscess was already reported due to the retention of the rubber dam into the gingival sulcus.[26,27] In addition, the use of metallic retainer retractors in areas with a narrow width of keratinized gingiva can contribute to the occurrence of gingival recession.[28] Actually, both methods can cause injuries to the periodontium to some degree, although it is worth noting a high repair capacity of the gingival tissue, causing almost no pain or discomfort for the patients.[29]

The present study observed gingival laceration and bleeding immediately after placement of the restoration, but this was not restricted to the RD group. These findings are in agreement with a recent study published by Daudt and others[15] in which the authors demonstrated that the use of a rubber dam in NCCL restoration resulted in a significantly higher gingival recession only immediately after isolation; this is not statistically different from the results of the CR/RC group one week after the restorative procedure.

Table 5: *Comparison of Gingival Insertion Level Between the Study Groups, Median (Minimum/Maximum)*

Groups	Median of Gingival Insertion Level		p-Value*
	Baseline	1 wk After	
RD	1 (0/3)	1 (0/3)	0.78
CR/RC	1 (0/3)	1.25 (0/3)	0.48
p-Value*	0.82	0.56	

Abbreviations: CR/RC, cotton rolls/retraction cord; RD, rubber dam.
** Wilcoxon test for independent samples ($\alpha = 0.05$).*

Table 6: *Number of Evaluated Restorations for Each Experimental Group Classified According to the World Dental Federation (FDI) Criteria*[24,25]

FDI Criteria	RD				CR/RC			
	Baseline	6-mo	12-mo	18-mo	Baseline	6-mo	12-mo	18-mo
1.Marginal staining								
VG	30	26	21	15	30	25	20	16
GO	—	2	4	4	—	2	3	4
SS	—	—	—	—	—	—	—	—
UN	—	—	—	—	—	—	—	—
PO	—	—	—	—	—	—	—	—
2. Fractures and retention								
VG	30	28	25	19	30	27	23	20
GO	—	—	—	—	—	—	—	—
SS	—	—	2	3	—	1	3	2
UN	—	2	3	8	—	2	4	8
PO	—	—	—	—	—	—	—	—
3. Marginal adaptation								
VG	30	24	20	14	30	23	19	16
GO	—	4	5	5	—	4	4	4
SS	—	—	—	—	—	—	—	—
UN	—	—	—	—	—	—	—	—
PO	—	—	—	—	—	—	—	—
4. Postoperative (hyper-) sensitivity								
VG	30	28	25	19	30	27	23	20
GO	—	—	—	—	—	—	—	—
SS	—	—	—	—	—	—	—	—
UN	—	—	—	—	—	—	—	—
PO	—	—	—	—	—	—	—	—
5. Recurrence of caries								
VG	30	28	25	19	30	27	23	20
GO	—	—	—	—	—	—	—	—
SS	—	—	—	—	—	—	—	—
UN	—	—	—	—	—	—	—	—
PO	—	—	—	—	—	—	—	—

Abbreviations: CR/RC, cotton rolls/retraction cord; GO for clinically good; PO for clinically poor; RD, rubber dam; VG for clinically very good; SS for clinically sufficient/satisfactory; UN for clinically unsatisfactory.

This means that the damage caused by both isolation methods is reversible and not persistent; the gingival tissue can be readily repaired in a way such that fewer than half of the patients complained of pain or discomfort one week after the procedure. The gingival insertion one week after the restorative procedure was similar to the baseline for both groups, which adds evidence that the damage produced by the isolation is reversible.

Contrary to the belief that the use of rubber dam isolation is more time consuming,[6,7,9] the present study demonstrated that the time required for the placement of NCCL restorations was not affected by the choice of the isolation method. This is related to the fact that the operators who placed the restorations in the present study are resident dentists with more than five years of clinical experience in operative dentistry. In addition, the restorations were all placed in the university environment, although there are some concerns regarding the difference between this kind of clinical trial and the results found in practice-based research.[30] Unfortunately, this hypothesis needs to be tested for restorations placed by operators with different levels of experience and in different environments.

The preferences of the patients for the isolation methods were similar. There is a widespread belief that RD isolation is more effective for preventing

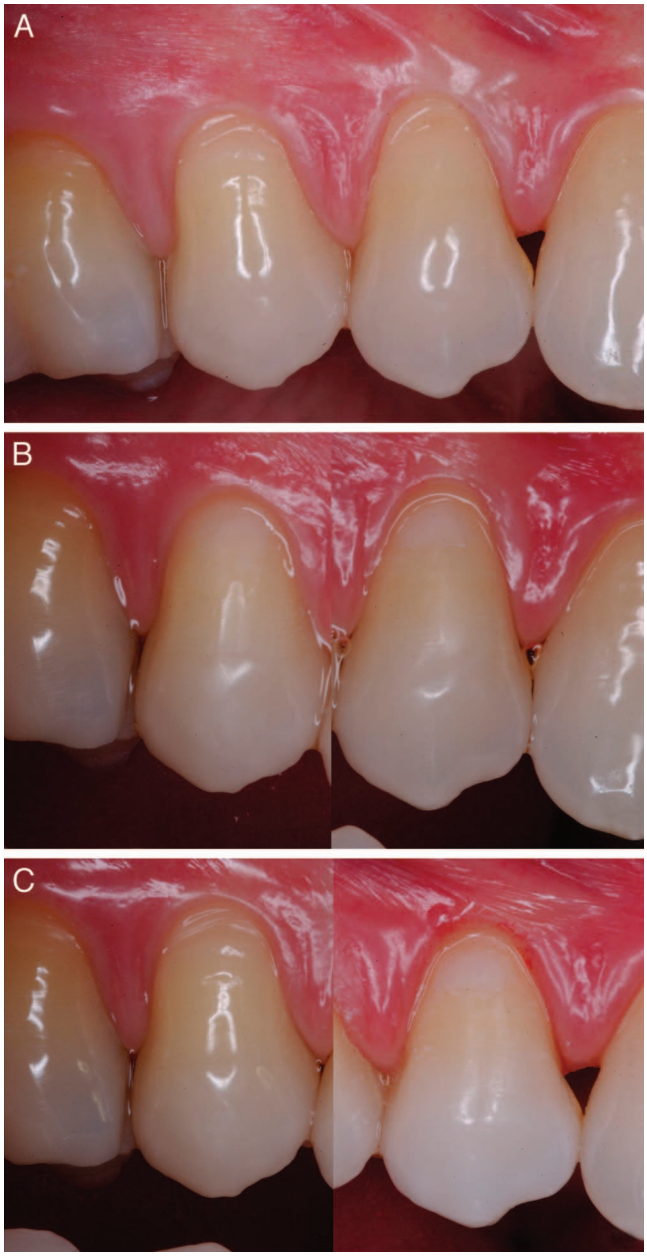

Figure 1. *Noncarious cervical lesions on upper first and second premolar (A): before and (B): after restorations and (C): at the 18 month clinical evaluation. Observe the lack of retention after 18 months for the restoration of upper second premolar (cord retraction).*

contamination of the operative field. However, this was not observed in the present investigation. Regarding the adhesive performance, we expected to find a better clinical performance with the use of a rubber dam because in theory, its use provides a cleaner and contamination-free surgery field, without saliva and gingival fluid. However, the results of the present study did not prove this hypothesis, leading us to accept the null hypothesis.

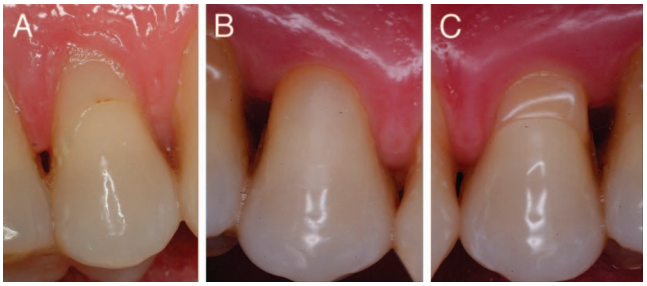

Figure 2. *Noncarious cervical lesions on upper second premolar (A): before and (B):after restorations and (C): at the 18 month clinical evaluation (cord retraction). It was classified as clinically good in marginal adaptation and marginal discoloration (see enamel margin).*

The results are in line with a recently published meta-analysis of NCCL clinical studies.[12] Heintze and Rousson[12] evaluated 105 studies, and a rubber dam was used in 47. Although the clinical success rate of restorations applied with a rubber dam

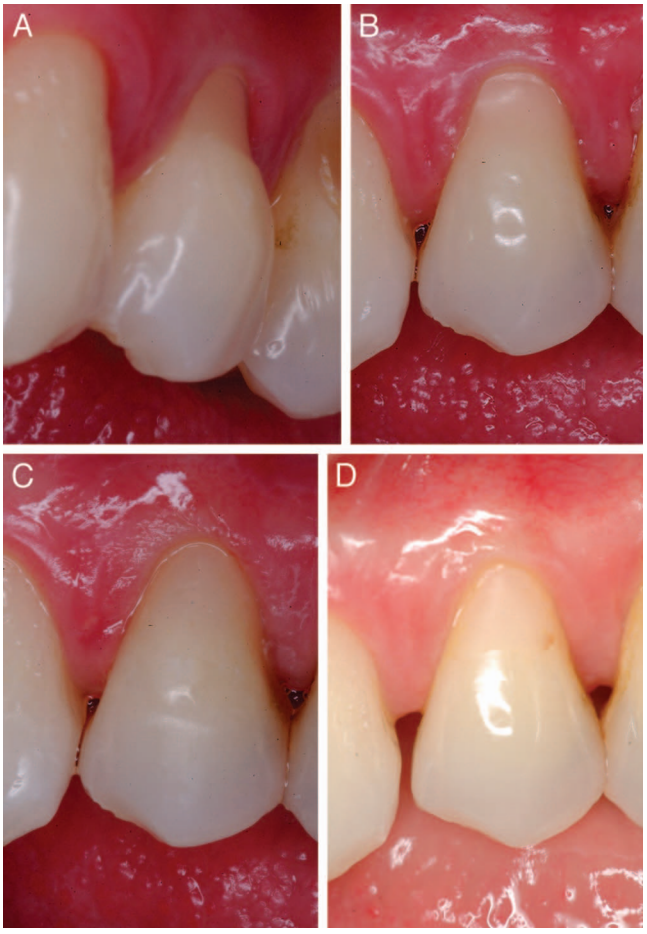

Figure 3. *(A): Lateral and (B): frontal view of noncarious cervical lesions on upper first premolar before and (C): after restorations (cord retraction) and (D): at the 18 month clinical evaluation. It was classified as clinically good in the marginal discoloration (see enamel margin).*

compared with cotton rolls/retraction cords showed a trend toward increased retention, the difference did not reach statistical significance. One cannot rule out the fact that experienced and trained clinicians placed the restorations in the present and in Daudt and others' study,[15] meaning that a surface free of contamination from saliva and the gingival fluid was also obtained with the CR/RC group. Again, whether this would be applicable in practice-based research, where clinicians are not extensively trained on both methods, or in other types of dental cavities, where the moisture control is more challenging, is yet to be addressed and deserves further investigation.

Previous systematic reviews of the literature have pointed out that one-step self-etch systems have higher annual failure rates than do other adhesive strategies.[12,31] This was also observed in laboratory studies: relatively low bond strength values to enamel and dentin were usually observed for this type of bonding strategy.[32-35] This is probably due to the more complex chemistry they require, because hydrophilic and hydrophobic monomers, water, solvent, and others components need to be blended in a single bonding solution.[36,37]

Nonetheless, the overall retention rates of the adhesive GO! in the present study were much lower than the average commonly reported by the literature for one-step self-etch adhesives. Almost 27% of the restorations debonded after a short 18-month follow-up. Although this was not the main objective of this clinical trial, it provided clinical information about a specific brand of adhesive that is clearly relevant to clinical practitioners.

In a recent laboratory study, the adhesive GO! produced the lowest microtensile bond strength values to dentin,[35,38] with a high percentage of premature failures. In addition, it was already reported that most of the teeth from the GO! group debonded completely during preparation for the microtensile bond strength test.[35,39] Under the manufacturer's instructions, a higher amount of silver nitrate was also observed in the hybrid layer produced with GO! in a previous study.[39]

Hass and others[39] hypothesized that a less than average performance in the laboratory tests could be due to the presence of a high acetone content, which resulted in the formation of a very thin hybrid layer.[40] The thinner the adhesive layer, the more susceptible it is to polymerization inhibition by oxygen.[41] In the same study, a low degree of conversion was observed inside the hybrid layer for the adhesive GO!.[39]

Altogether, the laboratory findings suggest that the poor polymerization and bonding of this material can be related to an inadequate equilibrium among the chemicals included in the GO! formulation. For instance, GO! is a 2-hydroxyethyl methacrylate (HEMA)-free adhesive. Some research has revealed that HEMA-free one-step adhesives are prone to phase separation, which may also account for their lower bonding effectiveness.[42,43]

However, one cannot omit mentioning that the quite low retention rates of the adhesive GO! in the present study were not observed in a study by Burrow and others.[44] Those authors reported an overall retention rate of 85% after three years. Different from the present study, Burrow and others[44] light-cured the adhesive for 20 seconds instead of the 10 seconds recommended by the manufacturer. The higher exposure time could have led to an increased degree of conversion[39] and thus improved its clinical performance.

Another difference from both studies is that selective enamel etching was performed before adhesive application in the study by Burrow and others.[44] Although selective enamel etching has not been associated with increased retention rate but only with reduced marginal discrepancies,[45,46] this technique was only evaluated for two-step self-etch adhesives.[45,46] Perhaps for one-step adhesives, selective enamel etching may also aid in restoration retention, but this should be the focus of future investigations.

Although one might suppose that cavity preparation would increase the retention rates of the materials, we did not perform any cavity preparation in this clinical trial basically for three reasons: 1) randomized clinical trials that compare adhesive performance in cavities with or without dentin roughening did not show significant differences.[47,48] 2) randomized clinical trials that compared enamel beveling with no bevel also demonstrated similar findings[49,50]; and last, the American Dental Association guidelines for testing the adhesive performance in clinical studies does not recommend any cavity preparation.[23]

The results of this study can be generalized only to patients with low caries risk. In the present study we have excluded patients with poor oral hygiene and active caries lesions because the restoration of NCCLs would be not the priority for these patients. Other preventive measures, such as oral hygiene instructions, dental sealing, and restoration of active lesions, would have been necessary before such

patients could have met the inclusion criteria for this clinical trial.

Since the introduction of the FDI criteria,[24,25] few studies have attempted to compare this instrument with the traditional criteria from the United States Public Health Services (USPHS). [51,52] A recent study that compared both methods concluded that the FDI criteria are more sensitive than the USPHS criteria for detecting small variations in clinical outcomes,[51,52] and this is the reason why we used only the FDI criteria in the present study.

CONCLUSIONS

The use of cotton rolls/retraction cord was shown to be similar to the use of rubber dam isolation in terms of patient's preference, gingival damage, chairside time, and retention rates of adhesive restorations in NCCLs.

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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 COEP/UEPG. The approval code for this study is 06231/09. This study was conducted at the State University of Ponta Grossa.

Conflict of Interest

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

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Two-year Randomized, Controlled Clinical Trial of a Flowable and Conventional Composite in Class I Restorations

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Clinical Relevance

Flowable composites may be an acceptable restorative material for small load-bearing posterior restorations.

SUMMARY

Objectives: This study evaluated the two-year clinical performance and volumetric wear of a flowable resin composite compared to a conventional highly filled composite resin in Class I restorations.

Methods and Materials: In this single-center, single-blinded, comparator-controlled clinical study (Institutional Review Board approved), 120 carious teeth distributed in 60 patients were randomly assigned to four calibrated practitioners who placed occlusal restorations (n=60 flowable and n=60 conventional composite). Direct and indirect assessment at baseline, six months, one year, and two years occurred during which the modified Cvar and Ryge criteria were evaluated. Volumetric wear was determined by superimposition of profilometer scans of baseline and two-year casts.

Results: At two years, there was no significant difference in anatomic form (p=0.80), color match (p=0.08), marginal adaptation (p=0.89), marginal discoloration (p=0.79), surface integrity (p=0.18), secondary caries (p=0.66), cold sensitivity (p=0.522), occlusal sensitivity (p=0.818), or volumetric wear (p=0.661) between materials. Both materials showed a decrease in all criteria except secondary caries (p=0.95) over time. Two-year mean volumetric wear was 3.16 ± 2.38 mm^3 for the flowable

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composite and 3.43 ± 2.50 mm^3 for the conventional composite.

Conclusions: The flowable and conventional composites used in this study have similar clinical efficacy after two years of service when placed as Class I occlusal restorations having isthmus widths less than one-half the intercuspal distance.

INTRODUCTION

Flowable composites were first introduced in the 1990s.[1] The handling characteristics and syringe delivery system of flowable composites remove some of the challenges encountered with placing composite resin in small to medium-sized preparations. Even though these materials are widely used by practicing dentists, their clinical applications have been limited by the mechanical limitations measured in early-generation flowable composites.[1,2] Early-generation flowable composites were filled at concentrations ranging from 50wt% to 70.5wt% (most studies only report wt% filler; however, vol% filler gives a better representation of composite microstructure).[1] Laboratory studies have shown that the increased filler content in current-generation flowable composites has improved several of the mechanical properties of these materials.

Filler content has been shown to influence the polymerization shrinkage of resin composites. Unlike resin, filler particles do not contract upon polymerization and, therefore, help decrease the polymerization shrinkage of composite resins. In studies[3,4] of experimental composites filled with varying concentrations of filler particles, filler concentration was inversely correlated to shrinkage and shrinkage stress. Labella and others[4] reported that the volumetric shrinkage of early-generation flowable composites (filler concentration 53wt%-68wt%) was higher (4%-6%) than that of conventional composites (approximately 2%). A study by Baroudi and others[5] concluded that flowable composites with a higher filler concentration (ranging from 55wt% to 71wt%) produced less shrinkage strain. The clinical repercussion of polymerization shrinkage is tooth-composite marginal opening, possibly leading to leakage around and ultimately under a restoration. An *in vitro* study by Bonilla and others[6] reported increased leakage under flowable composite occlusal restorations compared to restorations with a conventional composite.

Another criticism of flowable composite is that removing reinforcing particles from a composite decreases its strength. An *in vitro* evaluation of an experimental composite by Hosseinalipour and others[7] demonstrated that increasing hybrid filler content up to 65.2vol% (approximately 80wt%) increased flexural strength; and a study of 72 commercially available composites by Ilie and Hickel[8] showed that materials with filler content of ~78wt% had maximum flexural strength. Early-generation flowable composites (filler concentration 50wt%-70.5wt%) demonstrated lower flexural strength than did comparable conventional composites (75wt%-80wt%).[1] Studies by Irie and others[9] and Sumino and others[10] of current-generation flowable composites (65wt%-70wt% and 61%-71%), however, reported higher flexural strength than was associated with their conventional counterparts (56wt%-77wt% and 58wt%-69wt%). Similarly, early-generation flowable composites had lower fracture toughness than conventional composites,[1] while more highly filled flowable composites demonstrated similar fracture strength compared with conventional composites.[11]

In vitro studies have also suggested that flowable composites are less wear resistant than conventional composites. Filler particles protect the weaker resin matrix during abrasive wear.[12] In studies by Condon and Ferracane[13] and Lim and others[14], experimentally filled composites experienced less wear with increased filler content, and filler concentrations below 48vol% (approximately 60wt%-65wt%) showed considerably more wear than did more highly filled materials. *In vitro* wear studies by Schultz and others[15] and Clelland and others[16] of commercially available composites reported more wear on flowable composites than on highly filled materials. In these studies, the filler content of the flowable composites ranged from 59wt% to 68wt% and from 65wt% to 80wt%, while the conventional composites ranged from 77wt% to 80wt% and from 79wt% to 87wt%. An *in vitro* study by Sumino and others,[10] on the other hand, reported less wear with flowable composites; however, the flowable composites in their study had higher filler concentrations (61wt%-71wt%) than did the comparative conventional composites (58wt%-69wt%).[14]

As a result of their perceived mechanical limitations, flowable composites have traditionally been used clinically for restorations with minimal occlusal loading, such as liners, small Class I and II cavities, and Class V lesions. A clinical trial of small Class I restorations restored with two flowable composites demonstrated that marginal discoloration and marginal adaption worsened at three years after base-

Table 1: *Composition of Test Materials*

Material	Manufacturer	Filler Content	Filler Type
Filtek Supreme Ultra Flowable Restorative	3M ESPE	65wt% 46vol%	Silica nanoparticles Silica/zirconia nanoclusters
Filtek Supreme Ultra Universal Restorative	3M ESPE	72.5wt% 56vol%	Silica nanoparticles Silica/zirconia nanoclusters
Adper Single Bond Plus Adhesive	3M ESPE		Silica nanoparticles

line. The presence of secondary caries, anatomical form, retention, polishability, and color match of the restorations did not significantly change over the three-year period.[17,18] A two-year clinical trial compared conventional and flowable composites in Class II restorations and observed no difference between the materials.[19] One three-year and three two-year clinical trials compared conventional and flowable composites in cervical lesions for marginal discoloration, marginal adaptation, secondary caries, surface texture, color match, and anatomic form.[20-23] No difference was found between materials in any study except for one study[20] that reported that the conventional material had superior marginal adaptation.

The increased filler content of newer-generation flowable composites and corresponding improvements in properties warrant the further evaluation of these materials in load-bearing restorations. The purpose of this clinical trial was to evaluate the effectiveness of a new flowable resin composite (Filtek Supreme Ultra Flowable Restorative, 3M ESPE, St Paul, MN, USA; filler content: 65wt% and 46vol%) when used in Class I restorations compared to a conventional highly filled composite resin (Filtek Supreme Ultra, 3M ESPE; filler content: 72.5wt% and 55.6vol%) in Class I restorations. Our null hypothesis was that there would be no difference in any Cvar and Ryge criteria or clinical wear between the two composites. This was a single-center, single-blinded, comparator-controlled, randomized clinical trial of 24 months in duration.

METHODS AND MATERIALS

Prior to patient enrollment, an Institutional Review Board approved the clinical trial protocol. Inclusion criteria for patients in the study included the following: 1) 19 years or older, 2) good general health, 3) available for follow-up visits, and 4) have at least 28 teeth. The following exclusion criteria were used: 1) rampant uncontrolled caries, 2) advanced untreated periodontal disease, 3) >2 cigarette packs/d or equivalent chewing tobacco, 4) systemic or local disorders that contraindicate dental procedures included in this study, 5) evidence of xerostomia, 6) evidence of severe bruxing, clenching, or temporomandibular joint disorder, 7) pregnancy at the time of screening or tooth restoration, and 8) known sensitivity to acrylates or related materials. Inclusion criteria for restorations in the study included 1) at least one contact with an opposing natural or crowned tooth or a fixed partial denture, 2) a minimum of 1.5 mm in depth, 3) confined to occlusal pits and fissures, 4) initial restoration or an amalgam replacement, and 5) isthmus width from one-quarter to one-half the intercuspal distance. Exclusion criteria of the teeth were 1) periapical pathology or symptoms of pulpal pathology, 2) nonvital or previous root canal therapy, 3) previous pulp cap, 4) tooth hypersensitivity, 5) near exposures on preoperative radiographs, 6) severe periodontal disease, and 7) excessive biting forces. Sixty patients were enrolled in this study. The nature and purpose of the study, the clinical procedures, and the expected duration of participation were explained to each potential subject and informed consent was obtained.

Each enrolled patient possessed two teeth that met the inclusion criteria. Of these 120 teeth, 60 were allocated to the comparator group (Filtek Supreme Ultra) and 60 were allocated to the experimental group (Filtek Supreme Ultra Flowable). Manufacturer's information for all materials used in this study is presented in Table 1. The four participating clinicians were calibrated for placement and evaluation of the restorations prior to initiation of the study. During calibration, restorations were placed in typodont teeth exactly as described in the protocol to standardize all clinical procedures and familiarize the dentist with the materials. Patients were given local anesthesia as needed, and the teeth were isolated using nonlatex rubber dams. Shade selection was performed with the Vita shade guide. Conservative Class I cavity preparations were made with a high-speed handpiece, limiting tooth removal to no more than one-half the distance of the cusp tips on the occlusal surface of the prepared tooth. Any tooth with a pulpal exposure was excluded from the study. Any

Table 2: *Modified Cvar and Ryge Scoring Criteria for Clinical Assessment of Composite Restorations*

	Anatomic Form
A =	Restoration is continuous with existing anatomic form
B =	Restoration is discontinuous with existing anatomic form (undercontoured) but missing material is not sufficient to expose dentin or lining.
C =	Sufficient material is lost to expose dentin
Color Match	
A =	Restoration matches adjacent tooth structure in shade and/or translucency
B =	Mismatch in shade and/or translucency is within normal range of tooth shades
C =	Mismatch in shade and/or translucency is outside normal range of tooth shades
Marginal Adaptation	
A =	Explorer does not catch or slight catch with no visible crevice
B =	Explorer catches and crevice is visible but no exposure of dentin or base
C =	Explorer penetrates crevice and defect extended to enamel-dentin junction
D =	Restoration is fractured, mobile, or missing in part or *in toto*
Marginal Discoloration	
A =	No visual evidence of marginal discoloration
B =	Marginal discoloration present but has not penetrated in a pulpal direction
C =	Marginal discoloration has penetrated in a pulpal direction
Surface Integrity	
A =	Smooth surface with no irregularities
B =	Slightly rough or pitted—can be refinished
C =	Deeply pitted or grooved (not related to anatomy)—cannot be refinished
D =	Surface fracture or flaking
Secondary Caries	
A =	No caries present
D =	Caries present associated with the restoration

cavity preparation judged to be within 1 mm of pulpal tissue either clinically or radiographically was lined with Dycal (Dentsply Caulk, Milford, DE, USA), a calcium hydroxide containing liner or Vitrebond (3M ESPE), a resin-modified glass ionomer base. The preparations were etched with the 37% phosphoric acid applied initially to the enamel and then to dentin for 15 seconds, rinsed for 15 seconds, and dried using a cotton pellet or minisponge. Two or three consecutive coats (no curing between coats) of Single Bond Plus adhesive (3M ESPE) were applied to the enamel and dentin for 15 seconds, air-dried for five seconds, and light-cured for 10 seconds using the Elipar Freelight 2 LED curing light (3M ESPE, output=1000 mW/cm^2). Each resin composite was placed in 2-mm increments and cured for 20 seconds per increment. If dentin or A6B and B5B shades were used a 40-second cure was applied for each increment. Carbide finishing burs (7404, OS-1, OS-2, Brasseler, Savannah, GA, USA) were used to remove gross excess and to adjust the occlusion; this step was followed by finishing and polishing with Sof-Lex (3M ESPE) and Enhance/PoGo (Dentsply Caulk) points, cups, and discs.

The composite material used was determined by assigning the lowest numbered tooth to the material randomly assigned based on a computer-generated list with a block size of 2. The material used for either tooth was blinded to the patient; however, the difference in handling properties of the materials prevented blinding of the restoring dentist. Each patient was assigned a unique identification code, which was used to record the material used in each tooth.

Each restoration was evaluated directly and indirectly at baseline (one week after restoration placement), six months, and one and two years post-restoration. The direct clinical evaluations were performed using the modified Cvar and Ryge criteria[24] presented in Table 2. These criteria include anatomic form, marginal adaptation, surface texture, color match, marginal discoloration, secondary caries, sensitivity to cold, and sensitivity to biting. Evaluators were calibrated for evaluation of each Cvar and Ryge criteria by examination of photographs and cast representative of various scores. Anatomic form, marginal adaptation, surface texture, and secondary caries were determined by visual and tactile examination. Digital images were taken at every recall to document color match and marginal discoloration of the composite restoration to the tooth (Figure 1). Sensitivity to cold was measured by applying a cotton pellet soaked with pulp vitality refrigerant spray (Endo Ice, Coltene/Whaledent, Cuyahoga Falls, OH, USA) to the tooth for three seconds. Sensitivity to biting was measured by having the patient bite on a cotton roll for five seconds. After each test, the subject was asked to place an "X" on a 10-mm line labeled "1" on the left and "10" on the right. Patients were told that a "10" represents the worst pain they can imagine (ie, childbirth, major surgery, or kidney stone) and that "1" represents no sensation at all. All clinical assessments were performed by two trained examiners other than the operating clinician, and a consensus agreement was established for all clinical assessments. Examiners were blinded to the material used for each restoration.

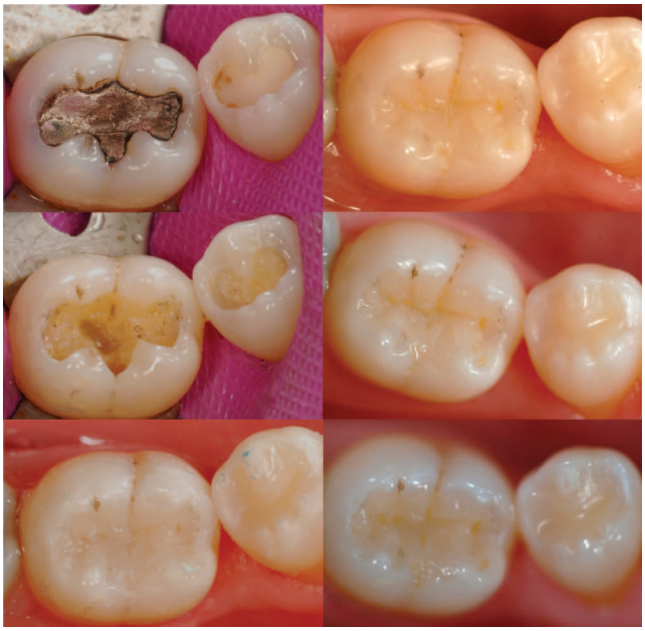

Figure 1. *Restoration of first mandibular molar with conventional composite and second mandibular premolar with flowable composite at (top left to bottom right) preoperative, preparation, baseline, six-month recall, 12-month recall, and 24-month recall.*

At the baseline and two-year evaluations, impressions were taken of the restored tooth with light-bodied polyvinylsiloxane (PVS) material around the tooth and heavy-body PVS in the tray (Imprint 3, 3M ESPE). The impressions were poured with vacuum-mixed gypsum stone (Silky-Rock, Whip Mix Corp, Louisville, KY, USA). The occlusal surfaces of the stone casts were scanned with a noncontact three-dimensional light profilometer (Proscan 2000, Scantron Industrial Products Ltd, Taunton, UK) with a resolution of 20 μm (mesial-distal) × 20 μm (buccal-lingual) × 75 nm (occlusal-gingival). The depth of focus was 2.5 mm for the profilometer. The baseline and two-year scans were superimposed, and the volumetric difference between scans was measured with Proform software. The two-year scans were modified to remove all data points aside from those approximately 0.5 mm outside of the margins of the restoration. This area was removed to eliminate any volumetric differences not present on the restoration (ie, errors on the cast, tooth wear). If the restoration could not be visualized on the cast, the clinical photograph of the preparation was used to determine restoration margins. All profilometry and superimposition were performed by the same trained investigator.

Each Cvar and Ryge outcome was compared between materials over time with a repeated measures analysis of variance (ANOVA) (α=0.05). Repeated-measures ANOVA was chosen instead of chi-square analysis in order to model these data as a repeated measure of correlated observations, which cannot be accomplished with 2 × 2 tables. Mean volumetric wear was compared between materials with a Mann-Whitney test (α=0.05).

RESULTS

At the two-year recall, 49 teeth remained in the flowable group (82% retention) and 49 teeth in the conventional group (82% retention). The majority of the missing data are due to noncompliance of the patient for recall evaluations. Within the flowable group, one restoration was replaced by the patient's local dentist and one restoration failed as a result of restoration chipping that extended slightly to the lingual surface that could not be smoothed and had to be replaced. In the conventional group, one restoration was replaced with a crown by the patient's local dentist. These three teeth were given failing scores (C or D) at each evaluation subsequent to failure. In the flowable group, 47 restorations were randomly placed in molars and 13 in premolars. In the conventional group, 51 restorations were randomly placed in molars and nine were placed in premolars.

Percent perfect Cvar and Ryge scores and visual analog pain scale recordings (mean±standard deviation [SD]) for the conventional and flowable composites are displayed in Table 3. Performance of materials for anatomic form, color match, marginal adaptation, marginal discoloration, surface integrity, and secondary caries was compared for each criterion by collapsing data into two groups (as a result of a limited number of non-alpha values). Each category was analyzed separately as a repeated measure over four time periods using a binomial distribution model with a logistic regression. Because values at the two-year follow-up were assumed to be less correlated to baseline measurements, a first-order autoregressive covariance matrix was selected for the repeated measures.

Pairwise comparisons of estimated marginal means using a sequential Bonferroni (α=0.05) was used to compare the performance of each material over time. Some degradation in performance occurred for each criterion over time, but no differences were noted in material performance at two years. Overall, no differences were noted between materials for anatomic form (p=0.80), color match (p=0.08), marginal adaptation (p=0.89), marginal discoloration (p=0.79), surface integrity (p=0.18), and secondary caries (p=0.66).

Table 3: *Percent Perfect Cvar and Ryge Scores and Visual Analog Pain Scale Recordings (Mean±Standard Deviation [SD]) for Conventional and Flowable Composites*

		Percent Alpha Score or Visual Analog Pain Scale Recording (Mean±SD)			
		Baseline	**6 Mo**	**12 Mo**	**24 Mo**
Anatomic form	Conventional	100.0%	98.3%	94.6%	86.0%
	Flowable	100.0%	100.0%	96.4%	89.8%
Color match	Conventional	88.3%	91.7%	83.9%	78.0%
	Flowable	96.6%	96.6%	92.7%	85.7%
Marginal adaptation	Conventional	98.3%	88.3%	78.6%	80.0%
	Flowable	96.6%	93.2%	81.8%	83.7%
Marginal discoloration	Conventional	98.3%	95.0%	91.1%	80.0%
	Flowable	98.3%	96.6%	90.9%	85.7%
Surface integrity	Conventional	96.7%	90.0%	78.6%	78.0%
	Flowable	98.3%	96.6%	92.7%	79.6%
Secondary caries	Conventional	98.3%	96.7%	94.6%	94.0%
	Flowable	100.0%	100.0%	94.5%	93.9%
Sensitivity to cold	Conventional	1.6 ± 2.0	1.8 ± 1.7	1.7 ± 1.7	1 ± 1.3
	Flowable	1.6 ± 1.6	1.5 ± 1.4	2.1 ± 2.2	1.5 ± 1.9
Sensitivity to biting	Conventional	0.7 ± 0.7	0.8 ± 0.8	0.3 ± 0.5	0.1 ± 0.3
	Flowable	0.5 ± 0.6	0.6 ± 0.7	0.3 ± 0.5	0.2 ± 0.5

Sensitivity was measured on a visual analog scale and was modeled as a normally distributed continuous variable. Pairwise comparisons of estimated marginal means were performed. Some changes in this value occurred over time but were not significant using the sequential Bonferroni post hoc *p*-value (α=0.05).

Biting values ranged from 0 to 5. These data were collapsed into three ordinal categories and modeled with the multinomial distribution with a cumulative logistic regression. The first-order autoregressive covariance matrix was employed for the repeated measures. No significant difference in materials was noted over the observation period, although bite levels of both materials changed significantly at each time period with respect to baseline measurements.

Two-year mean volumetric wear was 3.16 ± 2.38 mm^3 for the flowable composite and 3.43 ± 2.50 mm^3 for the conventional composite. Normality of the volumetric wear data was evaluated with a Shapiro-Wilk test and found to be nonparametric ($p<0.001$). A Mann-Whitney test determined that there was no significant difference between the volumetric wear of the flowable and conventional composites (p=0.661)

DISCUSSION

This study compared the clinical efficacy of a flowable (Filtek Supreme Ultra Flowable) and a conventional (Filtek Supreme Ultra Universal) composite. This prospective, single-blind, randomized, controlled clinical trial was conducted to determine the clinical performance of a flowable composite in a conservative Class I restoration. Regarding the clinical effectiveness of the flowable composite at two years, it had similar or superior properties to the conventional composite. There was no difference in anatomic form (p=0.80), color match (p=0.08), marginal adaptation (p=0.89), marginal discoloration (p=0.79), surface integrity (p=0.18), secondary caries (p=0.66), cold sensitivity (p=0.522), biting sensitivity (p=0.818), or volumetric wear (p=0.661) between materials. Both materials showed a decrease in perfect scores for all criteria except secondary caries (p=0.95) over time.

Ideally, the distribution of premolars and molars in each group should be kept even as a result of the clinical differences between their location and size. Premolar restorations would be expected to have lower occlusal forces than molars as a result of their distance from the fulcrum of the jaw. Premolars also have a smaller occlusal surface area, leading to more narrow preparations and higher c-factor. The incidence of premolar restorations was 22% in the flowable group and 15% in the conventional group. The allocation of premolars in each group is relatively even considering the random assignment of teeth to treatment groups.

The results of this study contradict the predictions that could be made for flowable composites based on *in vitro* data. Flowable materials have more poly-

merization shrinkage than conventional composites[4] and, therefore, should have pulled away from the walls of the restoration. This would have been observed in the present study as an increased prevalence of marginal discoloration, marginal discrepancies, and possibly secondary caries. These results were not seen in the present study. It is possible that the polymerization shrinkage that is observed in the laboratory is not sufficient to generate adequate stress to separate the bond between the composite and the tooth. The material tested in this study had lower polymerization shrinkage than did many comparable commercial flowable composites (mean vol% shrinkage: 3.3%±.4% for Filtek Supreme Ultra Flow and 3.9%-5.5% for other flowable composite materials tested).[25] Additionally, the lower modulus of elasticity of flowable materials has been theorized to reduce stresses generated at the adhesive interface.[19,22] It is also possible that marginal discrepancies were too small to be observed or felt and that they will grow with time. New research,[26] however, has shown that polymerization stresses may relax over time in a wet environment.

Another expected predicted result based on laboratory studies would have been increased wear of the flowable composite. Several clinical studies[27-31] have also quantitatively measured wear of composite resins using laser profilometry on casts. Some studies reported wear depths for posterior composite restorations that ranged from 22 to 91 μm/y,[27] 12 μm/y,[28] and 42-54 μm/y.[29] Other studies also reported volumetric wear as measuring 0.023 mm^3/y,[28] 0.09-0.132 mm^3/y,[30] and 1.14-1.51mm^3/5 y.[31] The wear values measured in this study (3.16±2.38 mm^3/2 y flowable; 3.43±2.50 mm^3/2 y conventional) are larger than values presented in previous studies. For example, one study[31] reported a wear rate of Filtek Supreme of 1.14 mm^3/5 y.[31] There is no current American Dental Association specification to recommend the maximum amount of volumetric composite wear; the most recent specification for vertical contact wear requires a maximum of 50 μm/y.[32]

The lower wear rate reported in previous studies may have been attributed to the protocol used for superimposition. In two of the studies, wear data at the cavo-surface margin was removed either by manually deleting the wear at beveled margins[31] or by discarding data attributed to marginal fractures.[28] In another study,[30] contact wear was measured by multiplying the contact areas by the mean vertical wear. In the present study, all wear was considered. As can be observed in Figure 2 (lower right frame), the greatest amount of wear was often observed at the cavo-surface margins. Other variation in the wear volumes reported between studies may be related to differences in the sizes of the composite restorations, impression and cast-making techniques, accuracy of scanning devices, and precision of the superimposition software. Variation could also be attributed to patient-related factors such as biting forces, the number of remaining teeth, and the positon of the restored tooth in the arch. Regardless of the magnitude of the wear, our study shows no difference in the wear of the flowable and conventional composites, which is in agreement with the qualitative wear measurement in a similar clinical study.[19]

A recent *in vitro* comparison of Filtek Supreme Ultra flowable and conventional reported similar flexural strength (133.3 MPa flowable and 116.1 MPa conventional) and fracture toughness (1.07 $MPa/m^{1/2}$ flowable and 1.03 $MPa/m^{1/2}$ conventional) and lower elastic modulus of the flowable (7.03 GPa) than the conventional (12.61 GPa) composite.[33] Only one restoration fracture was noted in the present study (although the two restorations replaced by other dentists may have been due to restoration fracture). These results indicate that the strength and toughness of the flowable material should be adequate for load-bearing restorations. It is important to note that the results can only be applied to the particular materials used in this study and restorations having isthmus widths of less than one-half the intercuspal distance.

Limitations of this study include the relatively small sample size and high patient drop-out rate. Wear measurement was hindered by the limited accuracy of the impression material, imperfections in the casts, and operator subjectivity in the manipulation of the superimposition software. The differences in the surface profilometry and superimposition protocols in this study and those of previous studies make direct comparison of wear data irrelevant. Future studies will explore digital impressioning for quantitative wear measurement and positioning matrices that can be worn by patients during scanning to aid in scan superimposition.

CONCLUSIONS

The flowable composite used in this study had clinical efficacy after two years of service that was similar to that of a conventional composite when placed as Class I occlusal restorations having

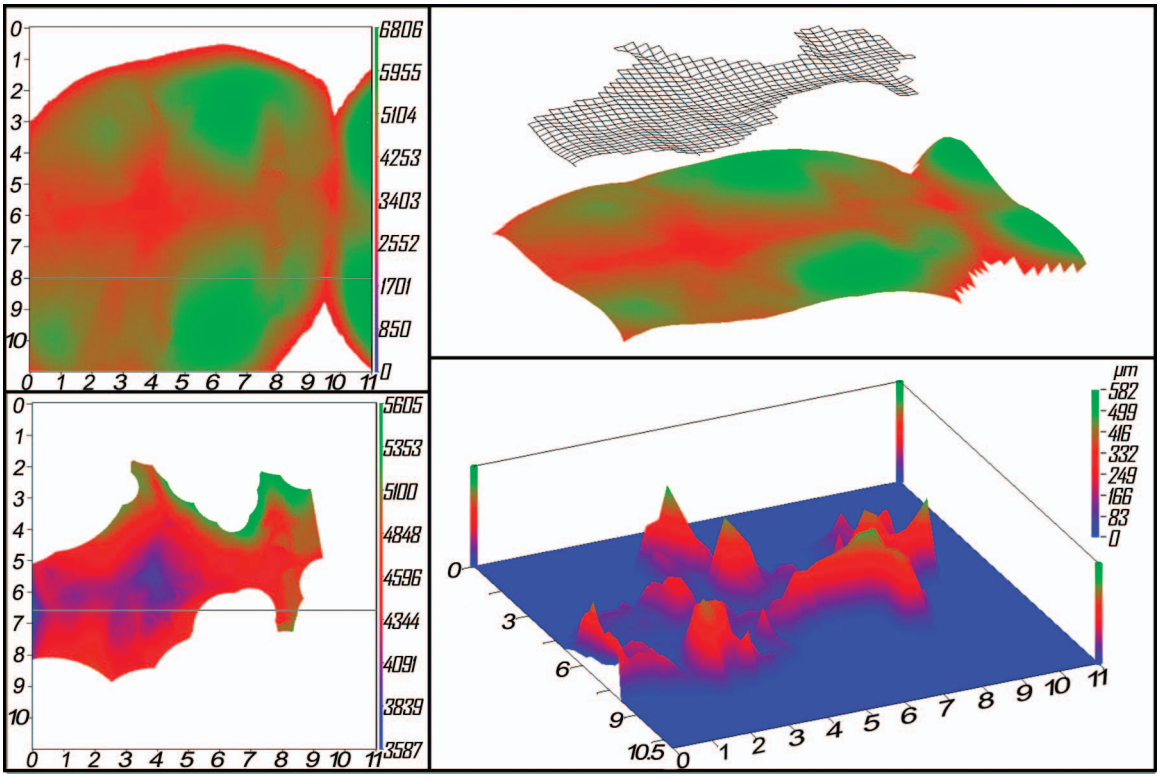

Figure 2. *Representative superimposition of baseline and two-year clinical scans. Baseline occlusal scan (top left frame), two-year occlusal scan trimmed to 0.5 mm from restoration margin (bottom left frame), superimposition of two-year mesh scan on baseline scan (top right frame), and difference between baseline scan and two-year scan (bottom right frame).*

isthmus widths of less than one-half the intercuspal distance.

Acknowledgment

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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 WIRB. The approval code for this study is CR-10-013.

Conflict of Interest

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

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Laboratory Research

Mechanical Properties and Simulated Wear of Provisional Resin Materials

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Clinical Relevance

Erosive wear behavior as a function of time is an important parameter for the selection of a provisional restoration.

SUMMARY

The purpose of this study was to determine flexural properties and erosive wear behavior of provisional resin materials. Three bis-acryl base provisional resins—1) Protemp Plus (PP), 2) Integrity (IG), 3) Luxatemp Automix Plus (LX)—and a conventional poly(methylmethacrylate) (PMMA) resin, UniFast III (UF), were evaluated. A resin composite, Z100 Restorative (Z1), was included as a benchmark material.

Six specimens for each of the four materials were used to determine flexural strength and elastic modulus according to ISO Standard 4049. Twelve specimens for each material were used to examine wear using a generalized wear simulation model. The test materials were each subjected to wear challenges of 25,000, 50,000, 100,000, and 200,000 cycles in a Leinfelder-Suzuki (Alabama) wear simulator. The materials were placed in custom cylinder-shaped stainless-steel fixtures, and wear was generated using a cylindrical-shaped flat-ended stainless-steel antagonist in a slurry of non-plasticized PMMA beads. Wear (mean facet depth [μm] and volume loss [mm^3]) was determined using a noncontact profilometer (Proscan 2100) with Proscan and AnSur 3D software. The laboratory data were evaluated using two-way analysis of variance (ANOVA; factors: 1) material and 2) cycles) followed by Tukey HSD post hoc test (α=0.05).

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The flexural strength ranged from 68.2 to 150.6 MPa, and the elastic modulus ranged from 2.0 to 15.9 GPa. All of the bis-acryl provisional resins (PP, IG, and LX) demonstrated significantly higher values than the PMMA resin (UF) in flexural strength and elastic modulus ($p<0.05$). However, there was no significant difference ($p>0.05$) in flexural properties among three bis-acryl base provisional resins (PP, IG, and LX). Z1 demonstrated significantly ($p<0.05$) higher flexural strength and elastic modulus than the other materials tested. The results for mean facet wear depth (μm) and standard deviations (SD) for 200,000 cycles were as follows: PP, 22.4 (5.0); IG, 51.0 (6.5); LX, 63.7 (4.5); UF, 70.5 (8.0); and Z1, 7.6 (1.2). Volume loss (mm^3) and SDs for 200,000 cycles were as follows: PP, 0.311 (0.049); IG, 0.737 (0.074); LX, 0.919 (0.053); UF, 1.046 (0.127); and Z1, 0.111 (0.017). The two-way ANOVA showed a significant difference among materials ($p<0.001$) and number of cycles for both facet depth and volume loss. The post hoc test revealed differences ($p<0.05$) in wear values among the tested materials examined in this study. The findings provide valuable information regarding the flexural properties and the relative wear behavior of the provisional resins examined in this study.

INTRODUCTION

Provisional restorations are used to prevent damage from occurring during the interim period between tooth preparation and fitting a definitive restoration. Besides the immediate protective, functional, and stabilizing value, interim restorations are useful for diagnostic purposes in which the functional occlusal and esthetic parameters are developed to identify an optimum treatment outcome before the completion of definitive procedures.[1-4] Occasionally, however, interim treatment has to function for extended intervals and provide long-term tooth protection and stability while adjunctive treatment is accomplished. Therefore, maintaining long-term stability of provisional restorations in the oral environment is required for complex treatment, including dental implant therapy.[5-8]

In the past decade, bis-acryl base resins containing inorganic fillers have been made available to provide long-term stability due to their enhanced mechanical properties in the oral environment. These newly developed provisional restorative materials have a different curing mechanism when compared with conventional poly(methylmethacrylate) (PMMA) provisional resins.[6] Also, this type of provisional restoration purportedly reduces the exothermic setting reaction and is easier to manipulate than conventional PMMA resins.[9] Although there are several published research reports regarding their mechanical properties,[10-15] little is known about their erosive wear behavior. To select the optimum provisional materials, evaluating the performance of wear characteristics is an important parameter. In addition, dental clinicians need more information about the clinical relevance of the improved mechanical properties of newer provisional restorative materials and the relationship of these properties to wear resistance. However, trying to relate clinical and laboratory wear data is a significant challenge because adequate clinical data are not available.

Recently, Barkmeier and others[16-18] recommended that a benchmark resin composite material (Z100 Restorative, 3M ESPE Dental Products, St Paul, MN, USA), which has exhibited good clinical and simulated wear performance, be used as a standard for laboratory wear comparison of other restorative materials. The recommendation is further strengthened by the good agreement of clinical and laboratory wear rates of the Z100 Restorative material.[17,18] The purpose of this study was to determine flexural properties and erosive wear behavior of bis-acryl base resins containing inorganic fillers. During the course of this research, the flexural properties and the wear behavior of bis-acryl provisional resins were compared with a PMMA conventional resin and a benchmark resin composite (Z100 Restorative). The hypothesis proposed was that bis-acryl–based resins will demonstrate significantly higher flexural properties and wear resistance than a PMMA conventional resin.

METHODS AND MATERIALS

Study Materials

Flexural properties and wear resistance of three bis-acryl base provisional resins—1) Protemp Plus (PP; 3M ESPE Dental Products), 2) Integrity (IG; DENTSPLY Caulk, Milford, DE, USA), and 3) Luxatemp Automix Plus (LX; DMG Chemisch-Pharmazeutishe Fabrik GmbH, Hamburg, Germany)—and a conventional PMMA resin, 4) UniFast III (UF; GC Corp. Tokyo, Japan), were examined. A resin composite, Z100 Restorative (Z1; 3M ESPE Dental Products), was also included as a control material.

Table 1: *Study Materials*

Provisional Materials	Shade	Manufacturer	Main Components[a]	Code
Protemp Plus (Lot No. B 498865, Lot No. C 495535)	A2	3M ESPE Dental Products (St Paul, MN, USA)	Base: dimethacrylate (BISEMA 6), silane-treated amorphous silica, reaction products of 1,6-diisocyanatohexane with 2-[(2- methacryloxy) ethyl]6-hydroxyhexanoate, 2-hydroxyethyl methacrylate (DESMA), silane-treated silica Catalyst: ethanol, 2,2′-[(1-methylethylidene) bis(4,1-phenyleneoxy)]bisethyl diacetate, silane-treated silica, benzyl-phenyl-barbituric acid, tert-butyl peroxy-3,5,5-trimethylhexanoate	PP
Integrity (Lot No. 100630)	A2	DENTSPLY Caulk (Milford, DE, USA)	Barium boron alumino silicate glass, hydrophobic amorphous fumed silica, polymerizable dimethacrylate resins	IG
Luxatemp Automix Plus (Lot No. 451192)	A2	DMG Chem-Pharm Fabrik GmbH (Hamburg, Germany)	Acrylic resin, glass powder, silica, urethane dimethacrylate, aromatic dimethacrylate, glycol methacrylate	LX
Unifast III (Lot No. Powder 1209253, Lot No. Liquid 1209121)	A2	GC Corporation (Tokyo, Japan)	Powder: ethyl-methyl methacrylate polymer, polymethylmethacrylate, barbituric acid derivative, organic copper compound, pigments Liquid: methyl methacrylate, *N,N*-dimethyl-*p*-toluidine trimethylolpropane, ethylene glycol dimethacrylate	UF
Resin composite				
Z100 Restorative (Lot No. N416713)	A2	3M ESPE Dental Products (St Paul, MN, USA)	Bisphenol A glycidyl dimethacrylate, triethylene glycol dimethacrylate, zirconia/silica	Z1

Abbreviations: IG, Integrity; LX, Luxatemp Automix Plus; PP, Protemp Plus; UF, UniFast III; ZI, Z100 Restorative.
[a] *Manufacturer's information.*

The test materials and their components are listed in Table 1.

Flexural Strength and Elastic Modulus Measurement

Flexural properties of the provisional resins were tested according to procedures specified in ISO International Standard 4049. The provisional resins (PP, IG, LX, UF) were mixed according to the manufacturers' instructions and placed into stainless-steel molds (25 mm × 2 mm × 2 mm) with a condenser. The resin composite paste (Z1) was placed into the mold in three increments. Each increment was light cured for 30 seconds with a Spectrum 800 Curing Light (DENTSPLY Caulk) set at 600 mW/cm^2. After the removal of the cured hardened specimens from the mold, all six sides were wet ground with No. 1200 SiC papers (Struers Inc, Cleveland, OH, USA). The specimens were then stored for 24 hours in distilled water at 37°C before the mechanical tests. The specimens were kept moist during testing to avoid desiccation. After the storage time, six specimens per test group were subjected to the three-point bending flexural strength test using an MTS Insight machine (MTS Systems Corporation, Eden Prairie, MN, USA) at a cross-head speed of 0.75 mm/min until the specimen fractured. The specimens were positioned on a three-point bending apparatus with a span length of 20.0 mm. The peak breaking stress and the modulus of elasticity were determined from the stress-strain curve using TestWorks 4 software (MTS Systems Corporation).

Generalized Wear Simulation

Twelve specimens of each provisional restorative material (PP, IG, LX, UF) and Z1 resin composite were prepared for simulated generalized wear (contact-free area (CFA) wear) using custom stainless-steel fixtures with a cavity in the center of the surface 4.5 mm in diameter and 4 mm in depth. The provisional resins were mixed and placed into the stainless-steel fixtures according to the manufacturers' instructions. For the resin composite material (Z1), two increments (approximately 2 mm in thickness) were each placed and cured for 40 seconds with the Spectrum 800 Curing Light. After 24 hours, all of the specimens were polished flat to 4000 grit using a sequence of silicon carbide papers (Struers Inc).

A Leinfelder-Suzuki wear simulation device (Alabama machine) was used for wear simulation in this study. The simulator has a plastic water bath, and the custom wear fixtures were mounted inside the four-station bath. A brass cylinder was then placed

around each fixture in the bath to serve as a reservoir for the abrasive media (water slurry of unplasticized PMMA with an average particle size of 44 μm). The media were placed inside the brass cylinders to cover the surface of the testing materials in the custom fixtures. The water slurry of PMMA inside the brass cylinders was approximately 6 mm in height over the surface of the test materials.

The antagonist for the generalized wear simulation (CFA) was a stainless-steel cylinder (diameter 6.5 mm) with a flat tip. The antagonist tips were mounted on spring-loaded pistons to deliver the wear challenges. During the application of the load, the antagonists rotated approximately 30° as the maximum force was reached (maximum load of 78.5 N at a rate of 2 Hz) and then counterrotated back to the original starting position as the load was relaxed to complete the cycle. Each specimen was subjected to wear challenges of 25,000, 50,000, 100,000, and 200,000 cycles in the wear simulator.

Wear Measurements

Prior to wear simulation, the surface of each test specimen was profiled using a Proscan 2100 noncontact optical profilometer (Scantron Industrial Products, Ltd, Taunton, England) with Proscan software. These profiles provided the pretest digitized contours (12 test specimens each for the four provisional resins and one resin composite material).

Following the wear challenge cycles, the specimens were ultrasonically cleaned (L&R Solid State Ultrasonic T-14B, South Orange, NJ, USA) in distilled water for three minutes and then profiled again using the Proscan 2100 unit. The X, Y, and Z coordinates of the before and after scans from the Proscan software were exported to another computer for analysis with AnSur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA). The X, Y, and Z coordinates generated with the Proscan software were saved as PRN files and then imported into the AnSur 3D program.

Scanning Electron Microscopy Observations

Specimens were prepared for argon-ion etching and scanning electron microscopy (SEM) examinations. The surfaces of the cured test materials were ground with abrasive discs (Fuji Star Type DDC, Sankyo Rikagaku Co Ltd, Saitama, Japan) followed by a series of diamond pastes down to 0.25-μm particle size (DP-Paste, Struers, Ballerup, Denmark) to bring the surfaces to a high gloss. The prepared surfaces were then subjected to argon-ion beam etching (IIS-200ER, Elionix, Tokyo, Japan) for 45 seconds with the ion beam directed at the polished surface (accelerating voltage of 1.0 kV, ion current density 0.4 mA/cm^2). The surfaces were then coated in a vacuum evaporator with a thin film of Au. Observations were done with an SEM (FE-8000, Elionix) at an operating voltage of 10 kV.

SEM examinations were also completed on the wear facets of the five test materials. Following the wear analysis, representative samples of each material were sputter coated with Au and Pd (Emitech SC7620 Mini Sputter Coater, Quorum Technologies, Ashford, UK). The coated specimens were then examined with a TM3000 Tabletop Microscope (Hitachi-High Technologies Corporation, Tokyo, Japan) using an accelerating voltage of 15 kV.

Statistical Analysis

A one-way analysis of variance (ANOVA) and Tukey post hoc test were used for analysis of flexural strength and elastic modulus. The wear simulation data (mean facet depth and volume loss) were analyzed using a two-way ANOVA and Tukey post hoc test. Factors for the ANOVA tests were 1) test materials and 2) number of cycles. Linear regression analysis of mean depth and volume loss data was used to examine the relationship of the two variables in the wear simulation tests: 1) test materials and 2) number of cycles. The association strength between the variables, R^2 (square of the correlation coefficient), was determined for each test material at the four cycling periods (25,000, 50,000, 100,000, and 200,000). A regression line was also developed to predict wear rates and volume loss rates for the test materials.

RESULTS

Flexural Strength and Elastic Modulus Measurement

The results of this study for flexural properties of the tested materials are shown in Table 2. The flexural strength ranged from 68.2 to 150.6 MPa, and the elastic modulus ranged from 2.0 to 15.9 GPa. The one-way ANOVA revealed that a significantly higher flexural strength and elastic modulus were found for Z1. For PP, IG, and LX, there was no significant difference ($p>0.05$) in flexural strength and elastic modulus among the materials. For UF, a signifi-

Table 2: *Flexural Strength and Elastic Modulus Mean (SD)*

Code	Flexural Strength, MPa	Post Hoc Test Group[a]	Flexural Modulus, GPa	Post Hoc Test Group[a]
Z1	150.6 (5.2)	a	15.9 (1.2)	a
LX	87.7 (4.7)	b	3.3 (0.3)	b
PP	84.0 (4.3)	b	3.0 (0.1)	b
IG	80.6 (6.9)	b	3.2 (0.2)	b
UF	68.2 (5.5)	c	2.0 (0.1)	c

Abbreviations: IG, Integrity; LX, Luxatemp Automix Plus; PP, Protemp Plus; UF, UniFast III; ZI, Z100 Restorative.
[a] *Same lowercase letter indicates no difference at the 5% significance level.*

cantly ($p<0.05$) lower value was found among the tested materials in flexural strength and elastic modulus.

Generalized Wear Simulation

The ANOVA results for wear simulation are presented in Tables 3 and 4. The two-way ANOVA of generalized wear data, for both mean depth and volume loss, revealed a significant effect for the factors of test materials ($p<0.001$) and number of cycles ($p<0.001$) as well as for the interaction of test materials and number of cycles ($p<0.001$).

The generalized wear values (mean wear depth and volume loss) for the five test materials at the four cycling periods (25,000, 50,000, 100,000 and 200,000) are summarized in Tables 5 and 6. The statistical differences ($p<0.05$) for mean depth and volume loss for each material at the four cycling periods, as well as differences among the materials at each cycling period, are also presented in Tables 5 and 6. As the number of cycles increased, the occurrence of significant differences ($p<0.05$) for the individual test materials also increased, except for Z1. The data also showed differences ($p<0.05$) among materials at the various cycling periods.

Regression lines for wear depth and volume loss vs cycling periods for the five test materials are presented in Figures 1 and 2. The strength of association (R^2) between the variables of the tested materials and number of cycles for both mean depth and volume loss are presented in Table 7. A strong association was found between the variables for both mean depth and volume loss. Predicted wear rates and volume loss rates determined by linear regression are also presented in Table 7.

Table 3: *Analysis of Variance: Facet Depth*

Factor	Sum of Squares	*df*	Mean Square	*F* Ratio	*p*
Material	46,761.31	4	11,690.33	853.34	<0.001
Cycles	24,734.61	3	8244.86	601.84	<0.001
Materials × Cycles	11,627.73	12	968.98	70.73	<0.001

Table 4: *Analysis of Variance: Volume Loss*

Factor	Sum of Squares	*df*	Mean Square	*F* Ratio	*p*
Material	10.37	4	2.60	1241.96	<0.001
Cycles	5.07	3	1.69	809.30	<0.001
Materials × Cycles	2.50	12	0.21	99.73	<0.001

SEM Observations

SEM examinations of the five test materials evaluated by argon-ion etching are presented in Figures 3a-e. The argon-ion etching demonstrated clear differences in filler particle size, shape, and distribution. For PP, approximately 50- to 200-nm size spherical filler particles were observed (Figure 3a). For IG and LX, 0.5- to 2-μm irregular-shaped glass filler particles were observed (Figure 3b,c). The conventional PMMA resin UF contains approximately 20-50 μm round polymer particles. The SEM observations of the resin composite (Z1) showed aggregated nanosize spherical filler particles, which are more densely distributed than the other test materials.

SEM examinations of the generalized wear facets (Figures 4a-e) after 200,000 cycles also demonstrated differences in the filler systems of the materials tested in this study. After wear testing of PP, the SEM examinations revealed relatively flat and smooth surfaces (Figures 4a), and there were some fine wrinkles observed on the worn surfaces. IG and LX exhibited a similar wear pattern with some plucking of irregular-shaped glass filler particles from the surface (Figure 4b,c). For UF, obvious cracks were observed on the worn surfaces, and some evidence of plucking of polymer particles was found (Figure 4d). Z1 showed a relatively flat and smooth worn surface, and some plucking of the fine filler particles was observed from the simulated generalized wear testing (Figure 4e).

DISCUSSION

Provisional restorations are required to fulfill several functions for the duration of their use in the oral environment. The mechanical properties of provisional materials may influence the integrity of provisional restorations *in situ* when exposed to functional loads.[10-15] One of the major causes for the replacement of provisional restorations have been

Table 5: *Generalized Wear: Facet Depth (SD)*[a]

Cycle	Mean Facet Depth, μm				
	Z1	PP	IG	LX	UF
25,000	5.0 (1.3) aA	14.1 (1.7) aB	19.9 (1.3) aC	23.2 (2.1) aCD	25.3 (2.8) aD
50,000	5.7 (1.2) aA	14.4 (2.4) aB	25.0 (3.1) bC	26.3 (3.3) aC	33.2 (3.8) bD
100,000	6.3 (1.5) aA	17.1 (3.0) aB	34.8 (3.6) cC	40.5 (3.8) bD	41.6 (4.2) cD
200,000	7.6 (1.2) aA	22.4 (5.0) bB	51.0 (6.5) dC	63.7 (4.5) cD	70.5 (8.0) dE

Abbreviations: IG, Integrity; LX, Luxatemp Automix Plus; PP, Protemp Plus; UF, UniFast III; ZI, Z100 Restorative.
[a] *Groups in vertical columns with the same lowercase letter are not different at the 5% significance level. Groups in different columns with the same number of cycles and same capital letter are not different at the 5% significance level.*

cited as fractures in the body and at the margins.[6,19] Interim restoration failures are a concern for both the clinician and patient because of additional cost and time associated with these complications. Fracture-related material properties, such as fracture resistance, elasticity, and marginal degradation of materials under stress, have typically been evaluated by determining material parameters such as flexural strength, flexural modulus, and fracture toughness.[20,21] Clinicians need to be knowledgeable regarding the flexural properties of provisional materials in order to deliver optimal treatment, especially when patients are required to use the provisional restorations for an extended time due to complicating conditions such as severe periodontal disease, parafunctional disorders, or dental implant therapy.[5-8] Although laboratory flexural strength values under constant loading may not reflect intraoral conditions, these values are nevertheless helpful in comparing materials under controlled situations and may be a useful predictor of clinical performance.[13]

The results of this study showed that bis-acryl provisional materials demonstrated significantly higher ($p<0.05$) flexural strength than conventional PMMA resin. These results are consistent with those of past studies that compared bis-acryl provisional materials with conventional PMMA resins.[12,14] Bis-acryl provisional resins contain multifunctional monomers, which increase strength due to cross-linking with other monomers and also include inorganic fillers that may have the capability of distributing the load stress and inhibiting the progress of crack propagation.[12,13] The bis-acryl provisional materials used in this study were in cartridge-based dispensing systems, which may contribute to superior flexural strength because of a more accurately proportioned and consistent mix.[22] In addition, methacrylate-based resins typically used in provisional materials are not cross-linked, and without polymerization under pressure, air entrapment may occur and result in lower strength values.[10]

The flexural modulus or the modulus of elasticity is a measure of the material stiffness: the higher the modulus, the stiffer the materials. Marginal breakdown and loss of marginal seal are more likely to occur in products with a lower modulus of elasticity. In the present study, the bis-acryl provisional resins (PP, IG, LX) did not show a higher elastic modulus value than the resin composite (Z1); however, they demonstrated a significantly higher elastic modulus than the PMMA resin (UF). Therefore, the hypothesis that bis-acryl base provisional resins will demonstrate significantly higher flexural properties than the PMMA resin was not rejected.

The SEM observations of the polished surfaces after argon-ion etching revealed that bis-acryl provisional resins showed a heterogeneous structure due to the inclusion of silane-treated inorganic fillers

Table 6: *Generalized Wear: Volume Loss (SD)*[a]

Cycle	Volume Loss, mm^3				
	Z1	PP	IG	LX	UF
25,000	0.067 (0.018) aA	0.206 (0.020) aB	0.297 (0.016) aC	0.341 (0.031) aCD	0.377 (0.037) aD
50,000	0.081 (0.020) aA	0.218 (0.018) aB	0.352 (0.024) bC	0.386 (0.049) aC	0.501 (0.035) bD
100,000	0.091 (0.022) aA	0.249 (0.034) aB	0.578 (0.034) cC	0.586 (0.050) bD	0.635 (0.039) cD
200,000	0.111 (0.017) aA	0.311 (0.049) bB	0.737 (0.074) dC	0.919 (0.053) cD	1.046 (0.127) dE

Abbreviations: IG, Integrity; LX, Luxatemp Automix Plus; PP, Protemp Plus; UF, UniFast III; ZI, Z100 Restorative.
[a] *Groups in vertical columns with the same lowercase letter are not different at the 5% significance level. Groups in different columns with the same number of cycles and the same capital letter are not different at the 5% significance level.*

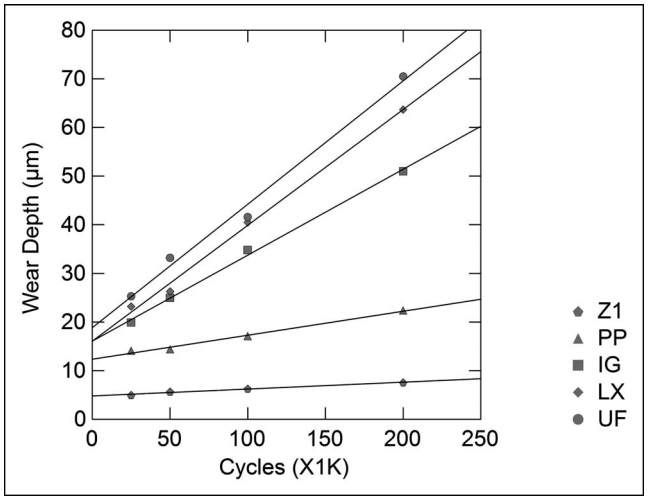

Figure 1. *Wear depth (μm) of tested materials vs cycles (X1K).*

or glass fillers (Figure 3a-c). The inorganic fillers contribute to enhancing not only the strength but also elastic modulus. A previously reported study of resin composites revealed correlations between filler content, filler size, distribution of filler particles, and material strength and elastic modulus.[23] However, a comparison among the three bis-acryl provisional resins in the present study did not show a significant difference ($p>0.05$) in flexural strength and elastic modulus among the three materials. PP employs approximately 50- to 200-nm size spherical silica filler particles (Table 1); IG and LX employ 0.5- to 2-μm irregular-shaped glass filler particles (Figure 3b,c). Although increasing the filler load of resin materials purportedly increases the ultimate strength and elastic modulus, there does not seem to be a clear relationship in the present study

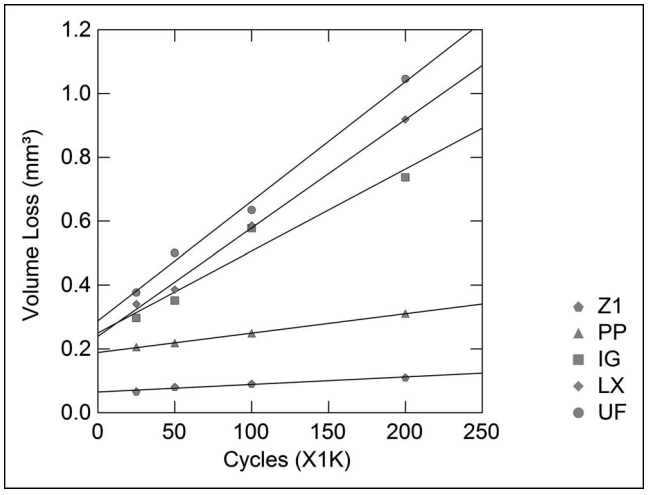

Figure 2. *Volume loss (mm³) of tested materials vs cycles (X1K).*

Table 7: *Regression Analysis: Wear (μm) and Volume Loss (mm³) per 100,000 Cycles and R^2 Value*

Code	Wear Rate	R^2	Volume Loss Rate	R^2
Z1	1.4	0.973	0.023	0.942
PP	4.9	0.983	0.061	0.999
IG	17.7	0.996	0.257	0.919
LX	23.8	0.993	0.339	0.994
UF	25.4	0.987	0.374	0.991

Abbreviations: IG, Integrity; LX, Luxatemp Automix Plus; PP, Protemp Plus; UF, UniFast III; ZI, Z100 Restorative.

regarding filler size or filler shape and mechanical properties of bis-acryl provisional resins. It is speculated that the type of resin matrix, degree of conversion, surface treatment of filler, and interparticle spacing of bis-acryl materials might contribute to mechanical property characteristics in the same manner as resin composites.[24]

Even if provisional restorations are used for a limited time period, it is important from a wear perspective to maintain occlusal relationships over the time span of the interim restoration. Loss of occlusal anatomy and support changes the vertical dimension of restored teeth, which may result in parafunction. In addition, wear progression of provisional restorations may lead to deteriorated surface texture and provide foci for crack propagation from the masticatory function.

To date, various approaches have been taken by researchers to fill the void in clinical data by conducting wear simulation studies in the laboratory for resin composites. Although trying to relate clinical and laboratory wear data is still a significant challenge, Barkmeier and others[17,18] have examined the relationship of simulated localized and generalized wear to CFA clinical wear using Z1 as benchmark resin composite. They reported that Z1 showed 17.0 μm of cumulative wear (CFA) after three years in the oral cavity, and there was good agreement between the relationship of simulated and clinical wear.[16,17] However, there is little information available regarding the relationship between clinical wear data and laboratory data of provisional materials. Therefore, to evaluate the relationship between the simulated and clinical wear of provisional materials, Z1 was employed as a benchmark material in the present study. From the results of generalized wear testing, the wear rates of the provisional resins tested appeared to be dependent on the material and the number of cycles. UF demonstrated significantly higher wear values than the three bis-acryl resins in wear

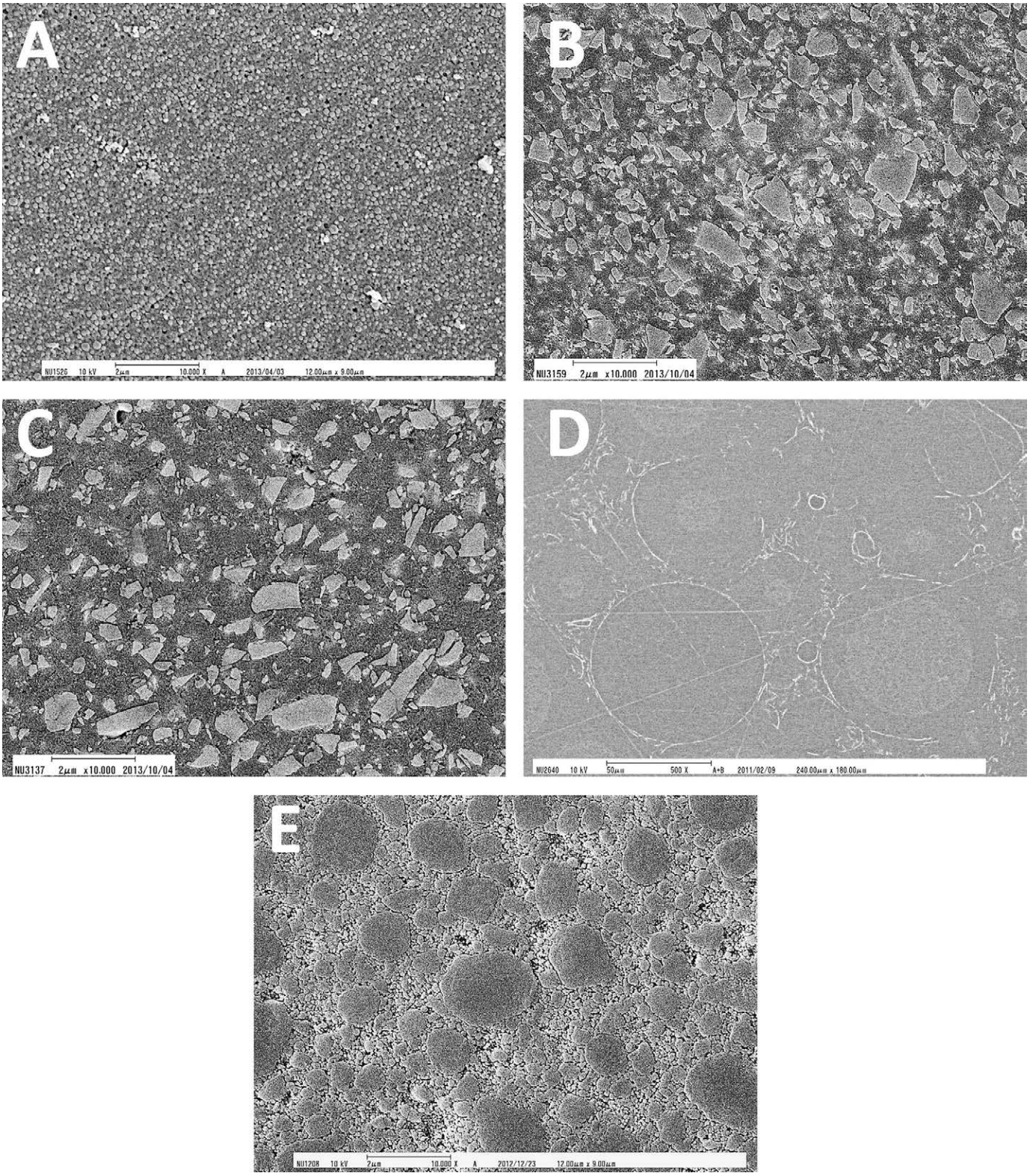

Figure 3. *(A): Protemp Plus, argon-ion etched surface (10,000×). (B): Integrity, argon-ion etched surface (10,000×). (C): Luxatemp Automix Plus, argon-ion etched surface (10,000×). (D) Unifast III, argon-ion etched surface (500×). (E) Z100 Restorative, argon-ion etched surface (500×).*

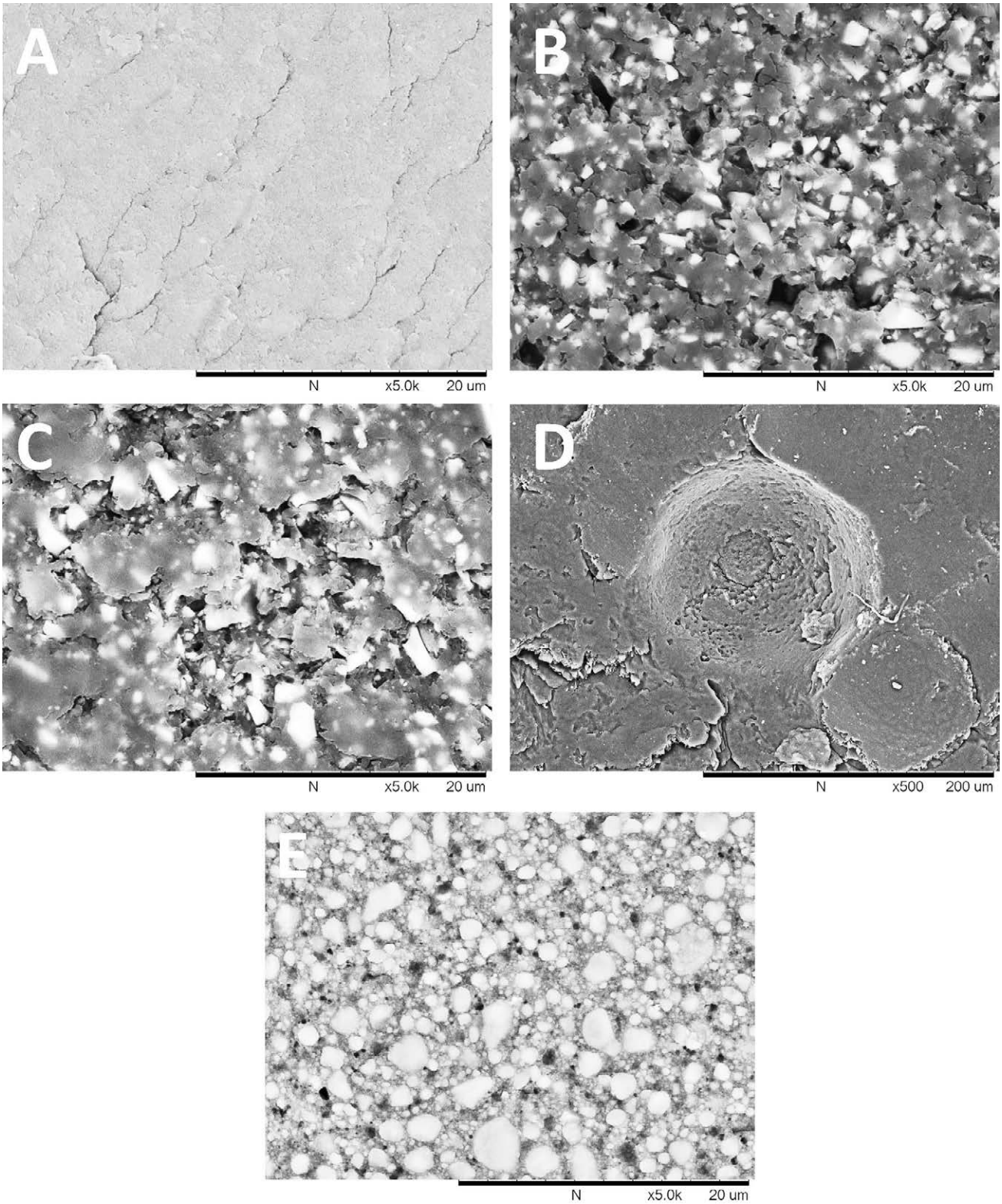

Figure 4. *(A): Protemp Plus, generalized wear near center of facet (5000×). (B) Integrity, generalized wear near center of facet (5000×). (C) Luxatemp Automix Plus, generalized wear near center of facet (5000×). (D) Unifast III, generalized wear near center of facet (500×). (E) Z100 Restorative, generalized wear near center of facet (5000×).*

mean depth and volume loss at 200,000 cycles. Therefore, the proposed hypothesis that bis-acryl provisional resins would demonstrate significantly higher wear resistance than the PMMA resin was not rejected.

PMMA provisional resins primarily consist of polymer particles and monomers that are considered as a relatively homogenous composition. Therefore, the wear of PMMA resins might be expected to occur uniformly in generalized wear simulation. However, the SEM observations in this study revealed that some polymer particles appeared to pull out from the surface, resulting in dark holes in the worn areas (Figure 4d). It is speculated that there were flaws between the polymer particles and resin matrix interfacial bond due to incomplete mixing that led to pull out of the PMMA particles.

On the other hand, bis-acryl provisional resins consist of inorganic fillers and multifunctional monomers, which possess different hardness characteristics. The wear behavior of this type of material may induce plucking and uncovering of filler particles, which project from the surface due to selective abrasion of the resin matrix. Although there was no significant difference between PP and the other bis-acryl resins in flexural properties, the mean wear depth and volume loss of PP was significantly lower ($p<0.05$) than the other bis-acryl–based resins for all of the cycling periods. The difference in wear rates between PP and the other bis-acryl resins is most likely related to inorganic filler size, shape, and distribution. From the SEM observations, PP and Z1 demonstrated relatively flat and smooth worn surfaces after wear testing compared with the other provisional resins (Figure 4a-e). PP and Z1 are made by the same manufacturer and appear to employ similar spherical nano-sized filler particles, which have the benefit of maintaining the surface texture even if plucking of fillers from the resin matrix occurs.[25,26] In addition, finer particles for a fixed-volume fraction of filler have been suggested to result in decreased inter-particle spacing and reduced wear.[27]

Linear regression was used to provide predicted wear rates for the five materials evaluated in this study (Table 6). The wear rates of the materials in this study, as defined by the slopes, can be categorized into three groups (Figures 1 and 2) for both wear depth and volume loss. Z1 exhibits the lowest wear rate (depth and volume loss); IG, LX, and UF exhibit similar wear rates and were in the highest wear category; and PP shows a wear rate between the two groups. When comparing the reported clinical data for Z1 after three years in oral cavity[16,17] to the wear rate of mean depth in the present study, the data suggest that provisional resins would reach the same wear depth reported for Z1 in 2 to 10 months. Therefore, there is a marked difference in wear resistance between the resin composite (Z1) and the provisional resins evaluated in this study, regardless of the inorganic fillers and multifunctional monomers incorporated into the provisional resin materials.

There may be a number of features affecting the selection of provisional restorative materials by clinicians; however, the flexural properties and the predicted rates for wear and volume loss, as described here, should provide valuable information for the selection of a provisional restorative resin material.

CONCLUSIONS

The results of this study demonstrated significantly higher ($p<0.05$) flexural strength and elastic modulus values for three bis-acryl provisional resins when compared with a conventional PMMA resin, and there was no significant difference ($p>0.05$) among the three bis-acryl base provisional resins in flexural strength and elastic modulus. Simulated wear of bis-acryl provisional resin materials also demonstrated significantly higher ($p<0.05$) wear resistance when compared with a conventional PMMA resin. The wear rates of bis-acryl provisional resins tested appeared to be dependent on the material and the number of cycles. The benchmark resin composite material, Z1, exhibited the highest flexural strength properties and the least amount of wear in generalized wear simulation.

The study results provide valuable comparisons of physical property values and wear resistance of a resin composite to three bis-acryl and one PMMA provisional restorative resin materials. This study augments both clinical and laboratory wear data of a benchmark resin composite (Z1) and provides valuable comparisons with provisional resin materials. The data reported provide guidance for clinicians in the selection of provisional resin materials.

Conflict of Interest

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

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Spectrophotometric Evaluation of Potassium Nitrate Penetration Into the Pulp Cavity

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Clinical Relevance

The tooth is permeable to potassium nitrate–containing formulations with an application time of 30 minutes.

SUMMARY

Objectives: The aim of this study was to evaluate the penetration level of potassium nitrate–containing desensitizers or whitening materials into the pulp cavity with regard to the concentration and viscosity of the formulation.

Methods and Materials: Fifty extracted human molar teeth were prepared and randomized into five groups of 10 specimens each. The control received a 30-minute treatment without any treatment material; the other four groups corresponded to treatment with DayWhite, a 14% hydrogen peroxide whitening material containing potassium nitrate; Previ-Dent 5000 Sensitive, a desensitizing toothpaste; Relief ACP, a desensitizing gel; or UltraEZ, a desensitizing gel. Potassium nitrate penetration levels were measured spectrophotometrically based on the Griess assay method. Treatment materials were measured for viscosity as a function of shear rate through the use of a cone-and-plate rheometer.

Results: Nitrate penetration levels were significantly different among the five groups ($p<0.0001$, Kruskal-Wallis test). After adjustment for multiple comparisons using an overall 0.05 level of type I error, the distribution of nitrate penetration values was found to differ significantly among all groups with the exception of DayWhite (median: 10.72 μM) and Ultra-

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EZ (median: 9.22 μM), which differed significantly from other groups but not from each other. The highest levels of nitrate penetration value were observed for PreviDent (median: 27.61 μM) followed by Relief ACP (median: 19.64 μM). The lowest penetration level was observed for the control group (median: 3.41 μM). Stable end-point viscosities of 11.43 ± 0.67 Pa/s, 1.33 ± 0.06 Pa/s, 0.85 ± 0.09 Pa/s, and 0.40 ± 0.01 Pa/s were observed for UltraEZ, ReliefACP, DayWhite, and PreviDent, respectively.

Conclusion: Potassium nitrate included in different formulations can penetrate the enamel and dentin within 30 minutes. The level of potassium nitrate penetration is influenced by concentration and may also be partly affected by the viscosity of the material as well as other constituents of proprietary preparations.

INTRODUCTION

Tooth whitening represents the most common elective dental procedure as a growing number of people envision a Hollywood smile highly driven by media emphasis on beauty and health.[1] It has proven to be safe and effective when properly diagnosed, treatment planned, and supervised by the dentist. The wide range of techniques available—in-office whitening, dentist-supervised at-home whitening, over-the-counter whitening, and do-it-yourself whitening—reflects its popularity.[2] The efficacy and side effects are related to the techniques, with tooth sensitivity being the most frequently reported side effect associated with all of the approaches.[3]

Whitening-induced tooth sensitivity commonly presents itself as generalized sensitivity to cold stimuli but often also occurs as a spontaneous sharp, shooting pain limited to one or few teeth.[4] The incidence of tooth sensitivity ranges from 0% to 75%, and the degree can vary from mild to intolerable, leading some patients to discontinue treatment.[5] A clinical trial by Jorgensen and Carroll[6] concluded that about half of the patients performing whitening will experience mild sensitivity, 10% will have moderate sensitivity, and 4% will have severe sensitivity. Despite the fact that sensitivity is only transient and will resolve almost immediately on completion of the whitening procedure, it adversely affects patient compliance and satisfaction with the whitening experience. Therefore, efforts to elucidate the etiology and develop strategies for the prevention and treatment of whitening-induced tooth sensitivity continue to be a central issue to be addressed in tooth-whitening procedures.

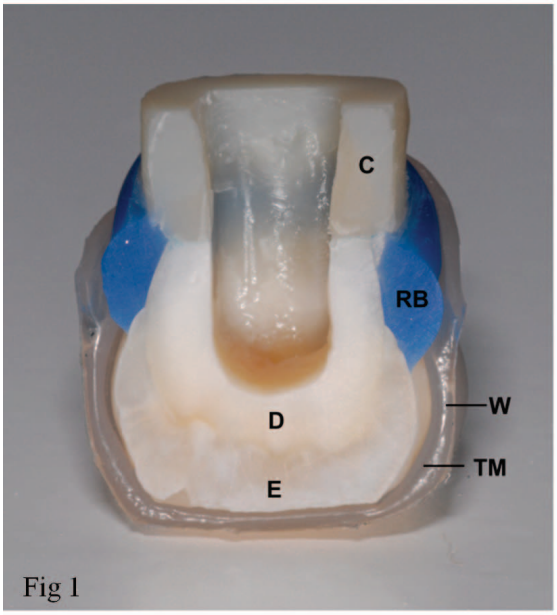
The management of tooth sensitivity related to whitening has been addressed using a variety of approaches based on hydrodynamic theory. According to Brännström's theory, dentinal fluid expands, contracts, or flows within dentinal tubules under the influence of thermal, evaporative, or osmotic changes and stimulates pressure-sensitive nerve receptors (A-δ fibers) that transmit the stimulus and produce the perception of pain.[7] Agents such as fluoride and amorphous calcium salts are generally used to occlude the permeability of dentinal tubules, preventing the flow of dentinal fluid. Another common desensitizing approach is to decrease the activity of dentinal sensory nerve fibers and prevent pain signal transmission to the central nervous system.

The mode of desensitizing action of potassium nitrate is that it affects the extracellular potassium ion concentration, which is the principal determinant of the nerve resting electrical potential. When the potassium ion concentration is increased above the normal physiologic level, the cell depolarizes, creating a period of inactivation. However, because of the properties of membrane gates, this period does not last long, and the action potentials begin to occur again.[8,9]

It has been demonstrated that hydrogen peroxide in the whitening gel readily penetrates into the pulp cavity within 5 to 15 minutes when applied to the external surface of the tooth.[10,11] The penetration is facilitated by the use of higher peroxide concentration, heat, light, and younger age of the tooth.[11-14] It may be assumed that potassium nitrate will penetrate into the tooth structure similar to hydrogen peroxide to exert the depolarizing effect on the dental nerve fibers.

Currently, potassium nitrate is available in varying concentrations in desensitizing gels or toothpastes, usually in conjunction with sodium fluoride or amorphous calcium phosphate. Several manufacturers have also incorporated potassium nitrate in their whitening formulations. These materials need to flow easily on insertion but should have a viscosity at low stresses to stay in place on teeth that is related to the rheological properties of the material.[15] However, little information is available regarding the penetration potential of potassium nitrate into the pulp with regard to the concentration and physical characteristics of the gels and pastes used.

The purpose of this study was to evaluate the potential penetration of potassium nitrate–contain-



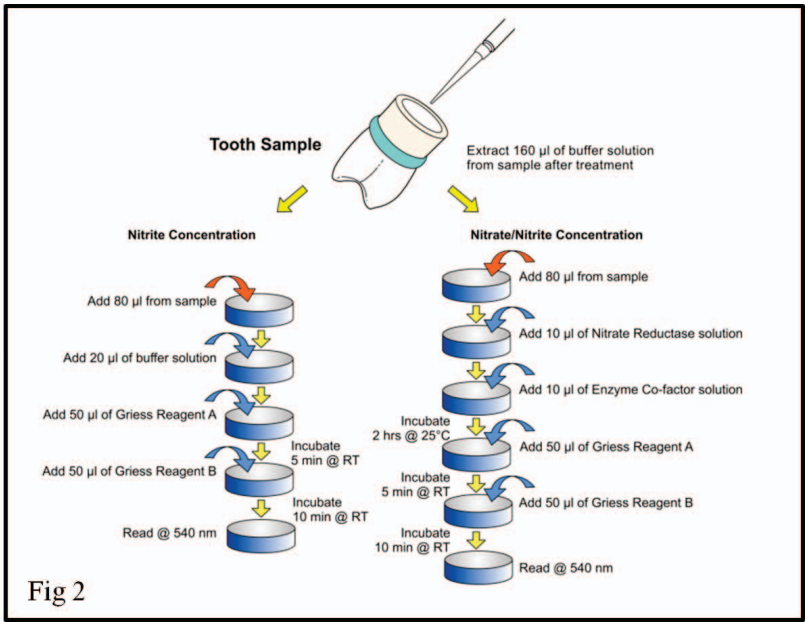


Figure 1. *Photograph showing cross section of tooth specimen preparation and material application.*
Figure 2. *Diagram of colorimetric assay.*

ing desensitizers or whitening materials into the pulp cavity. The hypotheses to be evaluated were the following:

1. There will be significant potassium nitrate penetration into the pulp cavity when compared to the control.
2. Potassium nitrate penetration into the pulp cavity will be concentration dependent.
3. Potassium nitrate penetration into the pulp cavity will be dependent on the viscosity of the treatment material.

METHODS AND MATERIALS

Sample Selection and Preparation

Fifty extracted human molar teeth were collected prior to the study and stored in 0.2% thymol (Sigma-Aldrich, St Louis, MO, USA) in distilled water at 4°C. Calculus was removed with a sickle scaler and any surface debris cleaned with plain pumice (Preppies, Whip Mix Corporation, Louisville, KY, USA) and purple prophy cups (Young Dental, Earth City, MO, USA). All teeth were observed for the absence of developmental anomalies, caries, existing restorations, deep crack lines, or severe attrition. The roots were trimmed 3 mm apical to the cemento-enamel junction, the pulp was removed, and the pulp chamber was enlarged with a pointed tapered diamond bur (NeoDiamond, Microcopy, Kennesaw, GA, USA) to maintain a standardized outer wall tooth thickness of 2 mm. Tooth thickness was measured from the outer surface to the outer boundary of the pulp cavity at the cross-sectioned root 3 mm below the cemento-enamel junction using an electronic digital caliper (Harbor Freight Tools, Pittsburgh, PA, USA). Molar teeth were selected for the study to enable the retention of 200 µL of phosphate-buffered saline in the pulp cavity that was required for the spectrophotometric assay of nitrate and nitrite. Additionally, the root surface was built up with flowable resin (Estelite Flow, Tokuyama Corp, Tokyo, Japan) to aid the retention of phosphate buffer and prevent any spilling out of the pulp chamber. The occlusal pit and fissures were sealed with flowable resin to prevent any leakage of the buffer out of the cavity. A resin barrier (OpalDam, Ultradent Products, Inc, South Jordan, UT, USA) was placed 2 mm apical to the cemento-enamel junction and light cured for 20 seconds (Elipar S10 LED curing light, 3M ESPE, St Paul, MN, USA) to limit the treatment material to the coronal surface of the tooth (Figure 1).

Application Protocol by Group

Specimens were randomized into five groups of 10 specimens each and are summarized in Table 1. Each treatment material (0.3 mg) was applied on the external coronal surface and covered with a low-density polyethylene wrap (Saran Premium Wrap, S.C. Johnson & Son, Inc, Racine, WI, USA) to

Table 1: *Desensitizers and Whitening Agents Used by Group*

Group	Brand Name	KNO_3 (%)	Viscosity (Pa/s)[a]
Control	NA	NA	NA
DayW	DayWhite	2.9	0.85 ± 0.09
PrevD	PreviDent 5000 Sensitive	5	0.40 ± 0.01
Relief	Relief ACP	5	1.33 ± 0.06
Ultra	UltraEZ	3	11.43 ± 0.67

Abbreviations: KNO_3, potassium nitrate; NA, not applicable.
[a] *Stable viscosity (10-point average ending at 400 L/s).*

simulate the placement of a custom-fabricated tray (Figure 1). The control received a 30-minute treatment with the wrap but without any treatment material applied. Group DayW was treated with DayWhite, a 14% hydrogen peroxide whitening material containing potassium nitrate (Philips Oral Health Care, Stamford, CT, USA); group PrevD with PreviDent 5000 Sensitive, a desensitizing toothpaste (Colgate-Palmolive, New York, NY, USA); group Relief with Relief ACP, a desensitizing gel (Philips Oral Health Care); and group Ultra with UltraEZ, a desensitizing gel (Ultradent Products).

The pulp cavities were rinsed twice with 100 μL of distilled water and dried with coarse paper points prior to the placement of 200 μL of buffer solution. The buffer solution acted as a stabilizing agent of potassium nitrate that might have diffused into the pulp cavity. All teeth were kept in a closed humid chamber (General Glassblowing Co, Richmond, CA, USA) during the treatment time (30 minutes) at 37°C with 100% relative humidity.

Spectrophotometric Assay of Nitrate/Nitrite

A nitrate/nitrite assay kit (Sigma-Aldrich) composed of nitrate and nitrite standard solutions, buffer solution, nitrate reductase, enzyme cofactors, and Griess dyes was used for the colorimetric determination of nitrate in the samples. The standard solutions were mixed with the buffer solution to yield final concentrations of 0, 25, 50, and 100 μM of nitrite and nitrate/nitrite to establish the calibration curves.

Figure 2 illustrates the colorimetric nitrate/nitrite assay procedure. Retrieving 80 μL of buffer solution from the pulp cavity and transferring it to a 96-well cell culture plate determined the nitrite concentration in the sample. Subsequently, 20 μL of buffer solution and 50 μL of Griess reagent A and B were added and mixed. The mixture was incubated for 10 minutes at room temperature and the absorbance read at 540 nm with a microplate reader (Power Wave X-I, BioTek, Winooski, VT, USA). The nitrite concentration in the sample solution was determined from the nitrite calibration curve.

The nitrite and nitrate concentration in the samples was determined by retrieving 80 μL of buffer solution from the pulp cavity and transferring it to a 96-well cell culture plate. Subsequently, 10 μL of nitrate reductase solution and 10 μL of enzyme cofactor solution were added, mixed, and incubated at 25°C for 2 hours. After incubation, 50 μL of Griess reagent A and B were added and mixed. The mixture was incubated for an additional 10 minutes at room temperature and the absorbance read at 540 nm with a microplate reader. The nitrate and nitrite concentration in the sample solution was determined from the nitrate/nitrite calibration curve. The final nitrate concentration in the sample was obtained by the following equation:

$$[\text{Nitrate}] = [\text{Nitrate/Nitrite}] - [\text{Nitrite}]$$

Measurement of Viscosity

Samples were measured for viscosity as a function of shear rate through the use of a cone-and-plate rheometer (Haake Rheostress 1, Thermo Scientific, Nashville, TN, USA) with temperature controls. For viscosity testing, 0.8 mL of sample was probed using a 35-mm-diameter/4-degree-angle stainless-steel cone plate at a gap height of 0.140 mm. Controlled shear rate ramp-ups were performed, a method that continuously raises the frequency of plate rotation while recording the sample viscosity in predefined time increments. These flow ramp-ups were performed over 3 minutes over a range of shear rates from 0.1 to 400 L/s, allowing for the viscosity curves to stabilize at higher shear rates. Sample temperature was controlled at 37°C throughout experimentation. Viscosity curves were generated for each of the four samples, and stable viscosity values were obtained.

Data Analysis

The nonparametric Kruskal-Wallis test was used to assess group differences because of nonnormality and variance heterogeneity associated with the distribution of potassium nitrate penetration values among the five treatment groups. All possible pairwise comparisons of treatment groups were made using the modification of the Tukey method as modified by Conover[16] to adjust for multiple comparisons in conjunction with an overall 0.05 level

Table 2: *Nitrate Penetration Descriptors by Group (μM).*

Group	Control	DayW	PrevD	Relief	Ultra
n	10	10	10	10	10
Mean	4.41	11.14	30.15	19.64	9.71
Standard deviation	2.93	2.49	8.20	3.49	3.88
Median	3.41	10.72	27.61	19.64	9.22
Minimum	0.39	6.82	18.07	11.45	3.64
Maximum	8.51	15.97	43.76	24.72	17.94

of type I error. Descriptive statistics and box plots were generated for each treatment group.

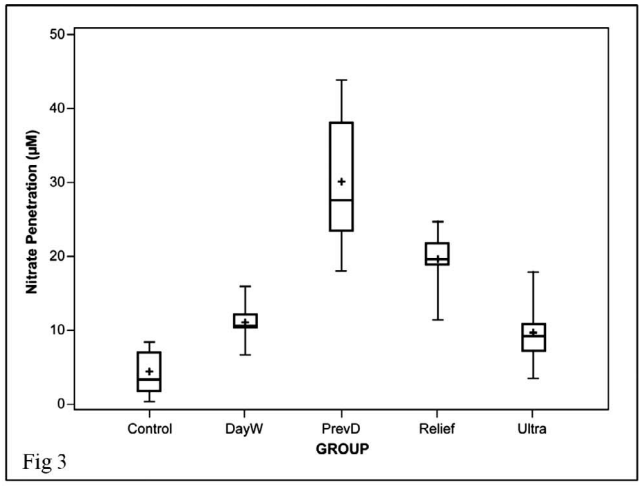
RESULTS

Potassium Nitrate Penetration

Descriptors of nitrate concentration are given for each of the five treatment groups in Table 2, and nitrate distributions are characterized graphically by box plots in Figure 3. The data provided strong evidence of differences in the distribution of potassium nitrate penetration among the five treatment groups ($p<0.0001$, Kruskal-Wallis test). After adjustment for multiple comparisons using an overall 0.05 level of type I error, the distributions of nitrate penetration values were found to differ significantly among all groups with the exception of DayWhite and UltraEZ, which could not be said to differ from each other, although each differed significantly from the other three groups. The highest levels of nitrate penetration value were observed for PreviDent (median: 27.61 μM); this distribution significantly differed from that of Relief (median: 19.64 μM). Both differed significantly from both DayWhite (median: 10.72 μM) and UltraEZ (median: 9.22 μM), although results for DayWhite and UltraEZ could not be said to differ from each other. The lowest penetration level was observed for the control group (median: 3.41 μM), which was significantly lower than for all the other treatment groups.

Viscosity

Figure 4 shows the average sample viscosity curves (n=3) for UltraEZ, ReliefACP, DayWhite, and PreviDent plotted against the shear rates of the ramp-up experiments. Stable end-point viscosities of 11.43 ± 0.67, 1.33 ± 0.06, 0.85 ± 0.09, and 0.40 ± 0.01 Pa/s were observed for UltraEZ, ReliefACP, DayWhite, and PreviDent, respectively (Table 1). Shear thinning behavior was observed for all samples. This decreasing order of sample viscosities can be observed over all shear rates investigated, with the exception of the similarities between DayWhite and



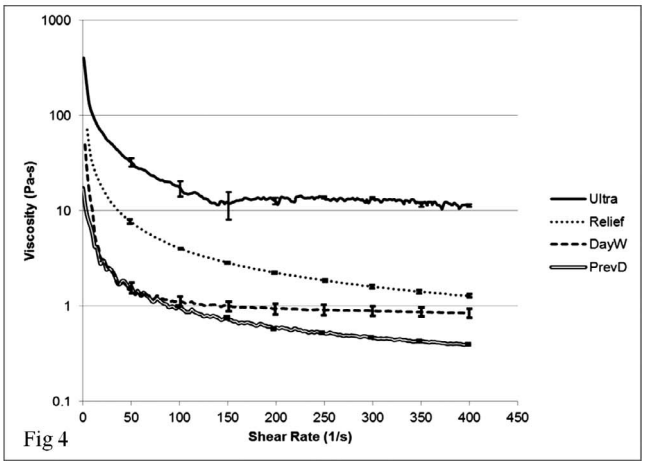


Figure 3. *Box plots of nitrate penetration by group.*
Figure 4. *Controlled shear rate viscosity ramp-up curves by treatment group.*

PreviDent viscosities from 0.1 to 100 L/s. Intra-sample standard deviations were relatively small for viscosity curve data over all shear rates investigated.

DISCUSSION

It is evident that whitening materials have to penetrate throughout the tooth to exert the whitening effect on chromogens within the enamel and dentin. Although numerous *in vitro* studies have shown that hydrogen peroxide readily penetrates into the pulp cavity, considering the potential toxic effects that hydrogen peroxide might have on the pulp, it is still debatable whether a high penetration amount is actually desired or needed for superior whitening efficacy.[17] The penetration of potassium nitrate into the tooth structure is expected to be similar to hydrogen peroxide to reach the pulp and exert its calming action on the nerve receptors located mainly in the inner dentin. However, there

has been no study in the literature reporting on the assay of potassium nitrate–containing whitening agents or desensitizers. This is somewhat surprising considering the variety of currently marketed delivery methods of potassium nitrate–containing dentrifices, desensitizers for professional use, and whitening gels.

This study explored the penetration potential of potassium nitrate with different concentrations and formulations. The findings supported the hypothesis that there would be significant penetration as compared to the control group. There was also higher penetration with increasing potassium nitrate concentration so that the second hypothesis was also supported. It is important to mention that even the control group exhibited trace amounts of nitrate (median: 3.41 μM). This may be explained by the fact that potassium nitrate is commonly used in food preparation, specifically cold meats and other delicatessen products.[18] There is also some nitrate (maximum contaminant level goals [MCLG]: 10 ppm) and nitrite (MCLG: 1 ppm) in our drinking water so that accumulation of trace amounts of potassium nitrate may be expected in human extracted teeth.[19] The results related to concentration confirm that the flux of molecules follow Fick's second law, in which diffusion is proportional to surface area, the diffusion coefficient, and concentration and inversely related to diffusion distance. It is also in agreement with studies that showed that penetration of hydrogen peroxide was concentration dependent.[10,12] The highest potassium nitrate concentration of 5% used in this study is also the maximum concentration allowed by the U.S. Food and Drug Administration to be used in desensitizing toothpastes because this material and concentration have the best scientific evidence for treating tooth sensitivity.[4]

The current protocol for the treatment and prevention of whitening-induced sensitivity can be either passive or active. Passive treatment involves using a lower concentration of whitening agent, decreasing the treatment time, or increasing the treatment interval, whereas active treatment involves the application of 3% to 5% potassium nitrate in the tray for 10 to 30 minutes before or after bleaching.[20-22] Brushing with a potassium nitrate–containing toothpaste for 2 weeks prior to the whitening and continuing this brushing throughout the treatment has been shown to have an additional benefit in the reduction of sensitivity.[23] Tray delivery of desensitizing toothpaste such as Previ-Dent 5000 Sensitive can also be effective; however, caution has been advised because toothpastes contain sodium lauryl sulfate (SLS), the primary ingredient in cleaning and hygiene products that creates foaming. SLS has been associated with increased aphthous ulcers and contact gingival irritation in some patients and also has been shown to remove the smear layer, inviting more sensitivity.[4] Manufacturers have also incorporated potassium nitrate into their whitening formula, and studies have shown that this formula indeed showed significantly less sensitivity with regard to incidence and severity.[24-27] Our study confirmed that all approaches are valid in terms of potassium nitrate penetration with an application time of 30 minutes. However, like any other medication to relieve pain, it is important to determine the onset, duration, and proper dosage of potassium nitrate for maximum efficacy, and these considerations should be further investigated in future studies.

There are also other factors to consider when evaluating potassium nitrate penetration levels, one of which is the viscosity of the material. Wille and others[15] suggested that whitening agents should fulfill specific rheological properties to withstand shear stress so that they do not flow out of the tray and are not easily washed away or swallowed. Therefore, with the use of a square root transformation of potassium nitrate penetration values to achieve conformance to model assumptions, we also explored possible relationships between potassium nitrate penetration and the potassium nitrate concentration and viscosity of the four noncontrol proprietary treatments to address our third hypothesis of whether potassium nitrate penetration will be viscosity dependent. No clear trends to support our third hypothesis could be identified based on these explorations using linear modeling, as there was evidence of significant interaction between potassium nitrate concentration and viscosity.

Although DayWhite and UltraEZ contain comparable concentrations of potassium nitrate (2.9% vs 3%, respectively), DayWhite has a lower viscosity than UltraEZ (0.85 vs 11.43 Pa/s). On the basis of the viscosity difference, it would have been expected that diffusion through DayWhite would have been more rapid than diffusion through UltraEZ; however, the concentrations of nitrate found in the buffer in the pulp cavities were nearly identical. The fact that each proprietary product is distinct in a variety of ways suggests that other factors may be exerting a modulating influence. One difference between these two preparations is that DayWhite contains hydrogen peroxide, while UltraEZ does not. Hydrogen

peroxide is sufficiently volatile as to produce local evaporative cooling. This potential thermal effect could slow diffusion in DayWhite and provide a plausible explanation to our findings. It is also noteworthy to point out that within the desensitizers (PreviDent, Relief ACP, and UltraEZ), there was a strong tendency of viscosity dependency: the lower the viscosity, the higher the penetration levels. This is in accordance with a study that evaluated the effect of viscosity on the penetration depth of resin-based materials into the tooth structure. The study showed deeper penetration into the tooth structure with the material of lower viscosity.[28]

This *in vitro* model is representative of the *in vivo* process, although it is not known how closely it would compare to the *in vivo* absorption of potassium nitrate in teeth with vital pulps exhibiting positive pulpal pressure during the application process. It is also noteworthy to point out that teeth were mechanically altered to encompass 200 μL of phosphate-buffered saline in the pulp cavity that may have affected the permeability of potassium nitrate. Based on these findings, further studies should be employed to evaluate the significance of potassium nitrate penetration levels at the nerve endings and the clinical significance of these penetration levels. The best potassium nitrate delivery method and application time should be assessed to ultimately suggest a whitening protocol with minimal tooth sensitivity and maximum whitening efficacy.

CONCLUSIONS

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

1. Potassium nitrate included in desensitizers, dentrifices, and whitening materials, when applied to the external surface of the tooth, can penetrate the enamel and dentin within 30 minutes.
2. The level of potassium nitrate penetration is influenced by concentration and may also be partly affected by the viscosity of the material as well as other constituents of proprietary preparations.

Acknowledgment

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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 University of Iowa. The approval code for this study is 201404750. This study was conducted at the University of Iowa.

Conflict of Interest

The authors of 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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Cervical Interfacial Bonding Effectiveness of Class II Bulk Versus Incremental Fill Resin Composite Restorations

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Clinical Relevance

When cervical margins of Class II cavities are inevitably located in cementum, bulk-fill and silorane based restorations might be preferable. When possible, restorations should be bonded using a total-etch approach.

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SUMMARY

Cervical interfacial bonding quality has been a matter of deep concern. The purpose of this study was to analyze microtensile bond strength (MTBS) and cervical interfacial gap distance (IGD) of bulk-fill vs incremental-fill Class II composite restorations. Box-only Class II cavities were prepared in 91 maxillary premolars (n = 7) with gingival margin placement 1 mm above the cementoenamel junction at one side and 1 mm below it on the other side. Eighty-four maxillary premolars were divided into self-etch and total-etch groups and further subdivided into six restorative material subgroups used incrementally and with an open-sandwich technique: group 1, Tetric Ceram HB (TC) as a control; group 2, Tetric EvoFlow (EF); group 3, SDR Smart Dentin Replacement (SDR); group 4, SonicFill (SF); group 5, Tetric N-Ceram Bulk Fill (TN); and group 6, Tetric EvoCeram Bulk Fill (TE). Groups 2-6 were bulk-fill restoratives. Tetric N-Bond Self-Etch (se) and Tetric N-Bond total-etch (te) adhesive were

used in subgroups 1–5, whereas AdheSE (se) and ExciTE F (te) were used in subgroup 6. In an additional group, Filtek P90 Low Shrink Restorative (P90) was used only with its corresponding self-etch bond. The materials were manipulated, light-cured (1600 mW/cm^2), artificially aged (thermal and occlusal load-cycling), and sectioned. Two microrods/restoration (n = 14/group) were tested for MTBS at a crosshead-speed of 0.5 mm/min (Instron testing machine). Fracture loads were recorded (Newtons), and MTSBs were calculated (Megapascals). Means were statistically analyzed by the Kruskal-Wallis test, Conover-Inman post hoc analysis for MTBS (multiple comparisons), and Mann-Whitney U test for IGD. The ends of the fractures were examined for failure mode. One microrod/restoration (n = 7/group) was investigated by scanning electron microscopy (×1200) for IGD.

MTBS values for SF/te, P90 in enamel, and TC+SDR/te in enamel and cementum were significantly higher compared with those for the control TC/te and TC/se in cementum. Most of the failures were mixed. IGDs were generally smaller at enamel margins, and the smallest IGDs were found in P90 at both enamel and cementum margins. Bulk-fill and silorane-based composites might provide better cervical interfacial quality than incremental-fill restorations.

INTRODUCTION

A strong challenge for resin composite restorations is their questionable adaptability to cavity walls and margins, particularly in the long-term scale of clinical service. This lack of adaptability is due to the inherent limitations of polymerization shrinkage and resultant shrinkage stresses, the mismatch in the coefficients of thermal expansion and contraction, the mismatch of the moduli of elasticity to that of the tooth structure, and the long-term chemical instability of the restorative material and adhesive joints in clinical service. These factors compromise the effectiveness of tooth-restoration interfacial bonding.[1-3]

The clinical reliability and longevity of intracoronal adhesive restorations in stress-bearing areas in the posterior teeth depend on the ability of these materials to sustain polymerization contraction stress. Moreover, such restorations should be able to endure complex chemical and mechanical oral environmental challenges, such as endogenous collagenolysis, hydrolytic degradation, functional loading, thermal and pH cycling, and bacterial biochemical activities. Currently, no single *in vitro* test can simultaneously simulate all of these parameters.[2-7]

Recent investigations have shown that the initial bonding effectiveness of contemporary adhesives is quite favorable regardless of the approach used. However, in terms of long-term clinical service, the bonding effectiveness of tooth restoration interfacial joints is questionable.[7]

The correlation between *in vitro* and *in vivo* data revealed that, currently, the best-validated method for assessing adhesion durability involves the aging of biomaterials that are bonded to either enamel or dentin. The literature shows that artificial aging can be carried out by storage in water for different periods, thermal cycling, and/or occlusal load cycling.[7]

A durable and reliable bond between the restoration and the remaining tooth structure should uniformly seal the interfaces against the microleakage of fluids, molecular movements, and ingresses of bacteria and nutrients that may lead to postrestoration hypersensitivity, marginal discoloration, recurrent caries, and adverse pulpal consequences.[2,7-11] Furthermore, the bond should be able to reinforce the remaining tooth structure by effectively cross-linking the discontinuity and efficiently transferring and distributing the functional reactionary stresses throughout the restorative complex that is formed by the remaining tooth structure, the restoration, and the adjoining bonds.[5,6] The effectiveness of bonded interfaces has long been investigated using assessments of microleakage and bond strength.[7,12]

One of the weakest parts in Class II composite restorations is leakage at the gingival margin of the proximal boxes. This leakage is due to the absence of enamel at the gingival margins, which implies a less stable and less uniform cementum-dentin substrate for bonding. This conjecture is supported by the findings of Ferrari and others, who experimentally demonstrated the presence of an outer layer of 150–200 μm that is partially formed by cementum and located below the cementoenamel junction (CEJ) and does not allow for the microretention of adhesive materials.[10,11]

The orientation of dentinal tubules can negatively affect the quality of hybridization and, thus, favor leakage in resin-based restorations that are placed in deep interproximal boxes.[11] Different techniques

and materials have been introduced to improve the performance of resin composite materials and the quality of interfacial bonding to the tooth structure. These techniques and materials include the introduction of nonmethacrylate silorane-based composites, nanofiller technology, and modifications of the triethyleneglycol dimethacrylate diluents and photoinitiators.[13] Recently, Smart Dentin Replacement has been marketed as a flowable bulk-fill base with reduced polymerization contraction stresses.[14-17] Furthermore, the SonicFill resin composite system uses sonic energy to provide bulk-fill resin composite restorations and has been reported to improve performance and reliability.[18,19]

Therefore, this study was designed to assess the effectiveness of these materials and techniques by investigating MTBS, failure modes, and interfacial gaps at the cervical interfaces of artificially aged Class II direct composite restorations. The null hypothesis was that the bulk-fill resin composites would not significantly affect the MTBS or the interfacial gaps.

METHODS AND MATERIALS

For this study, 91 caries-free human maxillary premolars that were freshly extracted for orthodontic reasons were used, and seven premolars were used for each study group. Only the teeth that were free of caries and exhibited no cracks or developmental defects were selected for the study. The teeth were collected after approval was obtained from the local biomedical research ethics committee.

Each tooth was covered coronally with wax to a level of 2 mm below the CEJ and then dipped in gum resin once (Anti-Rutsch-Lack, Wenko-Wenslaar, Hiden, Germany). After the gum resin dried, the excess apical resin was trimmed with a lancet to produce a uniform thickness of gum resin of approximately 0.25 mm that simulated the periodontal membrane. The teeth were then embedded in self-curing acrylic blocks in a vertical orientation to a level of 2 mm below the CEJ (Self-curing liquid and powder Major.Ortho, Major Prodotti Dentari S.p.A., Moncalieri, Italy).

Class II mesial and distal box-only cavities were created on each tooth using a round tungsten-carbide bur (No. 1, HM 1010, Meisinger, Neuss, Germany) to gain access through the enamel, and a cylindrical diamond abrasive with a flat end (No. 835012, Meisinger) was used to complete the preparation. New burs and abrasives were used after the creation of every five cavities. The preparations were performed using high-speed ranges under abundant air-water coolant.

The cavities were prepared with standardized dimensions such that the buccolingual dimension was 4 mm. Measurements were taken with digital calipers (Digital Vernier Caliper, Clarke International, Essex, England) with an accuracy of 0.01 mm, and a pencil was used to mark the outline. On each tooth, the gingival margin of the cavity was positioned 1 mm above the CEJ on the proximal side and 1 mm below the CEJ on the other side, and the axial pulpal depth was 1.5 mm as measured at the gingival wall using a graduated periodontal probe (1011 Duralite ColorRings, Nordent Manufacturing Inc, Elk Grove Village, IL, USA). All cavity margins were butt joint to deliver comparable results with previous experiments.[15]

The 84 samples were randomly divided into six study groups according to the resin composite restoration used. Each group was then subdivided into two subgroups according to whether a self-etch (se) or a total-etch (te) adhesive system was used. For comparison purposes, an extra group of seven teeth (group 7) was added and restored with a low-shrinkage silorane-based composite with its corresponding self-etch adhesive system. All study materials are listed in Table 1, and the study variables are shown in Figure 1.

A metallic matrix band tied to a universal matrix retainer (Tofflemire Retainer-Universal, Dentsply, Mount Waverley, Australia) was applied to each tooth so that the cervical end of the band extended beyond the gingival cavity margin. The matrix was tightened and the band was finger supported at its cervical end against the tooth surface to avoid an undue pressure of the fingertip. This was done to prevent the creation of gross marginal discrepancies during material insertion and curing that might compromise the results.

All study materials were used according to the manufacturers' instructions. A high-intensity output light curing unit (Ortholux Luminous Curing Light, 3M Unitek, Monrovia, CA, USA) was used to provide maximum conversion of the test resin composite restorative materials and adhesives upon curing. Each restorative material increment was light-cured for 20 seconds, and the adhesives were light-cured for 10 seconds from an occlusal direction. The Ortholux Luminous Curing Light is a fast-curing cordless light-emitting diode (LED) light with an output energy of 1600 mW/cm^2 (independent of the battery power level) that provides a wavelength of

Table 1: *Materials Used in the Study*

Material	Manufacturer	Lot No.	Description
Tetric Ceram HB	Ivoclar Vivadent, Schaan, Liechtenstein	N03283	Light-cured fine-particle microhybrid material based on a moldable ceramic
Tetric EvoFlow	Ivoclar Vivadent, Schaan, Liechtenstein	R36640	Incremental light-cured, flowable, microhybrid composite
SDR Smart Dentin Replacement	Dentsply, Milford, DE, USA	1011002185	Bulk-fill flowable composite base material that allows the curing of layers up to 4-mm thick
SonicFill Composite	Kerr, Orange, CA, USA	4252654	Bulk-fill low-shrinkage composite that allows the curing of layers up to 5-mm thick and uses sonic energy during insertion
Tetric N-Ceram Bulk Fill	Ivoclar Vivadent, Schaan, Liechtenstein	R65898	Bulk-fill resin composite material that allows the curing of 4-mm-thick layers
Tetric EvoCeram Bulk Fill	Ivoclar Vivadent, Schaan, Liechtenstein	R56348	Bulk-fill resin composite material that allows the curing of 4-mm-thick layers
Filtek P90 (Filtek LS), Low Shrink Posterior Restorative	3M ESPE, Seefeld, Germany	N 281586	Low-shrink silorane-based resin composite material
Tetric N-Bond Self-Etch	Ivoclar Vivadent, Schaan, Liechtenstein	P48222	Light-cured, one-step all-in-one self-adhesive
Tetric N-Bond	Ivoclar Vivadent, Schaan, Liechtenstein	R52704	Light-cured primer and adhesive, total-etch adhesive
AdheSE	Ivoclar Vivadent, Schaan, Liechtenstein	69346	Two bottle self-etch adhesive primer and bond
ExciTE F	Ivoclar Vivadent, Schaan, Liechtenstein	R50336	Primer and adhesive, total-etch adhesive
P90 System Adhesive, Self-Etch Primer & Bond	3M ESPE, Seefeld, Germany	N 281586	Two bottle self-etch primer and bond
Fine Etch, etchant	Spident, Incheon, Korea	FE1242	37% phosphoric acid gel

430–480 nm and a peak of 455 ± 10 nm. The details of the bonding procedures are presented in Table 2.

In groups 1–5, Tetric N-Bond Self-Etch adhesive (Ivoclar Vivadent, Schaan, Liechtenstein) was used for the se-subgroups, and Tetric N-Bond (Ivoclar Vivadent), a total-etch adhesive, was used in the te-subgroups. After the bonding procedure, the composite restorations were inserted according to the assigned study groups.

In group 1, Tetric Ceram HB (TC) resin composite (Ivoclar Vivadent) was inserted in horizontal increments of approximately 2-mm thickness each using a metallic plastic instrument (stainless steel, G. Hartzell & Son, Concord, CA, USA). Each increment was light-cured for 20 seconds before inserting the next increment until the cavity was completely filled. Before curing the most superficial increment, the plastic instrument was used to provide the proper occlusal anatomic form, then light-cured for 20 seconds.

In group 2, the open-sandwich technique was used, and Tetric EvoFlow (EF) (Ivoclar Vivadent) was used in a horizontal increment as a base of

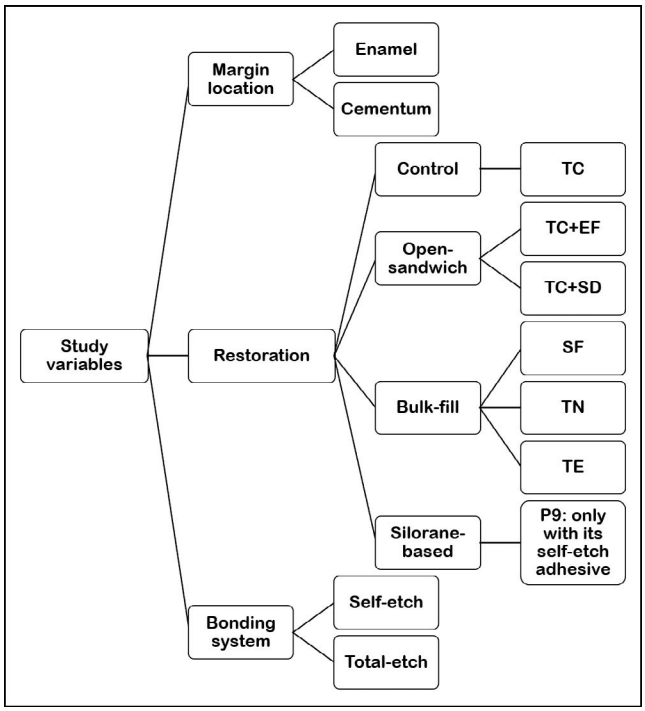

Figure 1. *Overview of the study variables.*

Table 2: *Summary of the Bonding Procedures*

Bonding System	Material Used With	Bonding Procedure
Tetric N-Bond Self-Etch (self-etch)	Tetric Ceram HB incremental Tetric Ceram HB underlined with Tetric EvoFlow Tetric Ceram HB underlined with SDR Smart Dentin Replacement SonicFill restorations Tetric N-Ceram Bulk Fill	Cavity was water-washed and air-dried after preparation, thick layers of Tetric N-Bond Self-Etch were applied to the enamel and dentin surfaces of the preparation and brushed in for 30 s. Excess Tetric N-Bond Self-Etch was dispersed with a strong stream of air and light-cured for 10 s.
Tetric N-Bond (total-etch)	Tetric Ceram HB incremental Tetric Ceram HB underlined with Tetric EvoFlow Tetric Ceram HB underlined with SDR Smart Dentin Replacement SonicFill restorations Tetric N-Ceram Bulk Fill	Cavity was water-washed and air-dried after preparation, etched for 15 s, and washed with vigorous water spray. Excess moisture was removed. Thick layers of Tetric N-Bond were applied to the enamel and dentin using an application brush, air-thinned, and light-cured for 10 s.
AdheSE (self-etch)	Tetric EvoCeram Bulk Fill	Cavity was water-washed and air-dried after preparation. One drop of primer and one drop of adhesive were dispensed individually. Primer was applied to the enamel and dentin for 30 s with a microbrush. Adhesive was applied with a microbrush and air dispersed with a strong air stream and light-cured for 10 s.
Excite F (total-etch)	Tetric EvoCeram Bulk Fill	Cavity was water-washed and air-dried after preparation, etched for 15 s, and washed with vigorous water spray. Excess moisture was removed. Thick layers of Excite-F bond were applied to the enamel and dentin using an application brush. Excess adhesive was dispersed with a strong stream of air and light-cured for 10 s.
P90 System Adhesive (self-etch)	Filtek P90 (Low Shrink Posterior Restorative System)	Cavity was washed and dried. The self-etch primer was applied to the enamel and dentin and massaged into the entire surface for 15 s. A gentle stream of air was applied until the primer was spread into an even film. The primer was light-cured for 10 s, then the adhesive was applied to the entire area of the cavity and a gentle stream of air was applied until the bond was spread into an even film, then light-cured for 10 s.

approximately 2 mm under the Tetric Ceram HB (Ivoclar Vivadent) (TC + EF), followed by light-curing for 20 seconds. TC was then incrementally inserted until the cavity was completely filled in a manner similar to group 1.

In group 3, the open-sandwich technique was used with SDR Smart Dentin Replacement (SDR) bulk-fill flowable resin composite (Dentsply International, Milford, DE, USA) as a base under TC (TC + SDR). A compule tip gun was used to eject the SDR into the cavity to form a base of approximately 4-mm thickness before light-curing for 20 seconds. The rest of the cavity was then incrementally filled with TC.

In group 4, SonicFill (SF), a Sonic-Activated Bulk Fill Composite System (Kerr, Orange, CA, USA) was applied using the SonicFill Handpiece (Kavo, Biberach, Germany). The handpiece was used to automatically dispense rheologically matched filling materials contained in SonicFill Unidose tips into the cavity via the action of sound and pressure. The SonicFill Handpiece works at a frequency of 5–6 kHz and was connected to the turbine hose of the dental unit through a multi-flex coupling device. The material was inserted in a first bolus of approximately 5-mm thickness and light-cured from the occlusal direction for 20 seconds. A second, thinner horizontal increment was then inserted to completely fill the cavity and was light-cured for 20 seconds after reestablishing the occlusal anatomic features as mentioned previously.

In group 5, Tetric N-Ceram Bulk Fill (TN) was used (Ivoclar Vivadent). An increment of approximately 4-mm thickness was inserted into the cavity using a plastic instrument (G. Hartzell & Son) and light-cured from the occlusal direction for 20 sec-

onds. A second increment of TN was inserted until the cavity was completely filled, anatomically contoured, and light-cured from the occlusal direction.

In group 6, Tetric EvoCeram Bulk Fill (TE) was used (Ivoclar Vivadent) in a manner similar to that used for group 5. However, AdheSE (Ivoclar Vivadent), a self-etch adhesive, or Excite F (Ivoclar Vivadent), a total-etch adhesive, were used in this group following the recommendations of the manufacturer.

In group 7, Filtek P90 (P90), a silorane-based composite (3M ESPE, Seefeld, Germany) was applied using a plastic instrument in increments of approximately 2.5-mm thickness. Each increment was light-cured for 20 seconds. Before the light-curing of the last increment, the occlusal anatomic features were reestablished. Only the P90 System Adhesive, a self-etch adhesive, was used in this group following the instructions of the manufacturer.

After the restorations were completed, a surgical scalpel blade (No. 15, Swann-Morton, Sheffield, England) was used to remove the gross marginal overhangs. Finishing and polishing were performed with 13-mm Sof-Lex XT discs (Sof-Lex XT Finishing and Polishing System, 3M ESPE, St. Paul, MN, USA) beginning with the coarser grit disc and ending with the superfine grit. The discs were mounted on a Sof-Lex finishing and polishing disc mandrel and were used at a slow speed range under abundant air-water spray.

All samples were exposed to artificial aging via thermal and occlusal load-cycling. The test specimens were placed in mesh bags and subjected to thermocycling for 5000 cycles in water baths between 5 ± 2°C and 55 ± 2°C with a dwell time of 30 seconds in each bath and a transfer time of 15 seconds between baths (Thermocycling machine, Proto-Tech, El Segundo, CA, USA). The specimens were then submitted to intermittent vertical occlusal loads between 25 and 100 N at 20 cycles/minute (20 HZ) for 1000 cycles using the chewing simulator CS4.2 (SD Mechatronik GmbH, Westernham, Germany) with a round-end piston that was 5 mm in diameter and touching the tooth and restorations at the buccal and lingual internal cuspal inclines.

After aging, a sawing machine (Isomet 5000 Linear precision saw, Buehler Ltd, Lake Bluff, IL, USA) was used at the lowest blade speed with water lubrication to section the teeth. Each tooth was serially sectioned longitudinally approximately 3–4 mm short of the acrylic block base in the mesiodistal direction to produce slabs that were approximately 0.8-mm thick and that contained restorations. An additional buccolingual longitudinal cut was made in the center of the tooth next to the axial walls of the restorations to approximately 1 mm short of the end of the acrylic block. After machine sawing, the samples were hand-split into two longitudinal proximal halves. The rods containing the restorations in both halves were cut apically short of the root apices using a needle-shaped diamond abrasive at high speed with an abundant air-water spray. Before, during, and after the cutting procedure, the rods with restorations at the gingival margins in the enamel were identified and separated from those with gingival cementum margins using a magnifying lens. Two microrods per restoration (n = 14/group) were tested for MTBS, and one microrod of each restoration (n = 7/group) was used to assess the cervical IGD under a scanning electron microscope (SEM) (×1200). The details of the work flow are shown in Figure 2.

MTBS

During the MTBS testing, each microrod was measured at the bonding interface in the buccolingual direction using a digital caliper (Clarke International). Each microrod was then fixed to a modified microtensile testing device (ie, an attachment jig) that allowed for loading in the vertical direction. Two layers of bonding agent (Tetric N-Bond) were applied to the two sides of the testing device, the microrod was longitudinally seated in line with the testing load direction, and the bonding agent was light-cured. A flowable composite was applied to each end of the microrod and light-cured to ensure strong rod-fixing. After that, an hourglass shape was produced at the bonded interface in the mesiodistal direction using a needle diamond abrasive in the high-speed range under abundant air-water coolant. The mesiodistal side of the microbar was measured using a digital caliper to the nearest 0.01 mm. The surface area of the bonded interface of each rod was calculated. The samples were subjected to microtensile testing at a crosshead speed of 0.5 mm/min using a universal testing machine (Instron 8871 Universal Testing Machine, Instron, Shakopee, MN, USA). The fracture loads were obtained in newtons and divided by the surface areas of bonding, thereby obtaining microtensile bond strength values in megapascals. Immediately after testing, all samples were examined under a stereomicroscope (Stereozoom 250 Microscope, Luxo Microscopes, Elmsford, NY, USA) at ×40 magnification to determine the failure mode. The failure modes were categorized as follows:

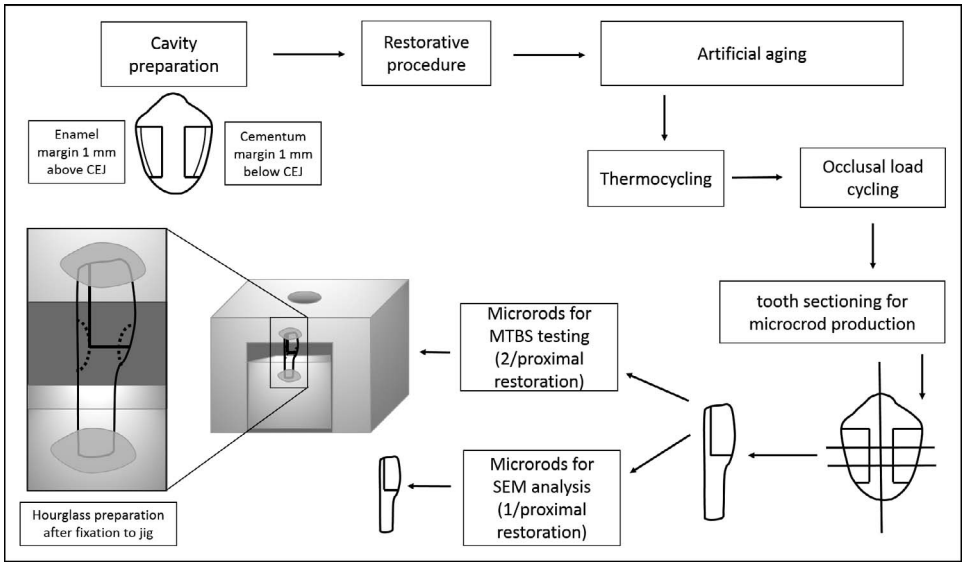

Figure 2. *Work flow of the microrod prepration for microtensile bond strength and intercial gap distance testing.*

a. Adhesive failure along the tooth/restoration interface
b. Cohesive failure in the resin involving the resin composite material and/or the bonding layers
c. Cohesive failure of the tooth
d. Mixed failure involving adhesive fracture and cohesive fracture in the composite with or without fracture in the tooth.

SEM examinations (×500) were performed on two of the failed microrods from each study group.

All data analysis for MTBS was conducted using R 3.0.2.[20] MTBS values were subjected to the Shapiro-Wilk confidence test, which proved that data were not normally distributed. Therefore, comparisons between groups for differences in MTBS were performed using a nonparametric Kruskal-Wallis test followed by a Conover-Inman post hoc test for pairwise comparisons. In addition, a Bonferroni correction was applied to correct for multiple comparisons. Differences with p values <0.05 were considered significant. All pretest failures were recorded as zero but were not included for the statistical analysis. For the statistical testing, only one out of two microrods per restoration was randomly chosen to avoid considering microrods from the same restoration as independent samples as proposed by Eckert and Platt.[21] Statistical analyses with randomly chosen

Table 3: Mean MTBS values, SD, minimum, maximum, and medians

Group	MTBS Values (MPa) at Enamel Margin						MTBS Values (MPa) at Cementum Margin					
	N	Mean	SD	Min	Max	Median	N	Mean	SD	Min	Max	Median
TC/se	14	14.7	4.1	6.8	20.4	14.8	12	7.6	4.0	2.2	17.4	7.0
TC/te	14	15.8	3.6	9.6	22.4	17.1	13	7.6	2.7	3.2	11.8	7.9
TC+EF/se	13	18.9	4.2	8.0	24.4	19.6	12	9.5	3.5	3.7	13.8	10.2
TC+EF/te	13	20.5	6.5	12.8	37.8	20.4	13	12.6	3.9	8.6	22.7	11.5
TC+SDR/se	14	13.8	4.3	1.9	18.0	15.0	12	12.7	8.6	3.7	38.5	10.5
TC+SDR/te	14	24.2	9.5	12.0	48.4	25.1	13	23.2	15.4	5.4	57.9	21.7
SF/se	14	13.1	2.0	10.8	18.0	12.4	13	10.8	3.6	5.8	18.5	11.2
SF/te	14	23.8	5.2	17.1	34.7	23.4	13	14.0	2.1	10.3	17.1	13.2
TN/se	13	16.2	8.2	3.3	37.0	16.8	13	7.6	3.8	1.6	13.2	6.4
TN/te	14	15.9	8.5	5.6	35.0	15.1	12	9.6	4.9	3.5	18.6	10.0
TE/se	12	13.6	5.7	6.1	21.3	13.9	12	9.4	3.6	5.1	16.4	8.6
TE/te	12	12.0	5.6	2.6	19.4	13.0	12	8.6	2.2	6.2	12.2	7.8
P90/se	14	23.6	4.1	12.5	28.0	24.6	13	18.6	7.9	9.1	32.2	17.3

Abbreviations: EF, Tetric EvoFlow; Max, maximum; Min, minimum; MTBS, microtensile bond strength; P90, Filtek P90 Low Shrink Restorative; SD, standard deviation; SDR, SDR Smart Dentin Replacement; se, self-etch; SF, SonicFill; TC, Tetric Ceram HB; te, total-etch; TE, Tetric EvoCeram Bulk Fill; TN, Tetric N-Ceram Bulk Fill.

Table 4: *Significantly Different Groups With p<0.05 in More Than 80% of Statistical Comparisons of Randomly Chosen Microrods (One Microrod per Restoration)*

Significantly Different Groups (>80% With $p<0.05$)		
P9/se/enamel	vs	SF/se/cementum
P9/se/enamel	vs	TC+EF/se/cementum
P9/se/enamel	vs	TC/se/cementum
P9/se/enamel	vs	TC/te/cementum
P9/se/enamel	vs	TE/se/cementum
P9/se/enamel	vs	TE/te/cementum
P9/se/enamel	vs	TN/se/cementum
P9/se/enamel	vs	TN/te/cementum
SF/te/enamel	vs	SF/se/cementum
SF/te/enamel	vs	TC+EF/se/cementum
SF/te/enamel	vs	TC/se/cementum
SF/te/enamel	vs	TC/te/cementum
SF/te/enamel	vs	TE/se/cementum
SF/te/enamel	vs	TE/te/cementum
SF/te/enamel	vs	TN/se/cementum
SF/te/enamel	vs	TN/te/cementum
TC/EF/se/enamel	vs	TC/se/cementum
TC+EF/se/enamel	vs	TC/te/cementum
TC+EF/te/enamel	vs	TN/se/cementum
TC+EF/te/enamel	vs	TC/se/cementum
TC+EF/te/enamel	vs	TC/te/cementum
TC+EF/te/enamel	vs	TE/te/cementum
TC+EF/te/enamel	vs	TN/se/cementum
TC+SD/te/cementum	vs	TC/te/cementum
TC+SD/te/enamel	vs	TC/se/cementum
TC+SD/te/enamel	vs	TC/te/cementum
TC+SD/te/enamel	vs	TE/se/cementum
TC+SD/te/enamel	vs	TE/te/cementum
TC+SD/te/enamel	vs	TN/se/cementum
TC+SD/te/enamel	vs	TN/te/cementum

Abbreviations: EF, Tetric EvoFlow; MTBS, microtensile bond strength; P90, Filtek P90 Low Shrink Restorative; SDR, SDR Smart Dentin Replacement; se, self-etch; SF, SonicFill; TC, Tetric Ceram HB; te, total-etch; TE, Tetric EvoCeram Bulk Fill; TN, Tetric N-Ceram Bulk Fill.

samples were repeated 10,000 times. Groups were considered significantly different when more than 80% of the repetitions showed p values <0.05.

IGD

To study the cervical interfacial micromorphology, one microrod from each restoration (n=7/group) was randomly selected and processed for SEM examination of the cervical interfaces (×1200) in a manner similar to the technique used by Duarte and others.[22] The widest interfacial gap in each specimen was measured and recorded in microns.[22] Statistical analysis was performed using the Kruskal-Wallis nonparametric test and post hoc Mann-Whitney U test in IBM SPSS Statistics 21 (IBM Corporation, Armonk, NY, USA).

RESULTS

MTBS

Table 3 shows the mean MTBS values, standard deviations, minimum, maximum, and median of the samples in each group. Table 4 shows significantly different groups with $p<0.05$ in more than 80% of comparisons. In all study groups, the mean MTBS values were higher at the enamel than at the cementum margins, but there was no statistical significance. At the enamel margins, the highest mean MTBS values were 24.2 ± 9.5 MPa for TC+SDR/te, 23.8 ± 5.2 MPa for SF/te and 23.6 ± 4.1 MPa for P90/se. At the cementum margins, the highest values were 23.2 ± 16.1 MPa for TC+SDR/te, 18.6 ± 7.9 MPa for P90/se, and 14.0 ± 2.1 MPa for SF/te; the lowest values at the cementum margins were 7.6 ± 4.0 MPa for TC/se, 7.6 ± 2.7 MPa for TC/te, and 7.6 ± 3.8 MPa for TN/se. All test restorations, with the exception of the TE, exhibited better mean bond strength values when tested with the total-etch rather than the self-etch bonding approach. TC+SDR/te at the enamel and cementum exhibited significantly higher MTBS values than control group TC/te and TC/se at cementum margins ($p<0.05$).

Similarly, SF/te at enamel margins exhibited significantly better mean values than the control group TC/te and TC/se at cementum margins ($p<0.05$). Furthermore, P90 exhibited significantly higher values than the control group TC/te and TC/se at cementum margins ($p<0.05$).

In contrast, TN and TE and TC+EF/se and TC+EF/te exhibited MTBS values that were not significantly different from those of the control group TC/te and TC/se ($p>0.05$) at enamel and cementum. However, at the enamel margins, TC+EF/te exhibited a significantly higher value than the control group TC at cementum.

Study of the failure modes of the failed ends of the microrods revealed that they were either mixed, adhesive, or cohesive failures in the resin composites. No cohesive failures in the tooth structures were observed in any of the tested samples. Most of the failures were mixed fractures. No cohesive failures in the resin were observed in TC+EF/se,

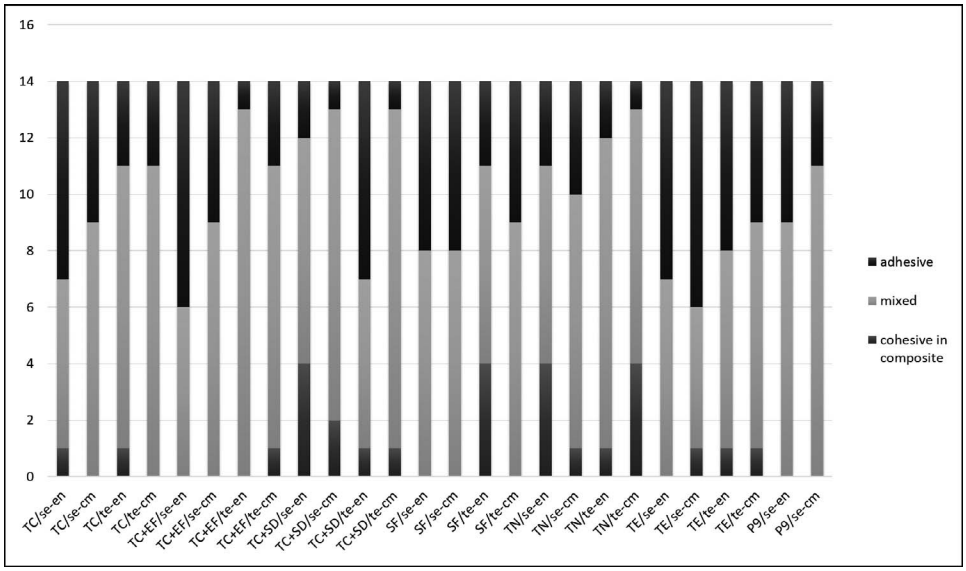

Figure 3. *Graph of the adhesive, mixed, and cohesive failure modes of microrods upon microtensile bond strength testing.*

SF/se, or P90, regardless of cervical margin location (Figures 3 and 4).

IGD

SEM examination of the cervical interfaces of the restorations revealed different areas of perfect cervical interfacial bonding zones and gaps that varied with the different restoration and bonding techniques. Table 5 presents the mean IGD and standard deviations of the different study groups. Figure 5 illustrates the statistically significant ($p<0.05$) difference in interfacial gap distances between P90 at the enamel and cementum margins and the control group TC/se at the cementum margins. The bulk-fill composites did not significantly improve the adaptations compared with the control group TC ($p>0.05$; Table 5).

DISCUSSION

In vitro testing of the effectiveness of interfacial tooth restoration bonds has long been performed in marginal sealing and MTBS studies to predict the clinical reliability of these bonds. Artificial aging via immersion in different media for different periods and thermal and occlusal load cycling has been used to mimic oral environmental conditions.[7,11,12,23,24]

In this study, the samples were tested for MTBS after thermal and load cycling because our pilot study and previous reports indicated that the results of immediate testing of restorations are less signif-

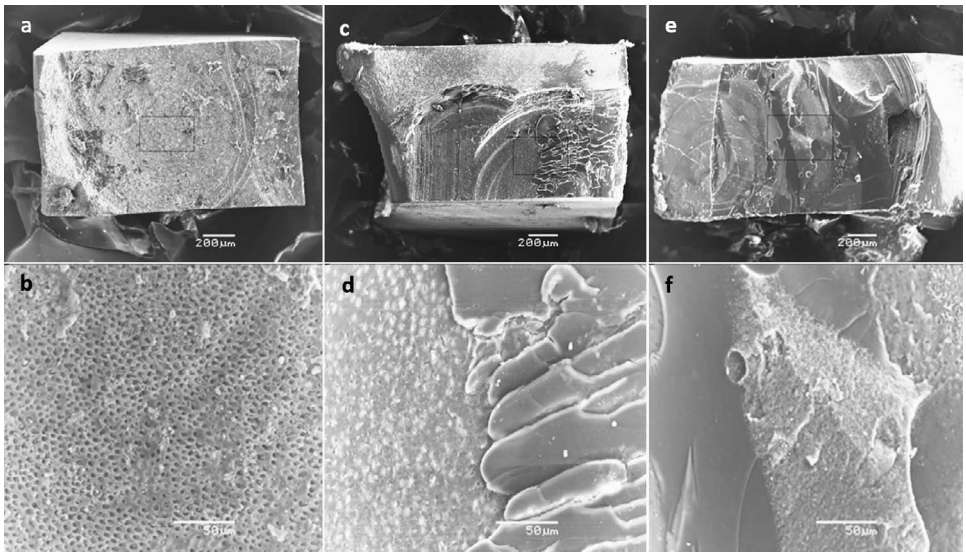

Figure 4. *Scanning electron microscope images (×40; x500) of the different failure modes: adhesive failure (a, b), mixed failure (c, d), and cohesive failure (e, f).*

Table 5: Mean Values of the Interfacial Gap Distances and Standard Deviation (SD)

Group	Mean Gap Distance at Enamel Margin (μm)[a]	SD	Mean Gap Distance at Cementum Margin (μm) [a]	SD
TC/se	7.3[b]	5.6	15.5[a]	8.4
TC/te	3.4[b]	4.3	9.5	6.7
TC+EF/se	9.1	6.1	16.3[a]	5.0
TC+EF/te	6.1[b]	4.3	13.4	7.0
TC+SD/se	8.8	6.9	12.6	6.2
TC+SD/te	6.4[b]	4.7	8.4	6.7
SF/se	6.9[b]	6.5	11.4	5.2
SF/te	6.8[b]	6.1	14.0	5.2
TN/se	8.7	7.8	18.4[a]	2.8
TN/te	6.7[b]	5.9	14.4	5.8
TE/se	6.8[b]	5.3	14.6	7.3
TE/te	9.9	4.6	14.9	5.7
P9/se	2.5[b]	2.9	7.7[y]	3.8

Abbreviations: EF, Tetric EvoFlow; MTBS, microtensile bond strength; P90, Filtek P90 Low Shrink Restorative; SD, standard deviation; SDR, SDR Smart Dentin Replacement; se, self-etch; SF, SonicFill; TC, Tetric Ceram HB; te, total-etch; TE, Tetric EvoCeram Bulk Fill; TN, Tetric N-Ceram Bulk Fill.
[a] Different letters indicate significant difference ($p<0.05$).

icant.[25] Bulk-fill resin composites that have been reported to exhibit reduced polymerization contraction stress were tested.[14-19]

Maxillary premolars with proximal box-only preparations were used to simulate cuspal deflections with challenging interfacial stresses on occlusal cyclic loading.[26] The testing of Class II box-only restorations provides higher C-factors that lead to greater polymerization contraction stresses compared with bonding to flat dentin surfaces.[27-30] High-intensity LED curing (1600 mW/cm^2) was used to rapidly provide a high degree of conversion to increase the challenging polymerization contraction stresses.[18,31-34] Therefore, the specimens were confronted with challenges of rapid intense contraction and thermal and occlusal load fluctuation stresses.

In this study, the modified testing device (ie, an attachment jig) used by El Zohairy and others[35] was

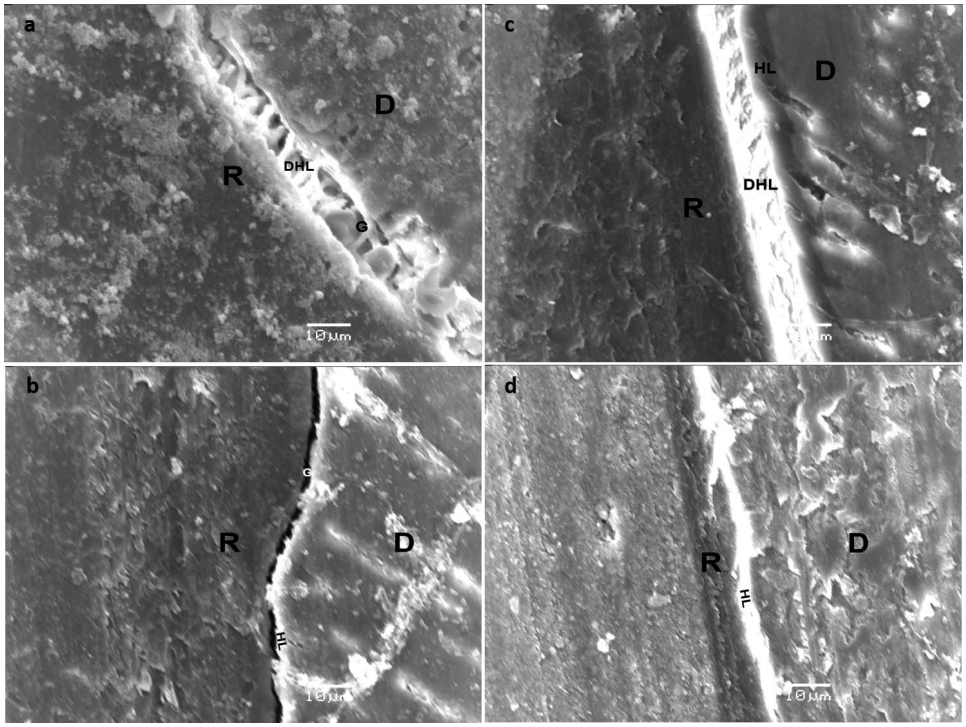

Figure 5. *Scanning electron microscope images (×1200) of the interfacial gap distance (IGD): interfacial gap in TC/te at the cementum margin (a), in TC+EF/se at the cementum margin (b), in TC+SDR at the cementum margin (c) and in P90/se at the enamel margin (d). Abbreviations: D, dentin; DHL, disrupted hybrid layer; EF, Tetric EvoFlow; G, gap; HL, hybrid layer; P90, Filtek P90 Low Shrink Restorative; R, resin; SDR, SDR Smart Dentin Replacement; se, self-etch; TC, Tetric Ceram HB; te, total-etch.*

used for MTBS testing. The microrod production and hourglass shape preparations were the least traumatic and minimized pretest failures. The numbers of microrods that have been used in MTBS vary widely between studies.[12,23,27,28] In the current MTBS testing, two microrods were used in each restoration, and 14 microrods per group were tested in either the enamel or cementum margins. For the evaluation of MTBS values, only one out of two microrods was randomly chosen for multiple comparisons to avoid treating microrods obtained from the same restoration as independent samples as recommended by Eckert and Platt.[21] For this reason, the number of samples considered at each comparison was restricted to seven, which constitutes a limitation of the current study.

All pretest failures were recorded and given a value of zero megapascals but were not included in the multiple statistical analyses, which is similar to the approach of Takahashi and others,[25] who recorded all pretest failures but did not include any in their statistical analyses. They used the freehand technique to prepare hourglass shapes; this technique can be traumatic and increase the pretest failures. However, this trauma was avoided in the preparation of our microrods.[21]

MTBS studies have been reported to effectively and reliably discriminate between adhesive bonding systems. Additionally, MTBS studies have also stated correlations between retention and many influencing factors, such as the diameter of the stick, the type of testing device, trimming into an hourglass shape, the handling of pretest failures, and the artificial aging technique.[11,21] The lack of adequate consistency between MTBS studies and the desire to clearly understand the correlation between a particular bond strength test and clinical performance have prompted recommendations to clarify the specimen fabrication details.[21,36-39] In the current study, microrods were trimmed into hourglass shapes before MTBS testing to concentrate the stresses at the sites of bonding and to correlate failures to bonding interfaces with fewer incidences of cohesive failures, although this practice may have generated stress concentrations and increased pretest failures.[39]

The higher MTBS values at the enamel margins compared with the cementum margins found in all study groups were not significant, regardless of the bonding technique. This can be explained by similarity in effectiveness of the adhesives/restorations used at both enamel and cementum margins. This is contrary to the reports that the cementum at the outer part of the interface and the orientation of the dentinal tubules in the deep proximal cavities might interfere with proper micromechanical interlocking and effective hybridization.[11]

Takahashi and others[25] studied MTBS in Class II restorations in molars that were restored after thermo-load cycling using Scotchbond Multipurpose, Adper Scotchbond1, Clearfil SE, and Clearfil Tri-S and incrementally placed resin composite Clearfil AP-X at the enamel and dentin margins. They found statistically significant differences among all groups at the enamel and dentin margins.[25] The differences in the materials and test protocol might explain the variations in the results between studies.

Findings in this study showed that EF liner did not significantly improve MTBS and that SDR combined with total-etch bonding produced an MTBS that was significantly higher than that of the control group TC regardless of margin location or bonding approach. This is in agreement with previous findings that confirmed that the effects of flowable liners on MTBS are specific to the material and the bonding system.[27,28,40,41]

Our results did not confirm that the use of total-etch bonding produced significantly higher MTBS values than those achieved by self-etch bonding in all comparisons. Although in terms of stability and degradation resistance, the total-etch bonding technique has been reported to produce more reliable resin-dentin hybrid layers than self-etch adhesives, particularly at the enamel margin,[2,7,11,24,28] reliability also seems to be affected by the performance of the specific adhesive bonding/restorative material.

SDR is a bulk-fill base with a modified methacrylate resin (a polymerization modulator), a slow polymerization rate, and a filler loading of 68 wt% that produces significantly lower polymerization contraction stresses than those produced by conventional flowable composites.[14,15] Therefore, combining the benefits of SDR with the superior performance of total-etch bonding may have provided the best resistance to challenging stresses, which would explain the results.

Similarly, the superior performance of SF/te at the enamel margins can be explained by the combination of the benefits of total-etching and the heavily filled (83.5 wt%) material with the reduced polymerization contraction (1.6%) and contraction stresses.[19]

The use of the P90 with its corresponding adhesive system provided the second-highest MTBS results, compared with the control group TC, at the cementum regardless of the bonding approach. This finding

may be related to the low contraction stresses of this low-shrinkage silorane-based material[42,43] and is probably related to a superior resistance of this material to thermo-load cycling relative to that of methacrylate-based composites. It has been reported that the reduced polymerization shrinkage of silorane composite results in significantly less stress at the bonding interface and reduces the need for a very strong adhesive.[42-44]

Interfacial gap distance measurements of microrods have been previously performed. Duarte and others[22] used 1-mm thick slices obtained by sectioning restored human third molar teeth for SEM assessment of the interfacial gap distances in class V restorations. Heintze[12] also reported this method. Loguercio and others[45] used 0.8 mm^2 cross-sectional area sticks of resin composite bonded to dentin to measure interfacial gaps (×400) using light microscopy.

Results of the interfacial gap distance measurements were in agreement with MTBS results, where better results were obtained by P90 at both enamel and cementum margins. At enamel margins, IGD did not significantly differ from the control TC, from TC+SDR/te, or from SF. On the contrary, at cementum margins, P90 did differ significantly from all other study groups and showed the least gaps.

Variations in cervical micromorphologic patterns related to the presence of gaps and differential appearances of resin-dentin interdiffusion along the respective cervical interfaces of the test restorations confirm the multifactorial nature of adhesive joint effectiveness, degradation, and debonding.[1-3,7] The effectiveness of the adaptation of the silorane-based composite has previously been reported in other studies.[45] The reduced shrinkage silorane-based resins and improved resistance to bonding degradation of the Filtek P90 System Adhesive explain the significantly better adaptation at the enamel and cementum margins relative to the control group TC at the cementum margin.

Although the null hypothesis that bulk-fill resin composites do not significantly affect MTBS can partially be rejected based on the results of the current study, the results also confirm that the use of these resins does not affect the cervical interfacial gaps.

CONCLUSIONS

The following conclusions can be made based on comparisons to the incrementally applied control group TC. In the open-sandwich restorations, the use of SDR with the total-etch approach significantly improved the MTBS values, whereas the conventional flowable composite EF did not. SF with total-etch and P90 at enamel significantly improved MTBS relative to TC at cementum, whereas TN and TE did not. The cervical interfacial adaptation of P90 at both enamel and cementum margins was significantly better than that of TC at the cementum margins when bonded by the self-etch approach.

When cervical margins of Class II cavities are inevitably located in cementum, bulk-fill and silorane based restorations might be preferable. When possible, restorations should be bonded using a total-etch approach.

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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 University of Dammam institutional review board. The approval code for this study is 110/2012.

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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Surface Treatments of Zirconia to Enhance Bonding Durability

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Clinical Relevance

The description of the available surface treatments to Y-TZP ceramics provides a background for its clinical application. An efficient chemical interaction alone is desirable because of potential harm from air abrasion. Currently, silica coating associated with a 10-methacryloxydecyl dihydrogen phosphate primer seems the most common form of surface treatment to provide long-lasting bonding to zirconia.

SUMMARY

This article reviewed the surface treatments used most often to improve adhesion between zirconia and adhesive cements, focusing on their capacity to provide long-term bonding. Traditional and new treatments for zirconia bonding were searched. Some new treatments were discussed along with topographical views of the modified zirconia. New methods, such as selective infiltration etching and the low-fusing glassy porcelain application are promising, but more research is needed.

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INTRODUCTION

The progressive improvement in the properties of dental ceramics has led to an increase in metal-free restorations. These properties include improved esthetics, chemical resistance, hardness, compression resistance, and biocompatibility.[1-3]

Several studies[4-7] have investigated the bond strength and the durability of various bonding methods to dental ceramics. Bonding to traditional silica-based ceramics, generally employing both mechanical and adhesive retention, has been well researched, and bond strengths are predictable.

While hydrofluoric acid (HF) etching along with methacryloxypropyl trimethoxysilane (MPS)application is a commonly recommended method used to roughen the surface of silica-based ceramics and to increase their wettability,[6] zirconia is a polycrystalline nonetchable material.[8-12] Adhesion is still an

issue, since a very low and unstable resin bond is promoted when the Y-TZP ceramic is untreated or has received primer application only.[13-16] Clinically, crown debonding (loss of retention) is a type of failure of zirconia-based restorations, and the search for surface treatments that improve resin adhesion to zirconia has increased in intensity.[12] In spite of that, there is little information about the most effective and durable bonding methods,[15,16] namely the new treatments such as nano-film deposition of silicon oxides,[17,18] the glaze-on technique (application of a thin, low-fusing glassy porcelain layer rich in silicon oxides),[19,20] heating silanes,[21] and chemical etching used for bond improvement to zirconia.[22,23] A previous report[24] of the literature showed that zirconia bonding was durable when air abrasion and a 10-methacryloxydecyl dihydrogen phosphate (MDP) component were combined, but this report was less informative with regard to the new surface treatments.

Thus, the aim of this review article was to search for the long-term response of the surface treatments used on Y-TZP zirconia surfaces for adhesion, from the most traditional to the newest ones, so that clinicians can discern which can provide the most durable zirconia restorations. Medline database (PubMed) was used as the main source of information for this literature review. The terms used were "zirconia and bond strength durability" and "zirconia and surface treatments." We excluded articles that were not in the English language and those that had been published before 1999, as well as articles regarding the durability of bonding between zirconia and veneering ceramics.

TRADITIONAL TREATMENTS

So far, the surface treatments most indicated for dental zirconia are chemical modification of the surface, micromechanical interlocking through air abrasion, or a combination of both. Ideally, utilizing chemical adhesion in addition to mechanical retention is required for zirconia, mainly because of zirconium dioxide's (ZrO_2) nonpolar surface, which leads to a nonstable (hydrolyzable) interface.[18]

Inokoshi and others[25] evaluated the effect of mechanical (tribochemical silica coating/Cojet and Rocatec, 3M ESPE, Seefeld, Germany) and chemical (silane/MDP–combined ceramic primers) surface pretreatment on the bond durability of two composite cements (bisphenol A diglycidyl ether dimethacrylate [Bis-GMA]–based and MDP-based cements) to dental zirconia. The combined mechanical and chemical surface pretreatment of zirconia improved the bond durability of both composite cements bonding to zirconia after storage for six months.

Attia and Kern[26] investigated the durability of the bond strength of adhesive luting cement to zirconia ceramic after the application of different ceramic primers. They concluded that after storage for 150 days in water without thermal cycling, a new universal primer provided significantly better long-term resin bonding to zirconia ceramic than did a conventional silane. They also concluded that cleaning methods had little effect on long-term resin bonding to zirconia ceramic. They also showed that a combined mechanical and chemical pretreatment is the best recommendation for a durable bond to zirconia.

There is some evidence that a minimum bond to Y-TZP ceramics is obtained using resin cements with phosphate ester monomers (MDP) alone. Wolfart and others[2] evaluated the durability of the bond to zirconia with two resin cements (Bis-GMA–based and MDP-based) after using different surface conditioning methods. The use of the MDP-containing composite resin on air-abraded zirconia ceramic presented the highest bond strength to zirconia surfaces after 150 days of water storage and was recommended as a promising bonding method. On the other hand, when evaluating the shear bond strength between zirconia and the dual-cured resin cement, Yoshida and others[27] reported that the application of the mixture primer and zirconate coupler was effective.

As mentioned previously, air abrasion in combination with phosphate ester monomer (MDP)–containing luting agents results in high, durable bond strengths, because the phosphate ester group chemically bonds to metal oxides such as zirconium dioxide. Sarmento and others[28] also reported that when zirconia was air-abraded with aluminum oxide (Al_2O_3) (110 μm) it resulted in higher roughness values, but air-abrasion protocols with silicon dioxide (SiO_2) (110 μm; Rocatec, 3M ESPE) promoted better adhesion to MDP-based resin cement (Panavia F/Kuraray, Kurashiki, Okayama, Japan).

In addition to that, heat treatment of the zirconia pretreated with the silane primer also resulted in higher bond strengths and bonding durability than did those created with the use of acidic primers.[21]

Ferracane and others[29] compiled a series of studies about self-adhesive resin cements, showing that they have been extensively studied on zirconia surfaces. Among all cements, Unicem (3M ESPE, St Paul, MN, USA [currently marketed as RelyX

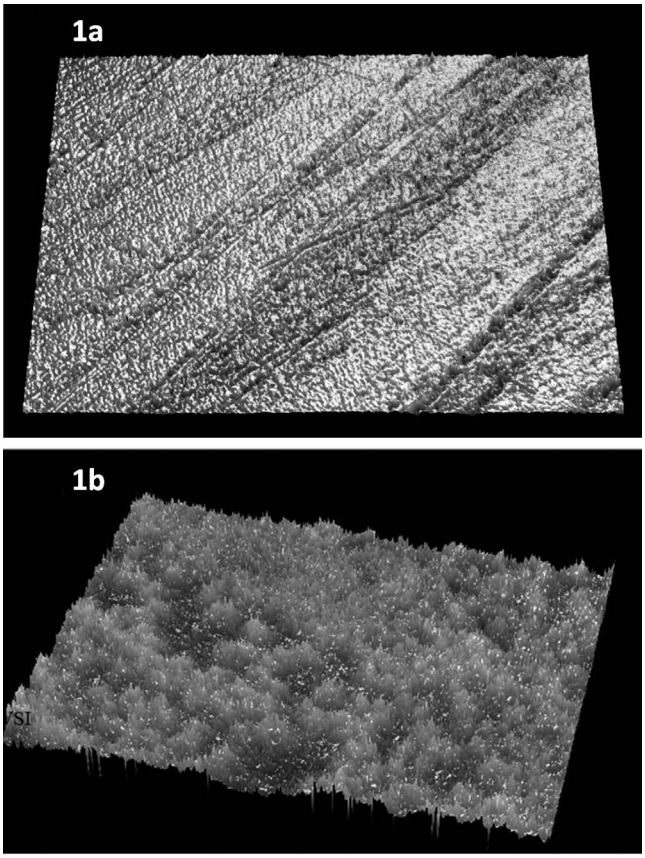

Figure 1. *Surface profilometry of control (a) and silica-infiltrated (b) Y-TZP surfaces.*

U200]), which was the first cement to contain a methacrylated phosphoric ester as a functional monomer, was the most promising material in terms of adhesion to zirconia, presenting results comparable to those of Panavia F (Kuraray America, New York, NY, USA). However, the authors[29] stated that the bonding stability is better when silica coating is used and that the decrease in bond strength depends on the aging protocol used.

NEW TREATMENTS

Some studies[19,20,30] reported that selective infiltration etching (SIE), based on ceramic infiltration by molten silica and posterior removal with hydrofluoric acid creating micromechanical irregularities, can enhance the zirconia ceramic-to-resin cement bonds. Aboushelib and others[31] evaluated the zirconia/resin bond strength and durability using this technique and observed that the bond strength of the SIE specimens was stable and strong after 26 weeks of water storage and thermal cycling (51.9 MPa). They also demonstrated a good seal against silver nitrate penetration across the zirconia/resin interface.

The idea of infusing silica on zirconia, making it chemically reactive to the Bis-GMA–based cements, is also present in several other methods that either lack long-term studies or were not sufficiently stable after aging. Examples of such methods are the pyrosilpen method, which promises a firmly fixed adhesive silicate coating after flame treatment[32]; the "glaze-on technique" that uses a thin intermediary coating of acid-etchable glasses[33,34]; the silica nanocoating; and[35] the sol-gel silica deposition.[36] A cold sol-gel process performed in the current authors' laboratory was used to infiltrate and grow a silica layer on pre-sintered zirconia surfaces that become etchable and treatable with silanes (Figure 1).

From the simplicity standpoint, the SIE looks very intricate and is still not accessible for more experimentation. The creator of the SIE method[20] stated "... SIE requires an investment of time and effort in order to achieve the required surface properties, and remains sensitive to the handling procedure during every step of the technique."

On the other hand, the "glaze-on," or vitrification, technique showed good bond strength results and can be easily performed, even though the vitrification of the intaglio surface of a Y-TZP–based crown might affect the seat of the crown, resulting in margin misfit. Unpublished results from this research group showed debonding of zirconia crowns with internal vitrification after 2 $x10^6$ cycles, apparently between the cement and the zirconia intaglio (Figure 2). Thus, both of these surface treatments still need more investigation.

The application of an experimental hot etching solution (methanol: 800 mL; 37% hydrochloric acid: 200 mL; and ferric chloride: 2 g at 100°C) changed zirconia surface morphology (Figure 3) and induced a significant improvement in surface roughness, comparable with those of the well-known surface treatment modalities. This was first reported by Casucci and others,[22] who studied the influence of different surface treatments on the microtensile bond strength of resin cement to zirconia ceramic. They reported that conditioning the high-strength ceramic surface with SIE and experimental hot etching solution treatments yielded higher bond strengths than were obtained with airborne particle abrasion or untreated specimens. To date there are no long-term studies involving this method.

Other options for increasing bonding to zirconia are just not suitable for clinical applications, mainly

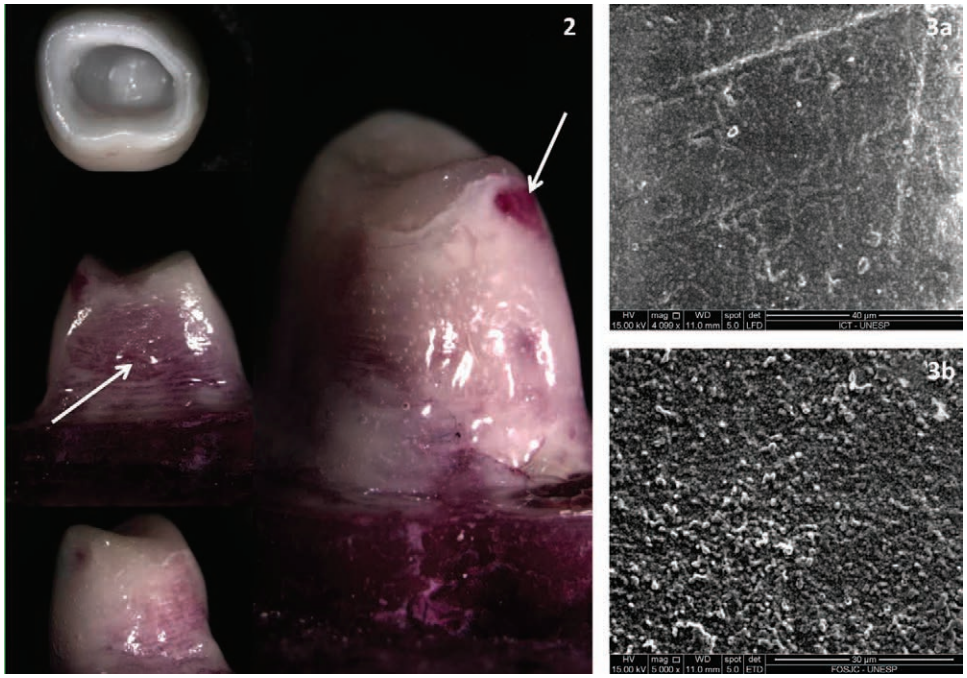

Figure 2. *Zirconia crown debonded after 2 x10^6 mechanical cycles, showing infiltration zones between the cement layer and the crown (arrows). The specimen had been previously submitted to internal vitrification, acid etching for 30 seconds, Monobond S (Ivoclar Vivadent, Zurich, Switzerland) silane treatment, and bonding with Variolink cement (Ivoclar Vivadent).*

Figure 3. *Nontreated (a) and treated zirconia surfaces (b). The treatment was the application of the hot etching solution proposed by Casucci and others[22] that caused surface modifications.*

because of the hazardous materials involved. An example is the Piranha solution (mixture of sulfuric acid and hydrogen peroxide) that was able to improve the hydroxylation and bond strength to adhesive monomers. But more intriguing than that was the use of 40% HF to improve bond strength.[37]

Table 1 lists some of the most recent studies about surface treatments of zirconia for improved bonding and their long-term performance.

The treatments inflicted on the ceramic surface are as diverse as the methods of aging and the testing protocols. The only study in which all specimens failed before testing, after aging, was the one in which the topography was not altered and bonding was only achieved by chemical interaction.[16] A few recent protocols had not been submitted to any type of aging at the time this review was performed.

With regard to the methods of testing bond strength, the tensile test was shown to be more sensitive in detecting differences among the bonding effectiveness of several surface treatments after aging.[38]

One study was found that involved testing the load for debonding of zirconia crowns supported by teeth or composite core after thermal cycling and under tensile testing. The zirconia surfaces had been submitted to silicatization, or glazing, of the internal surface and had been cemented with various types of cement. The results showed that the surface treatment effectiveness depended on the resin cement used, as glazing improved the retention force for a 2-hydroxyethyl methacrylate–based cement.[39]

A nondestructive method to treat the ceramic surface was the deposition of the oxide-fluoride phase on the zirconium oxide surface in a plasma reactor, which extracted HF in the presence of water and facilitated the Zr-hydroxylation. This improved the reaction with the silanes and, consequently, the bond strength, although no long-term results were presented. [40]

Although this review is about bond strengths to zirconia and their durability, it is important to recall that several studies observed that some surface treatments can affect the mechanical resistance of zirconia-based ceramics. Guazzato and others [41] investigated the influence of sandblasting, grinding, grinding orientation, and polishing on the flexural strength of zirconia ceramics. They observed that sandblasting and grinding increased the strength of dental zirconia. The increase in fracture strength after air abrasion was attributed to the residual compressive stress layer that promotes the transformation from the tetragonal to the monoclinic phase.[42,43] On the other hand, other studies [44,45] reported a decrease in strength as a result of surface damage after air abrasion.

Moreover, air abrasion leads to the tetragonal and then to the monoclinic phase change on the surface of zirconia that in the long term can be detrimental to the restoration, not only because of the defects it

Table 1: *Studies and the Best Outcomes of Surface Treatments for Zirconia Bonding, According to the Aging Condition*

Authors	Surface Treatment	Aging Method
Aboushelib et al. 2007[30]	SIE	1, 2, 3, and 4 wk of water storage and thermal cycling
Aboushelib et al. 2010[20]	SIE	10,000 thermal cycles, 4 wk or 26 wk of water storage
Aboushelib 2011[19]	SIE	4, 26, 52, and 104 wk of water storage and thermal cycling
Akgungor et al. 2008[47]	Air abrasion + MDP-containing primer/silane coupling agent mixture	24 h and 150 d with thermal cycling
May et al. 2010[13]	Airborne particle abrasion + MDP primer/silane coupling agent	24 and 90 d of water storage and thermal cycling
Passos et al. 2010[14]	Silica coating and silanization	90 d of water storage and 12,000 thermal cycles
Inokoshi et al. 2013[25]	Silica coating + MDP primer + MDP cement	Immersion in 37°C water for 10 d, 10,000 thermal cycles; or immersion in 37°C 6 mo water storage
Attia and Kern 2011[26]	Universal primer containing MDP	3 d without thermal cycling and 150 d with 37,500 thermal cycles
Ozcan et al. 2008[16]	No treatments (conventional and MDP cements) resisted the aging condition	6000 thermal cycles
Yoshida et al. 2006[27]		24 h of water storage and thermal cycling
Lehmann and Kern 2009 [48]	Air abrasion and MDP primers	3 and 150 d of water storage + 37,500 thermal cycles
Nakayama et al. 2010 [49]	MDP primers	10,000 thermal cycles
Oyagüe et al. 2009 [50]	Self-adhesive cement	24 h and 6 mo of water storage
Casucci et al. 2010[23]	Experimental hot etching solution	—
Casucci et al. 2011[22]	Experimental hot etching solution	—
Foxton et al. 2011 [51]	Airborne particles abrasion + erbium laser + conventional and MDP cements	6 mo of water storage
Shimoe et al. 2012 [52]	Air abrasion at various times alloy primer + subsequent heat treatment	Thermal cycling
Komine et al. 2013 [53]	Airborne-particle abrasion at a pressure of 0.1 MPa	20,000 thermal cycles
Lung et al. 2012[36]	3-Acryloxypropyltrimethoxysilane	Dry storage, 30 d of water storage, and thermal cycling
Piascik et al. 2012 [40]	Fluorination	—
Silva et al. 2012 [54]	No treatment (MDP resin cement + heating of ceramic primer at several temperatures) resisted the aging condition	24 h of water storage and thermal cycling
Aboushelib 2012 [55]	Fusion sputtering	6 mo of water storage
Queiroz et al. 2012 [56]	MDP cement Metal primers	6000 thermal cycles
Subaşı and Inan 2014 [57]	Airborne particles abrasion, silica coating, and combination of airborne particles abrasion and laser + MDP resin cement	6000 thermal cycles
Vanderlei et al. 2014[33]	Spray application of low-fusing porcelain glaze followed by etching with HF followed by tribochemical silica coating and brush application of low-fusing porcelain glaze + etching with HF + tribochemical silica coating	150 d of water storage and 12,000 thermal cycles
De Sá Barbosa et al. 2013 [58]	Self-adhesive cement	24 h and 1 y of water storage
Lung et al. 2013[59]	Sandblasting	6000 thermal cycles
Ha et al. 2013[21]	Silane heating	—

Abbreviations: HF, hydrofluoric acid; MDP, 10-methacryloxydecyl dihydrogen phosphate; SIE, selective infiltration etching.

creates [45] but also because of the low temperature degradation suffered by zirconia. [46] Thus, zirconia air abrasion with Al_2O_3 particles, large particles (>110 μm), and under high pressures (>3 bar) should be avoided, and an effective chemical component should be used. In the authors' search, no clinical studies about the specific effects of different surface treatments on zirconia restorations were found, signaling the urgent need for such trials.

CONCLUSIONS

Regarding the bond to zirconia, current literature states the following:

1. Silica coating of the surface via air abrasion of 30-μm particles associated to MDP-based primers or cements may result in more durable bonding;
2. SIE seems to be promising but not simple to perform;
3. The thin, low-fusing glassy porcelain application is also promising and simple to perform, even though new improvements should occur for preventing crown misfit; and
4. The methods of aging are very diverse, and the treatment efficiency may vary according to the study.

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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In Vitro Biocompatibility of Contemporary Bulk-fill Composites

WS Toh • AUJ Yap • SY Lim

Clinical Relevance

Despite manufacturers' claims, not all bulk-fill resin-based composites are biocompatible at 4 mm thickness. Bulk-fill composites based on pre-reacted glass ionomer (PRG) technology may be less biocompatible than non-PRG materials.

SUMMARY

This study evaluated the biocompatibility of contemporary bulk-fill resin-based composites (RBCs) including PRG (pre-reacted glass ionomer) materials based on the International Organization for Standardization 10993. In addition, the effect of composite thickness on cytotoxicity was also assessed. Two standard composites, two bulk-fill PRG RBCs, and three bulk-fill non-PRG RBCs were investigated. Block-shaped specimens of 2-mm and 4-mm thickness were cured with an irradiance of 700 mW/cm^2 for 20 seconds with a light-emitting diode curing light and eluted with culture medium at 37°C for 24 hours. L929 mouse fibroblasts were exposed to extracts at varying dilutions (1:1, 1:2, and 1:10) for 24 hours. Analyses were performed to assess cytotoxicity, phase contrast microscopy, and quantitative cell viability. Among the bulk-fill RBCs, extracts of PRG materials resulted in the lowest cell viability. At 4-mm thickness, undiluted extracts of bulk-fill non-PRG RBCs had significantly higher cell viability than the standard composites. Chemical composition, specimen thickness, and testing concentrations of extracts had significant effects on cell viability and morphology. Cytotoxic effects of composites on cell viability were parallel with cell morphologic changes. Not all bulk-fill RBCs demonstrated high cell viability (>70%) at 4-mm thickness despite manufacturers' recommendations of bulk placement and curing.

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INTRODUCTION

Amalgam has been the traditional material for restoring posterior teeth because of its effectiveness and cost.[1] In recent years, the use of amalgam has declined because of increased patient esthetic demands, fear of mercury toxicity, and environmental concerns after disposal. Resin-based composites (RBCs) are an esthetic alternative to amalgam. RBCs, however, have several disadvantages, including technique sensitivity, polymerization shrinkage, limited depth of cure, and lower physicomechanical properties compared with amalgam. The aforementioned may account for the higher failure rates and secondary caries associated with RBCs compared

with amalgam.[1] RBCs are usually placed in increments of less than 2 mm to ensure adequate light curing and reduce polymerization shrinkage.[2] Several manufacturers have introduced innovative bulk-fill RBCs that can be placed in a single increment to reduce the time and effort required for layering and adapting posterior composites.

An ideal bulk-fill RBC should have low polymerization shrinkage, high degree of conversion, superior depths of cure, and ample mechanical properties, and it should be biocompatible.[3-7] Through the use of novel proprietary resins, special modulators, unique fillers, and filler control, bulk-fill RBCs are said to have lower polymerization shrinkage and depths of cure up to 4 mm. At such cavity depths, curing light penetration may be compromised, thus leading to reduced monomer to polymer conversion, leaching of unreacted monomers, and biocompatibility issues. The latter may be more problematic with low viscosity flowable bulk-fill materials in view of their higher resin content.

Biocompatibility is the ability of materials to coexist with living tissues without causing harm. Nonbiocompatible or cytotoxic (ie, toxic to cells) restorative materials can cause short-term and long-term adverse tissue reactions ranging from postoperative sensitivity to irreversible pulp damage.[8] RBCs alone contributed to more than 12% of adverse reactions to dental materials.[9] In addition to the leaching of unreacted monomers, cytotoxicity can also be caused by the release of initiators and other additives from the organic resin as well as metal ions from the inorganic fillers. Proper curing of RBCs is important to ensure adequate mechanical properties and biocompatibility.[10,11]

Studies investigating the cytotoxicity of bulk-fill RBCs are still limited, and none have performed cytotoxicity testing of the recently launched bulk-fill PRG pre-reacted glass ionomer (PRG) RBCs. The latter, also known as giomers, are based on PRG technology in which acid-reactive fluoride containing glass is reacted with polyacids in the presence of water, freeze-dried, milled, silanized, ground, and used as fillers. In addition to fluoride release and demineralization inhibition, these materials also possess antiplaque formation properties.[12-15] Bulk-fill PRG RBCs are available in both regular (Beautifil bulk restorative [BBR], Shofu, Kyoto, Japan) and low (Beautifil bulk flowable [BBF], Shofu) viscosities. Recent studies have indicated cytotoxicity of fluoride to tissues by multiple mechanisms, including inhibition of enzyme activity, generation of reactive oxygen species (ROS), impairment of the antioxidant defense system, induction of inflammation, and apoptosis.[16,17] Kanjevac and others[18] have also reported positive correlation of cytotoxicity of glass ionomer cements with their fluoride release.

The objective of this study was to evaluate the biocompatibility of contemporary bulk-fill RBCs, including PRG materials based on the International Organization of Standardization (ISO) 10993.[19-21] The effect of material thickness (2 mm versus 4 mm) and extract dilutions were also investigated.

METHODS AND MATERIALS

Materials selected for this study included two standard composites (Filtek Z350 XT universal restorative [ZFR] and Filtek Z350 XT universal flowable [ZFF], 3M ESPE, St Paul, MN, USA), two bulk-fill PRG RBCs (BBR and BBF), three bulk-fill non-PRG RBCs (Smart Dentin Replacement bulk-fill flowable [SDR], Dentsply Caulk, Milford, DE, USA), EverX posterior (EXP) (GC Europe, Lueven, Belgium), and Tetric N-Ceram bulk-fill (TNC) (Ivoclar Vivadent, Schaan, Liechtenstein). The technical profiles and composition of the materials are shown in Table 1. L-929 mouse fibroblasts were purchased from American Type Culture Collection (Manassas, VA, USA). Dulbecco's modified Eagle's medium (DMEM) high glucose and fetal bovine serum (FBS) were acquired from Biowest SAS (Nuaillé, France), 0.05% trypsin/ethylenediaminetetraacetic acid and penicillin/streptomycin (P/S) were obtained from Life Technologies (Singapore), and an MTS [3-(4,5-dimethylthiazol-2-yl)-5-(3-carboxymethoxyphenyl)-2-(4-sulfophenyl)-2H-tetrazolium] cell viability assay kit was procured from Promega (Madison, WI, USA).

A custom-made black polyvinyl mold with a recess 4 mm long by 4 mm wide and variable depths of 2 mm and 4 mm was fabricated for specimen preparation. Four specimens per material were fabricated for each bulk-fill and standard RBC (n=4 per group). The mold was filled using a single increment, and excess material was removed by compressing the mold between two glass slides (1-mm thick). The specimens were then cured for 20 seconds through the glass slide using a BlueShot light-emitting diode (LED) curing light (Shofu) with an irradiance of 700 mW/cm^2 and an exit window of 8-mm diameter. Before each use, the intensity of the curing light was verified using an LED radiometer (Demetron LED radiometer; Kerr Corporation, Middleton, WI, USA). The cured specimens were sterilized by swabbing briefly with 70% ethanol before they were immersed in cell culture medium (DMEM-high glucose supple-

Table 1: *Composite Materials and Their Composition*

Material	Abbreviation	Shade	Matrix Composition	Filler % by Weight	Recommended Thickness (mm)	Recommended Curing Time and Light Intensity
Filtek Z350 XT universal restorative	ZFR	A2	Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA resin, silica filler, zirconia/silica cluster filler	78.5	2	20 s $\geq$400 mW/cm^2
Filtek Z350 XT flowable restorative	ZFF	A2	Bis-GMA, TEGDMA, procrylat resins, ytterbium trifluoride filler, silica filler, zirconia/silica cluster filler	65	2	20 s 400-1000 mW/cm^2 10 s 1000-2000 mW/cm^2
Beautifil bulk restorative	BBR	A	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, S-PRG filler based on fluoroboroaluminosilicate glass, polymerization initiator, pigments, and others	87	4	10 s $\geq$1000 mW/cm^2
Beautifil bulk flowable	BBF	Universal	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, S-PRG filler based on fluoroboroaluminosilicate glass, polymerization initiator, pigments, and others	73	4	10 s $\geq$1000 mW/cm^2
SDR posterior bulk-fill flowable base	SDR	Universal	Barium-alumino-fluoro-borosilicate glass, strontium alumino-fluoro-silcate glass, modified UDMA resin, EBPADMA, TEGDMA, camphorquinone photoinitiator, photoaccelerator, BHT, ultraviolet stabilizer, titanium dioxide, iron oxide pigments, fluorescing agent	68	4	20 s $\geq$550 mW/cm^2
EverX Posterior	EXP	Universal	Bis-GMA, TEGDMA, silicon dioxide, barium glass, glass fiber, polymethylmethacrylate, photo initiator	74.2	4	20 s $\geq$700 mW/cm^2 10 s $>$1200 mW/cm^2
Tetric N-Ceram Bulk-Fill	TNC	IVA (Universal)	Dimethacrylates, barium glass, ytterbium trifluoride, mixed oxide, additives, catalysts, stabilizers, and pigments	75-77	4	20 s$\geq$500 mW/cm^2 10 s $\geq$1000 mW/cm^2)

Abbreviations: BHT, butylated hydroxyl toluene; Bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA, bisphenol A glycidyl methacrylate; Bis-MPEEP, 2,2,bis (4-methacryloxypolyethoxyphenyl) propane; EBPADMA, ethoxylated bisphenol A dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate; S-PRG, surface pre-reacted glass ionomer; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

mented 10% FBS and 1% P/S) and incubated at 37°C in a humidified atmosphere of 95% air and 5% CO_2 for 24 hours. The extracts of the composite specimens were prepared at a surface area to volume ratio of 3 cm^2/mL culture medium following the guidelines of ISO10993.[19-21] All the composite specimens were prepared and extracted in culture medium within the same day. After incubation, the original extracts (1:1) were collected and then diluted in cell culture medium to obtain 1:2 and 1:10 dilutions before further testing. At least two independent experiments were performed for each test material.

The L929 mouse fibroblasts were seeded at a density of 2×10^4 cells per well in 96-well plates at 37°C in 5% CO_2 for 24 hours. The cells were allowed to attach for at least 15-18 hours before exposure to the composite extracts. The plating medium was removed and 100 μL of the extracts were added into each well for further incubation at 37°C for 24 hours. After incubation, cell morphologic changes

Table 1: *Composite Materials and Their Composition (ext.)*

Manufacturer	Batch Number
3M ESPE (St Paul, MN, USA)	N454576
3M ESPE (St Paul, MN, USA)	N452481
Shofu Inv (Kyoto, Japan)	51441
Shofu Inc (Kyoto, Japan)	71404
Dentsply Caulk (Milford, DE, USA)	1402000759
GC Europe (Lueven, Belgium)	1311301
Ivoclar Vivadent (Schaan, Liechtenstein)	R65894

were observed by phase contrast microscopy (DMI3000B, Leica, Wetzlar, Germany). Cell viability was assessed by MTS assay following the manufacturer's protocol. Briefly, 20 μL of MTS reagent was added to each well. The cells were incubated at 37°C in CO_2 for 2 hours, and the absorbance readings were taken at 490 nm and 650 nm (reference) using an Infinite 2000 plate reader (Tecan, Männedorf, Switzerland). Untreated cultures without any material served as a negative control. Percentage cell viability was calculated by normalization of the absorbance readings against that of the negative control (set as 100%).

Cytotoxicity testing of the materials was performed in quadruplicates (n=4 per material) and at least two independent experiments were performed to confirm the results. Cell viability data was analyzed using one-way analysis of variance and Scheffe's *post hoc* testing at a significance level of 0.05. The StatView software version 5.0 (SAS Institute Inc, Cary, NC, USA) was used to perform the statistical analysis.

Table 2: *Statistical Analysis of Cytotoxicity of Various Composite Materials* [a]

Dilution	Thickness (mm)	Cytotoxicity (From Most to Least Cytotoxic)
1:1	2	BBF > BBR, ZFR, TNC, ZFF, SDR, EXP
		BBR > ZFR, TNC, ZFF, SDR, EXP
		ZFR > SDR, EXP
		TNC > EXP
	4	BBR = BBF > ZFF, ZFR, TNC, EXP, SDR
		ZFF = ZFR > TNC, EXP, SDR
		TNC > SDR
		EXP > SDR
1:2	2	BBF > ZFR, BBR, SDR, ZFF, TNC, EXP
		ZFR > EXP
		BBR > EXP
	4	BBF > BBR, ZFR, ZFF, SDR, EXP, TNC
		BBR > ZFR, ZFF, SDR, EXP, TNC
		ZFR = ZFF > SDR, EXP, TNC
1:10	2	BBF = TNC = SDR = ZFR = EXP = BBR = ZFF
	4	BBF > BBR, SDR, ZFR, TNC, ZFF, EXP

Abbreviations: BBF, Beautifil bulk flowable; BBR, Beautifil bulk restorative; EXP, EverX Posterior; SDR, SDR posterior bulk-fill flowable base; TNC, Tetric N-Ceram bulk-fill; ZFR, Filtek Z350 XT universal restorative; ZFF, Filtek Z350 XT flowable restorative.

[a] *Results of one-way analysis of variance/Scheffe's* post hoc *($p<0.05$). > indicates statistical significance.*

RESULTS

Cell Morphology Analysis

Untreated L929 cells (negative control) were spindle shaped in appearance with extended cellular processes, filopodi, and lamellipodia (Figure 1). Varying degrees of morphologic alterations were observed with the various bulk-fill RBCs and standard composites, depending on chemical composition, specimen thickness, and extract concentrations (Figure 1). Undiluted extracts of ZFR and ZFF cured at 2-mm thickness had no obvious effect on the cell morphology compared with the negative control (Figure 1A). However, at 4-mm thickness, undiluted

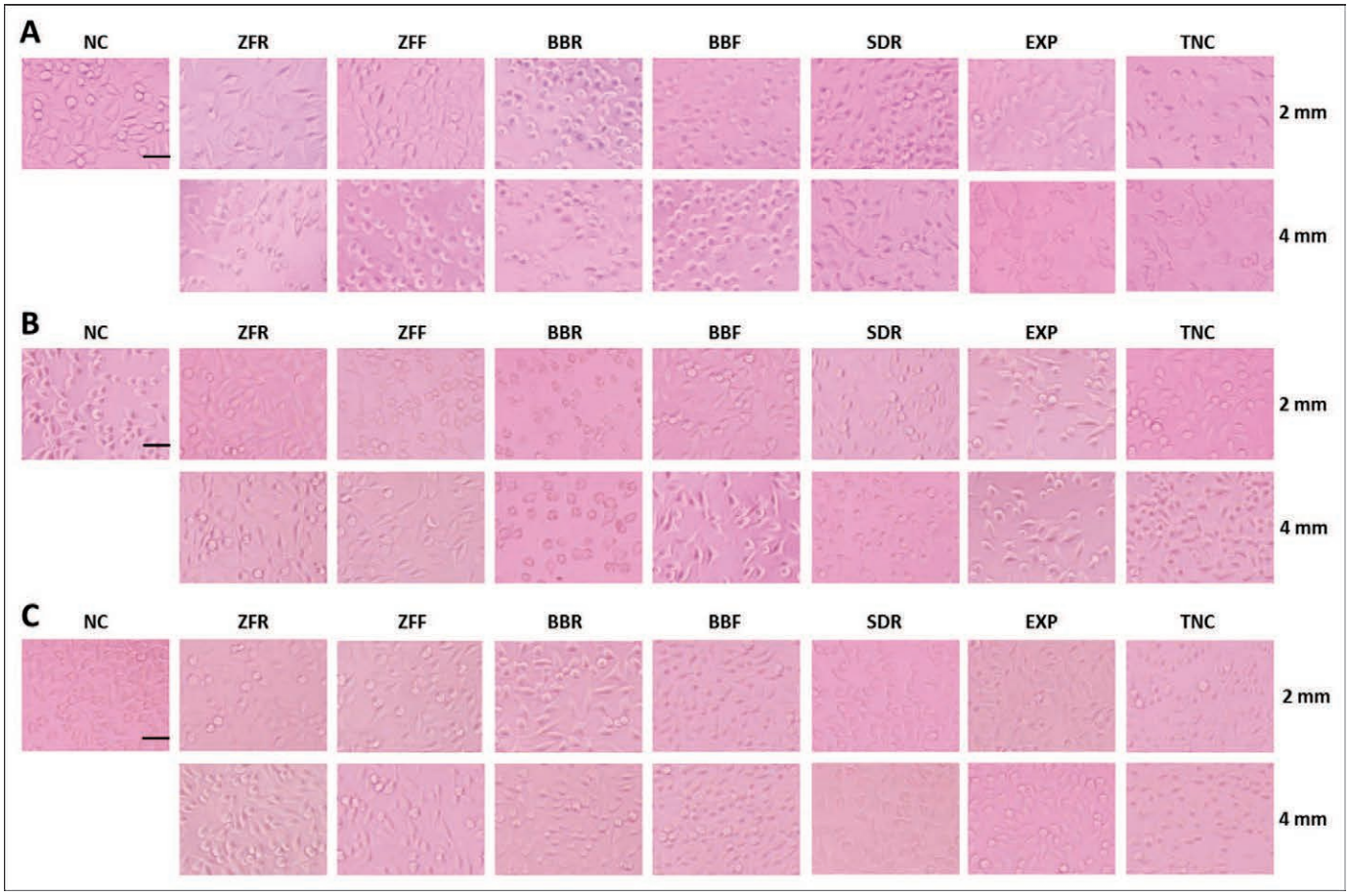

Figure 1. *Cellular morphology of L929 mouse fibroblasts after treatment with extracts of various resin composites. The cell cultures were exposed to (A): original extracts (1:1), (B): 1:2 diluted extracts, and (C): 1:10 diluted extracts at 37 °C for 24 hours, and cell morphology of treated and untreated cell cultures (negative control, NC) were observed under phase contrast microscopy. Representative images from four replicate cultures (n=4) were presented. Scale bar = 100 μm.*

extracts of ZFR and ZFF resulted in significant retraction and rounding of the cells.

Among the bulk-fill RBCs, undiluted extracts of bulk-fill PRG RBCs (BBR and BBF) at both 2-mm and 4-mm thickness caused the majority of cells to become small, retracted, and rounded, with condensed and fragmented nuclei morphology. The effects of BBR and BBF on cell morphology were concentration dependent (Figures 1B,C). Accordingly, there was a decrease in the number of retracted round cells; most cells remained spread out and spindle shaped when 1:10 diluted extracts of BBR and BBF were applied. Furthermore, non-PRG bulk-fill RBCs, including SDR, EXP and TNC, had little effects on the morphology of L929 cells; approximately 80% of cells remained spindle shaped when treated with the undiluted extracts. When the specimen thickness of these materials was increased to 4 mm, no significant effect on cell morphology was also observed.

Cell Viability Analysis

Varying levels of cell viability detected with extracts of the RBCs were normalized against the untreated cells set as the negative control at 100%. Differential cytotoxic effects were observed with the various bulk-fill RBCs (PRG and non-PRG) and the standard composites. Results were again dependent on chemical composition, specimen thickness, and extract concentrations (Table 2 and Figure 2). Undiluted (1:1) extracts of ZFR and ZFF at 2-mm thickness significantly reduced the cell viability to 66% ($p<0.0001$) and 79% ($p<0.05$), respectively (Figure 2a). Cell viability was further reduced to 36% and 28%, respectively, when specimen thickness of ZFR and ZFF was increased to 4 mm ($p<0.0001$).

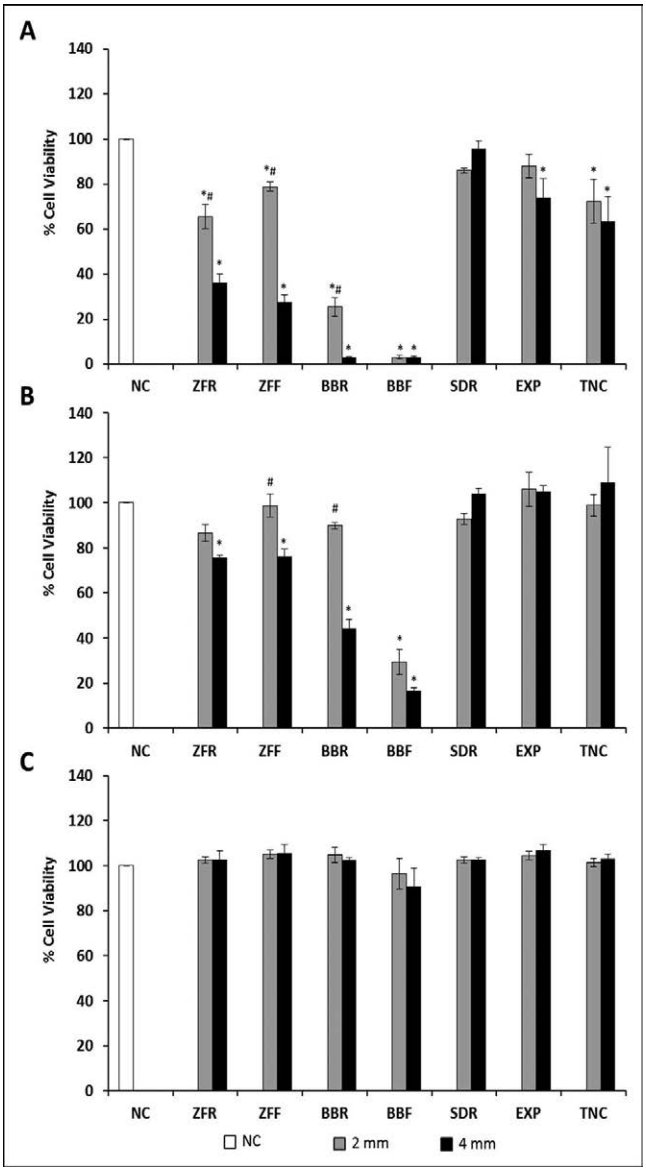

Figure 2. *Cellular viability of L929 mouse fibroblasts after treatment with extracts of various resin composites. The cell cultures were exposed to (A): original extracts (1:1), (B):1:2 diluted extracts, and (C): 1:10 diluted extracts at 37 °C for 24 hours, and cellular viability of treated and untreated cell cultures (negative control, NC) were determined by MTS assay in four replicate cultures (n=4). Results are normalized to the mean values of the negative control and presented as mean (± SD) percentage cell viability. * indicates significant decrease compared with the negative control; # indicates statistically significant differences between cell viability values of 2-mm and 4-mm composite extracts (analysis of variance, Scheffe's* post hoc *test, n=4).*

For the bulk-fill RBCs, undiluted extracts of bulk-fill PRG RBCs (BBR and BBF) at 2-mm thickness resulted in drastic decline of cell viability to below 30% ($p<0.0001$). Cell viability was further reduced to less than 5% when the cells were treated with BBR and BBF at 4-mm thickness ($p<0.0001$; Figure 2A). At 2-mm and 4-mm increments, BBR and BBF were significantly more cytotoxic than the non-PRG bulk-fill RBCs ($p<0.0001$) and standard RBCs ($p<0.01$; Figure 2A). Undiluted extracts of non-PRG bulk-fills, including SDR, EXP, and TNC, at 2-mm thickness reduced cell viability to 86% ($p\geq0.54$), 88% ($p\geq0.79$), and 72% ($p<0.001$), respectively. When the specimen thickness of these materials was increased from 2 mm to 4 mm, no significant difference in cell viability was observed ($p>0.05$). Ranking of material cytotoxicity (from most to least cytotoxic) based on the cell viability of L929 after 24-hour exposures to undiluted extracts were as follows: BBF, BBR, ZFR, TNC, ZFF, SDR, EXP (2-mm thickness) and BBR, BBF, ZFF, ZFR, TNC, EXP, SDR (4-mm thickness).

DISCUSSION

We compared the cytotoxicity of five bulk-fill RBCs and two standard composites using L929 mouse fibroblast cell line as specified in the ISO guidelines.[19-21] The use of immortalized cell lines, in this case L929, offers the greatest advantage of low interbatch variability and accuracy of response to toxic challenge.[22] A number of cell types, including human gingival fibroblasts,[23] periodontal ligament fibroblasts,[23,24] and dental pulp stem cells,[25] have been proposed for dental materials testing as these cell types are present in the oral tissues. The L929 mouse fibroblast cell line, however, remains to date one of the most common cell lines for cytotoxicity evaluation of dental materials.[22,26,27]

With the exception of bulk-fill PRG RBCs (BBR and BBF), bulk-fill RBCs generally have comparable or higher cell viability than the standard composites. Significantly lower cell viability was observed for RBCs cured at 4-mm thickness compared with 2-mm thickness, with the exception of SDR, EXP, and TNC at 1:1 and 1:2 dilutions. The differential cytotoxic effects observed in ZFR and ZFF at 2-mm and 4-mm thickness implied inadequate curing of these RBCs at 4 mm, resulting in the release of leachable toxic monomers. For the bulk-fill PRG RBCs (BBR and BBF), the differential cytotoxic effects between 2-mm and 4-mm specimens may be attributed in part to greater fluoride and other ion release in addition to the degree of conversion.[12,13] The degree of conversion for 4-mm-thick BBR and BBF specimens might also be compromised by the intensity of the LED curing light used. The manufacturer recommends an irradiance of 1000 mW/cm^2 for 10 seconds for both materials. Although the total LED (intensity×time) used in the study was greater than that

endorsed by the manufacturer, the intensity may not be sufficient to penetrate to 4-mm depths. The effects of RBCs on cell viability were paralleled by alterations of cell morphology. This was most evident in cells treated with BBR and BBF. In this instance, the cells demonstrated a transition from spindle shaped to a retracted and condensed morphology in a dose-dependent manner. The condensed nuclei morphology observed here is likely indicative of apoptosis, although further confirmative assay would be required.

BBF was found to be more cytotoxic than BBR. This was not unexpected as flowable materials are made less viscous by reducing filler content and adding diluents that have been shown to result in a more persistent mass leaching at toxic levels.[28] When the non-PRG materials were compared, the difference between regular (TNC and EXP) and low viscosity (SDR) products was also evident for 4-mm-thick specimens. Extracts of BBR and BBF induced morphologic alterations and cytotoxicity in a dose-dependent manner, linking cell morphologic changes with the cell viability. The cytotoxic effects of bulk-fill PRG RBCs (BBR and BBF) can be attributed to the release of fluoride and other ions, in addition to such monomers as triethylene glycol dimethacrylate (TEGDMA) and bisphenol A diglycidyl ether dimethacrylate (Bis-GMA), which are also present in other RBCs. Supplementary ions released from PRG fillers include aluminum, boron, sodium, silicon, strontium, and zinc[29,30] and are constituents of the fluoroaluminosilicate glass utilized in the PRG technology. Inhibition of enzyme activity, generation of ROS, impairment of the antioxidant defense system, induction of inflammation, and apoptosis by low concentrations of fluoride in L929 mouse fibroblasts,[31] human dental pulp stem cells,[18] and human gingival fibroblasts[32] have been reported. The correlation between concentration of fluoride ion released and cytotoxic response is shown to be high, positive, and significant.[18,33] The other bulk-fill RBCs (SDR, EXP, and TNC) also incorporate fluoride-containing glasses into their formulations (Table 1). Fluoride release with PRG-filled materials is, however, higher than for materials with fluoroaluminosilicate glass in the unreacted form.[34] The fluoride-containing glass must be reacted with polyacids for effective fluoride release. This explains in part the better biocompatibility of PRG RBCs compared with conventional and ceramic-reinforced glass ionomer cements.[27]

Undiluted extracts of SDR, EXP, and TNC at 4-mm thickness resulted in significantly higher cell viability (>60%) than that of the standard composites (ZFR and ZFF). The slight reduction in cell viability is most likely due to release of TEGDMA or Bis-GMA inducing cytotoxicity by generation of ROS, as previously described.[35-37] Among the bulk-fill RBCs, only SDR and EXP demonstrated acceptable cell viability (>70%) at 4-mm thickness based on the ISO cutoff of 70% cell viability.[20] Findings supported manufacturers' claims of curing at 4-mm thickness without causing cytotoxicity. It is important to note that not all bulk-fill RBCs can be adequately cured at 4-mm thickness.[38] Cell viability increases with extraction dilution. At 1:10 dilution, all RBCs, with the exception of BBR, showed almost 100% cell viability. Clinically, physical and mechanical properties, including polymerization shrinkage, must be considered in addition to biocompatibility. Flowable bulk-fill RBCs have been shown to shrink more than nonflowable ones.[38] The shrinkage stress can result in cuspal movement and microleakage, leading to postoperative sensitivity and possible pulpal inflammation.

Our study has several limitations. First, only a single cell line and test model were used for the assessment of cytotoxicity. It is important to note that cytotoxicity testing using an established cell line, in this case L929 mouse fibroblasts, would serve only as a general and preliminary assessment. Besides the differences in cell lines and primary cells, different test models including direct and indirect contact tests would also result in variability in cytotoxic responses.[26,39] Careful selection of more clinically relevant cell types as well as appropriate testing models and methods would be the next step to confirm the cytotoxic properties of biomaterials. We are currently investigating the use of neural crest stem cells and fibroblasts derived from human embryonic stem cells as human cell–based models and employing transcriptomic and proteomic analysis for cytotoxicity and genotoxicity testing of dental composites.[31,40,41] Comprehensive chemical analyses of culture medium extracts for organic and inorganic compounds are also necessary to identify plausible cytotoxic agents. In our study, the curing light distance was standardized at 1 mm away from the composite specimens. Clinically, this may be difficult to achieve because of curing light access and the presence of a matrix band and holder. Curing light-related parameters, including light type, distance, intensity, curing modes, and penetration may also influence the cytotoxicity of bulk-fill RBCs and warrants further in-depth investigations.

CONCLUSION

We evaluated the biocompatibility of contemporary bulk-fill RBCs based on the ISO 10993. Within the limitations of our study, we conclude that chemical composition, specimen thickness, and extract concentrations from RBCs have significant effects on cell viability and morphology. The cytotoxic effects of RBCs on cell viability paralleled changes in cell morphology. Among the bulk-fill RBCs, PRG materials BBR and BBF have significantly higher cytotoxicity. This may be attributed in part to the release of fluoride and other ions associated with the use of PRG technology. Only SDR and EXP demonstrated acceptable biocompatibility at 4-mm thickness based on the ISO cutoff of 70% cell viability.

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Regulatory Statement

This study was conducted in accordance with all the regulatory provisions, guidelines and policies of the National University of Singapore.

Conflict of Interest

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

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Microleakage of Class I and II Composite Resin Restorations Using a Sonic-resin Placement System

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Clinical Relevance

Bonding to intact enamel surfaces (Class I preparations) as opposed to dentin (cementum) surfaces appears to show superior results regardless of the material, C-factor, or insertion technique.

SUMMARY

Objectives: To determine microleakage of posterior Class I and II restorations using the SonicFill composite resin system.

Methods and Materials: Eighty previously extracted third molars were randomly assigned to four preparation/restoration groups (n=20): Group A: Class I preparations restored with SonicFill system/bulk fill; Group B: Class II preparations restored with SonicFill system/bulk fill; Group C: Class I preparations restored with Herculite Ultra composite resin/incremental technique; and Group D: Class II preparations restored with Herculite Ultra composite resin/incremental technique. Class I preparations were approximately 3.0 mm in width buccolingually and 3.0 mm in depth. Class II preparations were approximately 3.0 mm in width buccolingually, 1.5 mm in axial depth, and 4.0 mm in gingival depth. In all groups, the enamel and dentin surfaces were conditioned with Kerr 37.5% phosphoric acid, followed by application of Optibond Solo Plus adhesive system. Following restoration, the specimens were thermocycled, immersed in methylene blue dye, and embedded in acrylic resin. Specimen blocks were sectioned in the mesiodistal direction, with marginal dye pen-

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etration (microleakage) examined using a 20× binocular microscope. Class I and II restoration microleakage was scored separately using a 0-3 ordinal ranking system. Statistical analyses were conducted using nonparametric testing at the $p < 0.05$ level of significance.

Results: Significantly less microleakage was associated with both Class I restorative groups (A and C), SonicFill bulk fill and Herculite Ultra incremental fill, compared to the Class II restorative groups (B and D), SonicFill/bulk fill and Herculite Ultra/incremental fill.

Conclusions: According to the results of this study, the materials (SonicFill vs Herculite Ultra), C-factors, and insertion techniques (bulk vs incremental) did not appear to be significant influences with regard to marginal microleakage; however, the type of preparation cavity (Class I vs Class II) and the subsequent bonding surface (enamel vs dentin [cementum]) proved to be significant factors.

INTRODUCTION

One of the purposes of a dental restoration is to create a flawless seal at the material/tooth interface (margin), which should be resistant to contamination from oral fluids.[1,2] Composite resin has been increasingly utilized for the restoration of posterior cavity preparations in educational and private practice dental settings, as innovations regarding material sciences and preparation and insertion techniques have emerged.[3-5]

Leakage or microleakage occurs in conjunction with all dental restorations and has been defined as the "clinically undetectable passage of bacteria, fluids, molecules, or ions between a cavity wall and the restorative material applied to it."[1,6-8] This potentially destructive process is associated with several limiting factors, including material component (filler particle size/shape aggregates and resin loading) and physical characteristic variables (volumetric and postpolymerization shrinkage, modulus of elasticity, and thermal expansion); methods of polymerization; cavity preparation design parameters, and configuration factor, or "C-factor." Clinical variables include material manipulation and insertion techniques, isolation constraints (usage of dental rubber dam), and adequate knowledge of adhesive and composite resin science and applicable technique.[9-16] Composite resin, used as a posterior restorative, is significantly influenced by polymerization shrinkage with amounts ranging from 1.5% to 5%.[11] Shrinkage causes debonding of the material from tooth structure, precipitating clinical and radiographic sequelae including marginal staining and micro–gap formation (approximately 10 to 20 μm). This process, in turn, permits bacterial ingress with recurrent caries, sensitivity, possible pulpal inflammation, and, finally, restoration removal and reinsertion as final outcomes.[9,11,17]

Solutions for some of these specific material and clinical dilemmas have included the introduction of "packable" and "flowable" composites—allowing for enhanced adaptability of overlying materials; the usage of different insertion techniques (incremental vs bulk); and different restoration delivery methods (thermal and/or sonic energy).[15-23] However, many of these restorative solutions have shown conflicting results with regard to the effects on material shrinkage and marginal microleakage of contaminants.[15-23]

Recently, a composite delivery system together with a proprietary composite formulation has been introduced to the market to compensate for the misgivings (physical, material, and clinical) of composite resin science technology.[23-26] This system, SonicFill, a posterior restorative system, has been developed as a "bulk fill" (up to 5.0 mm) composite resin. SonicFill technology uses sonic energy, causing viscosity changes of the composite resin formulation for reportedly initial increased flowability and increased depths of cure.[23-27]

The specific aim of the present *in vitro* research project was to test both Class I and II posterior restorations using two composite resin systems, SonicFill and Herculite Ultra, considering the effects of material qualities, insertion techniques, "C-factors," and cavity classifications (bonding surfaces) on marginal microleakage. The hypothesis of the study was that the SonicFill system would show significantly less microleakage compared to Herculite Ultra when using the same experimental protocol.

METHODS AND MATERIALS

Tooth Selection and Study Preparation

Eighty previously extracted maxillary and mandibular third molars of similar size were selected for the present study. The teeth were cleaned of calculus, soft tissue, and other debris and were stored in a 1% chloramine T solution (Fisher Chemical, Fair Lawn, NJ, USA) consisting of 12% active chlorine diluted in tap water at room temperature. This study protocol, involving human research specimens (extracted teeth), was submitted to and approved by the

University of Tennessee Health Sciences Center Institutional Review Board for "Exempt" status review prior to study commencement. All teeth were examined macroscopically and microscopically (20×) to rule out the presence of fractures/fissures, carious lesions, abrasive/erosive lesions, and restorations. Teeth that did not conform to the inclusionary criteria were discarded. The teeth were then divided into four groups of 20 (n=20) and stored in tap water immediately prior to treatment.

Cavity Preparation

Experimental groups were based upon 1) restorative system—SonicFill (Kerr/Kavo, Bismarck, Biberach, Germany) and Herculite Ultra (Kerr Corporation, Orange, CA, USA); 2) class of cavity preparation (Class I or II) and cavity C-factor; and 3) insertion technique (bulk vs incremental). Class I preparations measured approximately 3.0 mm buccolingually and 3.0 mm in depth for each molar, while Class II preparations (extending the length of the tooth, mesiodistally) measured approximately 3.0 mm wide buccolingually and 1.5 mm in axial depth. The gingival floor was measured, 4.0 mm gingivally (depth), located slightly occlusal and/or apical in proximity to the cementoenamel junction (CEJ), depending upon the proximal surface anatomy of each tooth. All internal line angles were rounded, as appropriate for composite resin preparation design. The dimensions of the cavities were verified with a periodontal probe. All preparations were cut using a #245 tungsten carbide bur (Henry Schein, Melville, NY, USA), with margins beveled using a #368 finishing bur (Henry Schein), in a water-cooled, high-speed air turbine handpiece (Henry Schein). A new bur was used for every cavity preparation. One operator performed all cavity preparations, while another investigator verified the preparation parameters prior to restoration, to ensure accuracy and continuity. The teeth were then randomly divided into four groups based upon restorative system, insertion technique, and class of cavity.

Study Groups and Restorative Procedures

Group A—SonicFill Bulk Insertion, Class I preparations (20 teeth): SonicFill composite resin, shade A3, was used to restore each Class I preparation. The enamel and dentin surfaces were conditioned using 37.5% phosphoric acid etchant gel (Kerr Corporation) for 15 seconds, then rinsed and dried with an air/water syringe for 10-15 seconds. OptiBond Solo Plus (Kerr Corporation) adhesive agent was applied to all enamel/dentin surfaces for 15 seconds (air-dried) and light polymerized for 10 seconds. SonicFill composite resin was inserted into the preparation in one bulk increment, followed by light polymerization for 20 seconds.

Group B—SonicFill System/Bulk Insertion, Class II preparation (20 teeth): SonicFill composite resin, shade A3, was used to restore each Class II preparation. The enamel and dentin surfaces/margins were conditioned using Kerr 37.5% phosphoric acid etchant gel for 15 seconds, then rinsed and dried with an air/water syringe for 10-15 seconds. OptiBond Solo Plus adhesive agent was applied to all enamel/dentin surfaces for 15 seconds (air-dried) and light polymerized for 10 seconds. SonicFill composite resin was inserted into the preparation using one bulk increment, followed by light polymerization for 20 seconds.

Group C—Herculite Ultra Composite resin/Incremental Insertion, Class I preparation (20 teeth): Herculite Ultra composite resin, shade A3, was used to restore each Class I preparation. The enamel and dentin margins were conditioned using Kerr 37.5% phosphoric acid etchant gel for 15 seconds, then rinsed and dried with an air/water syringe for 10-15 seconds. OptiBond Solo Plus adhesive agent was applied to all enamel/dentin surfaces for 15 seconds (air-dried) and light polymerized for 10 seconds. Herculite Ultra was inserted into each preparation in three incremental layers (2 = base, 1 = anatomical). Each increment of the restoration was light polymerized for 20 seconds.

Group D—Herculite Ultra Composite resin/Incremental Insertion, Class II preparation (20 teeth): Herculite Ultra composite resin, shade A3, was used to restore each Class II preparation. The enamel and dentin margins were conditioned using Kerr 37.5% phosphoric acid etchant gel for 15 seconds, then rinsed and dried with an air/water syringe for 10-15 seconds. OptiBond Solo Plus adhesive agent was applied to all enamel/dentin surfaces for 15 seconds (air-dried) and light polymerized for 10 seconds. Herculite Ultra was inserted into each preparation in four incremental layers (2 = proximal [boxes], 2 = occlusal [anatomical]). Each increment of the restoration was light polymerized for 20 seconds.

A restoration template device, as shown in Figure 1, was utilized for insertion of the Class II composite resins. The template, modeled after a device from a study conducted by Bagis and others,[28] consisted of a quadrant of plastic typodont teeth mounted in acrylic tray material surrounded by a custom-fabricated stainless-steel frame using a separate

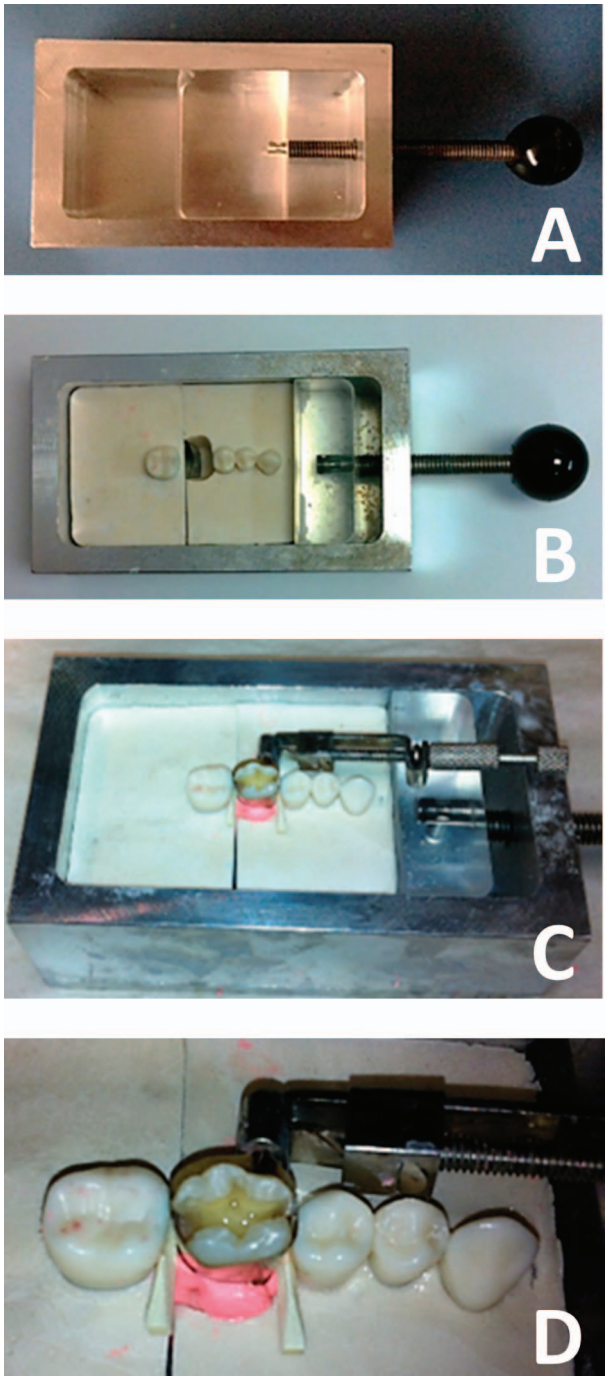

Figure 1. *Representative photographs (grouped) showing the manufactured device for insertion of the Class II composite restorations: (a)—custom-manufactured restorative device; (b)—device with plastic teeth in place and area removed for extracted tooth insertion; (c)—extracted molar with Class II preparation inserted into impression material with Tofflemire retainer and band positioned around MOD preparation; (d)—close-up photograph showing preparation ready for composite insertion, simulating a clinical patient environment.*

sliding unit with a screw/knob attached at one end of the apparatus. Each previously extracted tooth to be restored was implanted in heavy silicone-based impression material (simulating the periodontal ligament) and positioned into the acrylic mold by the sliding unit so that intimate contact was established on each proximal surface of the prepared tooth. This restoration adaptation technique was performed in order to approximate a realistic clinical situation. A Tofflemire universal metal circumferential matrix band/retainer was placed around each prepared tooth (Class II), and the respective materials were inserted according to each group's protocol and the manufacturers' instructions. One operator restored all cavity preparations, while another investigator verified the restoration parameters, to ensure accuracy and continuity.

All restorative materials were polymerized with a Schein (Henry Schein) quartz-tungsten halogen light. The light had been previously monitored with a radiometer and displayed adequate intensity ($\geq$800 mW/cm^2) levels. The composites were finished and polished using Schein (Henry Schein) finishing diamonds followed by Occlubrush (Kerr Corporation) polishing brushes. The specimens were stored in tap water at room temperature for seven days prior to leakage assessment.

Thermocycling and Assessment of Dye Penetration (Microleakage Scoring)

The specimens were subjected to artificial aging by thermocycling for 1000 cycles in separate water baths of 5°C and 55°C, with a dwell time of 60 seconds in each bath and a transfer time of three seconds. The root apices were sealed with utility wax, and the entire tooth surface was coated with two layers of commercial nail varnish to within 1.0 mm of the restoration. The specimens were immersed in a 1% aqueous solution of methylene blue dye for eight hours at room temperature, followed by thorough rinsing to remove any excess dye. The specimens were invested in clear autopolymerizing resin (Castin' Craft, Clear Plastic Casting Resin, ETI, Fields Landing, CA, USA) and labeled. A low-speed diamond saw (Buehler Isomet, Buehler Ltd, Evanston, IL, USA), cooled with water, was used to section each specimen block in a mesiodistal direction through the center of the restoration. Two sections were obtained from each block (20 blocks, or 40 readable surfaces per group), yielding dye penetration (microleakage) readings, examined at 20× magnification under a (Meiji EMT, Meiji-Labax Co, Tokyo, Japan) binocular microscope, with stan-

Table 1: *Table showing mean microleakage values of each experimental group.*

Group (Material + Class of Restoration)	Count, No.	Sum Ranks	Mean Ranks	Mean	Standard Deviation
A (SonicFill + I)	80	6920.000	86.500	0.000	0.000
B (SonicFill + II)	80	17,711.000	221.387	2.487	1.091
C (Herculite Ultra + I)	80	7747.000	96.838	0.175	0.671
D (Herculite Ultra + II)	80	18,982.000	237.275	2.775	0.795

dardized digital images obtained. Two observers scored each group blindly, and a consensus was reached if disagreement occurred. Microleakage scores (values) were determined based upon ordinal ranking (ranked 0-3) for each class of cavity preparation: 1) Class I: 0, no dye penetration; 1, dye penetration up to half of the restoration depth; 2, dye penetration greater than half of the restoration depth (to the pulpal floor); 3, dye penetration including the pulpal floor; 2) Class II: 0, no dye penetration; 1, dye penetration up to the full length of the gingival floor; 2, dye penetration up to half of the axial wall length; 3, dye penetration greater than half of the axial wall length.

Statistical Analysis

Microleakage values from all group specimens were statistically analyzed using Kruskal-Wallis and Mann-Whitney U-test nonparametric, multiple comparison tests. All data were submitted for statistical analysis at a predetermined value of $p < 0.05$ in terms of level of significance. The statistical calculations were performed using Statview 5.0 (SAS Institute, Cary, NC, USA).

RESULTS

All microleakage values are presented in Table 1. According to the Kruskal-Wallis testing, a significant difference ($p<0.0001$) was exhibited between the groups. Mann-Whitney U-test post hoc testing showed significant differences ($p<0.05$) between paired groupings (Table 2). All groups exhibited some degree of microleakage, except group A (SonicFill Bulk Insertion, Class I preparations) with a score of 0.000 ± 0.000. Group D (Herculite Ultra Composite resin/Incremental Insertion, Class II preparation) showed the greatest degree of microleakage (2.75 ± 0.795).

DISCUSSION

Composite resin has been rapidly replacing amalgam as the posterior restorative of choice for many dental patients.[4,5] However, the insertion of composite resin can be a very complex, challenging procedure for the dentist as a result of many material and clinical considerations.[5,9] This *in vitro* study utilized an objective means (dye penetration) of assessing marginal microleakage for posterior composite resin systems, utilizing different insertion techniques and cavity classifications (C-factors). Although the present study was conducted *in vitro*, careful attention was accorded to simulating a realistic clinical environment.

Specifically, this study wished to determine if the use of the SonicFill composite resin system (as compared to a universal, nanohybrid composite resin, Herculite Ultra) using an incremental-fill insertion technique had an effect on marginal microleakage of Class I and II cavity preparations. No study could be found that arrived at conclusions using different cavity preparation classes (C-factors) as a limiting variable.

The results attained in this study showed that the restoration of Class I and II cavity preparations using the SonicFill system did not prove to be a superior alternative for placement of posterior composite resins. The present study also revealed that neither the preparation configuration nor the "C-factor" played significant roles with regard to the microleakage incurred in the respective groups. The class of cavity preparation or, specifically, the bond to different tooth substrates (enamel, dentin [cementum]) was a more significant determinant of restoration microleakage. Groups A and C employing SonicFill and Herculite Ultra composite resin inserted into Class I cavity preparations revealed signif-

Table 2: *Table indicating statistical significance between paired groups.*

Group Pairings	*p*-Value	Statistical Significance ($p<005$)
A, B	<0.0001	S
A, C	0.1429	NS
A, D	<0.0001	S
B, C	<0.0001	S
B, D	0.0586	NS
C, D	<0.0001	S

Abbreviations: NS, not significant; S, significant.

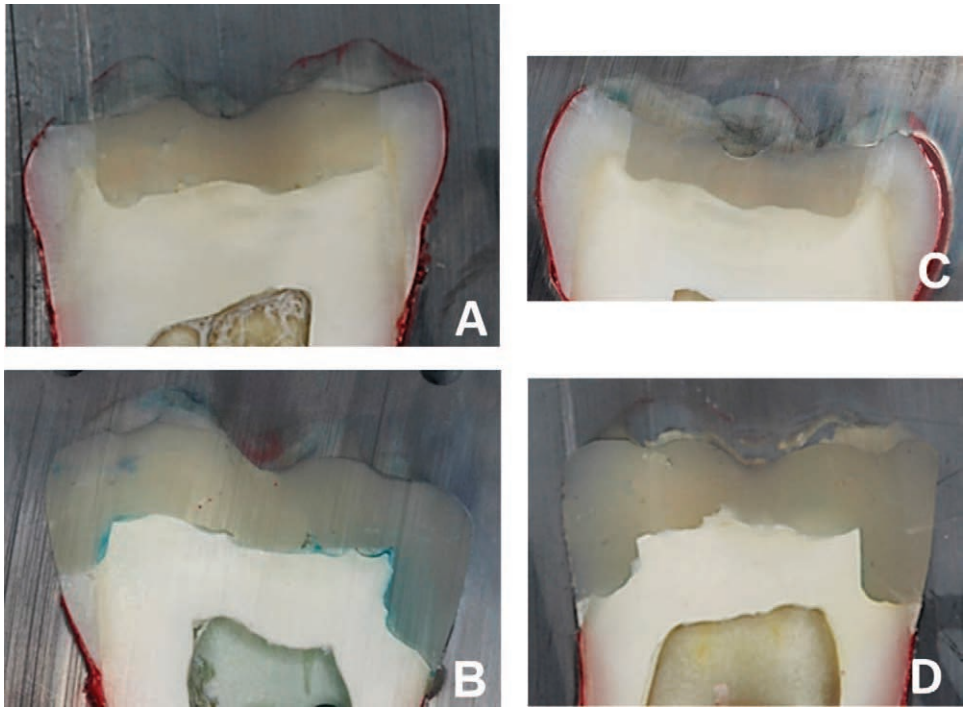

Figure 2. *Representative photographs (grouped) showing dye penetration (microleakage scores) relative to each experimental group: Group A (SonicFill™, Class I restoration) – showing no (score of "0") dye penetration (microleakage) around occlusal margins; Group B (SonicFill™, Class II restoration) – showing no (score of "0") microleakage on left (proximal) enamel surface coronal to the cementoenamel junction (CEJ) and microleakage (score of "3") on right (proximal) dentin surface apical to the CEJ; Group C (Herculite Ultra™, Class I restoration) – showing no (score of "0") microleakage around occlusal margins; Group D (Herculite Ultra™, Class II restoration) showing no (score of "0") microleakage at both proximal locations. Proximal surface preparations/restorations were located in enamel, thus revealing differences in tooth substructure (enamel vs. dentin [cementum]) adhesion.*

icantly less marginal microleakage compared to that seen in groups B and D, in which the same material systems were used, although inserted into Class II cavity preparations. This occurrence reemphasized the fact that the process of micromechanical adhesion of composite resin to enamel is a more efficacious process compared to the bond achieved by composite to dentin or cementum surface substructure.[1,29,30] Decreased levels of microleakage between the enamel surface and the adhesive/composite resin complex indicate that a quality seal has been attained; thus, a restoration with fewer marginal gaps and less subsequent clinical sequelae (stained margins, sensitivity, etc) is demonstrated.[1,2,29,30] Representative photographs (Figure 2) clearly demonstrate excellent ("0" scores) adhesion between the enamel surfaces and restorative materials (SonicFill and Herculite Ultra) for Class I restorations (groups A and C). Conversely, groups B and D (same materials, Class II restorations) clearly revealed marginal microleakage, as indicated by the dye penetration levels. Interestingly, as some of the Class II cavity preparations were cut slightly occlusal or gingival to the CEJ, dye penetration appears to originate from the proximal side where the gingival margin is located (ie, dentin [cementum]), as opposed to a corresponding enamel margin of the same restoration. In addition, the few Class II restorations in groups B and D that showed "0" scores on a single or both proximal surfaces were limited to preparations in enamel, occlusal to the CEJ (Figure 2, Group D). These occurrences add more support to the notion that superior bonds occur between enamel and the adhesive system/composite resin complex, even though the enamel margins (thinner enamel structure) are located in a proximal, gingival box for a Class II preparation.

The SonicFill restorative system utilizes sonic energy technology (changes in material viscosity) together with a composite material blend that, reportedly, creates a condition wherein the material closely adapts to the preparation margins using only one bulk increment.[23,24,26,27] Following deactivation of the sonic energy the material viscosity increases, allowing for increased depths of cure.[23,24,26,27] This system utilizes a formulation consisting of monomers (ethoxylated bisphenol A dimethacrylate, bisphenol A dimethacrylate, and triethyleneglycol), which is highly filled (barium glass and silicon dioxide) by weight (83.5%) and also includes special modifiers that react to the sonic energy, permitting a quicker "flow" and presumably better adaptability to the preparation walls.[23,24,26,27] Preliminary *in vitro* research[23,24,26] has reported polymerization shrinkage of 1.6%, increased flexural strengths, and a 5.0-mm depth of cure. These advertised claims have touted a more "user friendly," less time consuming composite resin (system) for use by the dentist.[23,24,26,27] Limited *in vitro* microleakage studies[31-34] using Class II cavity preparation/restoration criteria have shown no defin-

itive differences when comparing the SonicFill system to other composite systems or methods of insertion, although two studies[34,35] did reveal positive results using the SonicFill, as compared to other composite resin delivery systems.

Factors such as different adhesive systems and cavity preparation designs including low configuration or "C-factors" and using different insertion techniques (incremental), polymerization sources, and the use of liners (resin modified glass ionomer and flowable composites) can decrease the unfavorable material and clinical influences associated with polymerization shrinkage and, consequently, marginal microleakage.[13,17] The preparation "C-factor" can influence the magnitude of stress on posterior composites and has been defined as the ratio of the bonded to the unbonded surfaces of a cavity preparation.[12,14] A restoration with high "C-factor" would indicate a greater potential for bond interruption due to the forces imposed from polymerization shrinkage, with subsequent formation of marginal gaps and/or voids and, consequently, microleakage.[12,14] Class I preparations have the highest "C-factors" as a result of the number of bondable surfaces; therefore, they have the greatest potential for the detrimental effects of polymerization. Class II, III, IV, and V preparations have lower "C-factors" because of the ratio of bonded to unbonded surfaces and, therefore, predispose the restoration to a lower risk for possible clinical sequelae.[12,14] Contemporary insertion of composite resin into posterior preparations usually involves multiple increments, taking into account the material shrinkage and preparation design "C-factor."[11-14] Incremental insertion of composite resin into a posterior preparation is performed to compensate for the stresses induced into the material following light polymerization. Therefore, bulk-fill composite insertion can potentially result in increased shrinkage forces, gap formation at the tooth/material interface, and, in turn, microleakage.[36] A recently conducted study tested bulk-fill (including SonicFill) vs incremental-fill composite resins, using depth of cure, hardness, and shrinkage as testing parameters, and revealed that bulk-fill composites showed greater depths of cure and decreased levels of volumetric and polymerization shrinkage.[37]

As seen by the results of the present study, although Class I cavity preparations/restorations exhibited the highest "C-factors" (five bonded surfaces to one unbonded surface = "C-factor" of 5), Class I preparations filled with both SonicFill and Herculite Ultra (groups A and C) showed significantly less microleakage compared to Class II restorations (groups B and D) filled with the same restoratives. The Class II (MOD) preparations/restorations revealed a "C-factor" of 3. Thus, the Class I restorations showed a higher "C-factor" compared to the Class II restorations. Given these facts, it appears that bonding surfaces (enamel vs dentin [cementum]), rather than cavity design "C-factors" or novel material technologies, played a major role in terms of microleakage.

While bonding to enamel has been reported with consistently predictable results,[1,38-40] challenges have been encountered with dentin surface bonding because of the complex composition and histologic nature associated with dentin. An *in vitro* study conducted by Poggio and others[41] concluded that using different restorative techniques and materials did not positively influence microleakage penetration below the CEJ, at the dentin margin—further substantiating that dentin surface substructure is not as suitable a medium for bonding as is enamel. Etching enamel is efficient in terms of the removal of the smear layer, demineralizing the inorganic enamel surface, creating microporosities for a patent, mechanical bond.[1,42,43]

The degree of dye penetration as an *in vitro* method for determining marginal microleakage of dental restorations, including a myriad of material and/or cavity preparation variables, is a commonly used evaluation device and has been repeatedly performed and reported in the dental literature, although inconclusive results have sometimes been attained.[6,8,44-46] Microleakage studies provide adequate screening methods, possibly determining clinical success and longevity of a particular material or technique, although clinical, longitudinal studies are the best projectors of restoration performance.[6,8,44-46] The results attained from *in vitro* studies cannot necessarily be extrapolated to *in vivo* clinical outcomes; however, the authors agreed that the present results demonstrated that tooth surface (enamel) morphology was the most significant factor with regard to microleakage of both posterior composite resin restoration systems.

CONCLUSION

Within the limitations of the present *in vitro* study, the hypothesis was rejected that usage of the SonicFill system for restoration of Class I and II preparation cavities resulted in significantly less microleakage. Significant microleakage was encountered with specimens in both Class II restoration groups (groups B and D). However, very little or no microleakage was associated with the Class I insertions (groups A and C).

The results showed that the bonding surface (enamel and dentin), and not necessarily material technology and/or "C-factors," was the primarily limiting factor in terms of marginal microleakage.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Integrated Medical Research Informational System. The approval code for this study is: 479277.

Conflict of Interest

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

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Effect of High Irradiance on Depth of Cure of a Conventional and a Bulk Fill Resin-based Composite

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Clinical Relevance

For both RBCs, rapid photocuring using a PAC light for five seconds resulted in a shallower depth of cure than when the same radiant exposure of (37 J/cm^2) was delivered by a QTH or LED curing light used for 40 seconds or 20 seconds, respectively.

SUMMARY

Objectives: This study evaluated the effect of using three commercial light curing units (LCUs) delivering a range of irradiance values, but delivering similar radiant exposures on the depth of cure of two different resin-based composites (RBCs).

Methods: A conventional hybrid RBC (Z100 shade A2, 3M ESPE) or a bulk fill RBC (Tetric EvoCeram Bulk Fill shade IVA, Ivoclar Vivadent) was packed into a 10-mm deep semicircular metal mold with a 2-mm internal radius. The RBC was exposed to light from a plasma-arc-curing (PAC) light (Sapphire Plus, DenMat) for five seconds, a quartz-tungsten-halogen (QTH) light (Optilux 501, Kerr) for 40 seconds, or a light-emitting-diode (LED) light (S10, 3M ESPE) for 20 seconds and 40 seconds (control). The Knoop microhardness was then measured as soon as possible at the top surface and at three points every 0.5 mm down from the surface. For each RBC, a repeated measures analysis of variance (ANOVA) model was used to predict the Knoop hardness in a manner analogous to a standard regression model. This predicted value was used to determine at what depth the RBC reached 80% of the mean hardness achieved at the top surface with any light.

Results: The PAC light delivered an irradiance and radiant exposure of 7328 mW/cm^2 and 36.6 J/cm^2, respectively, to the RBCs; the QTH light delivered 936 mW/cm^2 and 37.4 J/cm^2 and in 20 seconds the LED light delivered 1825 mW/cm^2

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and 36.5 J/cm^2. In 40 seconds, the control LED light delivered a radiant exposure of 73.0 J/cm^2. For Z100, using 80% of the maximum hardness at the top surface as the criteria for adequate curing, all light exposure conditions achieved the 2.0-mm depth of cure claimed by the manufacturer. The LED light used for 40 seconds achieved the greatest depth of cure (5.0 mm), and the PAC light used for five seconds, the least (2.5 mm). Tetric EvoCeram Bulk Fill achieved a 3.5-mm depth of cure when the broad-spectrum QTH light was used for 40 seconds delivering 37.4 J/cm^2. It required a 40-second exposure time with the narrow-spectrum LED, delivering approximately 73 J/cm^2 to reach a depth of cure of 4 mm.

Conclusions: When delivering a similar radiant exposure of 37 J/cm^2, the QTH (40 seconds) and LED (20 seconds) units achieved a greater depth of cure than the PAC (five seconds) light. For both resins, the greatest depth of cure was achieved when the LED light was used for 40 seconds delivering 73 J/cm^2 ($p<0.05$).

INTRODUCTION

In recent years, there has been a global reduction in the use of amalgam resulting in an increase in the use of resins to the extent that, in Scandinavia, almost no amalgam restorations are placed.[1,2] This trend will only accelerate with the signing of the Minamata Convention that contains provisions to phase-down the use of dental amalgam.[2,3] Although some photopolymerizable resin-based composites (RBCs) have been reported to have excellent long-term results in clinical trials,[1,4,5] the results achieved in dental offices worldwide have been less promising.[6-8] If the RBC is undercured, the result will be suboptimal properties for the restorative material that could increase the probability of fracture of the restoration, encourage more secondary caries, increase the wear rate, and result in premature failure.[9] Undercuring is especially of concern for Class II restorations that commonly fail due to bulk fracture or secondary caries at the cervicogingival margin.[10] This part of the restoration is the furthest away from the curing light (LCU) and is the most difficult to photopolymerize.[11] Additionally, when dental RBCs are not adequately polymerized (and thus do not reach a sufficient degree of monomer conversion), they are more likely to leach chemicals into the mouth.[12-14] Arbitrarily increasing light exposure times in an effort to prevent undercuring is not the answer because this may cause unacceptable thermal trauma to the pulp and surrounding tissues.[15-17] Thus, it is important for the clinician to know the optimal exposure times for the RBCs being used in the office.

One method used to determine the potential clinical adequacy of photopolymerization is to measure the depth of cure (DOC) in a mold. The type and size of the mold, the RBC, and the light source used can all affect the measured DOC.[18-20] The test method described in the ISO 4049 standard can be used to evaluate the DOC.[21] This method uses a 4-mm diameter metal mold and evaluates the DOC at room temperature immediately after light exposure. The DOC is determined to be half the maximum length (depth) of hard RBC remaining after the soft RBC has been scraped away. The suitability of the ISO 4049 test to determine the DOC for bulk fill RBCs has been questioned because it has been reported to overestimate the true DOC.[22]

Depth of cure can also be assessed from microhardness measurements where a hardness value that is at least 80% of the maximum hardness is considered to be acceptable.[23-27] Two microhardness tests are commonly used, Vickers and Knoop. Both of these tests use loads less than 1000 g to make small indentations in the RBC, but the shape of the indentations is different for the two tests.[28] The Vickers test uses a square-based pyramid-shaped indenter to produce indentations that have approximately the same length on both axes. The lengths of the diagonals of the indentation are measured and averaged to calculate the hardness.[28] This test is most suitable for determining the hardness of brittle materials because, when this test is used to measure dental resins that exhibit some elastic recoil, elastic recovery occurs equally in both axes when the load is removed from the indenter, potentially resulting in an inaccurate measurement.[28] In contrast, the Knoop microhardness method uses a rhombic-shaped indenter to produce narrow indentations that have both a long and a short axis. Due to this unique shape, when the indenter is removed from the test material, elastic recovery (dimensional change) primarily occurs in the short axis leaving the length of the longer diagonal virtually unchanged. The Knoop hardness is calculated using only this longer axis and is virtually independent of the ductility of the tested material. This makes the Knoop test ideal for testing ductile materials such as RBCs.[28] Depth of cure values obtained by the Knoop test method may be more reliable[29] than the ISO 4049 test, and a strong positive correlation exists between the Knoop micro-

hardness values and degree of monomer conversion within each brand of RBC.[19,24,30-32]

Light-cured RBCs contain at least one photoinitiator, such as camphorquinone (CQ), with additional initiators sometimes included.[28,32-38] CQ is a type II photoinitiator that undergoes a bimolecular reaction. It has a maximum absorbance of light at a wavelength of 470 nm, but it is activated by a broad range of light from wavelength of approximately 510 nm down to well into the ultraviolet range below 360 nm.[28,34] In contrast, alternative photoinitiators such as monoacylphosphine oxide (TPO) and derivatives of dibenzoyl germanium (e.g. Ivocerin) are not activated by light above 460 nm but are very sensitive to light below 420 nm.[38-40] These type I photoinitiators undergo a unimolecular reaction upon irradiation. Consequently, they are more reactive to light compared to CQ and may require a lower radiant exposure when placed in thin increments.[34,37,38,41,42] However, due to the reduced penetration of shorter wavelengths of light, this advantage is lost as the thickness of the RBC increases.

Due to postirradiation polymerization, the time between curing a resin specimen and measuring the hardness can have a considerable impact on the results.[43-45] Although all RBCs exhibit some postirradiation polymerization, the amount depends upon the RBC used and the extent to which it has been polymerized.[43,45] After 24 hours, the increase in hardness can range from 12% for a well-polymerized RBC to over 350% for the same RBC when it is less well polymerized.[43] Despite the known effects of postirradiation polymerization, RBC specimens are often measured 24 hours or more after light curing[19,23,24,27,32,46,47] as the physical properties will likely remain stable during the time taken to make the measurements.[43] However, in a clinical situation the patient will not wait 24 hours before chewing. Thus, the bottom and sides of the RBC restoration may well be stressed soon after placement, requiring the RBC to reach an 80% bottom/top hardness ratio within a clinically relevant time.

There is a high demand from the dental profession to become more productive by shortening chairside procedures. Consequently, some manufacturers are suggesting that their high-output LCUs require much shorter exposure times than conventional lights.[48-51] It has even been calculated that, based on placing 2200 restorations in a year, a dentist could save enough time to produce US$26,399 more a year if five seconds of light exposure is used instead of 30 seconds under the same conditions.[52] Some contemporary LCUs claim to deliver irradiance levels up to 6 W/cm^2 and their manufacturers suggest that a very short (one to three seconds) exposure time will adequately cure the resin.[49] These recommendations have been challenged more than once, because for a given dose of energy, longer exposure times at a lower irradiance seem to be beneficial factors to ensure optimal RBC properties.[14,53-59] In addition, such high-output LCUs may cause more heating of the tooth and soft tissues, which raises the concerns of thermal damage.[15-17,36]

Another method to shorten chairside time is to bulk fill and cure RBCs in just one light exposure. According to the manufacturers, these bulk fill materials can be adequately cured in one 4- to 5-mm increment without having to extend the light exposure time.[38,60] Some reports have suggested that this is an acceptable technique,[25-27,46,47,61,62] but others have suggested that bulk filling and curing may produce undercured RBCs.[22,63,64] Bulk filling a cavity reduces some of the technical disadvantages associated with layering conventional RBCs, such as the incorporation of voids or contamination between the layers,[61,63] as well as improving chairside efficiency. However, as the thickness increases, exponentially fewer photons of light reach the bottom of the RBC, an effect which is more pronounced at the shorter wavelengths (410 nm) compared to longer wavelengths (460 nm).[33,65] To counteract this effect and increase the DOC, manufacturers have used different strategies, including using more reactive alternative photoinitiators and improving RBC translucency by reducing the amount of fillers or by matching the refractive indices of the fillers and resin matrix.[66] The advisability of rapid photocuring and bulk filling as an option for shortening chairside time needs to be examined, and the interaction of these two strategies requires further exploration.

The objective of this study was to evaluate the effect of using three commercial LCUs delivering a range of irradiance values, but similar radiant exposures, on the depth of cure of a popular version of a conventional hybrid and a bulk fill RBC. The tested null hypotheses were that: 1) when 37 J/cm^2 of radiant exposure was delivered from a plasma arc-curing unit (PAC), a quartz-tungsten-halogen unit (QTH), and a light-emitting diode unit (LED) light, both RBCs would achieve the manufacturer's claimed depth of cure; and 2) for each RBC, there will be no significant difference in depth of cure obtained using these different LCUs when the same radiant exposure was delivered.

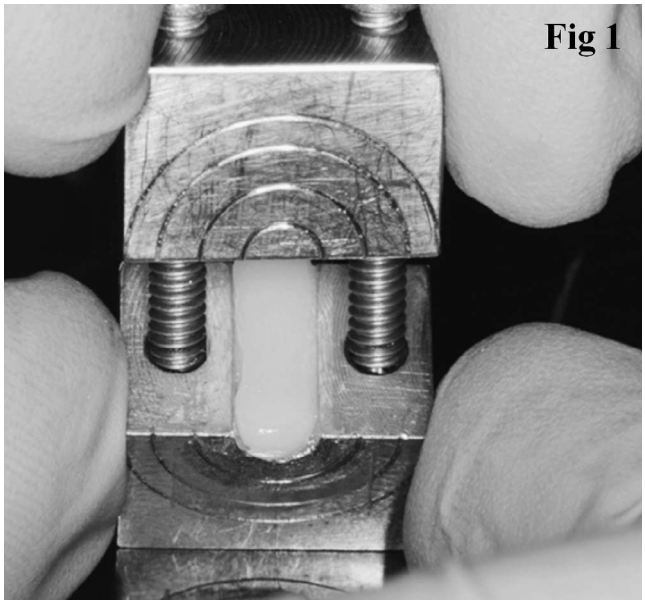

Figure 1. *The 10-mm deep half-cylindrical metal mold with a 2-mm internal radius filled with RBC.*

METHODS AND MATERIALS

A conventional hybrid composite (Filtek Z100 – shade A2, 3M ESPE, St Paul, MN, USA) and a bulk fill composite (Tetric EvoCeram Bulk Fill – shade IVA that is equivalent to shade A2 – A3, Ivoclar Vivadent, Amherst, NY, USA), which includes CQ and the alternative photoinitiator Ivocerin,[38] were used in this study. One example of three types of LCU was used to polymerize the samples: a PAC light; Sapphire Plus, DenMat, Lompoc, CA, USA), a QTH light; Optilux 501, Kerr, Orange, CA, USA), and a LED unit; Elipar S10, 3M ESPE). The PAC unit was used with its optional 4-mm "turbo" fiber-optic light guide, which is designed to deliver a high irradiance; the QTH unit was used with its 10-mm fiberoptic light guide; and the LED unit was used with its 10-mm fiberoptic light guide.

Sample Preparation

The Z100 and Tetric EvoCeram Bulk Fill materials were packed in a 10-mm deep half-cylindrical metal mold with a 2-mm internal radius (Figure 1). The top and bottom surfaces of the RBC in the mold were covered with a polyester strip and pressed flat. The LCU light tip was fixed and centered as if the mold were a cylinder just out of contact with the top polyester strip, and the RBC was exposed to light in one of four curing conditions: PAC for five seconds, QTH for 40 seconds, LED for 20 seconds, and LED for 40 seconds (Figure 2). The first three exposure times represented clinically relevant light exposure

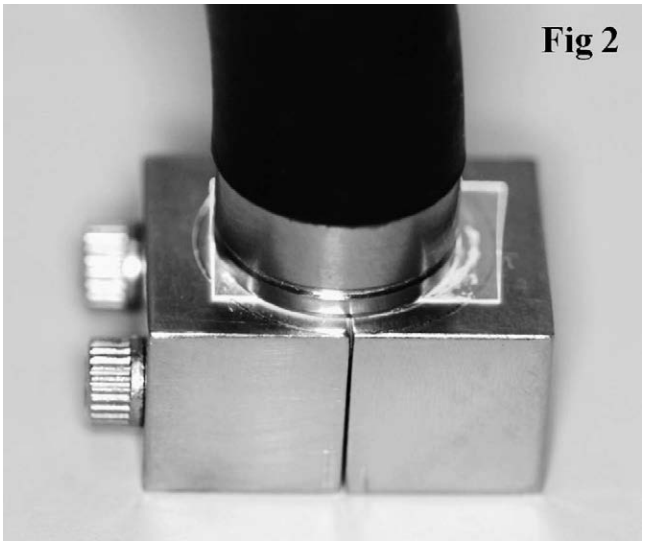

Figure 2. *The samples were polymerized with the tip of the light guide just out of contact with the Mylar strip.*

conditions and delivered similar radiant exposures to the specimens, all above 24 J/cm^2. The LED unit was used for an additional 40 seconds to provide an overexposure condition to act as a control. Five samples were prepared per RBC per irradiation condition for a total of 40 specimens (two RBCs * four LCU/time * five repetitions). All sample preparation and testing was conducted at ambient room temperature and humidity.

Depth of Cure Evaluation

The Knoop microhardness (KHN) was measured using an automated microhardness-testing device (HM 123, Mitutoyo Canada Inc, Mississauga, ON, Canada) applying a 50-gf load for eight seconds. The KHN was measured at nine points on the top surface and Figure 3 illustrates how, on each specimen, the hardness was also measured at three points on the lateral surface every 0.5 mm down from the top surface to the point where it was too soft to measure. The hardness measurements were started as soon as practically possible (termed near immediate measurement). All of the indentations were completed within 30 minutes of the end of light exposure.

Evaluation of Energy Delivered to the RBCs

The spectral radiant power output in milliwatts/nanometer (mW/nm) delivered to the specimens from each LCU was measured five times using a 6-inch integrating sphere (Labsphere, North Sutton, NH, USA) attached to a fiberoptic spectrometer (USB 4000, Ocean Optics, Dunedin, FL, USA), as previously described.[67] The integrating sphere had a 4.0-

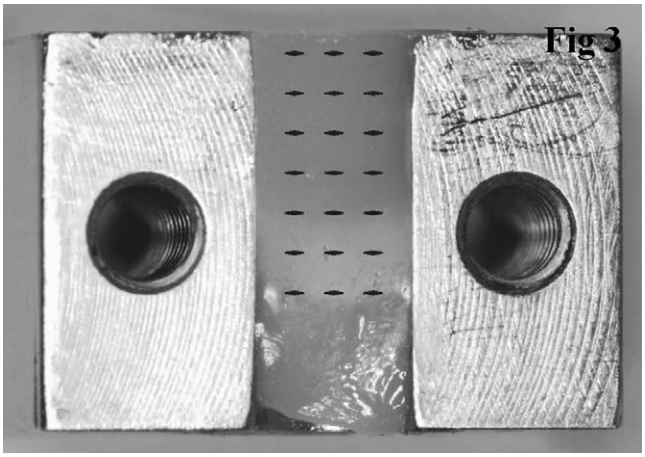

Figure 3. *Microhardness measured at three positions at each 0.5-mm increment down from the top surface of the RBCs.*

mm diameter entrance aperture (radius 2.0-mm), and the light tip was fixed and centered over this entrance. For each LCU, the mean radiant power (Watts) reaching the top surface of the semicircular mold was calculated as the average of the five radiant power values recorded by the integrating sphere divided by two. The irradiance (mW/cm^2) and radiant exposure (in J/cm^2) delivered to the RBCs was then calculated.

Statistical Analyses

For each RBC, Z100 and Tetric EvoCeram Bulk Fill, separate repeated measures analysis of variance (ANOVA) models were developed using the actual KHN measurements to predict the KHN over distance (depth) in a manner analogous to a standard regression model. The KHN measurements analyzed for each RBC constituted the five replicates that were measured under four different light/time conditions. A three by three grid on the top surface and three measurements at each of 18 depths (from 0.5 to 9.0 mm by 0.5 mm increments) constituted the 63 measurements that were modeled. Depth, light, and their interaction were the fixed effects in the statistical model that provided a predicted hardness at each 0.5-mm depth increment for each combination. This data analysis was carried out using SAS software (SAS, Cary, NC, USA), and statistical significance was set at $p \leq 0.05$.

RESULTS

The mean irradiance and radiant exposures delivered to the semicircular RBC specimens were respectively: PAC unit 7328 mW/cm^2 and 36.6 J/cm^2, QTH unit 936 mW/cm^2 and 37.4 J/cm^2, and LED unit (20 seconds) 1825 mW/cm^2 and 36.5 J/cm^2. In 40 seconds, the control LED delivered 73.0 J/cm^2 (Table 1). The spectral emissions from the LCUs through the 4.0-mm aperture into the integrating sphere are shown in Figure 4.

The mean KHN ± standard deviation measured at different depths for Z100 and Tetric EvoCeram Bulk Fill are reported in Figures 5 and 6, respectively. The level where the KHN was 80% of maximum hardness achieved at the top surface (100% line), irrespective of the choice of LCU, is shown on the Figures. Comparing Figures 5 and 6, it can be seen that the maximum top hardness of Tetric EvoCeram Bulk Fill was approximately 50% of the hardness of Z100. For Tetric EvoCeram Bulk Fill only, the broad-spectrum PAC and QTH lights produced harder KHN values down to a depth of 2.0 mm compared to the narrow spectrum LED light. At greater depths, the narrow spectrum LED unit used for 40 seconds produced harder KHN values for both RBCs (Figures 5 and 6).

In the statistical analysis, when considering the likelihood of the RBC to be cured at any point, depth was the most important predictor ($F[18,72]=2714.62$, $p<0.0001$). The choice of LCU had a smaller, but still very significant effect; however, this effect was not the same at all depths ($F[54,216]=59.95$, $p<0.0001$). Thus, it was not possible to directly compare the overall effect of the LCUs on the hardness. Instead, separate repeated measures ANOVA models were developed to predict a single hardness value for each combination of depth and light. For each RBC, these predictions were compared to the mean hardness value at the top surface achieved with all LCUs. The depth where the predicted value fell below 80% of the greatest top hardness value was used as the criteria for adequate curing. The greatest depth of adequate cure (5.0 mm) was obtained with Z100, whereas it was 4.0 mm for Tetric EvoCeram Bulk Fill (Table 1). For Z100, the LED used for 40 seconds achieved the best results, and the PAC used for five seconds, the worst. Plus or minus ties, the same is also true for Tetric EvoCeram Bulk Fill.

DISCUSSION

This study evaluated the effect of delivering different irradiances, between 936 and 7328 mW/cm^2, but very similar radiant exposures (approximately 37 J/cm^2) from three types of LCU, on the depth of cure of a conventional hybrid and a bulk fill composite. Using an automated hardness tester made near immediate hardness testing feasible and the test more clinically relevant.

Table 1: *Maximum Predicted Depth at Which the Knoop Hardness Number of the Resin-Based Composites (RBC) Achieved 80% of the Top Surface Value, Based on the Repeated Measures ANOVA Model ($p \leq 0.05$)*

RBC and Shade	Light and Exposure Time, s	Depth of Cure, mm	Irradiance Received by RBC, mW/cm^2	Radiant Exposure Received by RBC, (J/cm^2)
Z100 A2	PAC (Sapphire) for 5 s	2.5	7328	36.6
Z100 A2	LED (S10) for 20 s	3.5	1825	36.5
Z100 A2	QTH (501) for 40 s	4.0	936	37.4
Z100 A2	LED (S10) for 40 s	5.0	1825	73.0
Tetric EvoCeram Bulk Fill IVA	PAC (Sapphire) for 5 s	3.0	7328	36.6
Tetric EvoCeram Bulk Fill IVA	LED (S10) for 20 s	3.5	1825	36.5
Tetric EvoCeram Bulk Fill IVA	QTH (501) for 40 s	3.5	936	37.4
Tetric EvoCeram Bulk Fill IVA	LED (S10) for 40 s	4.0	1825	73.0

Tetric EvoCeram Bulk Fill did not cure to a greater depth than the conventional resin, irrespective of the LCU used (Table 1), and it did not reach the 4.47 mm DOC reported by Alrahlah and others[27] who used a similar split metal mold and the same S10 LED curing light for 20 seconds. These authors measured the Vickers microhardness of the RBCs after they had been stored for 24 hours at 37°C, and the test method and storage conditions would have affected the outcome.[30,43-45] The DOC results for Tetric EvoCeram Bulk Fill are much greater than previously reported (0.2 mm) in another study that also used a split metal mold and tested the hardness of the RBC immediately after irradiation.[22] However, the DOC is similar to the 3.32-mm DOC achieved for this RBC using the ISO 4049 scrape test in the same study.[22]

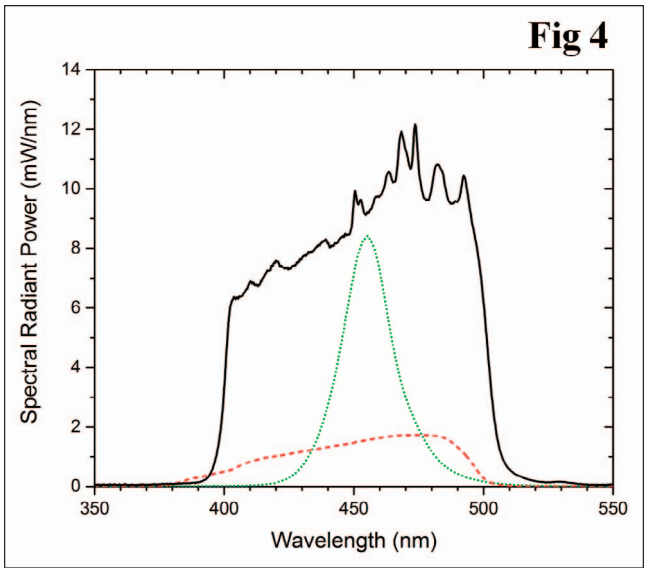

Figure 4. *Spectral radiant power from each LCU measured through a 4-mm diameter aperture placed at the entrance to an integrating sphere that was connected to a spectrometer.*

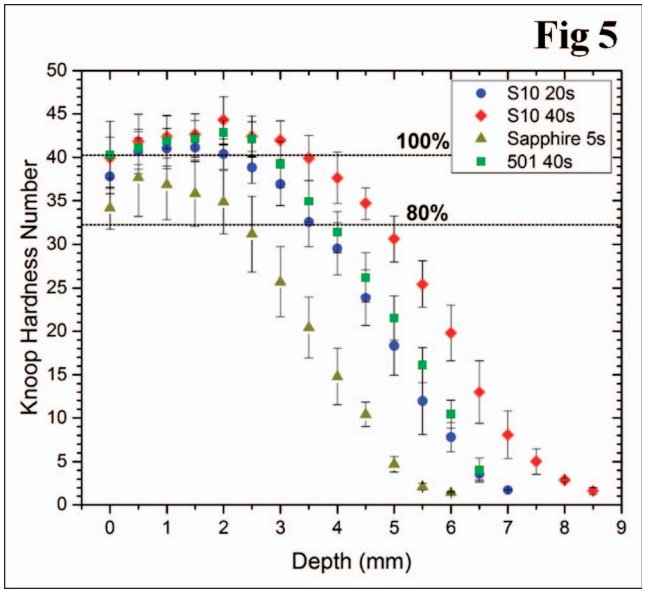

Figure 5. *Mean Knoop hardness number ± one standard deviation measured at each depth for Z100. Note the lines showing maximum top hardness (100%) and 80% value. Note also the different KHN scale compared to Figure 6.*

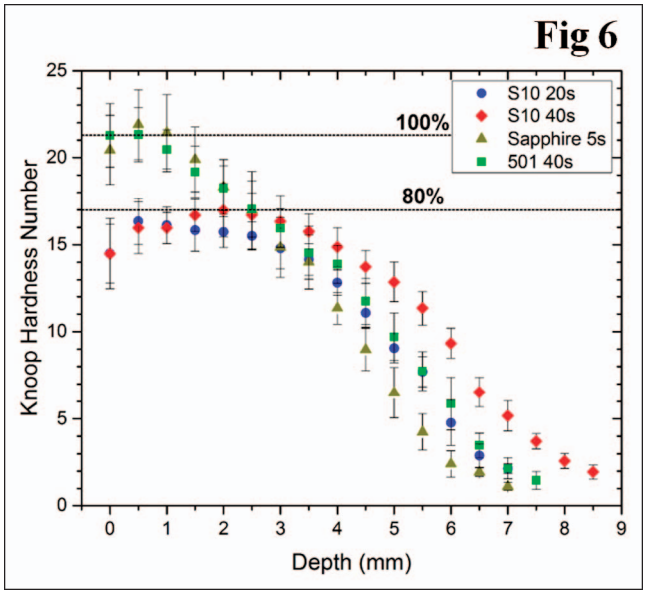

Figure 6. *Mean Knoop hardness number ± one standard deviation measured at each depth for Tetric EvoCeram Bulk Fill. Note the lines showing maximum top hardness (100%) and 80% value. Note also the different KHN scale compared to Figure 5.*

In view of the large differences in the microhardness values between the two RBCs, the analyses were carried out separately for each resin. The results show that, although the two RBCs were well cured at or near the surface when exposed to high irradiance levels for short times, as the depth increased, the microhardness values were negatively affected. When 37 J/cm^2 of radiant exposure was delivered from the QTH, LED, or PAC light, there were significant differences in depth of cure obtained using these different LCUs ($p<0.0001$). The conventional resin-based composite achieved the manufacturer's stated 2.0 mm depth of cure when any of the LCU units were used. The bulk filling RBC (Tetric EvoCeram Bulk Fill) only achieved a 4.0-mm depth of cure when the LED unit was used for 40 seconds, delivering twice as much radiant exposure, 73 J/cm^2, as the other test conditions. Photocuring using the PAC light for five seconds at 7328 mW/cm^2, resulted in the shallowest depth of cure for both RBCs. Thus, both the first and second hypotheses were rejected.

To minimize the effects of light beam inhomogeneity,[67] this study measured the wavelength and radiant exposure delivered from the LCU, centered over the 4-mm diameter aperture into an integrating sphere rather than the total energy delivered by the LCU. This minimized the effects of light beam inhomogeneity, and allowed for a close estimation of the radiant exposure received by the RBC specimens (~36.5 J/cm^2). This should have been more than enough for adequate photopolymerization.[28,38,68,69] Despite delivering a similar radiant exposure, Table 1 and Figures 5 and 6 show that the PAC light used for only five seconds produced the shallowest curing depth for both RBCs, although the PAC light produced the hardest resin to a depth of 1.5 mm for Tetric EvoCeram Bulk Fill (Figure 6). The irradiance from the turbo tip on the PAC light (7328 mW/cm^2) was much greater than that from both the QTH (936 mW/cm^2) and LED (1825 mW/cm^2) units (Table 1). These results corroborate previous findings that slower photopolymerization is more successful to rapid (five-second) photopolymerization of RBCs.[14,53-58,63]

In common with previous studies, this study used the 80% bottom/top threshold to define when the RBC is adequately cured.[22,24-27] It is interesting to note that using a metal mold under these test conditions, a conventional hybrid resin containing only CQ was able to achieve a 5.0-mm depth of cure when 73 J/cm^2 was delivered from the LED light in 40 seconds, whereas Tetric EvoCeram Bulk Fill only achieved a 4.0-mm depth of cure under the same conditions. Although the type I photoinitiators used in Tetric EvoCeram Bulk Fill undergo a unimolecular reaction upon irradiation and are therefore more reactive to light,[34,37,38,41,42] due to the effects of the Rayleigh scattering of light less light penetrates through the RBC as the wavelength decreases. Thus, little of the shorter wavelengths of light reach down into the depths of the RBC to initiate photopolymerization. This supports the observation that the depth of cure of CQ-based materials can be greater than that of TPO-based materials.[41]

As in a clinical situation at the bottom of the restoration, the RBC samples were not polished. The maximum microhardness was determined as the maximum hardness achieved by any LCU at the top surface. Figures 5 and 6 illustrate that both RBCs were harder just beneath the surface rather than at the surface. This observation has been reported previously[22] and may occur because in the subsurface region close to the irradiated surface, the RBC receives almost all the light from the LCU plus more of the beneficial effect of the exotherm from the RBC all around it. As the depth increases, the RBC receives fewer and fewer photons of light, and the amount of photopolymerization is less. Another possible explanation for this finding is that the polyester strip did not provide 100% protection from the air and thus the top surface of the resin contained an oxygen-inhibited layer. This effect requires further study.

To achieve a hardness value that was at least 80% of the maximum top value at 4 mm depth, the LED unit had to be used for 40 seconds for Tetric EvoCeram Bulk Fill. This is much longer than 10 seconds suggested by the manufacturer of this resin[38] for any LCU delivering >1000 mW/cm^2 (>10 J/cm^2), or the manufacturer of the LED unit.[48] This LED curing light has a relatively narrow spectral emission (Figure 4), and only the QTH and PAC lights delivered any spectral output below 420 nm. Thus, the TPO initiator used in the Tetric EvoCeram Bulk Fill may not have been efficiently activated by the LED light used in this study, and different results may have been achieved had a LED light delivering a broader spectral emission been used. However, since both the QTH and PAC lights are broad-spectrum lights and they delivered much more than the recommended amount of energy to the specimens, they should have been able to cure this RBC to a depth of 4 mm, but they failed to achieve this.

Similar to the ISO 4049 depth of cure scrape test guidelines,[21] the specimens were made in a metal mold at room temperature and they were tested almost immediately. The microhardness results obtained in the present study could have been influenced by the metal mold that completely blocked transmission of light outside the mold, unlike tooth material. However, the use of a metal mold has been recommended so as not to overestimate depth of cure.[20] The metal mold also created testing conditions closer to clinical conditions where a metallic matrix is placed around the proximal box in Class 2 preparations. In addition, had the specimens been examined at an intraoral temperature, or after 24 hours, a greater depth of cure would have been achieved.[30,43-45]

Despite these limitations, the results show that rapid photocuring using a broad-spectrum light source of a conventional RBC and a bulk fill RBC, which contains both Ivocerin and CQ, results in a shallower depth of cure. Future studies should evaluate the effect of using a broad-spectrum, polywave LED curing light on these RBCs and of using different combinations of exposure times and irradiance levels when photocuring other RBCs with similar amounts of energy.

CONCLUSIONS

Within the limitations of this study that used an 80% bottom/top hardness ratio to define adequate polymerization in a metal mold, it was concluded that:

1) When similar radiant exposures were delivered from the QTH, LED, or PAC light, there were significant differences in depth of cure depending on the LCU used.
2) The conventional resin-based composite (Z100) achieved greater than a 2-mm depth of cure when the PAC, QTH, or LED units were used delivering approximately 37 J/cm^2.
3) The bulk fill resin-based composite (Tetric EvoCeram Bulk Fill) achieved a 3.5-mm depth of cure when a broad-spectrum QTH light was used for 40 seconds. It required a 40-second exposure time with the narrow-spectrum LED delivering approximately 73 J/cm^2 to reach a depth of cure of 4 mm.
4) Rapid photocuring using the broad-spectrum PAC light for five seconds delivering 36.6 J/cm^2 at 7328 mW/cm^2 resulted in the shallowest depth of cure for both RBCs.

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Conflict of Interest

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

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Effects of Temperature and Aging on Working/Setting Time of Dual-cured Resin Cements

TA Pegoraro • R Fulgêncio • LE Butignon • AP Manso • RM Carvalho

Clinical Relevance: Clinicians must be aware that resin cements differ widely in their clinical handling and that performance may vary according to temperature and aging. As resin cements age, working time and setting time change significantly, and this may have a profound effect on clinical handling.

doi: http://dx.doi.org/10.2341/13-361-L

Effectiveness of Fluorescence-based Methods in Monitoring Progression of Noncavitated Caries-like Lesions on Smooth Surfaces

MB Diniz • PH Campos • ME Sanabe • DA Duarte • MTBR Santos
RO Guaré • C Duque • A Lussi • JA Rodrigues

Clinical Relevance: In this investigation, the authors observed that fluorescence-based methods were able to identify progressive enamel demineralization on smooth surfaces in the presence of biofilm.

doi: http://dx.doi.org/10.2341/15-036-L

Effect of Selective Etch on the Bond Strength of Composite to Enamel Using a Silorane Adhesive

L Bermudez • M Wajdowicz • D Ashcraft-Olmscheid • K Vandewalle

Clinical Relevance: The selective etch of enamel with phosphoric acid may improve the bond strength of silorane adhesives.

doi: http://dx.doi.org/10.2341/14-311-L

Influence of Staining Solution and Bleaching on Color Stability of Resin Used for Caries Infiltration

GSA Araújo • FS Naufel • RCB Alonso • DANL Lima • RM Puppin-Rontani

Clinical Relevance: A bleaching treatment of resin-infiltrated enamel lesions may improve appearance after staining.

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Splint Porcelain Laminate Veneers With a Natural Tooth Pontic: A Provisional Approach for Conservative and Esthetic Treatment of a Challenging Case

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Clinical Relevance: Under specific occlusion without incisal contact in centric occlusion, protrusive, or lateral movements, splint laminate veneers with a natural tooth pontic may be a conservative and esthetic approach as a provisional restorative treatment for severely discolored and apically resorbed anterior maxillary teeth.

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Effects of Temperature and Aging on Working/Setting Time of Dual-cured Resin Cements

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Clinical Relevance

Clinicians must be aware that resin cements differ widely in their clinical handling and that performance may vary according to temperature and aging. As resin cements age, working time and setting time change significantly, and this may have a profound effect on clinical handling.

SUMMARY

Objectives: To evaluate the effects of aging and temperature on working time (WT) and setting time (ST) of several dual-cured resin cements.

Methods: WT and ST were determined with a thermo-controlled stage oscillating rheometer. New cement kits were used for the study. Cements were mixed according to instructions and dispensed on the oscillating stage that was preset at 22°C or 37°C. Rheologic charts were generated from the beginning of mixing until no further oscillation was detected. After initial measurements, cement kits were aged at 37°C for 12 weeks, and WT/ST was determined again at both temperatures. Five samples were read for each material and condition. Data were analyzed with repeated measures analysis of variance and a Tukey test at α=5% for each individual material.

Results: The WT and ST of all cements were significantly affected by temperature and aging ($p<0.05$). In general, higher temperature accelerated WT/ST, but aging effects were material dependent. Some materials presented reduced WT/ST, whereas others showed increased WT/ST, regardless of the temperature.

Conclusions: The WT and ST were significantly affected by temperature variation and aging condition. Although temperature changes appeared to affect all materials similarly, aging effects were material dependent.

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INTRODUCTION

Resin cements have become clinically desirable because of their ability to bond to both the tooth structure and the restoration. They are often designed for specific applications, rather than general use, and are formulated to provide the handling characteristics required for particular applications.[1] Dual-cured resin cements have been considered the material of choice to lute esthetic indirect restorations and fiber posts.[2-4]

The setting mechanism of dual-cured resin cements is usually based on a redox reaction of benzoyl peroxide with aromatic tertiary amines (represented by catalyst and base paste, respectively). One or both pastes contain a light-sensitive compound (usually camphorquinone) that is responsible for initiating the light-cured setting mechanism. After the pastes are mixed, and until light is provided, working time is controlled by the ratio of inhibitors to peroxide and aromatic tertiary amines.[4] Both inhibitors and peroxides are organic chemical compounds that are susceptible to degradation upon storage; therefore, resin cements have a limited shelf life, and the setting mechanism of the cements may be influenced by changes in the chemistry of these components during storage time.[5] The implications of such changes on the mechanical properties of the resin cements are unknown; however, clinicians handling resin cements with altered working time (WT) and setting time (ST) may experience some clinical difficulties.

Chemical reactions are known to be generally accelerated by heat, and heat causes significant changes in the WT/ST of traditional and resin-based cements.[6,7] Moreover, a slower-setting resin cement is more likely to be affected by adverse reactions with acidic, permeable adhesive systems.[8,9] Consequently, the drawback with such materials and the chemical interactions resulting from polymerization and aging acting on physical properties becomes evident. Therefore, the aim of this study was to evaluate the effects of accelerated aging and temperature variation on the rheologic properties (WT/ST) of several dual-cured resin cements. The null hypothesis was that temperature variation during reaction or after accelerated aging would not affect the WT/ST of the materials.

METHODS AND MATERIALS

Six commercial resin cements were selected for this study (Table 1). One material is representative of an exclusive self-cure reaction, and the others are representative of dual-cure materials. The WT and ST were determined from the chemically initiated reaction of the materials alone, as no light activation was used. WT is defined as the period of time available for material manipulation and handling. ST starts as soon as the redox reaction (mixing the initiator and amine) is initiated and is the time the material takes to reach the limit viscosity, which corresponds to the formation of a solid mass and, consequently, a near complete polymerization.[10]

Preparation of the Samples

All specimens of each material were prepared from the same batches, and all materials were mixed according to the manufacturer's instructions. All handling and testing procedures were performed in a room with yellow light to prevent the ambient light from interfering in the reaction. Testing was performed with new resin cement kits within one month of manufacturing (checked against the printed expiration date), as the resin cements used in this study were received directly from the manufacturers and were used again after allowing the kits to age in a dark oven at 37°C.

The accelerated aging method was performed for 12 weeks, which corresponds to approximately 9 months of storage at room temperature.[11,12] It becomes necessary to use a mathematical formula to represent the period of correspondence between the accelerated aging and the period of considered normal storage. To estimate shelf life, it is necessary to multiply the elevated temperature period (3 months) by the acceleration factor (2.25), which will equal the accelerated time of approximately 6 or 7 months. The aging period will be estimated by the accelerated time (6 months) added to storage time (3 months), totaling and simulating a period of approximately 9 or 10 months of storage at room temperature.[11,12]

Catalyst and base pastes from each material were dispensed and proportioned (1:1 weight) in a precision digital balance (Bel Engineering, Onda Cientifica LTDA, Campinas, Brazil) before each data collection. Mixing was performed on mixing pads with plastic spatulas, except for the materials that used auto-mix tips per manufacturer's instruction.

Rheologic Measurements

The WT and ST were determined with the aid of a thermo-controlled oscillating rheometer (Sabri Dental Enterprises Inc, Downers Grove, IL, USA) coupled to a highly sensitive flatbed recorder with an electric pen lift and a chart paper with 10-mm

Table 1: *Six commercial resin cements selected for the study*

Resin Cement and Curing Process	Composition (Concentration Range %)[a]	Mixing Procedure per Manufacturer's Instructions [a]	Manufacturer and Lot Number [a]
C & B, chemically cured	*Base paste*: Bis-GMA (<21%), ethoxylated Bis-GMA (<16%), triethyleneglycol dimethacrylate (<11%), sodium fluoride (<4%)	Auto-mix tips provided by the manufacturer	Bisco Inc, Schaumburg, IL, USA
	Catalyst paste: Bis-GMA (<35%), triethyleneglycol dimethacrylate (<25%)		7E+08
Biscem, dual	*Base paste*: Bis-GMA (>10%), uncured dimethacrylate monomer (>20%), glass filler (>50%)	Auto-mix tips provided by the manufacturer	Bisco Inc, Schaumburg, IL, USA
	Catalyst paste: phosphate acidic monomer (>10%), glass filler (>50%)		7E+08
Calibra, dual	*Base paste*: barium boron fluoro alumino silicate glass (<70%), Bis-GMA (<20%), polymerizable dimethacrylate resins (<15%), hydrophobic amorphous silica (<5%)	Mix equal amounts of base and catalyst paste (1:1) for 20 to 30 s	Dentsply-Caulk, Milford, DE, USA
	Catalyst paste: barium boron fluoro alumino silicate glass (<70%), Bis-GMA (<20%), polymerizable dimethacrylate resins (<15%), hydrophobic amorphous silica (<5%), benzoyl peroxide (<5%)		Base: 070301 Catalyst: 060421
Duolink, dual	*Base paste*: Bis-GMA (<20%), triethyleneglycol dimethacrylate (<15%), urethane dimethacrylate (<18%), glass filler (<65%)	Auto-mix tips provided by the manufacturer	Bisco Inc, Schaumburg, IL, USA
	Catalyst paste: Bis-GMA (>20%), triethyleneglycol dimethacrylate (>20%), glass filler (>50%)		7E+08
Panavia F 2.0, dual	*Paste A*: 10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, DL-camphorquinone, catalyst, initiators[b]	Mix equal amounts of base and catalyst paste (1:1) for 20 s	Kuraray Medical Inc, Okayama, Japan
	Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments[b]		Base: A00246A Catalyst: B00039A
Variolink II, dual	*Base paste*: Bis-GMA (10% to 25%), urethane dimethacrylate (2.5% to 10%), triethyleneglycol dimethacrylate (2.5% to 10%)	Mix equal amounts of base and catalyst paste (1:1) for 10 s	Ivoclar-Vivadent, Schaan, Liechtenstein
	Catalyst paste: Bis-GMA (50% to 100%), urethane dimethacrylate (2.5% to 10%), triethyleneglycol dimethacrylate (2.5% to 10%), dibenzoyl peroxide (2.5%)		Base: K04431 Catalyst: K00889

Abbreviation: bis-GMA, bisphenol A glycidyl methacrylate.
[a] *According to manufacturer's information sheet.*
[b] *Ingredient composition not provided by the manufacturer.*

horizontal and vertical markers and a preset chart speed of 1 cm/min (Linseis Inc, Selb, Germany).

The chart recorder and a digital chronometer (Azula Marine, São Paulo, Brazil) were started exactly at the beginning of the mixing of the cements. After mixing per manufacturer's instructions, the material was transferred to the lower stage of the rheometer, which was preset at a temperature of 22°C or 37°C. The upper lever was lowered toward the material and reading continued until oscillation was completely restricted as detected in the chart, which was indicative of hardening of the material (Figure 1). Surface temperature of the oscillating rheometer was regularly monitored before and during the testing for each material with the aid of a laser thermometer (Neiko Tools USA,

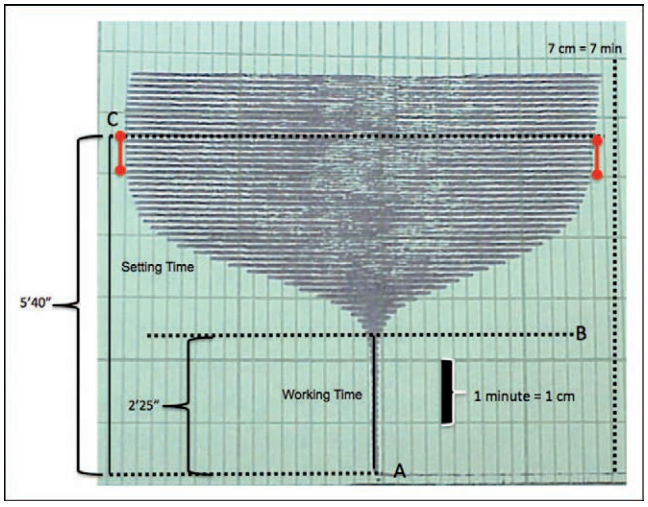

Figure 1. *Rheologic plot chalice chart illustrating the determination of working time/setting time.*

Homewood, IL, USA) pointed directly to the upper lever/lower stage interface and compared with the oscillating rheometer temperature indication to verify the accuracy of the readings. The WT and ST were determined directly from the chalice-like chart generated from each reading. The inflection points used, from the chart, to determine the WT and ST were 1) start of WT measurement during the begin of mixing per the manufacturer's instruction (vertical chart black line), 2) start of ST measurement (end of vertical chart black line), and 3) end of ST (restricted standardized horizontal chart lines indicative of material hardening; red line) (Figure 1).

Five mixes were prepared from each material for each of the reading temperatures, both before and after accelerated aging. The WT/ST results were expressed in minutes and seconds (x' y"±standard deviation) and relative percentage contribution of WT to the overall setting period. Repeated measures analysis of variance was used to analyze the mean differences between the effects of temperature and aging on WT and ST for each material and differences examined by Tukey test. All analyses were pre-established at $\alpha = 5\%$.

RESULTS

Both temperature and aging significantly affected WT and ST of the cements ($p<0.05$), but no significant interactions were observed ($p>0.05$). In general, the higher temperature always resulted in significant reduction of WT and ST for all materials, regardless of the aging condition (Table 2). Effects of aging varied widely among materials. Calibra, Panavia F 2.0, and Variolink II showed significant increases in both WT and ST after aging. Conversely, C&B and Duolink showed significant reductions in both WT and ST after aging. BisCem showed a significant reduction in WT but a significant increase in ST after aging. Panavia F 2.0 and Variolink II presented the longest WT and ST of all materials, regardless of temperature and aging. The relative percentages of WT over the entire ST of the cements were in the range of 52% to 63% for fresh cements cured at 22°C (Figure 2). Although the overall WT and ST were reduced at 37°C, it appeared that the increased temperature during setting affected WT and ST differently (Figure 2). That was even more evident for the aged groups (Figure 3).

Table 2: *Results of working time/setting time according to temperature and aging condition. Mean working time/setting time is expressed in minutes and seconds (x′y″) ± SD. n=5.**

Material	Temperature (°C)	Working Time		Setting Time	
		Fresh	Aged	Fresh	Aged
C & B	22	3′54″±12″	2′36″±5″	6′42″±1′54″	5′±1″
	37	1′54″±5″	1′30″±5″	3′54″±1′2″	3′1″±2″
Biscem	22	2′48″±5″	1′36″±5″	4′12″±1′2″	9′±1″
	37	1′54″±5″	54″±1″	3′48″±1′2″	6′54″±1′2″
Calibra	22	2′48″±5″	8′54″±3″	4′36″±1′2″	14′48″±2′1″
	37	1′24″±5″	4′1″±4″	2′30″±4″	8′42″±5″
Duolink	22	3′18″±4″	1′42″±1″	6′1″±3″	5′48″±1″
	37	1′54″±4″	54″±1″	3′36″±9″	2′48″±1″
Panavia F 2.0	22	8′42″±1′5″	19′48″±2′4″	13′18″±2′	34′1″±2′6″
	37	4′12″±1′1″	9′18″±1′1″	6′42″±1′2″	17′2″±1′1″
Variolink II	22	8′54″±3″	15′42″±1′2″	14′54″±1′2″	25′4″±3″
	37	3′48″±5″	6′36″±1′2″	6′36″±1′2″	10′54″±2′1″

** All comparisons between temperatures (22 vs. 37) and aging conditions (fresh vs. aged) were statistically significantly different for all materials ($p<0.05$).*

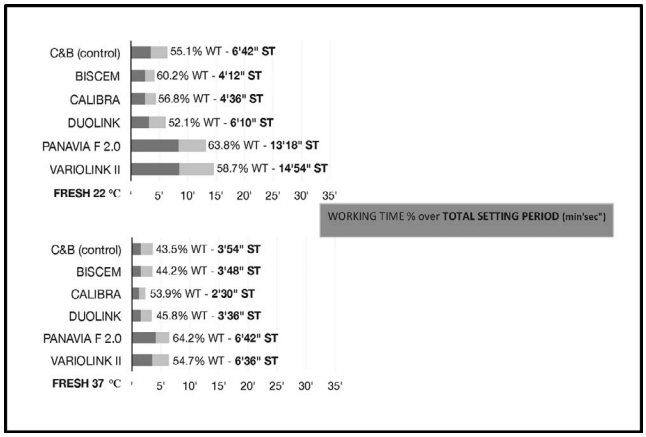

Figure 2. *Working time/setting time of fresh materials at 22°C and 37°C. Percent contribution of working time (dark portion of the bar) to the overall setting period of the materials.*

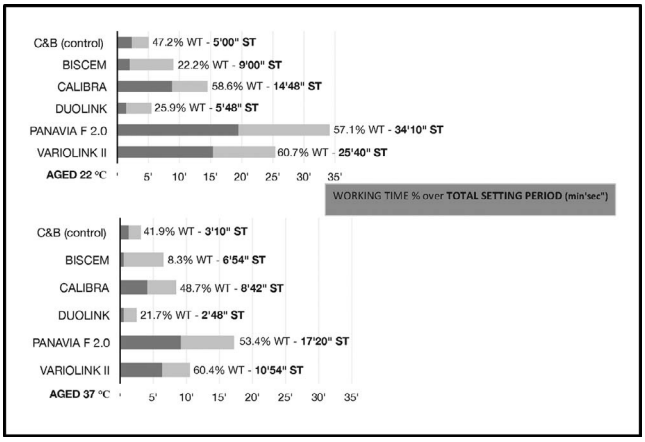

Figure 3. *Working time/setting time of aged materials at 22°C and 37°C: Percent contribution of working time (dark portion of the bar) to the overall setting period of the materials.*

DISCUSSION

The overall results demonstrate that the WT and ST of all tested cements were affected by temperature and aging. The anticipated null hypothesis should then be rejected. The WT for fresh materials ranged from 2′48″ to 8′54″ when maintained at 22°C. In general, a WT in the range of 1 to 2 minutes would be adequate for most of the luting procedures in clinical practice.[13] Panavia and Variolink had WTs above 8 minutes, but that should not be a clinical problem as both are dual-cure cements, and procedures can continue after cementation upon light activation of the cement. On the other hand, all other three dual-cured resin cements (Calibra, BisCem, and Duolink) showed WTs that were shorter than those for the self-cure C&B resin cement. This is of clinical importance because clinicians tend to believe dual-cure resin cements are preferable because they cure on demand and offer a longer WT. For all fresh materials, WT was roughly reduced by 50% when allowed to set at 37°C with most cements allowing less than 2 minutes before hardening takes place. This finding is in agreement with a recent study that also demonstrated a significant reduction in WT with an increase in temperature of dual-cured resin cements.[14] Considering that the materials will encounter body temperature when placed in the mouth, this may be of importance in such procedures as post cementations, for example. If the cement is first dispensed into the root canal and the insertion of the post is somehow delayed, body temperature will accelerate the reaction, so clinicians may encounter difficulties in inserting the post if hardening takes place. It is also important to note that clinicians may use the remaining material on the mixing pad to check for setting of the cement used to lute a crown, for example. It is not surprising that the cement on the mixing pad could still appear unset at room temperature while the portion in the mouth has already set because of the body temperature. Accelerated setting reaction of polymers when exposed to heat has also been demonstrated in other studies.[6,7,15] Of greater importance were the findings of WT after aging. It is a common belief that aging degrades the chemistry, thus resulting in slower reaction and longer WT. This, however, proved the opposite for C&B, BisCem, and Duolink, in which WT decreased significantly when materials were aged. Reductions in WT were evident for both temperatures, but dropped below one minute for BisCem and Duolink when setting at 37°C. Such a short WT may translate into clinical difficulties and limitation of procedures. Thus, it would be wise to check the working time of resin cements at chairside to prevent any unexpected shortened WT during a luting procedure, especially when the resin cement is not used often and is more than halfway to its expiration date. As expected, Calibra, Panavia, and Variolink resulted in increased WT after aging, and the latter two materials reached a WT in the range of 15 to 20 minutes when tested at 22°C, but not exceeding 10 minutes when tested at 37°C. Possible explanations as to why aging had different effects on the cements will be given later in the discussion.

With the exception of Panavia and Variolink, all other cements presented a ST that was within the clinically desirable range (4′00″ < ST < 7′00″). When heated to 37°C, ST dropped for all the materials, but never to a value that would compromise most clinical applications. Except for BisCem, which showed reduced WT and increased ST after

aging, all other materials followed the same trend for ST as for WT. For example, C&B and Duolink resulted in reduced ST, and Calibra, Panavia, and Variolink had an increased ST after aging. Of note was the markedly long ST for Panavia and Variolink. Although this long ST of the self-activated reaction is likely to be overlapped by the mandatory light activation of the material in the clinical set, areas where light cannot reach will probably undergo the delayed ST predicted in our findings. This may pose a more serious clinical implication whenever these cements are used in combination with a simplified, acidic adhesive. Adverse reactions and permeability issues between these adhesives and slow-setting, self-cure, or dual-cure resin cements are well known to dramatically compromise the bonding between the cement and the adhesive.[4,9,16-18] Variolink and Panavia have also been previously demonstrated to be highly dependent on light activation to achieve adequate properties.[4,19-28] It has been advised that these cements should only be used when clinicians can guarantee access to light exposure to ensure adequate curing.[4,22] As recently demonstrated, all-ceramic restoration thickness of 3 mm and above adversely affects the polymerization of dual-cured resin cements.[29] It was interesting to note that some resin cements presented a reduction in WT and ST after aging, others had the opposite behavior, and BisCem showed the odd reduction of WT in combination with an increase in ST. This different behavior can be explained by the chemistry involved in the formulation of the materials. The self-curing reaction is activated on mixing by the traditional acid-base reaction between benzoyl peroxide in the catalyst paste and aromatic tertiary amines in the base paste. Inhibitors usually control the speed of this reaction, and how well this chemistry functions determines WT and ST. The stability of WT and ST during aging is therefore dependent on the stability of these chemical compounds. It is well known that peroxides and inhibitors are not stable chemical compounds, as they are highly sensitive to heat. As the material ages during storage, particularly when exposed to heat, as in this study, both peroxides and inhibitors may undergo degradation.[30-32] The original concentration of peroxide and the ratio of inhibitors in the formula results in an unbalanced ratio over time because these chemicals degrade at different rates. If inhibitors degrade faster, peroxides will attack amines faster, and this will accelerate the reaction, reducing WT and subsequently ST if peroxides are still in active concentrations to react with all the remaining amines. This is likely what happened to C&B and Duolink. As BisCem presented a reduced WT and extended ST, it is likely that inhibitors degraded first, thus allowing for a faster reaction of peroxides with amines, but later the reaction slowed down to result in increased ST. It is possible that peroxides also degraded to a point that not enough amounts were available to react with amines and the reaction speed was reduced. We cannot rule out, however, that other mechanisms might be involved in this particular finding. BisCem was the only self-adhesive cement in the study, and these types of materials contain acidic monomers in their composition that might also play a role in governing the self-curing reaction. It is also interesting to note that the materials with similar behavior after aging are produced by the same manufacturer (Bisco). It is not known whether manufacturers tend to follow a standard chemistry when developing different products of the same class, but this coincidence warrants such speculation.

Conversely, it is likely that Calibra, Panavia, and Variolink had a faster degradation of the peroxides, which resulted in enhanced action of inhibitors, thus reducing the speed of reaction and increasing both WT and ST. It is probable that the chemistry involved in the determination and subsequent stability of the WT and ST of these resin cements is much more complex than the earlier explanation. This is beyond the scope of this study but warrants further investigation. Aging is an important clinical issue, and little literature is available that relates how it affects the clinical use of the products. Every product has an expiration date printed in the package. What is important to understand is that such shelf-life expectancy is determined by the manufacturer based on accelerated-aging in-house studies.[11,12] This means the actual detailed performance of the product has not necessarily been tested when estimating the shelf life, particularly when new products are launched. Moreover, as organic chemical compounds are expected to be unstable over time, changes in performance are naturally expected from a freshly made product versus an aged one. What is not clear is how such changes affect clinical handling and how clinical handling has to be adjusted to compensate for the changes. This study addressed these important issues to determine how unavoidable factors such as aging of the product and temperature differences may affect the clinical handling characteristics of WT and ST. Because alterations are material related, clinicians are

advised to take precautions when using these products. These could include accommodating purchases to permit the use of the freshest material possible and always storing peroxide-containing products in the refrigerator. Cold storage prolongs the stability of the chemistry and thus the stability of performance.

CONCLUSIONS

Both WT and ST were affected by temperature variation and aging condition. Some materials were demonstrated to have extended or shortened WT/ST periods after aging, despite temperature variation. Therefore, clinicians should be aware of such implications in clinical practice.

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Note

This study was developed as part of the requirements for Thiago Amadei Pegoraro to obtain a PhD degree in the graduate program at University of São Paulo, Bauru School of Dentistry, Sao Paulo, Brazil.

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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Effectiveness of Fluorescence-based Methods in Monitoring Progression of Noncavitated Caries-like Lesions on Smooth Surfaces

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Clinical Relevance

In this investigation, the authors observed that fluorescence-based methods were able to identify progressive enamel demineralization on smooth surfaces in the presence of biofilm.

SUMMARY

Although there has been a significant decrease in caries prevalence in developed countries, the slower progression of dental caries requires methods capable of detecting and quantifying lesions at an early stage. The aim of this study was to evaluate the effectiveness of fluorescence-based methods (DIAGNOdent 2095 laser fluorescence device [LF], DIAGNOdent 2190 pen [LFpen], and

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VistaProof fluorescence camera [FC]) in monitoring the progression of noncavitated caries-like lesions on smooth surfaces. Caries-like lesions were developed in 60 blocks of bovine enamel using a bacterial model of *Streptococcus mutans* and *Lactobacillus acidophilus*. Enamel blocks were evaluated by two independent examiners at baseline (phase I), after the first cariogenic challenge (eight days) (phase II), and after the second cariogenic challenge (a further eight days) (phase III) by two independent examiners using the LF, LFpen, and FC. Blocks were submitted to surface microhardness (SMH) and cross-sectional microhardness analyses. The intraclass correlation coefficient for intra- and inter-examiner reproducibility ranged from 0.49 (FC) to 0.94 (LF/LFpen). SMH values decreased and fluorescence values increased significantly among the three phases. Higher values for sensitivity, specificity, and area under the receiver operating characteristic curve were observed for FC (phase II) and LFpen (phase III). A significant correlation was found between fluorescence values and SMH in all phases and integrated loss of surface hardness (ΔKHN) in phase III. In conclusion, fluorescence-based methods were effective in monitoring noncavitated caries-like lesions on smooth surfaces, with moderate correlation with SMH, allowing differentiation between sound and demineralized enamel.

INTRODUCTION

Although there has been a significant decrease in caries prevalence in children from most developed countries, dental caries make up one of the most prevalent oral diseases.[1] In recent decades, major changes have occurred in the pattern of dental caries due to the widespread use and availability of fluoride. Thus, the slower progression of caries lesions[2] requires methods capable of detecting and quantifying lesions at an early stage.[3]

The early detection of smooth-surface caries lesions is important for determining the appropriate management and monitoring of dental caries at a time when preventive measures could still be introduced.[4] It is known that conventional methods for caries detection are subjective[5] and not capable of quantifying the mineral loss caused by the disequilibrium in the process of demineralization and remineralization of hard dental tissues.[6]

Thus, noninvasive quantitative methods have been evaluated to detect lesions at an initial stage and subsequently monitor lesion changes over time.[3] Fluorescence methods have received considerable attention as technology-based approaches to caries detection because bacterial porphyrins and other chromophores present on the demineralized dental tissues emit fluorescence when excited by a light source with a specific excitation wavelength.[3]

The laser fluorescence devices DIAGNOdent 2095 (LF, KaVo, Biberach, Germany) and DIAGNOdent 2190 pen (LFpen, KaVo) are based primarily on fluorescence absorption by bacterial by-products in porous carious lesions when the surface is illuminated by the device's diode laser with a wavelength of 655 nm.[3] Some studies have evaluated the performance of the LF and LFpen devices in detecting or monitoring caries development on smooth surfaces, with contradictory results.[7-16]

The intraoral fluorescence camera (FC, VistaProof, Dürr Dental, Bietigheim-Bissingen, Germany) was developed for the detection of caries and emits blue light at 405 nm to capture and digitalize images from the teeth while they are emitting fluorescence.[17] In incipient carious lesions, red porphyrin fluorescence is emitted, whereas such fluorescence is not emitted by sound enamel.[18] However, there is little evidence on the FC device's efficacy in detecting caries lesions on smooth surfaces. An *in vitro* study has shown good reliability of the FC device in detecting caries on smooth surfaces, similar to the reliability shown by the LF and LFpen devices.[19] In more recent studies, however, the FC device showed poor effectiveness in detecting demineralization and remineralization on smooth surfaces.[15,16]

Fluorescence-based methods have been proposed to aid caries detection, as they can offer objective assessments of the carious process.[20] However, there are still many questions regarding their performance when evaluating smooth surfaces. To date, no study has evaluated the efficacy of the LF, LFpen, and FC in monitoring the progression of caries lesions on smooth surfaces.

Therefore, the aim of this *in vitro* study was to evaluate the effectiveness of fluorescence-based methods (LF, LFpen, and FC) in monitoring progression of noncavitated caries-like lesions developed using a bacterial model. The null hypothesis is that there is no difference between the results obtained using the three different fluorescence-based devices on smooth surfaces.

METHODS AND MATERIALS

Sample and Specimen Preparation

Two hundred enamel blocks (4 × 4 × 2 mm) were obtained from bovine incisors and were stored frozen at −20°C. Each block was embedded in epoxy resin in order to expose only the buccal surface. This procedure was performed to allow polishing of the enamel surface as needed for an appropriate SMH analysis since such evaluation requires a stable specimen during the indentation process.[4]

All blocks were then stored individually at 100% humidity. The enamel surface was then sequentially polished with carbide papers (600, 1200, and 1500 grit) and diamond abrasive on a polishing paper, resulting in the removal of about 100 μm of the outer enamel, which was checked with a micrometer. Surface microhardness (SMH) analysis was performed using a microhardness tester (HMV-2, Shimadzu Corp, Tokyo, Japan) with a Knoop diamond under a 25-g load for five seconds.[6] Five indentations spaced 100 μm apart were made, and their average was recorded. Of the original 200 enamel blocks, only 120 with hardness of 310.9 ± 25.5 Knoop hardness (KHN) were selected. Each of the 120 enamel blocks was randomly allocated into one of two groups: control (n=60) or experimental (n=60).

The 60 enamel blocks in the experimental group were used to evaluate the effectiveness of fluorescence-based methods in monitoring the development of noncavitated caries lesions on smooth surfaces.

Experimental Design

This *in vitro* study involved three phases of treatment of enamel blocks in the experimental group: baseline (phase I), after the first cariogenic challenge (phase II), and after the second cariogenic challenge (phase III).

Measurement With Fluorescence-Based Methods

Each enamel block was assessed by two examiners using LF, LFpen, and FC. The examiners had experience handling the devices and had participated in previous published studies. The enamel blocks were removed from the 100% humidity storage environment, fixed in clear acrylic resin disks, and dried with a paper tissue.[6]

The LF and LFpen measurements were performed using a fiber-optic conical tip (tip B), specifically designed for smooth surfaces, and a cylindrical sapphire fiber tip, respectively, according to the manufacturer's instructions. Before each measurement, the devices were calibrated against a ceramic standard and were recalibrated after testing 10 blocks.[6,21] After calibration, the laser point was placed in the center of each enamel block and swept across the surface. The maximum fluorescence value detected by the devices was recorded. Each block was dried with a paper tissue and air-dried for five seconds and analyzed three times consecutively by each examiner, after which the mean values were calculated.[4,16]

The FC measurements were performed in a dark environment to block external light when examining the enamel blocks. The tip of the device was placed perpendicular to the enamel surface using a distance holder. After capturing the images of the enamel blocks, they were analyzed by the FC-specific software (DBSWIN, Dürr Dental), translating the red and green rate of fluorescence to numbers that correspond to the lesion severity.[17] The maximum value displayed by each sample was recorded for further analysis (Figure 1). The FC measurements were also done three times by each examiner, and the mean values were calculated.[16] The FC images were taken twice with a one-week interval.

Cariogenic Challenge

The enamel blocks in the experimental group (n=60) were used for the development of caries-like lesions using a bacterial model adapted from previous studies[22,23] and assessed by a previous study.[24] The experimental period for initial assessment of caries lesions was determined at eight days (phase II) and the second assessment after a further eight days (phase III).

Each enamel block was coated with a layer of epoxy adhesive and a layer of acid-resistant varnish, except for the buccal surface, leaving exposed a 16-mm^2 enamel window. Then each block was individually attached to orthodontic wire to allow the free enamel window to be immersed in 25 mL of distilled water in a Falcon tube without touching the tube walls and autoclaved at 121°C for 20 minutes. The 60 enamel blocks in the control group were used to evaluate the influence of the autoclave sterilization process on the enamel SMH.

The microorganisms used in this study were *Streptococcus mutans* (ATCC 25175) and *Lactobacillus acidophilus* (ATCC 4356). The organisms were grown overnight in brain heart infusion broth (Difco Laboratories, Detroit, MI, USA) under anaerobic

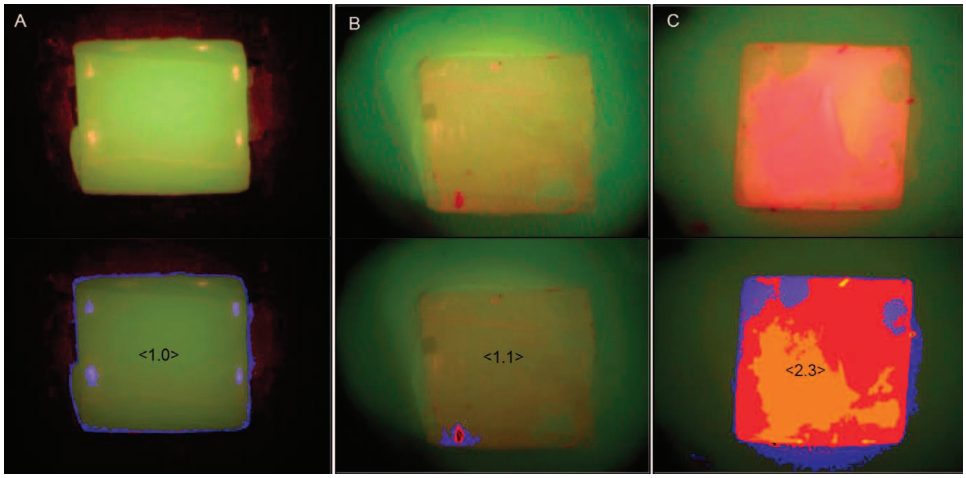

Figure 1. *Fluorescence images taken with an FC device from the buccal surface of an enamel block in all phases of the study. Upper pictures represent the digitalized images from the surface while it is emitting fluorescence. Lower pictures represent the fluorescence images analyzed by the FC-specific software DBSWIN. (A): At baseline (FC value = 1.0). (B): After the first cariogenic challenge (FC value = 1.1). (C): After the second cariogenic challenge (FC value = 2.3).*

conditions at 37°C. To standardize the inoculum density, a visible turbidity of McFarland 0.5 standard (equivalent to a bacterial quantity of 1.5×10^8 colony-forming units/mL) was applied. In addition, Gram staining was performed in order to differentiate between the two species of microorganism.

After sterilization, each enamel block was removed from the distilled water and transferred aseptically to another Falcon tube filled with 25 mL of a modified artificial caries solution (brain heart infusion culture medium supplemented with yeast extract, 0.5% glucose, 1% sucrose, and 0.5% young primary culture broth of *S. mutans* and *L. acidophilus*). The enamel blocks were incubated at 37°C in a candle jar. Every 48 hours, the specimens were transferred to another Falcon tube containing a new sterile artificial caries solution for a continuous cariogenic fresh supply.[22,23] The pH of the medium decreased from 7.0 to 4.5 within 48 hours of bacterial inoculation.

At the end of eight days (phase II), the biofilm was removed from the enamel blocks with sterile gauze, and the blocks were then washed with deionized water for 60 seconds. The samples exhibited a dull, whitish change resembling opacity under the microscope. SMH and fluorescence-based measurements were obtained. Then the enamel blocks exhibiting incipient caries lesions were autoclaved again and submitted to cariogenic challenge for a further eight days (phase III) in order to simulate the progression of caries lesions in the enamel. At the end of the experimental period, SMH and fluorescence-based measurements were obtained again. Steam autoclaving is effective for sterilizing the enamel to be used in cariogenicity tests and does not interfere with the demineralization pattern.[25]

Cross-Sectional Microhardness

After SMH analysis, all enamel blocks were longitudinally sectioned through the center of the exposed enamel for cross-sectional microhardness (CSMH) determination. Half of each block was embedded in acrylic resin, and the cut surfaces were exposed and polished. CSMH was determined according to the method described by Spiguel and others[6] using a Knoop indenter with a 25-g load for five seconds (Shimadzu HMV-2). Three rows of eight indentations at 10, 30, 50, 70, 90, 110, 220, and 330 μm from the outer enamel surface were made: one row in the central region of the exposed enamel and the other two spaced 100 μm from the first (one of these rows was 100 μm above the first row, and the second was 100 μm below it). The mean value of each distance indentation was calculated.[16]

Integrated hardness (KHN × μm) of sound and demineralized enamel was calculated to a depth of 220 μm using the trapezoidal rule[26] (GraphPad Prism, version 3.02, GraphPad Software Inc, La Jolla, CA, USA). The integrated loss of hardness (ΔKHN) was calculated by subtracting the demineralized integrated hardness from the integrated hardness of sound enamel.[6]

Polarized Light Microscopy

The other half of each enamel block was cut into sections of approximately 500-μm thickness using a diamond saw. The sections were then manually ground and polished to a thickness of 100 μm, mounted on slides with distilled/deionized water, and covered with a cover glass. The sections were examined by polarized light microscopy (Leica DM750, Leica Microsystems, Wetzlar, Germany) at 400× magnification. Three areas in the central

regions of the sections were analyzed by recording the thickness of the superficial enamel layer and the depth of the lesion using ImageJ 1.38x software (National Institutes of Health, Bethesda, MD, USA).[6,16]

Control Group

In order to evaluate the influence of the autoclave sterilization process on the enamel surface microhardness, the enamel blocks (n=60) were autoclaved using the same intervals used for the experimental group. There was no development of caries-like lesions in this group. There were three experimental phases in the control group: baseline (phase I), after the first autoclave process (phase II), and after the second autoclave process (phase III).

First, the enamel blocks were autoclaved at 121°C for 20 minutes and stored in distilled/deionized water for eight days (phase II). SMH and fluorescence-based measurements were obtained after this period. The enamel blocks were then autoclaved once more and stored in distilled/deionized water for a further eight days (phase III). SMH and fluorescence-based measurements were obtained again at the end of this period.

Statistical Analysis

The data were analyzed using the statistical software MedCalc for Windows, version 12.3.0 (MedCalc Software, Mariakerke, Belgium), and the level of significance was α=0.05. Outcome variables were the mean values of LF, LFpen, FC, SMH, and ΔKHN and the phases (I, II, and III) as variation factors.

The intraclass correlation coefficient (ICC) was used to assess intra- and interexaminer reproducibility for fluorescence-based methods. The ICC was considered poor when the values were below 0.40, fair for values between 0.40 and 0.59, good for values between 0.60 and 0.75, and excellent for values above 0.75.[27]

The Kolmogorov-Smirnov test was used to verify the normal distribution of the data. Nonparametric tests for paired samples were used because of the lack of normality of the data. In order to compare the three phases of the experiment for measurements made using SMH and fluorescence-based methods, the nonparametric Friedman test and multiple comparison tests were performed. The Wilcoxon test was used to compare the integrated hardness of sound and demineralized enamel.

The percentage change of SMH (%SMHC), determined in relation to the baseline measurement, was calculated for each enamel block according to the method of Cury and others: %SMHC = (SMH after demineralization − baseline × 100)/baseline.[26] The Mann-Whitney test was used to compare the %SMHC between phases II and III.

A receiver operating characteristic (ROC) analysis was conducted to evaluate the performance of each fluorescence-based method in monitoring the development of caries lesions in enamel. ROC analysis is a good statistical approach for methods with numerical values. The sensitivity is plotted as a function of 1 − specificity for various possible cutoff points. The area under the ROC curve (Az) can be calculated for each method, and the closer the curve is to the upper left corner, the greater is the overall accuracy of the test. With ROC analysis, the best cutoff points for discriminating between sound and carious teeth can be also calculated.[28] Thus, Az values and cutoff limits for differentiating between carious and sound teeth were calculated for each method in phases II and III. With these optimal cutoff points, sensitivity and specificity were also calculated for each method. The comparison between these values was performed by the McNemar test. In the present study, enamel blocks submitted to the cariogenic challenge were considered as carious ones (presence of noncavitated lesion), while enamel blocks that had not yet been submitted to the cariogenic challenge were considered as sound ones (absence of lesion).

Spearman's rank correlation coefficient (rho) was used to test the strength of a relationship between the different fluorescence-based methods and SMH, considering all phases or ΔKHN and lesion depth (μm), in phase III. The Spearman coefficient varies between −1 and 1; the closer these extremes, the greater the association between variables.

RESULTS

Table 1 represents the intra- and interexaminer reproducibility assessed by calculating ICC for LF, LFpen, and FC for the experimental group in all three phases. ICC values for intra- and interexaminer reproducibility indicated fair to excellent agreement for the fluorescence-based methods in phases I and II and good to excellent agreement in phase III.

With respect to the experimental group, the fluorescence-based methods showed significant differences between the three phases, with the highest values being recorded for phase III ($p<0.05$; Table 2). With regard to the SMH analysis of the enamel blocks, statistically significant differences ($p<0.05$)

Table 1: *Intraclass Correlation Coefficient (ICC) for Intra- and Interexaminer Reproducibility for LF, LFpen, and FC in the Experimental Group for All Phases of Treatment (n=60)*

Phase	Method	ICC (95% confidence interval)		
		Intraexaminer reproducibility		Interexaminer reproducibility
		Examiner A	Examiner B	
I	LF	0.93 (0.88-0.96)	0.87 (0.79-0.92)	0.61 (0.44-0.73)
	LFpen	0.85 (0.75-0.91)	0.84 (0.74-0.91)	0.70 (0.57-0.79)
	FC	0.76 (0.60-0.86)	0.74 (0.56-0.84)	0.49 (0.27-0.64)
II	LF	0.65 (0.41-0.79)	0.86 (0.77-0.92)	0.53 (0.32-0.67)
	LFpen	0.76 (0.60-0.86)	0.89 (0.81-0.93)	0.87 (0.81-0.91)
	FC	0.51 (0.18-0.71)	0.66 (0.43-0.80)	0.74 (0.63-0.82)
III	LF	0.87 (0.79-0.92)	0.94 (0.90-0.97)	0.90 (0.85-0.93)
	LFpen	0.92 (0.86-0.95)	0.94 (0.90-0.97)	0.93 (0.90-0.95)
	FC	0.90 (0.83-0.94)	0.82 (0.69-0.89)	0.82 (0.75-0.88)

were observed between the three phases, with the lowest values being recorded for phase III. The %SMHC was statistically significant between phases II and III ($p<0.05$). Integrated hardness (KHN×μm) was significantly different between sound enamel (88.101±10.489) and demineralized enamel (42.335±14.598) (Wilcoxon test, $p<0.05$). Integrated loss of hardness (ΔKHN) was 45.766.6 ± 16.067.6. With respect to the control group, there was no difference in the fluorescence values for LF, LFpen, FC and SMH values between all the three phases ($p>0.05$) (Table 2).

Table 3 shows sensitivity, specificity, area under the ROC curve (Az), and cutoff points for LF, LFpen, and FC in phases II and III of the treatment in the experimental group. Applying the best cutoff points to differentiate between sound and carious teeth, it was observed that FC and LFpen demonstrated statistically higher sensitivity, specificity, and Az values in phases II and III, respectively. In addition, LF showed the lowest values in phase II, and LF and FC presented similar values in phase III.

Spearman's rank correlation coefficients (rho) are shown in Table 4. There was a significant negative correlation between SMH and fluorescence values in phase I and a significant positive correlation between SMH and fluorescence values in phases II and III ($p<0.05$). There was also a significant positive correlation between fluorescence values and ΔKHN in phase III ($p<0.05$). No correlation was observed between fluorescence values and lesion depth ($p>0.05$). The highest correlation was found for LFpen in phase II (rho = 0.380), meaning that the higher the SMH values after the first cariogenic challenge, the higher the LFpen measurements.

Figure 1 shows the digital and the fluorescence images taken with the FC device from the surface of an enamel block in all phases of the study. An increase in fluorescence values was observed in each consecutive phase.

Figure 2 shows a polarized light photomicrograph after the second cariogenic challenge (phase III). The demineralized enamel seems dark under polarized

Table 2: *Fluorescence Values, Surface Microhardness (SMH) Values, and Percentage of SMH Change (%SMHC) in the Experimental (n=60) and Control (n=60) Groups for All Phases of Treatment*[a]

Group	Phase	Mean ± standard deviation				
		LF*	LFpen*	FC*	SMH* (KHN)	%SMHC**
Experimental	I	7.8 ± 2.5 A	15.9 ± 4.8 A	1.0 ± 0.0 A	310.9 ± 25.5 A	—
	II	12.3 ± 7.0 B	30.0 ± 11.3 B	1.2 ± 0.1 B	104.1 ± 43.6 B	−66.4 ± 5.0 A
	III	24.8 ± 10.7 C	60.2 ± 20.2 C	1.6 ± 0.3 C	43.6 ± 10.1 C	−85.9 ± 3.5 B
Control	I	6.8 ± 2.3 A	14.8 ± 4.5 A	1.0 ± 0.0 A	305.7 ± 45.4 A	—
	II	6.4 ± 2.0 A	14.9 ± 4.3 A	1.0 ± 0.0 A	305.4 ± 42.5 A	—
	III	6.9 ± 2.3 A	14.1 ± 4.9 A	1.0 ± 0.0 A	306.1 ± 43.2 A	—

[a] *Significant differences are represented by different letters within the same column (*Friedman and multiple comparison tests/**Mann-Whitney test;* $p<0.05$).

Table 3: *Sensitivity, Specificity, Area Under the Receiver Operating Characteristic Curve (Az), and Cutoff Points for LF, LFpen, and FC in the Experimental Group for Phases II and II of the Treatment (n=60)*[a]

Phase	Method	Sensitivity	Specificity	Az	Cutoff points
II	LF	0.68 A	0.69 A	0.757 A	>8
	LFpen	0.75 B	0.90 B	0.889 B	>21
	FC	0.99 C	0.88 B	0.983 C	>1
III	LF	0.89 A	0.98 A	0.983 A	>13
	LFpen	0.98 B	0.96 B	0.997 B	>25
	FC	0.87 A	0.99 A	0.989 A	>1.2

[a] *Significant differences are represented by different letters within the same column (McNemar test; p<0.05).*

light microscopy. The mean lesion depth was 103.9 ± 29.3 μm.

DISCUSSION

The present study evaluated the effectiveness of fluorescence-based methods in monitoring the development of noncavitated caries-like lesions on smooth surfaces. To our knowledge, this is the first study to use a bacterial model for caries generation to evaluate the ability of the LF, LFpen, and FC devices to detect initial caries-like lesions in enamel and monitor their progression. It should be emphasized that conventional methods for the detection of caries lesions do not comply with the criteria for an ideal caries detection method because they rely on subjective interpretation and are insensitive to early caries detection. Therefore, methods capable of detecting and quantifying early caries lesions are required as adjunct tools in clinical practice, allowing preventive intervention before irreversible destruction of tooth substance occurs.[3]

It was verified that the *in vitro* methodology used was capable of forming caries-like lesions in enamel using a bacterial model composed of *S. mutans* and *L. acidophilus*, simulating the process of dental caries.[24,29] The bacterial model offers the opportunity to evaluate the variability and exchangeability of the species involved in the carious process and factors such as lesion site or availability of fermentable carbohydrates.[30-32]

To evaluate the effectiveness of fluorescence-based methods for monitoring enamel caries lesions, an *in vitro* model using bacterial films is likely to be more realistic than chemical systems since the devices have the potential to identify bacterial metabolites such as porphyrins (fluorophores and other chromophores) produced by cariogenic bacteria.[3,17-19,33,34] Even though some studies have shown that cultures of selected oral bacteria, such as *S. mutans* and *Lactobacillus* species, seem to show no typical porphyrin fluorescence,[5,36] other studies demonstrated that *S. mutans* induced enamel lesions exhibited increased fluorescence in the red region.[37,38] Fluorescent properties of dental caries can be attributed to tissue demineralization and an increase in bacterial flora and its metabolism.[39,40] In addition, pH seems to have an important influence

Table 4: *Spearman's Rank Correlation Coefficient (rho) Between Fluorescence-Based Methods and Surface Microhardness (SMH), integrated loss of hardness (ΔKHN), and Lesion Depth (μm) in the Experimental Group for All Phases of Treatment (n=60)*[a]

Phase	Method	Spearman's rank correlation coefficient		
		SMH	ΔKHN	Lesion depth (μm)
I	LF	−0.235*	—	—
	LFpen	−0.328*	—	—
	FC	−0.198*	—	—
II	LF	0.328*	—	—
	LFpen	0.380*	—	—
	FC	0.191*	—	—
III	LF	0.297*	0.224*	0.091 ns
	LFpen	0.343*	0.246*	0.110 ns
	FC	0.356*	0.285*	0.114 ns

[a] *Variables statistically correlated.*
* *p<0.01.*
Abbreviation: ns, not significant.

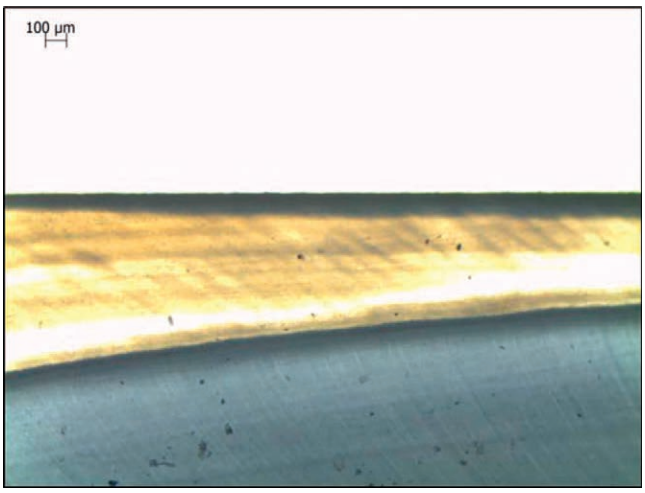

Figure 2. *Polarized light micrograph of an enamel block after the second cariogenic challenge. (Magnification = 400×). The demineralized enamel seems dark on the top surface.*

on the light absorption and emission of porphyrins.[41] In the present investigation, it could be suggested that the red fluorescence recorded by the devices was probably due to the synergistic effects between the carious process and the bacterial species and their environments.

In the present study, CSMH analysis was used in combination with polarized light microscopy to determine lesion mineral distribution and area of demineralization. Other studies on enamel demineralization have also used CSMH profiling for validation and evaluated the artificial caries lesion depth in a polarized light microscope.[6,16,24,42] It is important to point out that researchers have used a considerable number of analytical techniques to quantify changes in the mineral content of enamel during caries lesion formation.[43] Transverse microradiography (TMR) provides a quantitative measure of the mineral content, lesion depth, and attributes of the surface layer in enamel lesions.[44] CSMH measures the mechanical resilience (physical strength) of enamel, which cannot be determined by TMR.[45] According to ten Bosch and Angmar-Mansson,[46] SMH is a noninvasive technique and does not damage the enamel's macrostructure. SMH can be used in caries research, as it measures an important parameter with structural integrity and mechanical properties that cannot necessarily be derived from TMR variables.[43] The results of superficial microhardness analysis after the first and second cariogenic challenges showed that KHN values were significantly lower than the baseline values, demonstrating that the present methodology was able to create and cause progression of caries-like lesions, a finding confirmed by ΔKHN and polarized light microscopy. These results corroborate those of Spiguel and others[6] and Moriyama and others,[16] who used *in situ* methodologies to create caries-like lesions. It is known that polarized light microscopy is a good method for assessing lesion depth, and CSMH is an indirect method for the assessment of mineral loss in smooth-surface caries lesions.[47]

It should be emphasized that a caries detection method should offer good reproducibility, allowing consistent and reliable results to be produced by different evaluations and examiners. According to Lussi and Hellwig,[34] a high level of agreement for the LF device means that it could be useful for monitoring the carious process. In general, good to excellent agreement was observed for LF and LFpen in all phases for both intra- and interexaminer reproducibility, except for LF in phase II (interexaminer reproducibility). These results confirm Aljehani and others,[13] Bahrololoomi and others,[14], Moriyama and others,[16] and De Benedetto and others,[19] who also observed good to excellent reproducibility values for LF and LFpen devices for caries detection on smooth surfaces. In contrast, fair to good agreement was found for the LF device for intraexaminer reproducibility.[4] These differences might be attributed to the experimental design, which was an *in vitro* study using a buffer system to produce caries-like lesions, and to subjective errors in the measurements.

Generally, the FC device also showed good to excellent agreement for intra- and interexaminer reproducibility, supporting the findings of De Benedetto and others.[19] Fair interexaminer agreement at baseline might be explained by the specimen size and procedure for polishing of the enamel surfaces, which reflected the six light-emitting diode (LED) light sources, interfering with image capturing and fluorescence analysis by the different examiners.[16]

To date, no information about the effectiveness of different fluorescence-based methods in monitoring the demineralization process on smooth surfaces is available. In this study, the LF, LFpen, and FC fluorescence values showed significant differences between the three phases. This suggests that the devices were able to identify progressive enamel demineralization on smooth surfaces in the presence of biofilm.[3,6] In other words, an increase in fluorescence values might be attributed to bacterial endogenous porphyrins and related compounds present in enamel caries lesions. Moriyama and others[16] and Spiguel and others[6] also observed differences in fluorescence values after *in situ* enamel demineralization on smooth surfaces. The LF device showed a significant increase in fluorescence values after artificial demineralization, different from the *in vitro* studies of Mendes and others[21] and Diniz and others,[4] in which the caries-like enamel lesions were induced without oral bacteria and no differences found between sound and demineralized enamel for the LF values.

It was observed that the fluorescence values obtained with the LF and LFpen devices were different in all phases of this study. The LFpen fluorescence values were higher than the values recorded with the LF device, corroborating the findings of previous studies.[16,17,20,48] This finding could be attributed to the smaller diameter and architecture of the LFpen tip. Also, inside the tip of the LFpen device, the excitation and fluorescence follows the same optical path of propagation in

opposite directions, which is different in the LF device.[34]

When assessing the performance of fluorescence-based methods for caries detection, the cutoff points recommended by the manufacturers should be discussed because they could affect treatment decision making in clinical practice. It was observed that changes in LF fluorescence values at baseline and after the first and second cariogenic challenges were within the cutoff points proposed by the manufacturer. The manufacturer proposed that values of 0-10 indicate sound teeth and that values of 11-30 indicate enamel caries. In contrast, the values obtained with LFpen fluorescence did not fit within the cutoff points proposed by KaVo, which state that values of 0-14 indicate a healthy surface and that values of 15-20 indicate enamel caries. According to the manufacturer, the fluorescence values observed in phases II and III with the LFpen would indicate dentin caries lesions, and operative and preventive care is advised. Moriyama and others[16] observed that changes in LF values at baseline and in LFpen values at baseline and after *in situ* demineralization were within the cutoff points proposed by the manufacturer. These differences might be attributed to the different degree of demineralization obtained by the different methodologies. The present study was conducted *in vitro* using a bacterial model with no mimicry of the diverse conditions present in the oral cavity that might affect development of dental caries.

In regard to the FC device, according to the manufacturer, the numbers between 0.0 and 1.0 represent a healthy tooth, values from >1.0 to 1.5 indicate incipient enamel caries, and values from >1.5 to 2.0 indicate deep enamel caries. For Diniz and others,[20] values between 0.0 and 1.0 indicated a sound surface, and those between 1.1 and 1.2 indicated enamel lesion. In the present study, the FC values were statistically different between the phases and lend support to the cutoff points proposed by the manufacturer, allowing monitoring of the development of enamel caries on smooth surfaces. On the other hand, Moriyama and others[16] obtained FC values very close to each other at baseline and after *in situ* demineralization, making it difficult to monitor incipient caries lesions. It should be stressed that care must be taken in choosing to adopt the cutoff points proposed by the manufacturer and by other studies for interpreting the FC fluorescence values.

The VistaProof FC is a system with blue LEDs emitting at 405 nm (blue-violet light), and it is similar in design to the quantitative light-induced fluorescence (QLF) system, presenting the same excitation wavelength. The QLF device is considered a valuable instrument for early caries detection, capable of monitoring demineralization and remineralization and quantifying changes in the mineral content of noncavitated lesions. The fluorescence image of incipient caries lesions is digitized, and the fluorescence loss is quantified in comparison to the fluorescence radiance level of sound enamel. Three parameters are analyzed: fluorescence loss (ΔF; %), area of the lesion (A; mm^2), and fluorescence loss integrated over the lesion area (ΔQ; $\Delta F \times A$; $\% \times mm^2$).[49-51] The only significant difference is that QLF measures mainly the loss of intrinsic fluorescence of the dental enamel caused by demineralization, and VistaProof fluorescence camera is based on the increase in fluorescence of carious tissues due to the presence of bacterial metabolites, such as porphyrins.[17-19]

The results obtained from the control group verified that fluorescence and SMH values were statistically similar among the three phases of the present investigation. Thus, it was demonstrated that the autoclave sterilization process did not influence the fluorescence values and enamel surface microhardness. Parsell and others[52] reported that steam sterilization did not interfere with the enamel hardness of extracted teeth. By contrast, Chandler[53] showed by microhardness testing before and after autoclaving that some modification of enamel does occur under the influence of moist heat, pressure, and air drying. The differences found in Chandler's study[53] might be related to the autoclaving process, which was performed for five minutes at 132°C followed by air drying at subatmospheric pressure for 10 minutes.

According to the results of the ROC analysis, the optimal cutoff points to indicate initial enamel caries were >8 (LF), >21 (LFpen), and >1.0 (FC), and cutoff points to indicate deep enamel caries were >13 (LF), >25 (LFpen), and >1.2 (FC). With these cutoff points, the sensitivity and specificity values were high. Fluorescence-based methods were observed to perform well in monitoring the development of enamel caries lesions. The highest area under the ROC curve was found for FC in phase II and for LFpen in phase III. Mendes and Nicolau[10] and Mendes and others[11] reported good performance of the LF device in detecting incipient caries lesions since the area under the ROC curve was >0.8. Diniz and others[20] also described areas >0.9 for LF and LFpen devices in detecting occlusal caries lesions in

permanent molars. After the first cariogenic challenge, LF and LFpen devices showed lower values for sensitivity and specificity, whereas after the second cariogenic challenge, the values were greater. These findings support those of previous research by Mendes and others,[11] who observed that the less developed the caries lesions on a smooth surface, the worse the performance of the LF device. Thus, at inner enamel caries, the performance was better than at outer enamel caries in primary teeth.

There was a significant moderate correlation between SMH and fluorescence values for all methods in all phases of the study and between ΔKHN and fluorescence values after the second cariogenic challenge. At baseline, the correlation between SMH and fluorescence values was negative, indicating that higher SMH measurements lower the fluorescence values. These results show that the devices were able to monitor the development of noncavitated enamel lesions, in agreement with the results for the LF device reported by Mendes and Nicolau.[10] Spiguel and others[6] described a positive significant correlation between ΔKHN and LF values after *in situ* demineralization, as verified for LF, LFpen, and FC devices in the present investigation. Conversely, Moriyama and others[16] found no significant correlation between fluorescence values and SMH at baseline and after demineralization and between ΔKHN and fluorescence values after demineralization. These differences might be attributable to the *in situ* methodology used by Moriyama and others[16] to create caries-like lesions of the enamel. Also in line with Moriyama and others,[16] the present study found no significant correlation between fluorescence values and lesion depth. On the other hand, Mendes and others[11] found a good positive correlation between LF values and lesion depth on smooth-surface natural caries in primary teeth.

It is important to emphasize that fluorescence-based methods should be considered as adjunct tools to the visual examination for caries detection and monitoring of smooth surfaces.[16] A systematic review and meta-analysis have shown that fluorescence-based devices have similar overall performance; however, better accuracy in detecting more advanced caries lesions has been observed.[54] Further *in vivo* studies are needed to elucidate the efficacy of fluorescence-based methods in monitoring the development of enamel caries lesions on smooth surfaces.

CONCLUSION

It can be concluded that fluorescence-based devices were effective in monitoring the development of noncavitated caries lesions on smooth surfaces *in vitro*, using a bacterial model for caries induction. The FC device showed good performance with regard to indicating incipient noncavitated caries lesions, while the LFpen device performed better at indicating deep noncavitated caries lesions. The fluorescence values showed significant moderate correlation with SMH, allowing differentiation between sound and demineralized enamel.

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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 Cruzeiro do Sul University–UNICSUL.

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 Selective Etch on the Bond Strength of Composite to Enamel Using a Silorane Adhesive

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Clinical Relevance

The selective etch of enamel with phosphoric acid may improve the bond strength of silorane adhesives.

SUMMARY

An improvement in bond strength to enamel has been demonstrated with the use of phosphoric acid prior to bonding with self-etch methacrylate-based adhesive agents. No research has evaluated the effect of phosphoric-acid etching of enamel with a newer self-etch silorane adhesive. The purpose of this study was to evaluate the shear-bond strength of composite to enamel using the self-etch silorane adhesive compared to other self-etching methacrylate-based adhesives, with or without a separate application of phosphoric acid. Bovine incisors were sectioned using a diamond saw and mounted in plastic pipe. The bonding agents were applied to flattened enamel surfaces with or without the application of 35% phosphoric acid. The bonded tooth specimens were inserted beneath a mold, and composite was placed incrementally and light cured. The specimens were stored for 24 hours and six months in water and tested in shear. Data were analyzed with a three-way analysis of variance (ANOVA) to evaluate the effects of surface treatment, adhesive agent, or time on the bond strength of composite to bovine enamel (α=0.05). Significant differences were found between the groups based on surface treatment ($p<0.01$) or adhesive agent ($p<0.01$), but not on time ($p=0.19$), with no significant interactions ($p>0.14$). Phosphoric-acid etching of bovine enamel significantly increased the bond strength of the self-etch methacrylate and the silorane adhesives. The methacrylate-based adhesives had significantly greater bond strength to enamel than the silorane adhesive.

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INTRODUCTION

Enamel and dentin adhesive bonding agents have changed significantly over the past few decades with the trend towards simplification and ease of use and placement. Spanning seven generations, these ma-

terials require anywhere from one to three steps. Currently, there are two major categories of methacrylate-based adhesives: etch-and-rinse and self-etch.[1] Etch-and-rinse adhesives have been available since the early 1990s and are divided into three- and two-step systems. The three-step adhesives require a separate acidic conditioner, primer, and bonding resin. To reduce the placement time and complexity of three-step adhesives, manufacturers combined the primer and resin components to create a simplified two-step system. Self-etch adhesives were more recently introduced and are divided into two- and one-step systems. Two-step self-etch adhesives combine the acidic conditioner with the primer in the initial step and use a bonding resin in the second step. Even further reduction in the number of steps came with the introduction of one-step self-etch adhesives with the acidified primer and bonding resin placed in one simplified step.[1,2]

With the evolution of self-etch adhesive systems, there was a concern that the manufacturers were sacrificing the strength of the bond to enamel by using a weaker acidified primer in order to eliminate one step in the procedure. Laboratory studies have demonstrated that self-etch adhesives produce lower bond strength to enamel compared to etch-and-rinse adhesives.[3] More significantly, clinical studies have shown significantly less marginal defects and staining with selective etching of enamel with phosphoric acid when using a self-etch adhesive agent.[4] Van Meerbeek and Yoshihara[7] state that the use of phosphoric acid on enamel currently remains necessary to maintain the most durable bond to the interface and to protect the more vulnerable bond to dentin against degradation.

A completely different alternative to conventional methacrylate-based composites and adhesives has been introduced recently that is based on a ring-opening silorane (low-shrinking) monomer (Filtek LS, 3M ESPE, St Paul, MN, USA) formed by the chemical building blocks of siloxanes and oxiranes. According to the manufacturer, siloxane is characterized by hydrophobicity, and the oxirane polymer is known for low shrinkage.[8] In the polymerization process of silorane composites, the ring-shaped molecules open and approach the "neighbor" molecules. The expansion of the ring before polymerization has been shown to decrease the overall polymerization shrinkage to an average of 1.0% - 1.5%.[9] On the other hand, methacrylate-based resins connect by shifting closer together in a linear response, resulting in a greater loss of volume.[8] In a recent study, it was found that the silorane-based composite, Filtek LS, had the lowest polymerization shrinkage when compared to methacrylate-based composites, even though they shared similar physical properties.[9] The LS System Adhesive is a two-step self-etch bonding agent developed exclusively to work with the new silorane restorative composite. The LS System Adhesive Primer is hydrophilic and adheres to tooth structure. The LS System Adhesive Bond adheres to both the hydrophilic primed tooth structure and to the hydrophobic silorane-based Filtek LS composite.[9] The Filtek LS System Adhesive belongs to a similar two-step, self-etch adhesive strategy as other two-step self-etch methacrylate-based adhesives, such as Clearfil SE Bond (Kuraray, New York, NY, USA), and OptiBond XTR (Kerr, Orange, CA, USA). No research has been done evaluating the effect of selective etching using phosphoric acid on the bond strength of the new self-etch silorane adhesive and the unique low-shrinking Filtek LS silorane restorative material to enamel.

Recent systematic reviews of the literature reported the average annual failure rate of noncarious cervical lesions bonded with different dental adhesives and restored with composite resin. The mild two-step self-etch adhesives (eg, Clearfil SE Bond) were found to be the most effective clinically.[10,11] Clearfil SE Bond is composed of a mildly acidic self-etch primer containing 10-MDP (methacryloyloxydecyl dihydrogen phosphate), the active acidic functional monomer for demineralizing the enamel and the dentin. The acidic monomer is not as strong as phosphoric acid.[6] Although no significant difference in retention rates in restored noncarious lesions using Clearfil SE Bond with or without the selective etching of enamel were found in a recent 13-year clinical study by Peumans and others,[4] less marginal discoloration and better marginal integrity were found in the selective etch group. OptiBond XTR is a recently introduced two-step, self-etch adhesive that employs Kerr's ternary solvent system and a filled adhesive with an optimized formulation to reportedly produce outstanding adhesion for direct and indirect procedures.[12] Limited research is available evaluating this new two-step self-etching adhesive.

The purpose of this study was to evaluate the shear bond strength of composite to enamel using the two-step, self-etch silorane adhesive (LS System Adhesive) compared to other two-step self-etch methacrylate-based adhesives (Clearfil SE Bond, OptiBond XTR) with and without a separate application of phosphoric acid. The null hypothesis tested

was that there would be no difference in shear bond strength of composite restorative material to bovine enamel based on 1) surface treatment, 2) adhesive agent, or 3) time.

METHODS AND MATERIALS

One hundred twenty freshly extracted bovine incisors (Animal Technologies, Tyler, TX, USA) were stored in 0.5% chloramine-T and used within three months of purchase. The crowns were sectioned in a buccolingual direction at the cementoenamel junction to remove the root using a water-cooled diamond saw (Isomet 5000, Buehler, Lake Bluff, IL, USA). Retention cuts were placed in the lingual surface of the crown to prevent tooth dislodgement during shear testing. The teeth were mounted in polyvinylchloride pipe using dental stone and bis-acryl resin. After the stone and bis-acryl resin was set, a small area of the exposed enamel was flattened using a diamond wheel bur mounted in a drill press (Proxxon, Hickory, NC, USA) and smoothed using 600-grit silicon-carbide paper (Norton Abrasives, Worchester, MA, USA).

The enamel specimens were divided into six groups with 20 specimens each in order to compare the shear bond strength of the different adhesives and surface treatments over time. The adhesives were applied as a two-step, self-etch bonding agent with and without the selective enamel-etch technique. For the groups using a selective enamel-etch technique, 35% phosphoric-acid gel etchant (Kerr) was applied to the enamel for 15 seconds, rinsed with water for 15 seconds, then lightly air dried for three seconds. The adhesives were then applied according to the manufacturer's instructions (Table 1) and light cured with a visible light-curing unit (Bluephase G2, Ivoclar Vivadent, Amherst, NY, USA). Irradiance was determined with a radiometer (Bluephase Meter, Ivoclar Vivadent) and was considered acceptable if greater than 1000 mW/cm^2.

After application of the adhesive, the bonded specimens were placed in a jig (Ultradent Products, South Jordan, UT, USA) and secured beneath a white plastic mold. The bonded area was limited to the 2.4 mm circle determined by the mold. The teeth treated with LS System Adhesive were restored with Filtek LS composite, and the teeth treated with Clearfil SE Bond and OptiBond XTR were restored with Filtek Supreme Ultra (3M ESPE) composite. All materials were used according to manufacturer's instructions. The composite restorative materials were incrementally placed in two increments to a height of 3-4 mm. Each layer was polymerized as recommended by the manufacturer with the visible light curing unit. The specimens were stored for 24 hours and 6 months in distilled water at 37°C in a laboratory oven (Model 20GC, Quincy Lab, Chicago, IL, USA).

The bond strength was tested in shear mode with a knife-edge blade in a universal testing machine (Model 5943, Instron, Norwood, MA, USA) at a crosshead speed of 1 mm/min until failure. Shear bond strength values in megapascals (MPa) were calculated from the peak load of failure in newtons divided by the specimen cross-sectional surface area. The mean and standard deviation were determined per group. Following testing, the specimens were examined under a 10× microscope to determine the fracture mode as either: 1) adhesive fracture at the adhesive interface, 2) cohesive fracture in the enamel or composite, or 3) mixed (combination of adhesive and cohesive) in enamel or composite. Data were analyzed with a three-way analysis of variance (ANOVA) with Tukey post-hoc test to evaluate the effects of adhesive agent, surface treatment, or time on the bond strength of composite to bovine enamel (α=0.05).

RESULTS

Significant differences were found between the groups based on surface treatment ($p<0.01$) or adhesive agent ($p<0.01$), but not on time ($p=0.19$), with no significant interactions ($p>0.14$) as shown in Table 2. The Clearfil SE Bond and OptiBond XTR groups had significantly higher bond strengths to enamel than the LS System Adhesive group as shown in Figure 1. Etching the enamel with phosphoric acid prior to placement of the adhesives resulted in significantly higher bond strength. There was no significant difference in bond strength with any of the adhesives between 24 hours and six months of storage in water. Etching of the enamel was associated with more mixed or cohesive fractures (Figure 2).

DISCUSSION

An improvement in bond strength to enamel has been demonstrated with the use of phosphoric acid prior to bonding with self-etch methacrylate-based adhesive agents. No research has evaluated the effect of phosphoric-acid etching of enamel with the newer self-etch silorane adhesive, Filtek LS System Adhesive. The unique curing mechanism and chemistry of Filtek LS required the development of a dedicated adhesive. Filtek LS must be used with the LS System Adhesive, a two-step, self-etch bonding

Table 1: *Composition and Technique Guide for Filtek LS System Adhesive, Clearfil SE Bond, and OptiBond XTR as reported by the Manufacturers*

Adhesive System	Component	Composition	Application
LS System Adhesive	Primer	Hydroxyethyl methacrylate Bisphenol A glycidyl methacrylate Water Ethanol Phosphoric acid-methacryloxy-hexylesters Silane treated silica Hexanediol dimethacrylate Copolymer of acrylic and itaconic acid (Dimethylamino) ethyl methacrylate Camphorquinone Phosphine oxide	Apply with brush for 15 s Expose to air stream Cure 10 s
	Adhesive	Substituted dimethacrylate Silane treated silica Triethylene glycol dimethacrylate Phosphoric acid methacryloxy-hexylesters Camphorquinone Hexanediol dimethacrylate	Agitate bottle Apply to tooth surface Expose to air stream Cure 10 s
Clearfil SE Bond	Primer	Hydroxyethyl methacrylate Methacryloyloxydecl dihydrogen phosphate Hydrophobic aliphatic dimethacrylate Camphorquinone Water Accelerators Dyes Others	Apply for 20 s on tooth surface Evaporate volatile ingredients with a mild air stream
	Adhesive	Bisphenol A glycidyl methacrylate Hydroxyethyl methacrylate Methacryloyloxydecyl dihydrogen phosphate Hydrophobic aliphatic dimethacrylate Colloidal silica Camphorquinone Initiators Accelerators Others	Apply to tooth surface Expose to air stream Cure 10 s
OptiBond XTR	Primer	Glycerol phosphate dimethacrylate Hydrophilic co-monomers Camphorquinone Water Ethanol Acetone	Apply for 20 s; dry thin for 5 s
	Adhesive	Resin monomers Hydroxyethyl methacrylate Camphorquinone Inorganic fillers Ethanol	Apply with light brushing motion for 15 s; light cure for 10 s

Table 2: *Mean Shear Bond Strength and Statistical Analysis**

Bonding Agent	Shear Bond Strength (SD), MPa				
	Self-Etch		Etch-and-Rinse		Bonding Agent Total
	24 Hours	6 Months	24 Hours	6 Months	
Clearfil SE Bond	20.9 (2.9)	21.9 (4.8)	29.8 (4.2)	28.8 (6.5)	25.3 (4.6)[B]
LS System Adhesive	16.3 (3.8)	14.2 (3.3)	18.9 (3.5)	20.4 (4.8)	17.5 (3.9)[A]
OptiBond XTR	19.5 (2.6)	25.4 (7.3)	29.5 (3.2)	30.9 (7.9)	26.3 (5.3)[B]
Surface treatment total	19.7 (5.6)[a]		26.4 (7.0)[b]		

**Groups with the same upper case letter per column or lower case letter per row are not significantly different ($p > 0.05$).*

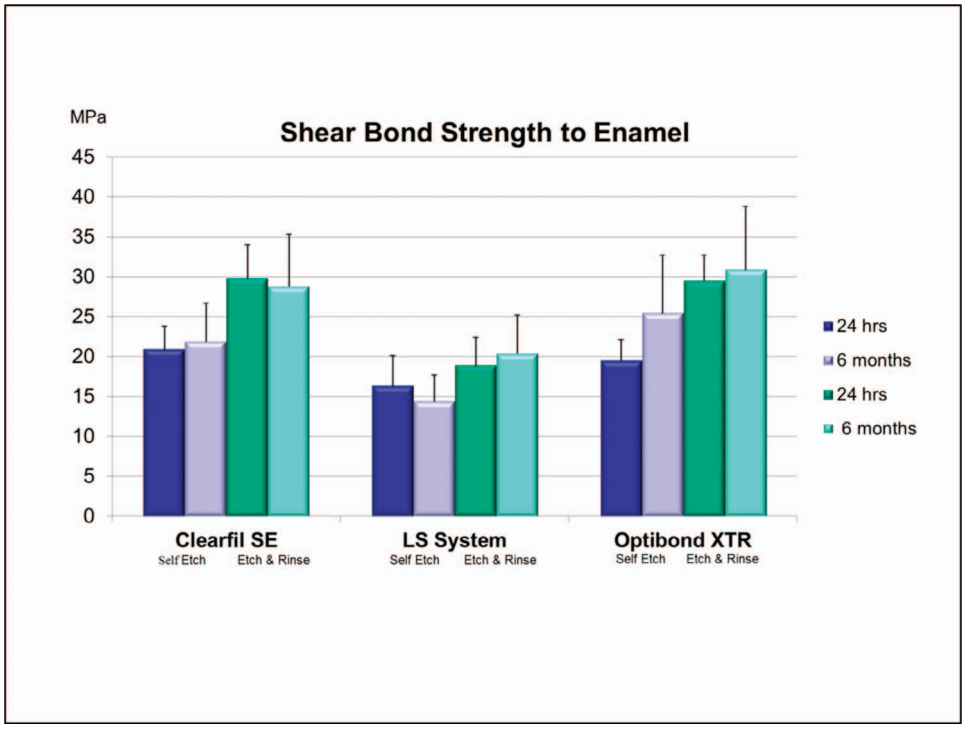

Figure 1. *Mean shear bond strength of adhesives applied in self-etch and etch-and-rinse modes over time. Error bars represent 1 standard deviation.*

agent. Self-etch adhesives have gained significant popularity recently due to their ease of use compared to the older generations of etch-and-rinse adhesives. However, the etch-and-rinse adhesives have been considered the "gold standard" when bonding to enamel due to the predictable etch pattern created from the relatively lower pH of the phosphoric acid.[13]

In this study, a significant difference was found based on surface treatment. Etching with phosphoric acid resulted in significantly higher bond strength to

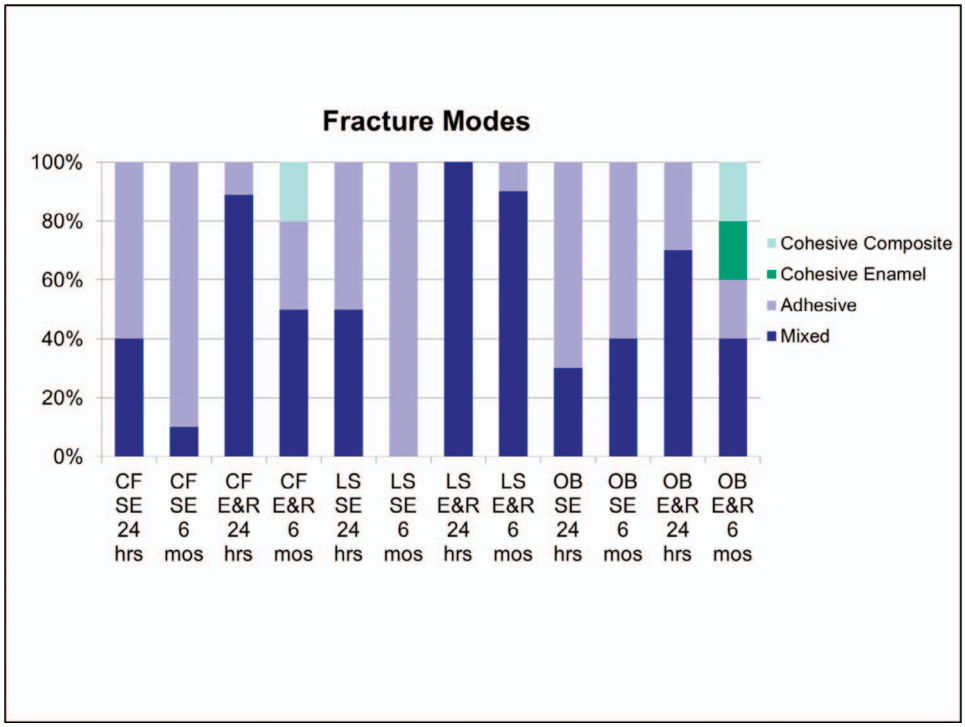

Figure 2. *Fracture mode of adhesive bonding agents over time.*

enamel, and therefore the first hypothesis was rejected. Also, phosphoric-acid etching of enamel resulted in more mixed and cohesive fractures, which is associated with a more stable interface.[14] Etch-and-rinse systems are characterized by an initial etching step with a stronger acid, usually 32%-37% phosphoric acid at a pH of approximately 0.9, to completely etch enamel and dentin. The alternative self-etch approach produces a shallower and less retentive enamel-etching pattern.[6] The degree of demineralization produced by self-etch adhesives depends largely on the acidity or etching aggressiveness of the functional monomer. Although not evaluated in this study, some strong self-etch adhesives (pH<1) create demineralized enamel that is similar to that created by phosphoric acid; whereas, some mild self-etching adhesives have demonstrated reduced bond strength to enamel in laboratory studies.[15] Although stronger self-etch adhesives may improve the bond to enamel, their high acidity may lead to overetching of dentin and subsequent loss of mineral content. Mild self-etch adhesives, as used in this study, may form ionic bonds between their functional monomers and the calcium of hydroxyapatite in dentin.[7] To maximize the bond to enamel without compromising the bond to dentin, selective etching of enamel with phosphoric acid prior to the application of a mild self-etch adhesive may be the most efficacious approach to adhesion to tooth structure.[7,16] According to Peumans and others,[4] selective etching of enamel may reduce marginal defects that can negatively influence the esthetic performance of direct composite restorations, especially in large visible enamel surfaces such as Class IV restorations.

A significant difference based on adhesive agent was also found in this study. The use of Clearfil SE Bond or OptiBond XTR resulted in significantly greater bond to enamel than the LS System Adhesive, and therefore the second null hypothesis was also rejected (Figure 1). Clearfil SE Bond and OptiBond XTR were chosen specifically for this study based on the fact that they are two-step self-etch adhesives similar to the LS System Adhesive. Clearfil SE Bond has been shown in laboratory studies to have consistently stronger bond strengths to enamel than other two-step, self-etch adhesive agents.[10,11] No clinical studies are available evaluating the clinical performance of the relatively newer OptiBond XTR adhesive. However, a recent laboratory study by Meharry and others[17] found no significant difference in bond strength to enamel between Clearfil SE Bond and OptiBond XTR similar to the results of this study. The LS System Adhesive has a higher pH (2.7) than Clearfil SE Bond (2.0) or OptiBond XTR (1.6).[8] The relatively lower pH of Clearfil SE Bond and OptiBond XTR may have contributed to their stronger bond to enamel compared to the LS System Adhesive.[17] OptiBond XTR is considered a mild self-etch adhesive with an initial pH of 2.4; but the pH drops to 1.6 during the primer application. The primer contains a three-part solvent of water, ethanol, and acetone that reportedly enhances the self-etch capability by facilitating penetration of the hydrophilic monomers into tooth structure.[17] Clearfil SE Bond contains the functional monomer, 10-MDP, vs the LS System Adhesive's Vitrebond copolymer, although both are stated to have the same purpose of bonding to tooth structure.[13] However, the monomer 10-MDP contains phosphate groups capable of producing ionic bonds with calcium in hydroxyapatite.[18]

No significant difference was found in bond strength based on storage time, and therefore the third null hypothesis was not rejected. Water sorption, in addition to masticatory stresses, salivary enzymes, and changes in temperature and pH, are thought to be major factors in destabilizing the adhesive-tooth interface over time.[17] Simplified bonding agents, with the hydrophilic primer and adhesive combined together, are potentially more susceptible to hydrolytic degradation over time. However, the adhesive bonding agents evaluated in this study are less susceptible to hydrolysis over time due to the separate primer and adhesive application.[2] Six months is a relatively short time to evaluate the effects of hydrolysis on bond strength. Longer storage times may be necessary to elucidate any changes in the more stable adhesive agents evaluated in this study.

Very limited *in vivo* research has been published evaluating the clinical performance of Filtek LS. The majority of short-term clinical studies have found similar results between Filtek LS and methacrylate-based materials in Class I and Class II restorations, suggesting no real benefit in the use of the low-shrinkage silorane-based composite restorative material.[13,19-23] Although the polymerization shrinkage of Filtek LS has been shown in laboratory studies to be the lowest among composite-based restorative materials, there is evidence to suggest that the higher elastic modulus or stiffness of Filtek LS may increase the polymerization shrinkage stress generated during placement in cavity preparations. Also, recent studies by Baracco and others[13,22] found that restorations restored using an etch-and-rinse adhe-

sive and methacrylate-based composite had better marginal adaptation than those restored with Filtek LS and the self-etching LS System Adhesive. No research has been completed evaluating the effect of selective etch on the clinical performance of Filtek LS with LS System Adhesive. The results of this laboratory study suggest that it may be advantageous to etch the enamel with phosphoric acid prior to the placement of the unique self-etch LS System Adhesive and Filtek LS composite restorative material. *In vivo* studies are necessary to determine if the greater bond strengths achieved with phosphoric-acid etching of the enamel prior to the placement of LS System Adhesive is clinically significant.

CONCLUSION

Phosphoric-acid etching of bovine enamel significantly increased the bond strength of the two-step, self-etch methacrylate and silorane adhesives. The methacrylate-based adhesives had significantly greater bond strength to enamel than the silorane adhesive.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of JBSA-Lackland, TX Institutional Review Board. The approval code issued for this study is FWH20130020A.

Disclosure

The views expressed in this article are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, or the United States Government.

Conflict of Interest

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

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Influence of Staining Solution and Bleaching on Color Stability of Resin Used for Caries Infiltration

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Clinical Relevance

A bleaching treatment of resin-infiltrated enamel lesions may improve appearance after staining.

SUMMARY

Objective: The objective of this study was to evaluate the color stability of Icon-infiltrated white spot lesions after staining and the bleaching effect on the infiltrated and stained surfaces.

Methods and Materials: Enamel-dentin specimens (N=30, 5 × 5 × 3 mm, 1-mm enamel + 2-mm dentin thickness) were prepared from bovine incisors and randomly allocated into three groups (n=10): control, demineralized, and infiltrated. Artificial enamel subsurface lesions were created using 50 mL of 0.05 M acetate buffer solution. Specimens were produced by Icon application in enamel caries-like lesions, according to the manufacturer's instruction. Baseline color readings were assessed using a spectrophotometer, and CIE L*a*b* measurements of each specimen were performed using a white background. To simulate extrinsic dietary staining, specimens were placed into a 4-mL coffee infusion, three times daily for 15 minutes, for 14 days. After the staining procedure, color measurements were performed again. Then, bleaching procedures were performed using 16% carbamide peroxide gel for four hours daily for 21 days, and a final color assessment was performed. To compare the baseline and final measurements, *t*-test was used (α =0.05). The statistical comparison between the groups was performed using the one-way analysis of variance and Tukey tests (α =0.05).

Results: Coffee staining provided a significant reduction of L* values and an increase of a* and b* in all groups (control, decayed, and infiltrated). The bleaching procedure provided

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a significant increase in L* and decrease of a* and b* values in all groups. There was no significant difference in ΔE values between decayed and infiltrated groups before bleaching, and after bleaching, the infiltrated group showed the lowest ΔE values.

Conclusion: It can be concluded that enamel infiltrated with Icon presents significant alteration of color after staining when compared with sound enamel. However, if there is discoloration of the infiltrant, the bleaching treatment can be used successfully.

INTRODUCTION

Orthodontic treatment with fixed appliances increases the risk of developing demineralized white spot lesions. The main reason for the development of caries is plaque stagnation around braces, mostly underneath the arch wires and between the bracket and gingival margin.[1] An established active white spot lesion has a chalky, opaque appearance, since the light is scattered mainly within the lesion body. Scattering is caused in interfaces between substances with different refractive indexes. Untreated white spot lesions may result in progression to cavities and severe esthetic problems.[2] In addition, sometimes during the remineralization of these white spot lesions, stains get incorporated into the lesion, leading to the creation of brown spots and thus increasing the esthetic problem. Hence, the treatment of these white spot lesions should aim both to prevent the caries progression and to improve esthetics by decreasing opacity and staining.[3]

Lately, a new approach has been launched to arrest noncavitated enamel lesions. After erosion of the pseudo-intact surface layer with hydrochloric acid, low-viscosity resins (Icon) penetrate within the lesion.[4] Thus, the porosities of carious lesions can be occluded, and the diffusion of acids and minerals is reduced. In this way, lesion progression is hampered, and caries progression is slowed down or even arrested.[4] A study conducted by Paris and Meyer-Lueckel[5] demonstrated that the infiltration technique might be an alternative to microabrasion and restorative treatment, particularly for white spot lesions of esthetically relevant teeth, as in cases of white spot lesions in facial surfaces.

According to the manufacturer, Icon is a TEGDMA-based resin matrix. Some authors have suggested that the addition of TEGDMA to a restorative material could increase water sorption,[6,7] decrease general mechanical properties,[8] and hinder color stability.[9] The discoloration of restorative materials might be attributable to water sorption and the hydrophilicity of the matrix resin. If the resin material can absorb water, it can also absorb other fluids, resulting in the alteration of color.[10] This process may cause plasticization and softening of the resin matrix and reduction of color stability. Discoloration of tooth-colored resin-based materials may be caused by intrinsic and extrinsic factors. Extrinsic factors such as adsorption of stains may also cause discoloration.[11] In this way, surfaces containing the infiltrant and submitted to colored solutions can be stained, since their composition contains a greater amount of TEGDMA. For infiltrated lesions (white spot lesions treated with Icon), this could be an esthetic problem.

To solve this problem caused by staining solutions, some recent studies reported polishing of the infiltrated lesions and concluded that polishing increases their resistance to staining challenges.[12,13] However, it seems clinically impossible to remove only the infiltrant because it penetrates the enamel. Over time, the infiltrant could stain again, and the polishing procedures would need to be performed again, resulting in greater wear and excessive loss of enamel.

Some physical and mechanical properties of infiltrants have already been studied,[14] but few studies have been published regarding their esthetic behavior.[13,15] Rey and others[15] showed that Icon showed a higher staining susceptibility compared with adhesive systems. Thus, it is necessary to develop a method to improve Icon appearance after staining. Borges and others[13] suggest repolishing the infiltrated lesions to minimize the staining effect. However, this polishing alternative may result in unnecessary enamel wear caused by abrasives.

A very common procedure is to reach esthetic conditions using bleaching. Bleaching methods have been developed, and peroxide compounds at different concentrations are used for tooth-whitening procedures. Contemporary bleaching agents are typically either hydrogen peroxide or carbamide peroxide. The procedure may be performed at a dental office or by applying the agent by the patient in gel form within the confines of a custom tray.[16]

As a highly hydrophilic material, the infiltrant can be stained, but its behavior in bleaching procedures is unknown. No study has assessed the staining behavior of infiltrated lesions after staining and bleaching. It is questionable whether bleaching treatment is effective after infiltrant staining. Thus,

the aims of this study were 1) to evaluate the color stability of white spot lesions with Icon after staining and 2) to evaluate the bleaching effect on these infiltrated and stained surfaces. The hypotheses tested were 1) coffee staining causes a color change in the infiltrated enamel and 2) a bleaching procedure provides color recovery for the infiltrated enamel.

METHODS AND MATERIALS

Specimen Preparation

Enamel-dentin specimens (N=30, 5×5×3 mm, 1-mm enamel + 2-mm dentin thickness) were prepared from bovine incisors. Enamel surfaces were polished (silicon carbide paper 1200, 2400, 4000), and dentin surfaces were covered with two layers of acid-resistant nail varnish (Colorama, São Paulo, Brazil), leaving a 4×4 mm^2 exposed area. All specimens presented the same enamel thickness, and using a simple drawing, they were randomly allocated into three groups (n=10): control, demineralized, or infiltrated.

Artificial enamel subsurface lesions on the unprotected areas were created by storing specimens in 50 mL of 0.05 M acetate buffer solution, considering a ratio of 2.0 mL/mm^2 of exposed enamel, pH 5.0, at 50% hydroxyapatite saturation, for 10 hours at 37°C.[17] Caries-like lesions were etched with 37% phosphoric acid gel Scotch Bond Etchant (3M ESPE, St Paul, MN, USA) for 60 seconds,[18] washed with water spray, and dried for 15 seconds. Then, the specimens were dried by immersion in 100% ethanol. Icon (DMG, Hamburg, Germany) was applied to the etched enamel surface using a microbrush, resting for 3 minutes, according to the manufacturer's instructions. Specimens were light cured for 40 seconds using Free Light 2 (3M/ESPE) with 1000 mW/cm^2 irradiance, measured with a power meter (Ophir Optronics Inc, Danvers, MA, USA). The infiltrant was applied for a second time, resting for one minute, and then light cured for 40 seconds. All specimens were immersed in artificial saliva for 24 hours.

Color Assessment

An initial color reading of the specimen (baseline) was performed using a spectrophotometer (CM-700d, Konica Minolta, Tokyo, Japan) in reflectance mode. The samples were positioned in a sample carrier in a light cabin (GTI Mini Matcher MM1e, GTI Graphic Technology Inc, Newburgh, NY, USA) to standardize the ambient light during the measurement process, and then the samples were subjected to a reading with the spectrophotometer. The CIE L*a*b* measurements of each specimen were performed using a white background. In the color space, L* indicates lightness (L+ = lightness and L– = darkness), the a* coordinate represents the red/green range (a*+ = redness and a*– = greenness), and the b* coordinate represents the yellow/blue range (b*+ = yellowness and b*– = blueness). The values of the coordinates a* and b*, when approaching zero, indicate neutral colors (white and gray) and an increase in magnitude for more saturated or intense colors. The L*a*b* color space includes all perceivable colors and is based on a cube root transformation of the color data. This detailed analysis through the study of the coordinates L* a* and b* separately helps to better understand which one was more responsible for the total color change (ΔE).

The total color change (ΔE) was calculated according to the following formula[19]: $\Delta E = [(L_1 - L_0)^2 + (a_1 - a_0)^2 + (b_1 - b_0)^2]^{1/2}$, using On Color QC Lite software (Konica Minolta) to generate spectral measurements as a function of wavelength for data processing and analysis.

Staining Procedure

To simulate extrinsic dietary staining, specimens were placed into 4 mL of coffee infusion (Tradition Nescafé, Nestlé, Araras, SP, Brazil) of 8 g (powder) to 100 mL boiled water. After preparation, the specimens were immediately placed in contact with the coffee. This procedure was repeated for 15 minutes three times daily for 14 days. After the staining procedure, color measurements were performed again.

Bleaching Procedure

For the bleaching procedure, specimens were fixed in a device, and approximately 1 mm of the bleaching agent was applied to the exposed enamel surface. The 16% carbamide peroxide gel (Whiteness Perfect, FGM, Santa Catarina, Brazil) was left in contact with the enamel surfaces for four hours daily for 21 days.

The samples were maintained in relative humidity until the final color assessment (after 21 days), which was performed following the procedure of the initial color assessment. The L*, a*, b* and ΔE parameters were subjected to statistical analysis. To compare the baseline and final measurements, a *t*-test was used ($p<0.05$). The statistical comparison between groups was performed using one-way

Table 1: *Means and Standard Deviations of L*, a*, and b* Parameters Observed for the Groups Considering Time and Treatment for Each Group (No Bleaching and Bleaching) Individually*[a]

	Color Parameter	Time	Control	Decayed	Icon Infiltrated
No bleaching	L	Baseline	88.20 (1.37) Ab	93.10 (1.53) Aa	87.60 (1.14) Ab
		After staining	75.91 (3.41) Ba	60.50 (3.71) Bb	62.54 (1.80) Bb
	a	Baseline	−0.09 (0.33) Bb	0.26 (0.14) Bb	0.96 (0.43) Ba
		After staining	3.42 (0.91) Ab	3.08 (0.69) Ab	8.45 (0.64) Aa
	b	Baseline	11.78 (1.77) Ba	5.07 (0.68) Bb	9.33 (1.13) Bc
		After staining	14.08 (1.27) Aa	10.41 (1.23) Aa	18.79 (1.50) Aa
Bleaching	L	Baseline	75.90 (3.41) Ba	60.49 (3.71) Bb	62.17 (1.86) Bb
		After bleaching	87.19 (2.12) Ab	91.65 (2.92) Aa	88.27 (1.30) Ab
	a	Baseline	3.42 (0.91) Ab	3.07 (0.69) Ab	8.34 (0.57) Aa
		After bleaching	0.40 (0.66) Bb	1.19 (0.79) Ba	0.62 (0.34) Bb
	b	Baseline	14.07 (1.27) Aa	10.41 (1.23) Aa	18.64 (1.28) Aa
		After bleaching	11.54 (2.72) Ba	2.48(2.33) Bb	10.18 (1.63) Ba

[a] *Capital letters indicate comparison between the baseline and final measurement of the column. Lowercase letters demonstrate comparison between groups in rows. Different letters indicate statistically significant differences (*$p<0.05$*).*

analysis of variance and Tukey tests ($p<0.05$). Statistical analyses were performed by Assistat software (Campina Grande, Brazil).

RESULTS

Comparison of Values Between Baseline and After Staining

Table 1 shows the L*, a*, and b* mean values of the baseline and after the staining procedures, before and after bleaching, and Table 2 shows the ΔE mean values for all groups.

Coffee staining provided a significant reduction of L* values for all groups (control, decayed, and infiltrated). After coffee staining, the control group presented the highest L* values compared with the other groups. There was a significant increase for the a* and b* values for all groups ($p<0.05$). Infiltrated enamel showed, at baseline, significantly higher a* and b* values than decayed and sound enamel. Despite a significant baseline difference among all groups concerning the a* and b* values, all groups showed significantly higher a* and b* values after coffee staining, but they did not show any differences among each other after staining.

The bleaching procedure provided a significant increase in the L* values for all groups (control, decayed, and infiltrated). After bleaching, decayed enamel presented the highest L* values compared with the other groups, but there was no significant difference between the control and infiltrated groups. There was a significant decrease for the a* and b* values for all groups ($p<0.05$). After bleaching, decayed enamel showed significantly higher a* values than infiltrated and sound enamel, and there was no significant difference between infiltrated and control groups for the b* values. The lowest b* values were exhibited by the decayed group.

The lowest ΔE values were observed in the control group ($p>0.05$) when considering baseline × staining. However, after bleaching, the stained groups showed the highest ΔE for the decayed group compared with the group infiltrated with Icon. There was no significant difference in the ΔE values between the decayed and infiltrated groups ($p<0.05$) before bleaching, indicating that the Icon-infiltrated group underwent a significant color change similar to the decayed group. However, after bleaching, the infiltrated group showed the lowest ΔE values.

DISCUSSION

Color changes in direct restorative materials, and more specifically in resin materials, have a direct influence on esthetics and therefore on the clinical longevity of a restoration. Enamel infiltrated by no-filler low-viscosity resin may become an esthetic

Table 2: *ΔE Values Observed by All Groups in All Experimental Periods*[a]

Group	ΔE	
	Baseline × Staining	Staining × Bleaching
Control	13.1206 b	3.7379 ab
Decayed	33.1940 a	5.6464 a
Infiltrated	41.1049 a	2.2845 b

[a] *Different letters indicate statistically significant differences (*$p<0.05$*) for independent comparisons considering baseline × staining and staining × bleaching individually.*

problem over time because, like any resin material, it can be subject to alteration of color. The extent of discoloration varies according to the habits of the patient, such as oral hygiene and diet.[20]

Coffee was chosen as a dye-testing substance in this study because it is frequently consumed. Coffee exhibits a strong potential for staining both tooth structure and resin materials.[21] The compatibility between the brown dye from coffee and the resin polymer chain has been suggested to facilitate the adsorption and penetration of the dye in the resin.[21]

This study was performed using bovine teeth instead of human teeth because Attia and others[22] found that bovine and human enamel substrates behave similarly in terms of staining and bleaching effect. Moreover, the composition, density, and microhardness of bovine substrate are very similar to those of human enamel.[23,24]

Many methods are currently used to assess tooth color. Since spectrophotometers allow an objective color assessment and provide precise quantitative data,[25] this was the method used in the present study. The color alteration measurements were evaluated using reflectance measurements with the CIE Lab Color coordinate system. According to Dietschi and others,[26] when the three coordinates of color dimensions are analyzed separately, the L* values, which represent the lightness of the object, appear to be the most relevant parameter for comparisons under experimental conditions. Also, the ΔE values were analyzed because they indicate the magnitude of the color change at two different moments.[12]

The first hypothesis tested in this study was supported, as it was verified that after staining there was a significant L* value decrease for all groups. According to Joiner,[19] the L* value is a measurement of the lightness of an object and is quantified on a scale such that a perfect black has an L* value of zero and a perfect reflecting diffuser has an L* value of 100. The L* values decreased, probably because of the incorporation of the dye present in coffee into the infiltrant. Moreover, the sound enamel presented the highest L* values, which were significantly different from those of decayed enamel infiltrated with Icon. This means that infiltrated enamel is able to incorporate more dye. Clinically, this could be an esthetic problem. In addition, a* and b* coordinates increased after the staining procedures. The values of the a* and b* coordinates approached zero, indicating neutral colors (white and gray) and an increase in magnitude for more saturated or intense colors.[19]

These alterations can be explained by the composition of Icon. Its matrix is TEGDMA based, with no filler particles.[4] Some authors have suggested that TEGDMA is a monomer that presents high water sorption and has a hydrophilic behavior compared with other monomers.[4,7] Thus, it can be assumed that Icon would easily absorb dyes that are present in beverages and food. The a* and b* coordinate changes drove the alterations of the ΔE values, as sound enamel exhibited the lowest ΔE values, and enamel infiltrated with Icon presented almost three times higher ΔE values.

The second hypothesis was also supported, as there were significant alterations of the color coordinates and the ΔE values. After staining, all groups submitted to the bleaching treatment showed increased L* values, indicating higher lightness because of the application of carbamide peroxide. Moreover, decreased a* and b* values indicate that there was dye neutralization. Joiner[19] assumed that, as the values of the a* and b* coordinates approach zero, they indicate neutral colors. The carbamide peroxide action is due to its own breakdown into hydrogen peroxide and urea. Urea further breaks down into ammonia and carbon dioxide, which accounts for the elevation of the intraoral pH. Hydrogen peroxide breaks down into water, oxygen, and free radicals, which result in oxidation of the pigments in teeth.[27] The ΔE values showed that enamel infiltrated with Icon provided similar alteration of color after bleaching when compared with sound enamel. These results indicate that it is possible to increase the brightness of the enamel infiltrated with Icon, making the use of polishing procedures unnecessary.

Borges and others[13] showed that demineralized enamel treated with resin infiltration showed significant staining when exposed to coffee and wine, consistent with the results presented in this study. They suggested that the repolishing of the specimens could minimize the staining effect. However, polishing procedures may remove unnecessary enamel structure, causing iatrogenic damage to the enamel. This study suggests a more conservative approach: the bleaching procedure. A bleaching procedure does not promote the removal of enamel, and according to the results presented in this study, after carbamide peroxide bleaching, the ΔE values of the demineralized enamel treated with resin infiltration were statistically similar to those of the control group. According to the CIE/Lab units, a ΔE of less than 1 is excellent, and a ΔE value less than 3.3 is considered clinically insignificant.[28]

The effects of staining and bleaching on composite resins have been reported. Villalta and others[29] observed that composite resins were affected by staining solutions, such as wine and coffee, after bleaching. After bleaching, discoloration was removed completely from the composite resins, probably because of the superficial cleansing of the specimens, which may explain the results of this study regarding resin infiltration.

CONCLUSION

Based on the results obtained on this study, it can be concluded that enamel infiltrated with Icon presents significant color alteration after staining when compared with sound enamel. Therefore, patients should avoid the consumption of colored beverages and foods to increase the longevity of the resin infiltration in esthetically important areas. However, if the discoloration of the infiltrant occurs, bleaching treatment can be used successfully. *In vivo* studies should be performed to assess more accurately the staining behavior of Icon.

Acknowledgment

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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 UNICAMP.

Conflict of Interest

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

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Splinted Porcelain Laminate Veneers With a Natural Tooth Pontic: A Provisional Approach for Conservative and Esthetic Treatment of a Challenging Case

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Clinical Relevance

Under specific occlusion without incisal contact in centric occlusion, protrusive, or lateral movements, splint laminate veneers with a natural tooth pontic may be a conservative and esthetic approach as a provisional restorative treatment for severely discolored and apically resorbed anterior maxillary teeth.

SUMMARY

Esthetic rehabilitation of discolored anterior teeth is always a great challenge, especially in the presence of pathology. Fortunately, conservative management in the esthetic zone has become more feasible in compromised cases because of the development of restorative materials and advances in dental adhesives. This report presents a complicated case of a patient with tetracycline-related discoloration, multiple root resorption, and a periapical lesion. Treatment was conservative and used a natural tooth pontic and splinted porcelain laminate veneers.

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INTRODUCTION

Minimally invasive treatment is undoubtedly the most valuable approach to increasing the longevity of natural teeth in esthetic restorations. The conservative approach in esthetic restorations is gaining popularity due to the development of restorative dental materials such as adhesives, resin composites, and porcelain. There is accumulating evidence of their durable bonding effectiveness from labora-

tory and clinical studies.[1-3] These advancements in adhesive dentistry enable clinicians to have various treatment options for a diversity of esthetic problems.

Tooth discoloration results from extrinsic or intrinsic factors.[4] Intrinsic discoloration generally arises from chromogens that chelate to the tooth structure and are often of systemic or pulpal origin, and the management of intrinsic discoloration is usually unpredictable.[5,6] Tetracycline (TC) staining is a typical intrinsic discoloration that rarely responds to bleaching treatment and is most effectively addressed with restorative procedures to mask the dark color.[7] Porcelain laminate veneers have been proposed as a viable restorative option for TC-stained teeth to conceal the color defect without extensive tooth reduction.[8-11]

Coexisting periapical lesions or periodontal problems of anterior teeth needing esthetic restoration may require a multidisciplinary approach and/or modification of the conventional treatment plan. Even when an abutment is periodontally compromised, it can still be used as a natural tooth pontic within a splinted crown restoration as it offers the benefits of being the right size, shape, and color.[12,13] The aim of this report is to present a provisional approach developed to address our patient's strong demand to retain a natural tooth while still achieving esthetic results in the presence of diverse esthetic and pathologic problems with the maxillary anterior teeth.

CASE REPORT

A 36-year-old woman presented with a chief complaint of recurring fistula of her left maxillary central incisor and desired esthetic rehabilitation of her anterior maxillary teeth. A clinical examination showed a generalized band-like grayish TC stain of the anterior teeth. Tooth #9 had a darker discoloration in the cervical area than the other anterior teeth. The tooth was tender to percussion, and a labial sinus tract was observed in the apical area. Unesthetic old resin composite restorations also existed on the mesial side of both canines. The patient had a slightly open bite with deep overjet, and anterior guidance was absent in protrusive movement. A lingual bonded retainer was observed in both the maxillary and mandibular anterior teeth. Radiographic examination revealed general root resorption in most of the teeth and severe root resorption in some incisors. Tooth #9 had a radiolucent apical lesion with an excessively shortened root (Figure 1). The patient's dental history indicated that orthodontic treatment for her open bite was completed a year previous to the examination, and a root canal treatment was performed on tooth #9 six months previous due to pain. The patient was four months post nightguard bleaching treatment to remove the discoloration on her upper anterior teeth, which was not effective in lightening the teeth.

The patient had a strong desire to save tooth #9 rather than have it extracted, even though she was well aware of the severe root resorption. Her treatment plan was thoroughly discussed, and intentional replantation was planned to maintain tooth #9 using a "natural tooth pontic concept." To manage the apparent TC discoloration on tooth #9, nonvital bleaching was performed prior to replantation. Splinted porcelain laminate veneers, as provisional restorations, were designed for teeth #8, #9, and #10 with consideration for the patient's occlusion and reinforcement splinting of the natural tooth pontic.

Tooth #9 was atraumatically extracted using flat-beak forceps to minimize damage to the periodontal tissues after repositioning guidelines were marked on teeth #8, #9, and #10 using a marking pen. The extraction socket was thoroughly debrided and degranulated. Granulation tissues and resorptive lesions of the tooth were removed using a periodontal curette and an ultrafine diamond point (Komet, Lemgo, Germany), and the apex was prepared for retrograde filling. The apical preparation was etched with 37% phosphoric acid, and a two-step etch-and-rinse dentin adhesive system was applied (Adper Single Bond 2, 3M ESPE, St Paul, MN, USA). The adhesive application area was carefully limited to just around the prepared canal to ensure there was no spillage out onto the root surface. The canal was then filled with flowable resin composite (Tetric N Flow, Ivoclar Vivadent, Schaan, Liechtenstein) that was subsequently light cured. The tooth was repositioned along the marked guidelines and splinted to the adjacent teeth using adhesives and resin composite (Adper Single Bond and Z-250, 3M ESPE). A prefabricated lingual retainer was placed to retain alignment of the anterior teeth (Figure 2). To improve the dark gray-brown discoloration of tooth #9, nonvital tooth bleaching was performed two weeks after surgery with sodium perborate and distilled water (Figure 3). After bleaching, the access cavity was filled using adhesive and resin composite (Adper Single Bond and Z-250, 3M ESPE).

Before tooth preparation for laminate veneers, a diagnostic wax-up was performed on a study model to assess the incisal interference and fabricate a

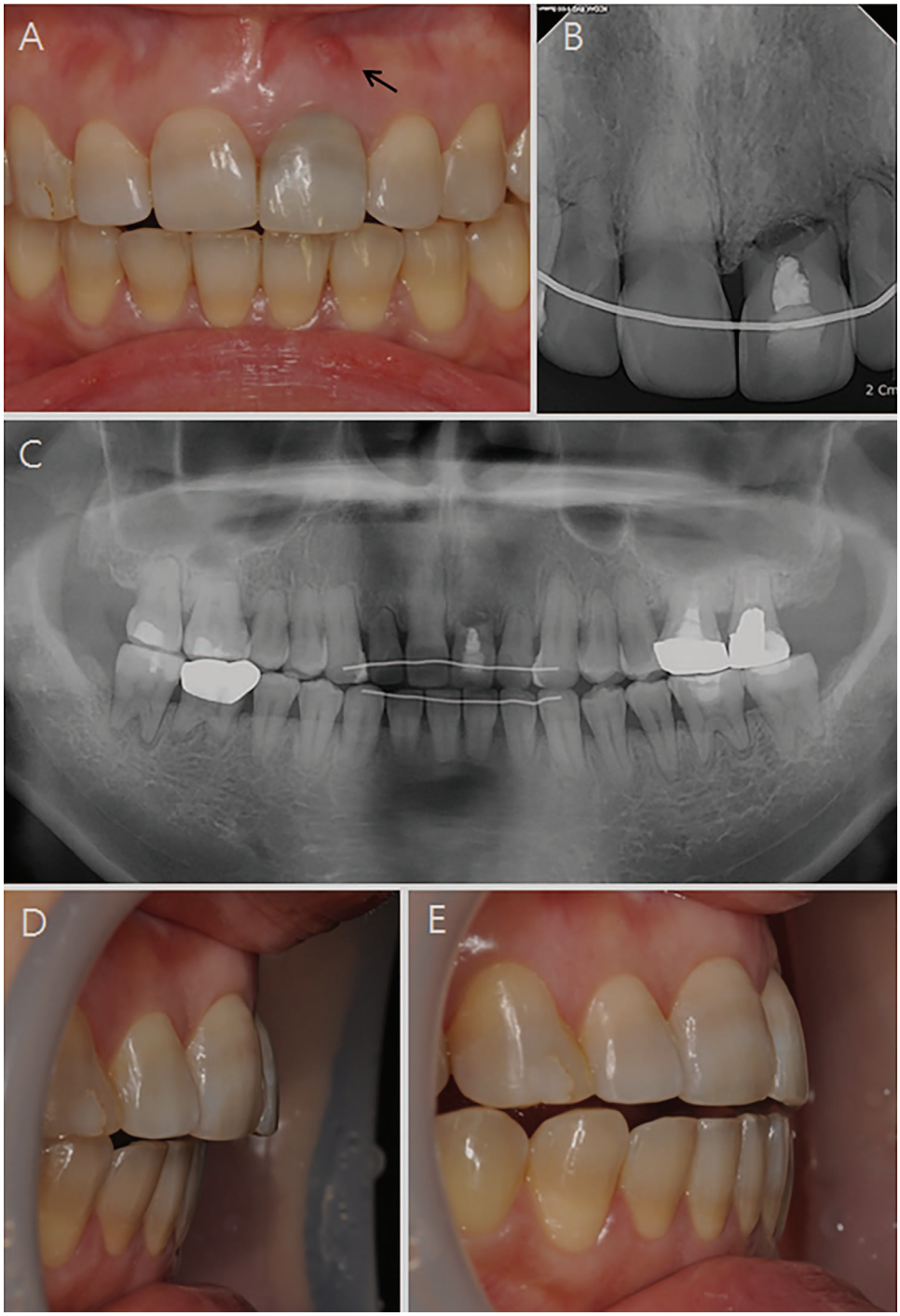

Figure 1. *Preoperative clinical and radiographic examinations. (A): Labial fistula on the apex of tooth #9 (arrow). (B and C): Periapical and panoramic radiographs. Note the shortened roots with external resorption on multiple teeth, especially tooth #9. (D and E): Intraoral photographs of protrusive movement showing the deep overjet without anterior guidance.*

silicone index as a preparation guide. Intraoral mockup with resin composite was also performed to simulate the shape of the final restorations (Figure 4). Six maxillary anterior teeth were prepared for porcelain laminate veneers with an incisal overlapping design. Labial reduction was performed with the dimensions of 0.5, 0.9, and 0.9 mm in each cervical, middle, and incisal third of the teeth, respectively, which is deeper than a standard laminate veneer case.[14,15] Preparation depth was checked using a depth gauge bur (Komet) and the silicone index (Figure 5A,B). Predesigned proximal preparation was carefully performed with consideration for the tooth #9 splint and the overall esthetic results. The interproximal margins between the canine and lateral incisor were extended to be at

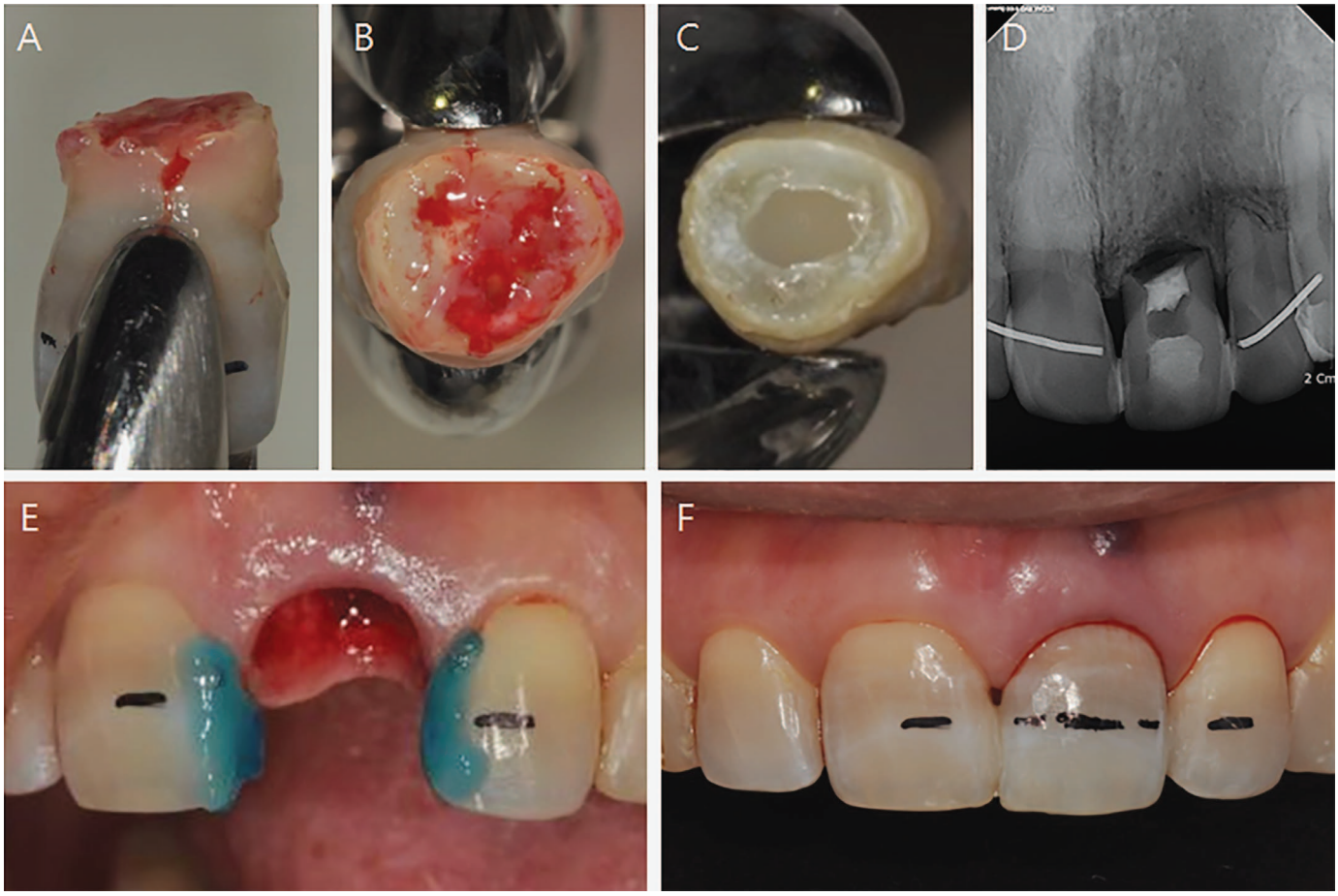

Figure 2. *Intentional replantation of tooth #9. (A and B): Severe apical root resorption. (C): Apical preparation and retrograde filling with resin composite. (D): Periapical radiograph after surgery. (E and F): Replanted tooth #9 was repositioned along the marked guide line and splinted with resin composite.*

the linguoproximal line angle because the natural contact was already lost by removing the old resin composite restorations. The proximal extension in the embrasure between #7 and #8 and the distal embrasures of the canines was prepared just short of breaking the contact area. To provide a natural embrasure appearance in the splinted laminate veneers, the interproximal margins between teeth #8 and #9 and between teeth #9 and #10 were further lingually extended into two thirds of the proximal surface, which had been splinted with resin composite. An estimated 0.5-1.0 mm of the incisal edge was reduced to create a butt joint with the palatal surface because we wanted to increase the incisal length 1.0-1.5 mm from the existing crown length, assuming a 1.5- to 2.0-mm incisal porcelain length in the final restorations to address the openbite. The gingival margin was prepared with a fine diamond chamfer bur to be positioned 0.5 mm subgingivally.

A final impression was taken (Exafine, GC Corporation, Tokyo, Japan), and provisional restorations (Luxatemp, DMG, Hamburg, Germany) were placed using noneuginol temporary cement (Figure 5C,D). Opaque lithium disilicate (IPS e.max Press LT, low translucency, shade BL3, Ivoclar Vivadent) was chosen as a porcelain ingot to mask the intrinsic discoloration with thin veneering material. The laminate veneers on teeth #8, #9, and #10 were splinted considering the unfavorable crown to root (C/R) ratio of tooth #9 and the occlusion that showed no anterior guidance on protrusive movement (Figure 6A).

After try-in, the internal surfaces were conditioned with 5% hydrofluoric acid (IPS ceramic etching gel, Ivoclar Vivadent) for 20 seconds, rinsed with water, and dried. Then, silane coupling agent (Monobond S, Ivoclar Vivadent) was applied for one minute and dried. The prepared teeth were etched with 37% phosphoric acid for 15 seconds,

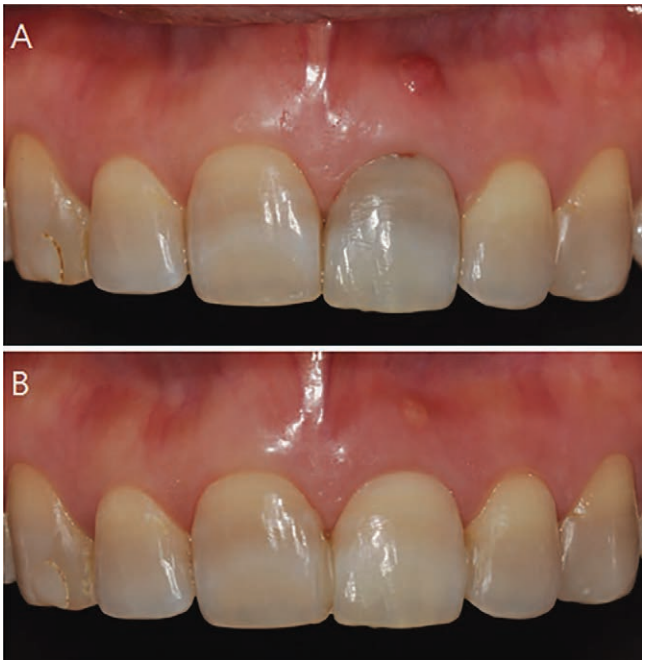

Figure 3. *(A): Before and (B) after nonvital bleaching of tooth #9 with sodium perborate and distilled water.*

rinsed, dried, and adhesive applied (Scotchbond Multi-Purpose, 3M ESPE). Transparent shade Variolink N resin cement (Ivoclar Vivadent) was applied as a luting agent. Excess cement was gently removed using an explorer and pre-engaged floss after tack-curing for 1-2 seconds with a light emitting diode (LED) light curing device (Bluephase 20i, Ivoclar Vivadent). Multidirectional final light curing was done for 60 seconds each. Finishing and polishing was performed with a 24-fluted carbide bur and ultrafine diamond bur (Komet).

Occlusion was checked in protrusive and lateral movements and revealed the absence of incisal contact, which was the same as before treatment (Figure 6B). The patient was very satisfied with her esthetic outcome after delivery of the final restorations (Figure 6C,D). At her one-year follow-up visit, the restorations had still maintained their esthetic appearance, and a stable periodontal tissue response was noted (Figure 6E,F).

DISCUSSION

Treatment planning decisions should be based not only on comprehensive examination and diagnosis but also an understanding of the therapeutic indications and possible complications.[16] Thus, therapeutic modalities, knowledge of restorative materials, and patient desires should be incorporated in decision-making to ensure a satisfactory treatment outcome. In the present case, treatment decisions were made using a systematic approach to resolve several clinical problems that were primarily due to periapical inflammation of tooth #9 with an unfavorable C/R ratio and generalized TC discoloration. Specifically, various clinical problems and the patient's strong desire to save a natural tooth led us to design splinted laminate veneers with a natural tooth pontic that might serve as a temporary restorative treatment.

Decision #01: Preserve or Extract the Compromised Tooth

The first decision with regard to resolving the periapical inflammation of tooth #9 was between extraction and surgical endodontic treatment. Because of the drastic root resorption that resulted in the unfavorable C/R ratio, extraction would have been a rather rational treatment option. The first option after tooth extraction was implant installation at the extraction site, and the second was placement of a three-unit fixed partial denture on teeth #8-#10. However, both options were rejected because the patient had a strong desire to save tooth #9, even though she recognized that saving the compromised tooth could lead to a provisional restorative treatment. On the other hand, preserving the natural tooth was advantageous not only for avoidance of psychologic trauma, but also for more predictable conservation of the hard and soft tissue around the anterior maxillary teeth compared with extraction.[17-19] The behavior of the alveolar bone after extraction complicates routine implant installation procedures and esthetic prosthetic restoration due to the reduced bone volume and gingival degeneration.[20] The biotype of the gingiva is thin, particularly in young female patients, and soft tissue recession and black triangle formation is inevitable. By keeping and using the natural tooth as a pontic, the cervical gingival contour could easily be maintained without noticeable gingival recession and shrinkage.

An apicoectomy or intentional replantation was considered to save tooth #9, but apicoectomy was ruled out due to the short length of the root and limited access to the extensive resorptive lesion. We adopted the natural tooth pontic concept for the treated tooth by replantation. Surgical curettage and replantation also enabled an extraoral radical operation to remove the periapical inflammation and external root resorption.

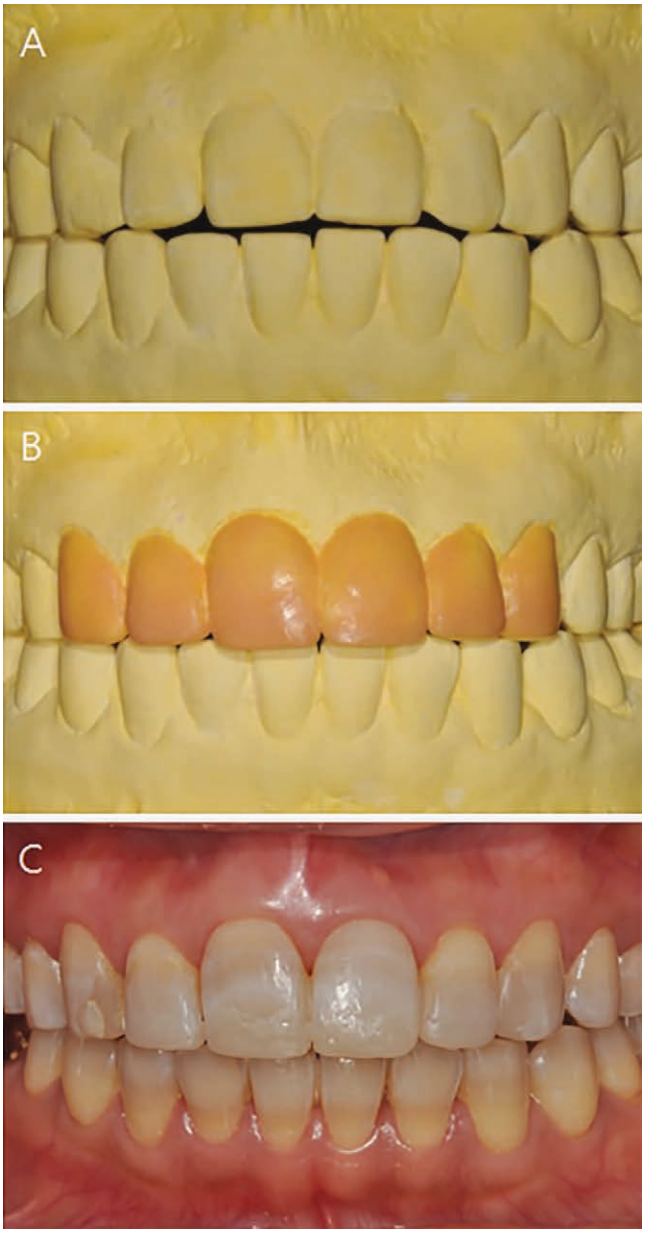

Figure 4. *(A): Preoperative study model. (B): Diagnostic wax-up. (C): Intraoral resin composite mockup.*

Decision #02: Management of the TC Discoloration of the Maxillary Anterior Teeth

Because of the severity of the discoloration, TC-stained teeth may discourage any attempt to improve the shade disharmony using bleaching agents, which usually do not remove all of the grayish and bluish undertones.[21,22] Our patient had a typical TC band and reported little effect of a bleaching treatment performed at a previous clinic. Therefore, the restorative options, ie, full veneer or laminate veneer restorations, were remaining options available to manage the discoloration. Full veneer restorations would have a more definitive effect in masking the intrinsic discoloration than laminate veneers and also would allow us to easily

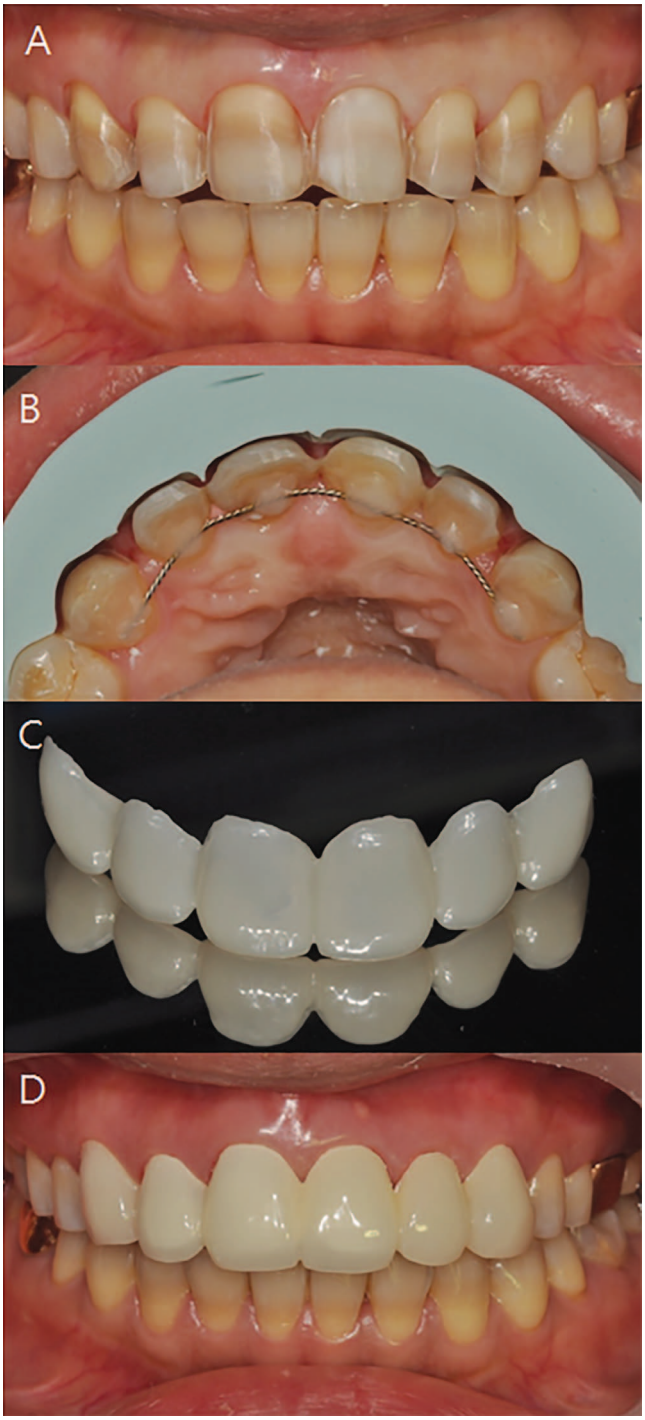

Figure 5. *Tooth preparation and provisionalization for laminate veneers. (A): Frontal view. (B): Occlusal view. The amount of reduction was checked using a silicone index, which was prefabricated from the diagnostic wax-up model. (C and D): Provisional restorations.*

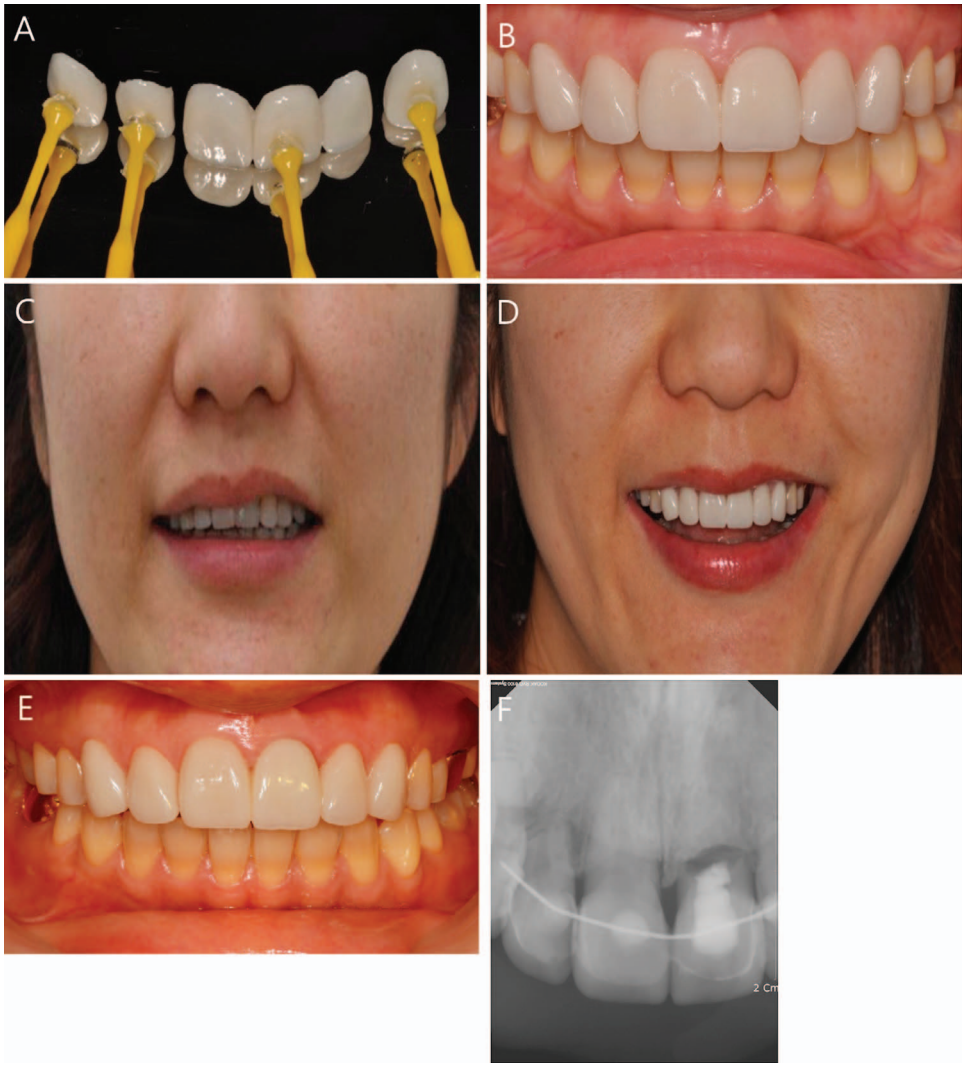

Figure 6. *(A): Porcelain laminate veneers. The prosthesis for teeth #8, #9, and #10 was splinted. (B): Intraoral photograph after cementation. (C): Pre- and (D) postoperative extraoral photograph. (E): One-year follow-up intraoral photograph and (F) periapical radiograph.*

correct the tooth shape and alignment: an open bite and excessive overjet in this case. However, they have shortcomings in terms of extensive reduction of tooth structure and the possibility of pulp irritation. In this case, the incisors had very short roots due to orthodontic complications. We instead chose laminate veneer restorations, which result in less destruction of the dentin and subgingival tooth structure.

The choice of laminate veneer restorations required us to consider several aspects in order to obtain shade improvement. First, the much darker discoloration of tooth #9, which was likely due to necrotic tissues or residual endodontic intracanal medicaments, needed to be removed to lessen the color difference reflected through the laminate veneers; the one-week nonvital bleaching removed the darker discoloration in the cervical area remarkably. With regard to the consideration of tooth preparations, a little more labial reduction was required to allow additional thickness for the ceramic veneers, which may have increased masking ability.[14,23,24] We also placed the gingival finishing line 0.5 mm subgingivally to avoid mismatching the shade of the porcelain and dark cervical tooth structure.[15] In addition, a high-value opaque lithium disilicate ingot (IPS e.max Press LT, Ivoclar Vivadent) was selected to mask the grayish discoloration effectively.[24-27]

Decision #03: Maintaining the Tooth That Had Poor Periodontal Support

Even though it was decided that tooth #9 be retained, it was a great challenge given its poor periodontal support due to the very short root and subsequent surgical treatment. Splinting was required because the replanted tooth was regarded as a natural tooth pontic. Splinted restorations are

often indicated for functional and esthetic rehabilitation in the setting of periodontally compromised dentition.[28] Splint crown restorations have also proven to be effective for stabilizing teeth and ensuring periodontal health.[29,30] In the present case, the resin splinting of teeth #8 and #10 and application of the lingual bonded retainer just after intentional replantation played a role in splinting tooth #9. Under normal occlusal conditions, there might not be a way to splint the tooth other than these two methods, even though preparations and placement of each laminate veneer would weaken the proximal resin splinting of tooth #9. However, our patient had excessive overjet and slight open vertical overlap, resulting in absent incisal contact in centric occlusion and protrusive movement. Therefore, we decided to reinforce the splinting of the replanted tooth using a splint laminate veneer, which is an exceptional restorative option. Under our patient's specific occlusion, the splint laminate veneer was believed to provide stability by splinting the tooth and minimizing the possibility of incidental failure associated with using only resin composite and wire.

The IPS e.max Press (Ivoclar Vivadent) used in this case is a reinforced lithium disilicate system with substantially improved physical properties including 400 MPa of flexural strength and greater esthetic characteristics with high translucency compared with other ceramic materials.[31,32] The minimally invasive treatment concept used in the present case was possible because of this improved dental porcelain and numerous reports of success in terms of the durability of bonded porcelain veneers.[33-36] The splint porcelain laminate veneer in this case provided more bulk in the splinted embrasures by slightly more tooth reduction in the corresponding embrasures of tooth #9, although literature supporting a standard splinting thickness could not be found. The proximal margins of the splinted embrasures were also placed more lingually to give a natural embrasure appearance.

Even though the splinted porcelain laminate veneer with a natural tooth pontic showed a very satisfying result on one-year follow-up, it needs to be observed as a temporary restorative treatment, because supporting literature on the expected longevity of this type of restorations was not found.

CONCLUSION

Using a systematic approach during the decision-making process, a comprehensive treatment plan was developed, and satisfactory esthetic results were achieved in the management of this complicated clinical case. Under a specific occlusion profile, which included the absence of incisal contact during central occlusion and protrusive and lateral movements, a splint laminate veneer with a natural tooth pontic was provisionally used as a conservative and esthetic treatment option to address the challenging conditions of the present case without complications from extraction or a full veneer crown. Periodic periodontal care would help to maintain the esthetic outcome and lengthen the service span of the splinted porcelain laminate veneers.

Conflict of Interest

The authors deny any conflicts of interest related to this study.

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