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

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Guest Editorial

Electronic Media and the Journal

K. Matis, Editorial Assistant

The following is being provided by one who far surpasses me in understanding electronic communications. Kevin Matis is our Editorial Assistant and I thank him for the contribution.

JA Platt, Editor

ltz jst sA dat Im frm d oldA gNR8N, n txt msgN, mob dvcs n tblt cmputAs seem vry "Star Trek" 2 M. It amzs me, thN, dat Op Dent S movN ryt alng N2 d 21st - or S dat d 22nd cntry...

Ahh, the text message – the secret language of today's youth. We recently had a conversation in an Editorial Staff meeting with one of the dental students here at Indiana University. He explained to us the news gathering processes of the current crop of students; needless to say, it had nothing to do with the newspaper or the 5 o'clock evening news broadcast. Today's news consumer gets their info from twitter, facebook, rss feeds and podcasts; for this group, the electron is mightier than the sword.

It hasn't been easy, but we figured that it is time to drag the journal, kicking and screaming, solidly into the electronic age. Sure we've dabbled, we have had manuscript submissions mainly electronic for 8 or so years now, we have published online for almost 6 years, and for the last 4 years we have done all our reviewing and manuscript processing exclusively online. Well, that isn't good enough now, it's time to really make this journal available to as many as desire to read it. With that in mind we would like to introduce you to some of our new and upcoming projects.

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Our Academy members have asked and asked, and it is here – a pdf of the entire journal. This pdf can be downloaded into your tablets, smartphones and other e-reader devices so you can take the journal with you wherever you go.

We have started a Newsletter. It isn't anything fancy, and it isn't anything that our online subscribers couldn't do for themselves; but it is one of the best read and most responded to newsletters in our industry! Many of you have seen the email – we have taken the table of contents of issue 3 a couple of days before it came out, and sent to all of our subscribers for whom we had an email address. The response was overwhelmingly positive: of the 2000 emails we sent out, 38.7% of you opened the email – compared with 22.7% industry wide, 12.6% of you clicked on the links to get to specific articles – compared with 1.5% industry wide! We are so proud of each of you for the interest that you take in staying current with new research and techniques that make you the excellent dentists that you are! If you did not get this Newsletter, and would like to, please visit our homepage at www.jopdent.org and fill in your email information at the bottom of the screen, choose the format you prefer to receive, then click subscribe, the next table of contents will be sent your way, with all the necessary links to access the articles.

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in Social Media. Facebook and Twitter are here to stay, and we are ready and (mostly) willing to "tweet", "retweet", "post to our wall" and be "liked" and "followed". We are still working on our twitter feed, but if you visit us on Facebook we are posting links to all of our online early content, so you can be the first to see new papers as they come out in the (almost) finished form. We intend to get twitter to do the same, but haven't worked all the bugs out quite yet. We encourage you to follow us on Twitter and Like us on Facebook, so you can get the most out of your subscription. We also recommend that you introduce these links to your friends and collegues. There are many out there that may only be interested in 2 –3 of our manuscripts a year and therefore a subscription would not be worthwhile, but having access to a feed of all the upcoming titles may pique their interest in some specific articles – a win-win for authors, the journal, and most of all for the profession.

Upcoming projects include a new mobile interface for the journal, so you can more quickly get to online content from your smartphones and tablets. It is an exciting thing for us seeing how this new programming will work. You can sit in your office, scan through the titles, mark articles you would like to read, then access them on-the-go in a link on your mobile device. You can be sitting in a plane, waiting for the door to close, pull up the list of articles, choose what you would like to read during the flight, and the device will quickly download the selected articles to your hard-drive so you can read them after you lose Wi-Fi.

We also expect that in the not too distant future, all our scientific content will be made available online. This will allow anyone to have the access they need to our past publications that include so much important foundational research for what we all do today.

I expect that sometime in the more distant future we will all be able to subscribe to inner-cranial subscriptions where we can have our journal downloaded directly to our cerebrum for instant access when we need it, but till that time – happy reading!

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Clinical Research

Comparison of Two At-home Whitening Products of Similar Peroxide Concentration and Different Delivery Methods

JB da Costa • R McPharlin • T Hilton
JL Ferracane • M Wang

Clinical Relevance

Tooth whitening products of similar peroxide concentrations provide similar whitening effects when applied for the same amount of time regardless of the delivery method.

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SUMMARY

Purpose: **This study compared the whitening efficacy, side effects, and patients' preferences/perceptions of two whitening systems of similar peroxide concentration but different formulation and delivery methods.**

Methods: **The tooth color change of 24 participants was measured using a shade guide (BSG) and a spectrophotometer (ES). Color difference was calculated: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. One whitening treatment was randomly applied to the right or left maxillary anterior teeth and the other was applied to the contralateral teeth, at-home with 35% carbamide peroxide in a tray (TW) or with 14% hydrogen peroxide in strips (WS). The tooth color was evaluated at baseline, 15 and 30 days (15 days postwhitening). Participants rated their tooth and soft tissue sensi-**

tivity (1–10 scale) and completed a questionnaire on their preferences. Results were analyzed by repeated measurement regression analysis/Tukey and Mann-Whitney ($p<0.05$).

Results: **At 15 days, the teeth treated with TW and WS presented ΔE* = 7 and 6, respectively (ΔBSG=3 for both), and at 30 days, they presented ΔE* = 7.5 and 6.5, respectively (ΔBSG=3 for both). There was no significant difference in tooth and soft tissue sensitivity between treatments. No participant reported tooth and gingival sensitivity at the postwhitening appointment. Of the participants, 83% preferred the TW over WS.**

Conclusion: **Both ΔE* and ΔBSG showed no significant difference in tooth color change between TW and SW at either time point. By the end of the study no participants reported tooth and gingival sensitivity. Participants preferred TW over SW.**

INTRODUCTION

The at-home whitening procedure was first published by Haywood and Heymann in 1989.[1] The 10% concentration of carbamide peroxide (CP) used in trays overnight has been considered the "gold standard." This technique is still the most common whitening procedure, and literature heavily supports the efficacy of this method.[1–14] Despite the great amount of research on at-home tray whitening with 10% CP, some patients do not desire to wear trays over a long period of time and still aspire to have whiter teeth faster. In order to achieve a faster whitening effect in a shorter period of time, dental product manufacturers have increased the peroxide concentration of the whitening products. Higher-concentration tooth whitening studies are scarce. Two studies evaluated whether higher concentration of peroxide, 20% CP vs 7.5% hydrogen peroxide (HP)[15] and 6% HP[16] could be used safely and effectively to reduce treatment time. Overall, these studies demonstrated the safety and effectiveness of these higher-concentration gels, with minor teeth sensitivity and oral tissue irritation. Recently, a manufacturer introduced to the market a new whitening system, Opalescence PF 35% CP (Ultradent, South Jordan, UT, USA). To date, there are no studies that have evaluated the efficacy of this high-concentration peroxide gel.

In 2000, a trayless whitening system, Crest Whitestrips (Procter & Gamble, Manson, OH, USA) was introduced on the market, in which a measured dose of HP gel was applied on the anterior teeth using a flexible polyethylene strip instead of a custom tray.[16] Initially, the whitening strips presented 6% HP, equivalent to 20% CP, and the manufacturer recommended applying them for 30 minutes twice daily. Several randomized controlled clinical trials showed the efficacy of this product.[13,17–24] Originally the strips were available over the counter. In 2004, the strips whitening gel concentration was increased, and they were designed to be professionally dispensed.[25] To date, there is one study comparing different professionally dispensed strips with 10% HP,[26] and there are a few studies that have evaluated the efficacy of higher concentration of whitening strips, 14% HP, Crest Whitestrips Supreme.[25,27,28]

The 14% HP Whitestrips Supreme, which corresponds to approximately 40% CP, is similar in concentration to the Opalescence PF 35% CP. Both products are dentist supervised and recommended to be performed at home, twice a day, for 30 minutes per each daily application. Unlike the strips, the 35% CP whitening gel is applied via a whitening tray. To the authors' knowledge, there are no studies that evaluated the efficacy of these high-concentration peroxide gels using different dispensing methods.

The objectives of this study were 1) to compare the whitening efficacy of two whitening systems of similar peroxide concentration using visual and instrumental shade matching methods, 2) to evaluate possible side effects such as gingival irritation and tooth sensitivity, and 3) to evaluate patients' preferences for and perceptions of both systems. The null hypotheses to be tested were that there is no difference in whitening effect (as measured by spectrophotometer and shade guide) and tooth and tissue sensitivity (as measured by visual analog scale) between Opalescence PF 35% CP and Crest Whitestrips Supreme 14% HP whitening systems over a 30-day evaluation period in maxillary anterior incisors and canines.

MATERIALS AND METHODS

This was a randomized, single-blinded, split-mouth design clinical study. One clinician gave instructions on materials to the patients, and another clinician evaluated the tooth color change. A total of 25 patients were selected for this study according to specific inclusion and exclusion criteria (Table 1).

During the screening appointment, the participants signed the institutional review board authorization and consent form. The Loe and Silness

Table 1: *Inclusion and Exclusion Criteria for Acceptance as Participants*

Inclusion Criteria	Exclusion Criteria
- Be at least 18 years old - Willing to sign a consent form - Willing to return for postwhitening evaluation - Presence of all six maxillary teeth equal or darker than 1M2 VITA Bleachedguide in the value order - Have no maxillary anterior teeth with more than one-sixth of the facial surface covered with a restoration	- History of any medical disease that may interfere with the study or require special consideration - Presence of gross pathology - Use of tobacco products during previous 30 days - Current or previous use of whitening agent - Loe and Silness[29] gingival score greater than 1.0 - Pregnant or lactating women - Tetracycline-stained teeth

gingival index[29] of the upper anterior teeth was used to ensure that participants did not have moderate to severe periodontal tissue inflammation. One impression of the maxillary arch was taken. The model was used for bleaching tray and positioning jig fabrication. A reservoir was placed on the stone teeth prior to tray fabrication using a block-out resin (LC Block-Out Resin, Ultradent) and the trays were scalloped. The positioning jig was fabricated in order to position the tip of the spectrophotometer in the same position at every color measurement. An impression of the tip of the probe of the spectrophotometer was made and a cast was fabricated. The spectrophotometer probe cast was used as a stamp guide to mark the positioning jig. The facial middle third of the maxillary teeth of the custom positioning jig was marked with the spectrophotometer tip cast using an ink pad. The facial marks were cut out of the jig, leaving an opening for placement of the spectrophotometer probe. Prior to color measurement, the custom jig was positioned in the patient's mouth, and the spectrophotometer probe was positioned into the jig opening. At the same appointment, the participants received a dental prophylaxis to remove any extrinsic stains.

At the baseline appointment, the participants received a nonwhitening toothpaste (Crest Cavity Protection, Procter & Gamble) and soft-bristled manual toothbrush (Oral B, Iowa City, IA, USA) and were asked to brush before and after the whitening procedure in addition to their standard home care regimen. The whitening treatments were randomly assigned, by flip of a coin, to the right (teeth nos. 6–8) or left (teeth nos. 9-11) maxillary anterior teeth. The 35% CP Opalescence PF (TW; Ultradent) was applied in a tray to the right or left maxillary anterior teeth, and the contralateral maxillary anterior teeth were treated with 14% HP Crest Whitestrips Supreme (WS; Procter & Gamble), which corresponds to 40% CP. The participants were instructed to whiten half the arch with the 40% CP strip and the other half with 35% CP in the whitening tray at the same time for 30 minutes. They were asked to whiten their teeth twice a day and to separate the times they whiten by at least three hours. They were instructed to whiten their teeth for two weeks. Participants only received half of the tray designated to be used with 35% CP gel, thus there would be no confusion as to which should be used with 35% CP. The tray was well adapted to the teeth, and the patients were asked to wipe off any excess material in order to prevent leakage to the adjacent tooth and to avoid gingival sensitivity. They were asked to adapt the strip to the teeth and not to overlap the tray. The participants also received a diary in which they indicated on a daily basis the level of tooth and gingival sensitivity that they experienced and the times they used the at-home whitening systems. They were asked to rate their tooth and soft tissue sensitivity experience using a visual analog scale (VAS) in one of 10 categories. The VAS is designed to present to the respondent a rating scale with minimum constraints. A 1 VAS corresponds to *no pain* and 10 VAS corresponds to *severe pain*.

At the baseline appointment tooth color was evaluated visually using the VITA Bleachedguide 3D-Master (BSG; Vita Zahnfabrik, Bad Sackingen, Germany) by one independent experienced evaluator and instrumentally using an intraoral spectrophotometer (ES; Vita Easyshade, Vident, Brea, CA, USA). Shade matching with the BSG was performed under a color-corrected light (Rite-Lite, Addent, Danbury, CT, USA), having a correlated color temperature of 5500 K that simulates northern-sky daylight. The ES measures the color of the teeth based on the CIELAB color notation system (Commission Internationale de l'Eclairage 2004), in which L* denotes lightness (achromatic), whereas a* and b* denote green-red and blue-yellow coordinates,

Table 2: *Questions to and Answers (by %) From the Study Participants*
1) Which method of treatment do you prefer?
• Tray whitening (83%)
• Whitening strips (4%)
• I have no preference (13%)
2) Which method is more comfortable for your teeth?
• Tray whitening (75%)
• Whitening strips (21%)
• Equally comfortable (4%)
3) Which method is more comfortable for your gums?
• Tray whitening (38%)
• Whitening strips (58%)
• Equally comfortable (4%)
4) Can you see a difference in tooth color between right and left teeth?
• Yes (25%)
• No (75%)
5) Was it easy to do the procedure twice a day?
• Yes (92%)
• No (8%)

respectively. ΔE^* is the total color difference or the distance between two colors. The total color difference was calculated using this formula[30]: $\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.

The color measurements were taken prior to tooth whitening (baseline), 15 days after the whitening treatment was started, and at 30 days (15 days postwhitening) in order to evaluate tooth color relapse.

At the last appointment the participants were asked to complete a questionnaire (Table 2), and if desired, they were given an upper whitening tray and whitening gel, Opalescence 10% CP (Ultradent Products), to keep whitening the upper teeth. The participants who wished to have the lower teeth whitened came back to have lower teeth impressions made and to produce trays for delivering the whitening gel.

STATISTICAL ANALYSIS

The results were analyzed with computer software (Sigmastat 3.1, Systat Software, Chicago, IL, USA) and SAS v.9.1 (SAS Institute, Cary, NC). A *t*-test ($p<0.05$) was used to compare the L*a*b* of the right and left upper anterior teeth obtained with the spectrophotometer at baseline. The Mann-Whitney rank sum test ($p<0.05$) was used to evaluate the BSG results of right and left upper anterior teeth at baseline.

The individual ΔL^*, Δa^*, Δb^*, and ΔE^* were computed by subtracting the baseline measurements from the follow-up measurements. The results were analyzed by repeated measurement regression analysis to evaluate color change over time for each treatment. A compound symmetry covariance structure was used to evaluate the variability and correlations between times and treatments. Data were adjusted for multiple testing by applying the Tukey test ($p<0.05$). The delta shade-guide rank results were analyzed by Mann-Whitney rank sum test. Both parametric and nonparametric tests evaluated two factors: whitening system (TW and SW) and time (baseline, 15 days, and 30 days).

The tooth and gingival sensitivity average score of tooth whitening were compared using Kruskal-Wallis one-way analysis of variance on ranks ($p<0.05$) for each whitening system and time.

The responses to the questionnaire are reported in percentages.

RESULTS

A total of 25 participants enrolled and 24 completed the study. One participant did not present for the last tooth-color evaluation. Of the participants, 12 were men and 12 were women, with an age range from 21 to 75 years. There were no statistically significance differences in the mean (ES) and median (BSG) baseline shade of the right and left teeth.

Both treatments had significant mean color change from baseline to after treatment (Table 3). The teeth became lighter, less yellow, and less red. There was no difference in ΔL^*, Δa^*, Δb^*, ΔE^*, and BSG values between time for each treatment and

Table 3: *Spectrometric and Visual Results of Both Whitening Treatments at 15 and 30 Days (15 Days Postwhitening), Mean ± Standard Deviation*

Treatment	Time, days	ΔL*	Δa*	Δb*	ΔE*	ΔBSG
TW	15	4.2 ± 2.6	−1.7 ± 1.2	−4.83 ± 2.5	7.0 ± 3	−3.0 ± 1.5
TW	30	4.0 ± 2.2	−1.7 ± 1	−5.6 ± 2.7	7.5 ± 3	−3.0 ± 1.4
WS	15	3.5 ± 2	−1.4 ± 1	−4.34 ± 2.6	6.0 ± 3	−3.0 ± 1.6
WS	30	3.42 ± 2	−1.5 ± 1	−4.7 ± 3	6.5 ± 3	−3.0 ± 1.6

Abbreviations: TW, tray whitening; WS, whitening strips.

between treatments at each time evaluated (Table 3).

According to the participants' diaries, five participants reported mild (1–3 in the VAS) tooth sensitivity and six participants reported mild soft tissue sensitivity on the side that was whitened with the TW. Four participants reported mild soft tissue and tooth sensitivity on the side that was whitened with the WS. There was no significant difference in tooth and soft tissue sensitivity between the two treatments. No participant reported tooth and gingival sensitivity at the postwhitening appointment.

Of the participants, 83% preferred the TW treatment; 75% found this type of treatment more comfortable to the teeth, though only 38% found it more comfortable to the soft tissue than WS (Table 2). Seventy-five percent of the participants could not see a difference in tooth color between the right and left upper incisors. The 25% of participants who noted a color difference reported that it was on the side that was whitened with the TW. Ninety-two percent found it easy to do the procedure twice a day.

DISCUSSION

According to the ES and BSG results, the hypothesis that both whitening treatments would provide similar results was accepted. HP is different from CP in composition (CP = HP plus urea), concentration (10% CP has 3.5% HP), and duration of activity. HP products are active for 30 to 60 minutes,[31] and CP products are active for two to 10 hours.[32] It was previously believed that an HP whitening agent would bring about faster results compared with a CP whitening agent of similar concentration. These claims were based on the fact that CP has to chemically break down into HP and urea in order to be effective.[15] However, like the present study, a previous study showed that CP and HP of similar concentrations applied for the same amount of time yielded similar whitening effects when evaluated with a shade guide.[15] The present study showed that the methods of whitening gel delivery, tray vs strip, did not influence the whitening effect. In addition to concentration, the degree of whitening is directly related to the amount of time that the agent is in contact with the tooth.[33]

At two weeks' postwhitening, the results were not significantly different from immediately after whitening treatment was ceased for both methods, therefore no color relapse was noted. According to Matis and others,[26] it appears that there is minimal reversal in color when wraps and strips are used. They commented that perhaps this is because the teeth's "inherent lightness potential" is not exceeded, and therefore, no color reversal occurs.[26] In their study, they evaluated wraps and strips containing 8% and 10% HP, and the time of application was similar to that used in this study. The common factor between Matis and others and this study was that the whitening gels were similar in concentration. Therefore it can be extrapolated that a higher concentration of whitening gels applied for several shorter periods of time can retain color better than low-concentration gels applied for several longer periods of time (eg, 10% tray whitening used overnight) or extremely high concentration applied for a short period of time (eg, in-office whitening).

Noteworthy is the low incidence of side effects with these products, especially in view of the relatively high peroxide concentration. Both whitening systems yielded minimal side effects in the study participants, with no differences between the methods. Of the participants, 21% reported mild tooth sensitivity when they used TW, and no symptoms

were reported at the last appointment. Also, 25% of the participants reported mild gingival sensitivity during TW use, and 16% reported mild transient gingival sensitivity during WS use. None of the participants reported gingival or tissue sensitivity two weeks after treatment for both systems. A study that evaluated the same strips reported that only two of the 13 participants in the experimental group reported mild tooth sensitivity, and only three reported mild soft tissue irritation.[27] The high-concentration CP products are relatively new on the market, and there are no studies showing its potential side effects.

The great majority of the participants preferred the TW over the WS. Most of them found the TW more comfortable on the teeth; nonetheless, 58% of them found the WS more comfortable on the soft tissue. Some participants commented that the strips are difficult to place and keep in place. Despite that, they were more gentle to the soft tissue, even though the trays were scalloped and the participants were instructed to wipe off any excess gel. A total of 92% found it easy to do the procedure twice a day.

CONCLUSION

Twice-daily use of 14% HP or 35% CP for two weeks resulted in significant improvement in tooth color relative to baseline that was sustained two weeks after treatment was finalized. There was no difference in effectiveness between treatments of similar peroxide concentration and different delivery methods. The tooth and gingival sensitivity was mild and transient for both groups. The great majority of the participants preferred tray whitening over strip whitening.

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Conflict of Interest Declaration

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

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Laboratory Research

Occluding Effect of Nd:YAG Laser and Different Dentin Desensitizing Agents on Human Dentinal Tubules *In Vitro*: A Scanning Electron Microscopy Investigation

LMS Al-Saud • HNA Al-Nahedh

Clinical Relevance

Varying degrees of dentinal tubule occlusion can be achieved with a Nd:YAG laser and the different desensitizing agents. The efficacy of these treatment modalities needs to be determined through clinical investigation.

SUMMARY

Objectives: This *in vitro* study aimed to microscopically evaluate and compare the occluding effect of the Nd:YAG laser and different dentin desensitizing agents on human dentinal tubules.

Materials and Methods: The Nd:YAG laser (SunLase™ 800) and four commercially available and professionally applied dentin desensitizers (Gluma® desensitizer, Tenure Quick®, Quell™ desensitizer, and VivaSens®) were investigated in this study. Sixty-four extracted intact human molars were used. Each dentin surface was divided by shallow indentation into two halves, one of which was used for treatment and the other of which served as a control. The dentin surfaces were etched to remove any smear plugs and to mimic the open dentinal tubules of sensitive dentin using 0.5 M ethylenediaminetetraacetic acid (pH 7.4) for two minutes (applied with a microbrush) and then rinsed with an air-water syringe for 30 seconds. The laser samples (n=16) were ran-

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domly divided into four groups of four samples each. These groups were the step-up technique group, the 14-day group, the one-minute group, and the two-minute group. Forty-eight samples were treated with the four tested desensitizing agents and were randomly divided into four groups (n=12/group). Each group was further subdivided into three subgroups (n=4). Samples of the first subgroup were treated for 14 days, while those of the second subgroup were treated once. Samples of the last subgroup were fractured longitudinally after a single treatment. All of the samples were then examined under a scanning electron microscope.

Results: The Nd:YAG laser–irradiated dentin showed reduction or complete obliteration of the dentinal tubule lumen; thus, the treatment modified the original dentinal structure. The lased dentin surface in the two-minute group showed bubble-like changes in the area of the dentinal tubules' orifices. Statistically, the two-minute group was found to have a significantly higher percentage of partially or fully occluded tubules than did the one-minute group. All of the studied desensitizing agents produced occlusion of the dentinal tubules; however, the appearance of the precipitates, the level of coverage, and the degree of dentinal occlusion varied among the tested products.

Conclusion: Throughout the specified period of this study, occlusion and/or narrowing of the open dentinal tubules have been successfully achieved with both treatment approaches.

INTRODUCTION

Dentinal hypersensitivity is one of the most painful and least predictably treated chronic conditions in dentistry.[1] It is defined as pain arising from exposed dentin, typically in response to chemical, thermal, or osmotic stimuli, that cannot be explained as arising from any other form of dental defect or pathology.[2]

The nature of the exposed dentin is of relevance, as not all patients exhibiting dentin exposure will develop sensitivity. The number and diameter of dentinal tubules at the tooth surface have been shown to be significantly increased in hypersensitive dentin.[3,4] While the exact mechanism of dentinal hypersensitivity is still controversial, the hydrodynamic theory is the most accepted hypothesis.[5] The dentinal tubules, which are open and wide, contain a fluid. According to this theory, this fluid expands when it is exposed to heat and contracts when it is exposed to cold or touch. The contraction and expansion change the pressure in the fluid phase, which in turn activates mechanoreceptor nerves close to the pulp. When nerve receptors are activated, sodium ions enter the dentin and potassium exits. This ion exchange polarizes the nerves and causes pain.[4] The concept of tubule occlusion as a method of dentin desensitization is a logical conclusion based on the hydrodynamic hypothesis.[6] The most commonly used agents in the treatment of dentin hypersensitivity can be broadly classified into the following groups: anti-inflammatory agents (corticosteroids), protein precipitants (formaldehyde, silver nitrate, strontium chloride hexa-hydrate), tubule occluding agents (calcium hydroxide, potassium nitrate, sodium fluoride), tubule sealants (resins and adhesives), and miscellaneous (laser).[1] Lasers may play a prominent role in treating hypersensitive dentin and providing reliable and reproducible results.[7,8] Usually a sequence of treatment methods are used, starting with the most conservative and switching to more aggressive treatment options if the original methods were not effective.

In clinical trials, several resin adhesives have demonstrated significant reductions in cervical dentin hypersensitivity. Gluma desensitizer is a resin material that was originally developed as the primer for a dentin-enamel adhesive system. This material's mode of action is based on resin and protein precipitation. It is believed that Gluma internally occludes the dentinal tubules, probably through coagulation of plasma proteins in the dentinal fluid.[9] According to the manufacturer, it achieves its effect by precipitation of plasma proteins, which reduces dentinal permeability and occludes the peripheral dentinal tubules. These inhibit the flow of fluid through the tubules that causes sensitivity.

Formulations containing potassium salts (eg, chloride, nitrate, citrate, and oxalate) are widely used for treating dentin hypersensitivity. In 2001, Gillam *and others*[10] evaluated four commercially available oxalate-containing products. Tenure Quick® (aluminum oxalate), Sensodyne Sealant® (ferric oxalate), and MS Coat® (oxalic acid) covered the dentin surface and occluded the tubules. However, ButlerProtect® (potassium oxalate) did not cover the surface to any great extent but provided some dentinal occlusion. The authors concluded that professionally applied in-office products containing oxalate are capable of covering the dentin surface and/or occluding the tubules to varying degrees.

Combining solutions of calcium chloride and potassium phosphate can result in the precipitation of amorphous calcium phosphate. Applied to exposed dentin, such a system could occlude dentinal tubules and reduce sensitivity.[11] Quell Desensitizer, which was used in this study, forms a gel on the surface of the tooth that deposits amorphous calcium phosphate (ACP) on the dentin surface and into the open dentinal tubules, according to the manufacturer.

The laser, by interacting with the tissue, causes different tissue reactions, according to its active medium, wavelength, and power density and based on the optical properties of the target tissue.[12] The lasers used for the treatment of dentin hypersensitivity are divided into two groups: low output power (low-level) lasers (helium-neon [He-Ne] and gallium/aluminum/arsenide [GaAlAs] (diode) lasers) and middle output power lasers (Nd:YAG and CO_2 lasers).[7] The laser interaction with the dental pulp causes a photobiomodulating effect, increasing the cellular metabolic activity of the odontoblasts and obliterating the dentinal tubules with the intensification of tertiary dentin production.[12]

It has been suggested[13] from experiments with lasers on hypersensitive teeth that the laser likely results in a melted dentin surface, with occlusion of open dentinal tubules. Cox and others[14] observed melted dentin, crazing on the surface, slight debris formation, and modification of dentin tubule structure where the tubule periphery had melted. The sealing of exposed dentinal tubules with melted and recrystallized dentin is caused by the thermal and occlusive effects of laser, which result in prolonged relief.[14] Dentin desiccation after laser irradiation is another possible mechanism, which could result in temporary relief of dentin hypersensitivity.[15] According to Sun and Tunér,[16] more difficult cases of dentin hypersensitivity can be treated by the use of lasers. A hypersensitive tooth that does not respond to 4 to 6 J per root in two or three sessions is indicated for endodontic treatment. They further recommend that the occlusal scheme is evaluated as part of the treatment protocol.[16]

The use of lasers may open up new dimensions in the treatment of dentinal hypersensitivity. Pashley and others[17] reported that CO_2 laser irradiation can fuse dentin and reduce dentinal permeability. Zhang and others[18] investigated the efficacy of the CO_2 laser in the management of dentinal hypersensitivity and found that the CO_2 laser (1 W in a continuous wave mode and irradiation time ranging from five seconds to 10 seconds) is useful in the treatment of cervical dentinal hypersensitivity without thermal damage to pulp.

The effectiveness of two types of lasers, the Nd:YAG laser (1 W and 10 Hz for 60 seconds at 1064 nm) and the 685-nm diode laser (25 mW and 9 Hz for 100 seconds), were evaluated in terms of dentin desensitization as well as both the immediate and late therapeutic effects on teeth with gingival recession. Both lasers were found to be effective in treating dentin hypersensitivity without adverse effect. However, the Nd:YAG laser was more effective than the diode laser for desensitization of teeth with gingival recession.[19] In another study,[20] the effectiveness of three types of lasers, Er:YAG (2940 nm, 60 mJ/pulse, 2 Hz, 20 seconds), Nd:YAG (1064 nm, 100 mJ/pulse, 15 Hz, 100 seconds), and GaAlAs (Diode; 808 nm, 100 mW, 20 seconds), as dentin desensitizers was evaluated. It has been found that Nd:YAG laser irradiation is more effective in the treatment of dentin hypersensitivity than are the Er:YAG laser and the diode laser.

The Nd:YAG is a near-infrared laser with a primary wavelength of 1064 nm. Other wavelengths exist with Nd:YAG but are not typically used in dentistry. It is very important to note that the Nd:YAG laser is non-ionizing in character and is therefore nonmutagenic. This wavelength is invisible, and, therefore, an additional laser beam or white light aiming beam is always added as an integral part of the system.[21] The ability to perform procedures at very low power settings is a unique and positive feature of the free-running Nd:YAG laser, since the aim is to use the smallest amount of energy to achieve therapeutic goals. The first use of Nd:YAG laser for the treatment of dentin hypersensitivity was reported by Matsumoto and others[25] in 1985.

The laser used in this study, SunLase™ 800 (Dental Laser System, SUNRISE Technologies), provides a constant beam of coherent, bundled, continuous monochromatic light with an emission wavelength of 1064 nm, which was delivered through a 320-μm optic fiber tip with a straight hand piece. The Nd:YAG laser was chosen in this study because it has been investigated in numerous studies and has been reported to be effective in treating dentin hypersensitivity *in vitro* and *in vivo*.[1,8,22,23] In addition, since this laser is one of the most widely studied lasers, it would be a good choice for comparison with other treatment modalities.

Since dentin permeability and hypersensitivity are both reduced when the dentinal tubules are obtu-

rated or occluded, techniques and/or agents that effectively occlude the dentinal tubules are extremely important tools in managing hypersensitive dentin. Many desensitizing treatments have been investigated both *in vitro* and *in vivo*.[24-32] However, no study to date has compared the effect of several different desensitizing agents with the effect that the Nd:YAG laser has on the dentinal tubule's morphology. The aim of this study was to quantitatively and qualitatively investigate and compare the occluding effect of the Nd:YAG laser and some desensitizing agents on human dentinal tubules using scanning electron microscopy (SEM).

MATERIALS AND METHODS

Sixty-four freshly extracted caries-free human first and second molars were used in this study. Immediately after extraction, they were stored in a dark glass container of 0.025% thymol solution at 4°C to inhibit microbial growth until use. The roots of the teeth were cut just below the root furcation area using a diamond disc (Rotary Dental Instrument, Kahla, Germany) on a straight hand piece (KAVO EWL type 4415, KAVO, West Germany), then the pulp chamber was closed with sticky wax to prevent seepage of pulpal tissues. Facial or lingual surfaces (depending on which of them is more flat) were ground flat at the cervical one-third of the crown to remove enamel and to expose superficial dentin using 240-grit and 320-grit aluminum oxide paper (HANDIMET II Roll Grinder, BUEHLER, USA).

Each dentin surface was divided into two halves. One half was marked by a dimple on the enamel surface and was exposed to the treatment, and then the other half was used as a control (Figure 1). Fractured specimens were prepared so that the entire surface received the treatment after a minimal groove had been made on the enamel surface vertically and parallel to the long axis of the tooth to aid the fracture procedure. Prior to treatment, the exposed dentin surfaces were polished with wet 400-grit and 600-grit aluminum oxide abrasive paper; an average grinding time of 30 seconds was used for each specimen. Then specimens were placed in an ultrasonicator (SONICER, Yoshida Dental Mfg Co Ltd, Osaka, Japan) filled with distilled water for 30 minutes to remove any debris. The dentin surfaces were etched to remove any smear plugs and to mimic the open dentinal tubules of sensitive dentin using 0.5 M ethylenediaminetetraacetic acid (EDTA) (pH 7.4) for two minutes (applied with a microbrush); samples were then rinsed with an air-water syringe for 30 seconds[28] (Figure 2).

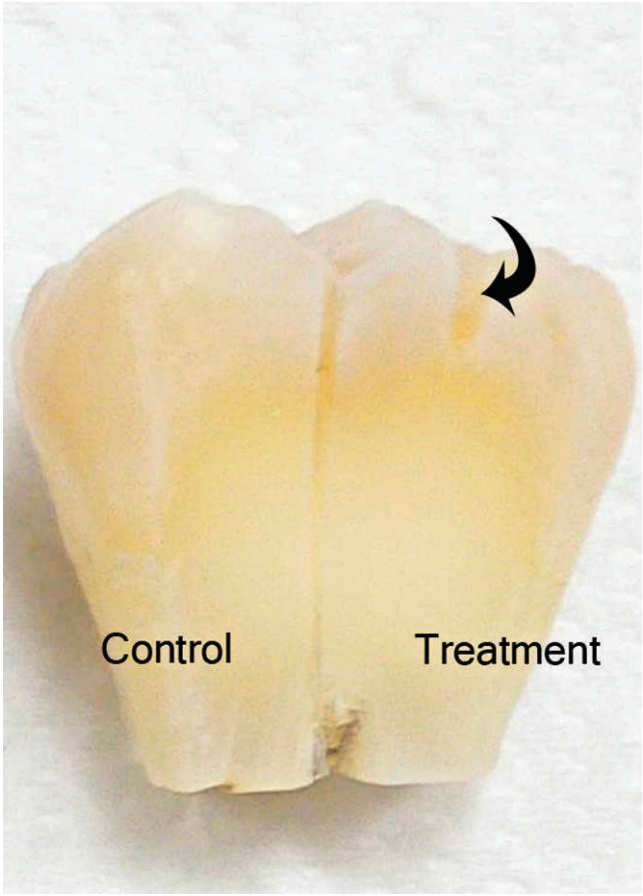

Figure 1. *Prepared tooth specimen with the dentin surface divided into two halves by shallow indentation. Note the dimple that indicates the treatment side (arrow).*

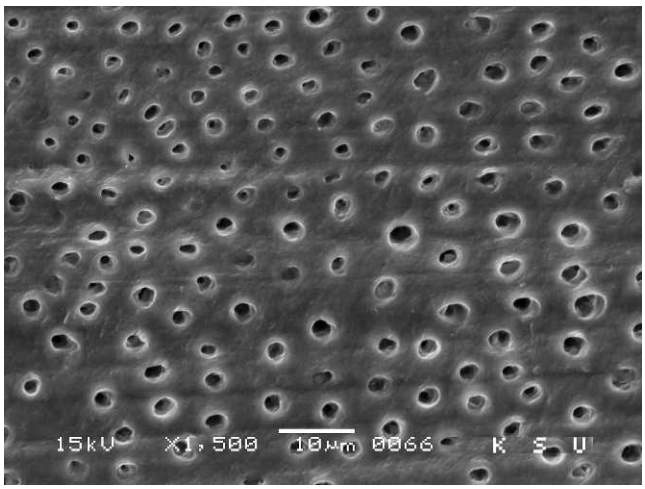

Figure 2. *Scanning electron micrograph (1500×) of dentin surface that has been etched with 0.5 M EDTA (pH 7.4) for two minutes to remove smear layer and to mimic the open dentinal tubules of sensitive dentin.*

Table 1: *Application Time, Techniques, and Power Densities for the Pulsed Nd:YAG Laser Group*

Fiber Type	PPS (pulses per second)	Application Technique	Power, W	Application Time
320 noncontact mode	10	Step-up technique	0.4	30 s for each degree of intensity, with 15-s lag between irradiations
			0.5	
			0.6	
			0.7	
			0.8	
		Single power	0.8	One min
		Single power	0.8	Two min

The samples were randomly divided into the following treatment categories. 1) Pulsed Nd:YAG Laser SunLase™ 800 (Dental Laser System, SUNRISE Technologies) was used to treat the dentin samples. The laser was applied in a "step-up" procedure (30 seconds for each degree of intensity), with a 15-second lag between irradiations for thermal relaxation of the tissue. The machine is supplied with a 40° radius cannula for 320-μm fiber. The laser tip was held perpendicular to the irradiated surface in noncontact mode 2–3 mm away from the tooth in a continuous flowing motion. The laser tip was swept in a zigzag pattern and in circular fashion, respectively; a distance of approximately 1 mm was maintained between consecutive lines to ensure complete surface coverage. According to the manufacturer, the maximum intensity for desensitization must not exceed 0.8 W per application. Application time, techniques, and power densities for the pulsed Nd:YAG laser group are shown in Table 1.

A total of 16 samples were randomly distributed into four groups, as follows: Group 1: Step-up group; four samples were lased with the Nd:YAG laser using the step-up technique mentioned above and were observed immediately; Group 2: Step-up 14 group; four samples were lased twice (days 1 and 14) with the Nd:YAG laser using the step-up technique mentioned above. In between laser applications, the samples were stored in artificial saliva solution until day 14, when the treatment was repeated;

Group 3: One-minute group; four samples were lased with the Nd:YAG laser using only 0.8 W for one minute. Two of the samples were frozen using liquid nitrogen and were fractured using a sharp chisel and a mallet, and the internal tubule morphology was observed immediately under SEM; and Group 4: Two-minute group; four samples were lased with the Nd:YAG laser using only 0.8 W for two minutes. Two of them were frozen using liquid nitrogen and fractured using a sharp chisel and a mallet, and the internal tubule morphology was observed immediately under SEM.

2) The desensitizing agents: The desensitizing materials used and their composition and method of use are summarized in Table 2. The treatment details were determined according to the manufacturer instructions supplied with each material. Group 1: Gluma® desensitizer (Heraeus Kulzer, Dormagen, Germany); Group 2: Tenure Quick® (Den-Mat Corporation, USA); Group 3: Quell™ desensitizer (Pentron Clinical Technologies, LLC, Wallingford, CT, USA); and Group 4: VivaSens® desensitizing varnish (Ivoclar Vivadent AG, FL-9494 Schaan/Liechtenstein).

For each of the four previously mentioned groups, a total of 12 samples were randomly distributed into three subgroups, as follows: i) Multiple applications: four samples received 14 days of treatment. The material was applied every fourth day (days 1, 5, 9, and 13). For the duration of the experiment, the samples were stored in artificial saliva at 37°C until day 14 and were then evaluated. The artificial saliva solution was changed after each treatment application; ii) Single application: four samples received a

Table 2: *The Desensitizing Materials Used in this Study, Their Composition and Method of Use*

Material/Manufacturer	Composition	Method of Use
Gluma® desensitizer	5% Glutaraldehyde, 2-hydroxyethyl-methacrylate (HEMA) purified water	Applied with a microbrush and left for 60 s, dried, then rinsed with water
Tenure Quick®	Aluminum oxalate–based agent	Six coats were applied with a microbrush, dried, then light-cured for 30 s; procedure was repeated once
Quell™ desensitizer	Amorphous calcium phosphate (ACP)–based agent	Using a microbrush, dentin surface was rubbed with part A solution for 10 s (repeated twice) and the surface was left wet. Then part B solution was applied in the same manner and repeated two times as well
VivaSens® desensitizing varnish	Liquid varnish (ethanol, water, and hydroxypropylcellulose) containing potassium fluoride, polyethyleneglycol dimethacrylate, and other methacrylates	Three drops (mixed with a brush precoated with organic acid) were applied by gentle surface rubbing, then gently air-dried for 10 se

single treatment with Gluma and were then observed immediately under SEM; and iii) Fracture samples: four samples received a single treatment application, and then they were frozen using liquid nitrogen and fractured using a sharp chisel and a mallet for observing the internal tubule morphology. Care was taken to avoid potential dissolution of the desensitizing agents on the dentinal surface, which may occur with conventional specimen preparation.

SEM Preparation and Evaluation

The specimens were washed and air-dried and then prepared for SEM examination. The specimens were mounted on SEM stubs and sputter-coated with approximately 300 Å of gold (Fine Coat JFC 1100, Teol Ltd, Tokyo, Japan) and examined under low-vacuum SEM (Jeol JSM-T330 A, Jeol Ltd). The treated dentinal surfaces were scanned and their micromorphology was evaluated. Additionally, the presence of any dentinal surface alteration, precipitation, or debris was noted and described. Representative scanning electron micrographs were taken at different magnifications. They were chosen based on the frequently observed appearance of the treated dentin surface, and they were judged by two evaluators to represent the treatment effect.

Quantitative Assessment

Following the pilot study, it was observed that only the Nd:YAG laser and the Gluma desensitizer groups were amenable to quantitative assessment (percentage of tubule occlusion), because all the other treatment surfaces in other groups were totally covered by the desensitizing materials and, thus, there were no dentinal tubules evident for counting.

The percentage of partially and/or fully occluded tubules was calculated for each representative micrograph from groups 1 and 2; four micrographs were evaluated for each specimen using the following simple formula:

$$\textit{Percentage of partially or fully occluded tubules} = \frac{\textit{Number of partially or fully occluded tubules} \times 100}{\textit{Total number of tubules}}$$

The fully and partially occluded tubules in each micrograph were marked using a computer program (Adobe® Photoshop® Elements 2.0, version 2.0.2, Adobe Systems Incorporated, Adobe, Tokyo, Japan), and the mean percentages were then calculated for both groups. All the micrographs calculated were at magnification 2000×, and the dimension of the picture side was 10 μm.

Using SPSS 12 for windows (SPSS Inc, Chicago, IL), statistical analysis was done using one-way analysis of variance (ANOVA) and the Tukey post hoc test for multiple comparisons at the 5% level of significance (95% confidence level) for the six subgroups (laser step-up , laser twice step-up, Gluma immediate, Gluma 14 days, laser one minute, and laser two minutes).

Qualitative Assessment

The micrographs were evaluated for their surface characteristics and for the patency or occlusion of the

dentinal tubules. Ranking criteria for surface characteristics of the micrographs (Figure 3) was conducted using the following descriptive categories (modified and adapted from Kumar and Metha[1]):

A. The dentinal tubules are partially occluded and dentinal orifices are slightly smaller, with little or no debris.
B. The dentinal tubules are mostly occluded; the dentinal surface is devoid of film or precipitate.
C. The dentinal tubules were mostly occluded; the dentinal surface is partially covered with film or precipitation or shows some surface alterations.
D. All the dentinal tubules are totally occluded and the surface is totally covered with precipitate and/or a resin film.

RESULTS

Quantitative assessment for the Nd:YAG laser and Gluma groups showed that the one-minute laser group had the smallest mean percentage of partially or fully occluded tubules, while the two-minute laser group had the highest mean percentage. One-way ANOVA revealed statistically significant differences (in the mean percentages of partially and fully occluded tubules) between at least two of the six groups (laser step-up immediate, laser step-up 14 days, Gluma immediate, Gluma 14 days, laser one minute, and laser two minutes) ($p<0.05$). The Tukey post hoc test for multiple comparison revealed a statistically significant difference ($p<0.05$) between the laser one-minute and the laser two-minute groups; however, for the other groups, no statistically significant differences were found ($p>0.05$).

Qualitative assessment of micrographs of dentin surfaces treated by the tested materials revealed different patterns of material deposition onto the dentin surfaces. The results demonstrated that all of the applied desensitizing agents as well as the laser produced occlusion of the dentinal tubules, although the level of coverage and occlusion varied between the products.

Nd:YAG Laser

The lased dentin surface in all treatment subgroups showed occlusion of open dentinal tubules in most areas, with evident narrowing and reduction in the diameter of the affected tubules. The dentin surface showed little or no alteration in the samples treated using the step-up technique; however, the majority of dentinal tubules were fully occluded, and some showed only a reduction in their diameter (Figure 4a). The same micromorphology was observed in the samples treated twice (during 14 days) with the step-up technique, although their dentinal surfaces showed some precipitations of surface debris (Figure 4b). In some irradiated samples, there appeared to be some banding or zigzag patterning at the dentinal surface in the areas where the laser beam had passed, and it reflected the method that was used in

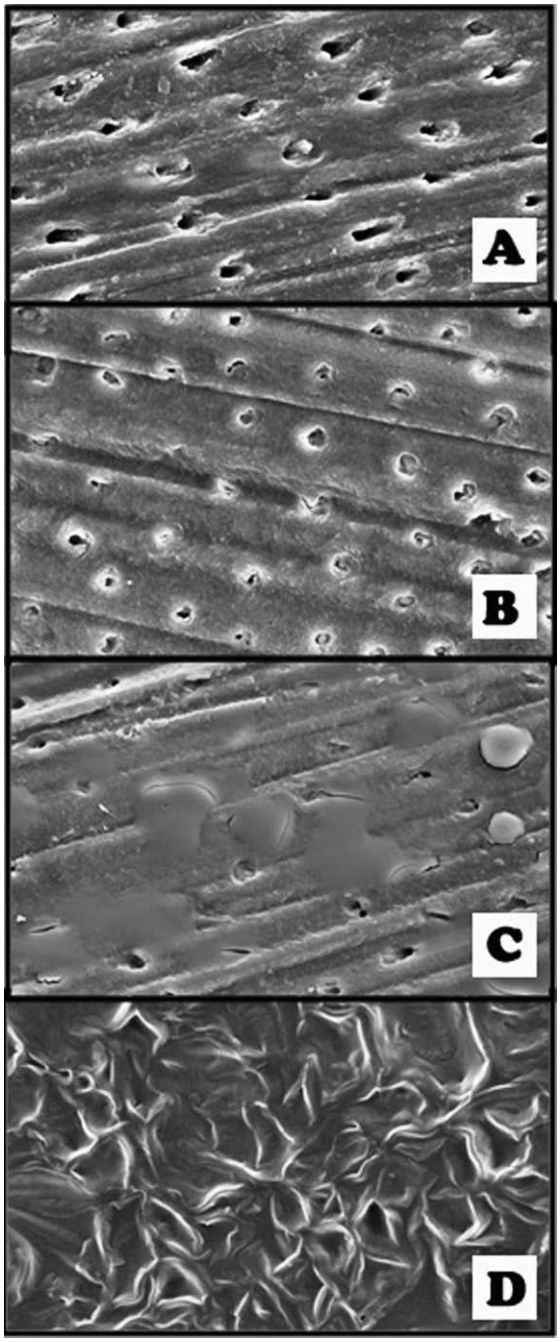

Figure 3. *Representative micrographs of the descriptive categories of the qualitative assessment (picture index).*

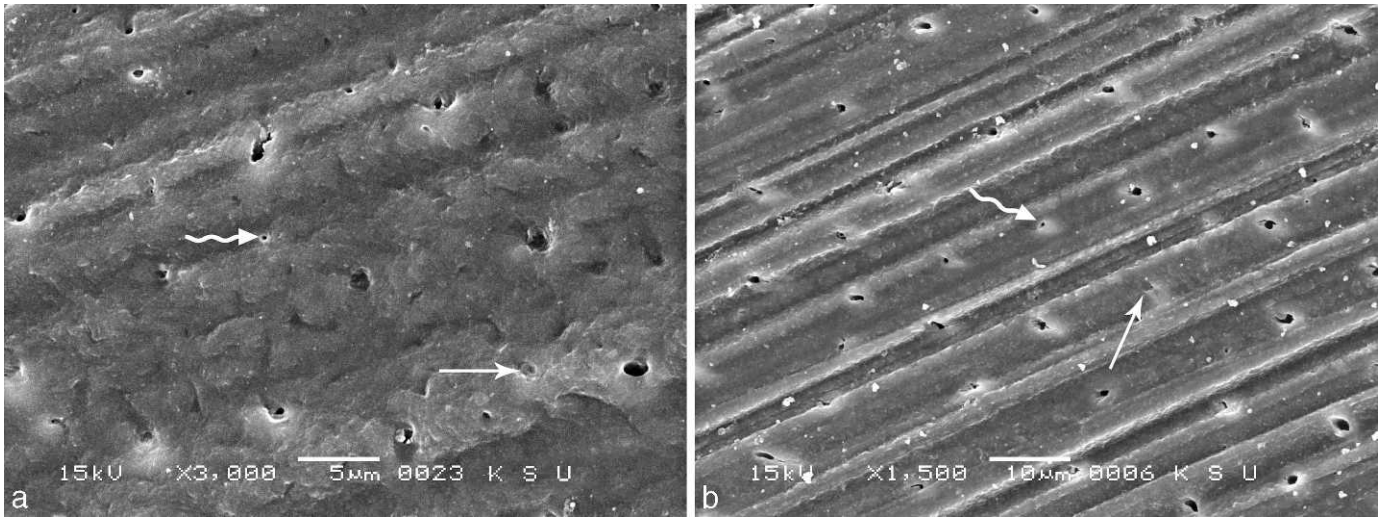

Figure 4. *Scanning electron micrographs showing (A) dentin surface treated once with Nd:YAG laser using the step-up technique(3000×) and (B) after multiple applications and storage in artificial saliva solution for 14 days (1500×). Note the fully occluded tubules (arrow) and the tubules with reduced diameter (curved arrow).*

the application of the laser beam. The samples lased for one minute using the power of 0.8 W showed many occluded tubules as well as some tubules with reduced diameter, and there were no precipitations or surface alterations on the treated dentinal surfaces (Figure 5a). The fractured samples of this subgroup showed some sealed dentinal tubule orifices (Figure 5b). The samples lased for two minutes using the power of 0.8 W showed not only dentinal tubules that were closed and sealed but also peritubular dentin that appeared to be melted. It was smooth and glossy compared to the surrounding dentin surface (Figure 6a), with round-elliptical, bubble-like changes at (and around) the area of the dentinal tubule orifices. The fractured samples of the laser two-minute group showed occluded tubules in some areas at the surface, and in some micrographs the melting of the dentin subsurface could be observed (Figure 6b).

Gluma®

Generally the samples treated with Gluma desensitizer showed some occluded dentinal tubules with a dentin surface that was devoid of debris but showed some intratubular material precipitations. No mi-

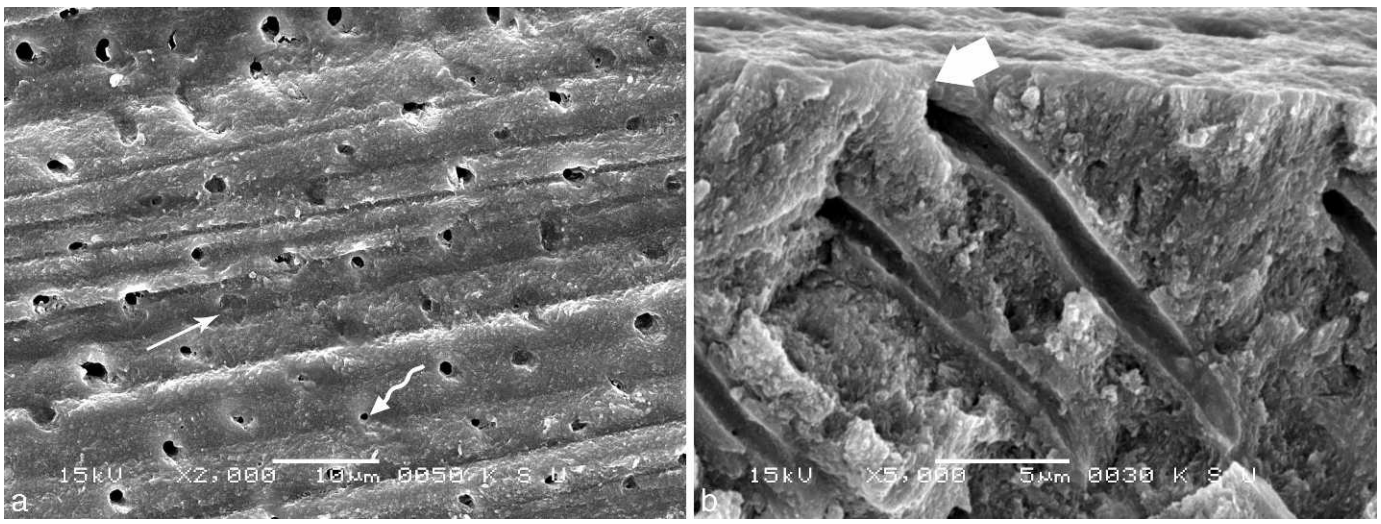

Figure 5. *Scanning electron micrographs showing dentin surface treated with Nd:YAG laser (0. 8W for one minute). (A) Treated dentin surface (2000×). (B) Fractured dentin surface (5000×). Note the fully occluded tubules (arrow) and the tubules with reduced diameter (curved arrow) and the occluded tubule orifice (thick arrow).*

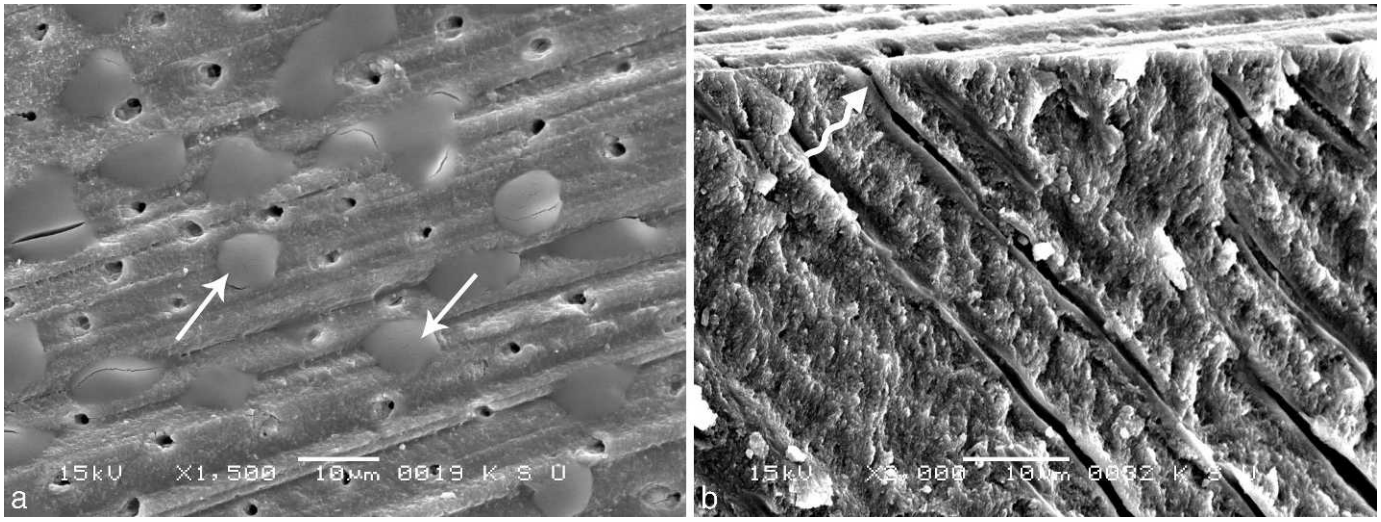

Figure 6. *Scanning electron micrographs showing dentin surface treated with Nd:YAG laser (0.8 W for two minutes). (A) Treated dentin surface (1500×). (B) Fractured dentin surface (3000×). Note the laser-induced surface changes, occluded tubules as well as melting of the area around the tubules (arrows), and the tubule orifices closure or (melting) (curved arrows).*

cromorphological differences were found between the single-application samples and the multiple-application (14-day) treatment samples. Some dentinal tubules were closed, some appeared open, and others showed reduced lumen diameter (Figure 7a). The fractured samples showed some tubular occlusion and tubule plugs. Frequently the precipitations were observed to be extended into the tubule lumen more than they were in any other treatment group, although the surface deposits were comparatively much smaller. In some areas of the fractured samples, the deposits were found to be extended 7.38 μm inside the tubule lumen (Figure 7b).

Tenure Quick®

Total coverage of the dentin surface was observed in all samples treated with this aluminum oxalate–based material, as the dentinal tubules were no longer visible. The treated dentinal surface appeared rough and irregular with multiple crystal-like structures. In some areas there were clusters of the material (highly charged and appeared intensely white). This micromorphology was observed in the single-application as well as the multiple-application (14-day) treatment samples (Figure 8a). When the treated dentin surfaces were fractured, it was observed that the material formed a thick (up to

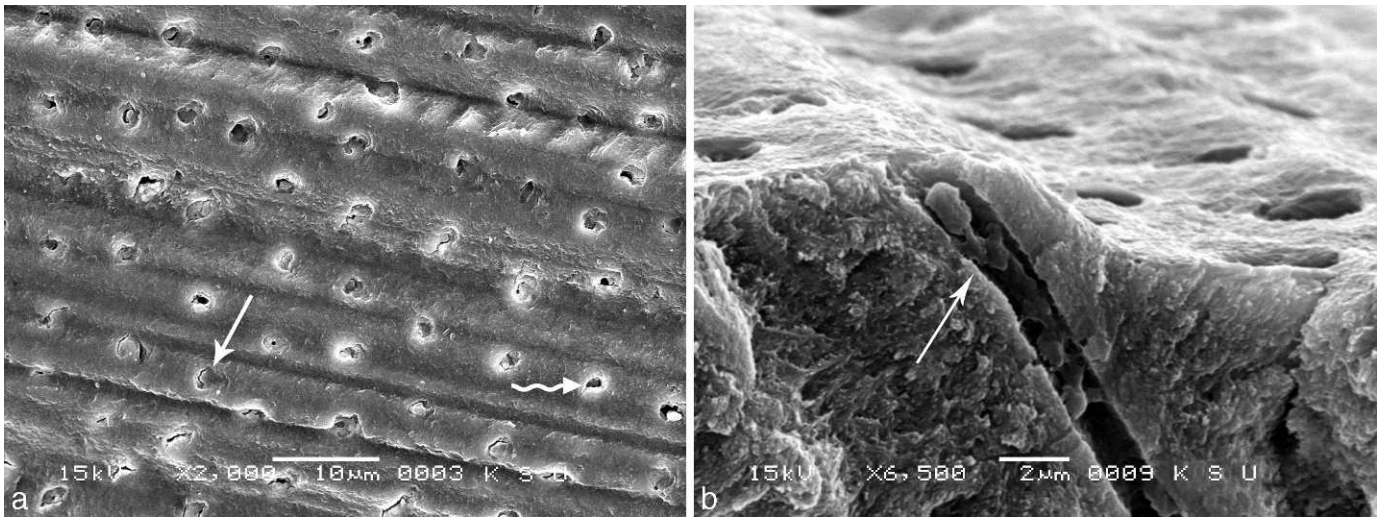

Figure 7. *Scanning electron micrographs showing (A) the dentin surface treated with Gluma (2000×). Note the fully occluded tubules (arrow) and the reduced diameter tubules (curved arrow). (B) The fractured dentin surface (6500×). Note that the material penetrated to a considerable depth into the dentinal tubule lumen (two-way arrow).*

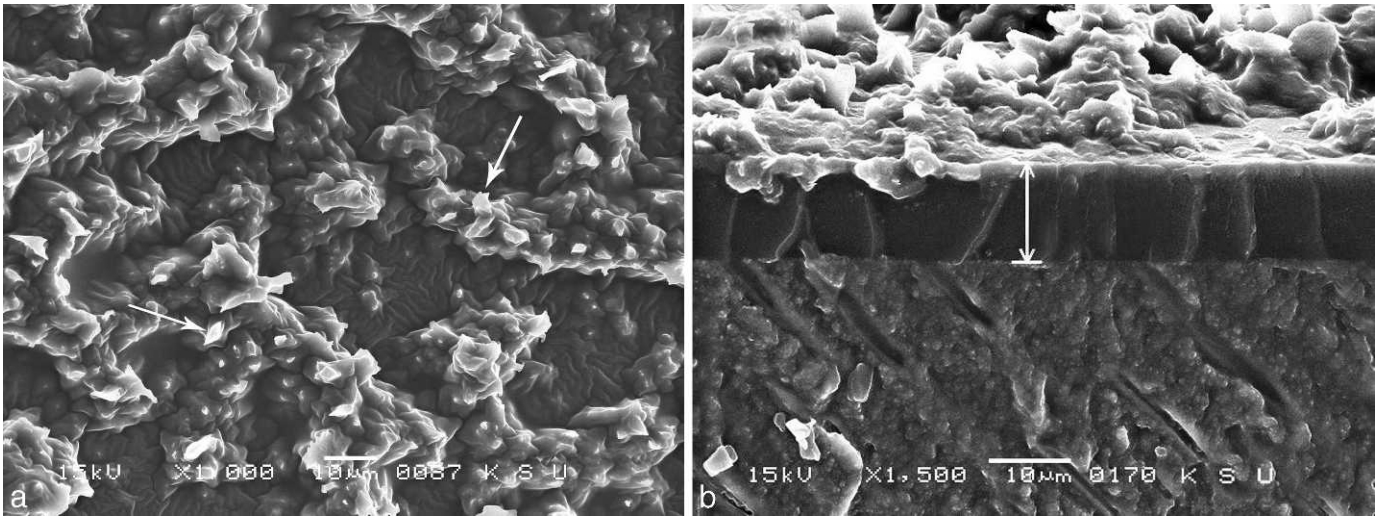

Figure 8. *Scanning electron micrographs showing (A) the surface deposition of Tenure Quick on dentin surface (1000×). Note the complete coverage of the dentinal tubule orifices and the crystal-like structures (arrows). (B) The dentin surface after fracture (1500×). Note the thick material deposit on the dentin surface (two-way arrow) and the completely plugged tubule orifices.*

11.58-μm) solid layer (Figure 8b) that was closely adhered to the dentin surface and that had numerous crystalline surface deposits. In some areas the material penetrated the tubule lumen to some extent.

Quell™

Scanning electron micrographs of dentin samples treated with amorphous calcium phosphate, the active ingredient in Quell desensitizer, showed that the dentin surfaces were totally covered with a thick, rough, porous woven layer, and the dentinal tubules were not visible at all. This micromorphology was observed in the single-application as well as the multiple-application (14-day) treatment samples (Figure 9a). When the treated dentin surfaces were fractured, it was evident that the material occluded the tubule orifice with crystal-like structures, and the rough, thin surface layer (1-1.5 μm) appeared to be closely adhering to the dentin surface. Some material crystals appeared to penetrate the tubule lumen to some extent (Figure 9b).

VivaSens®

All of the samples treated with VivaSens showed total coverage of the dentinal surfaces with an apparently smooth layer of wavy, fiber-like, glossy

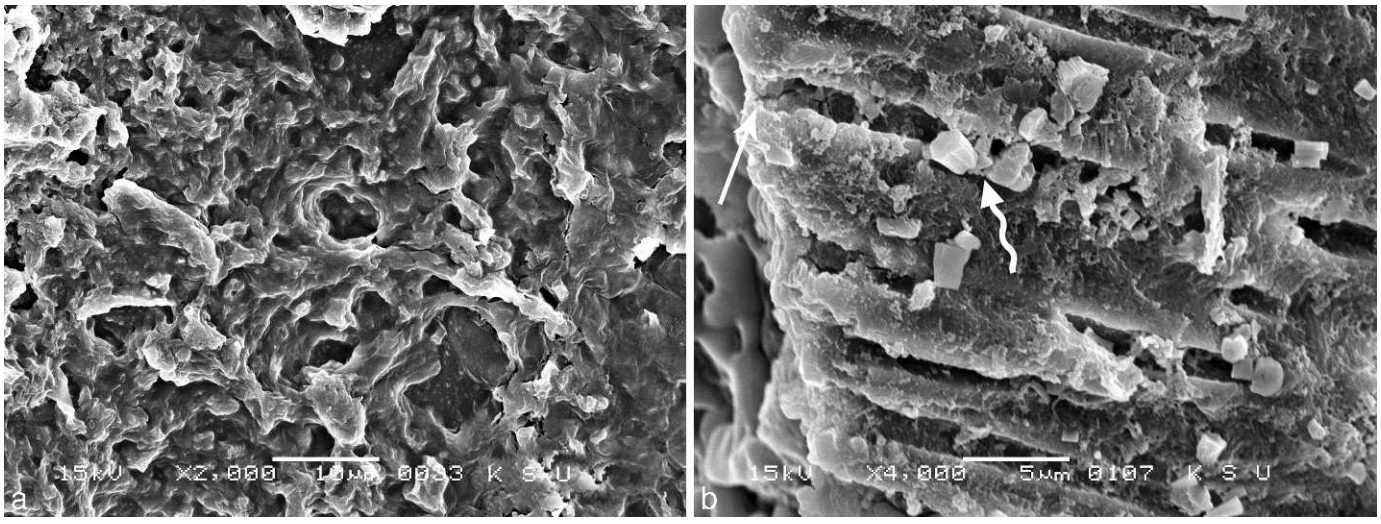

Figure 9. *Scanning electron micrographs showing (A) the surface deposition of Quell desensitizer on dentin surface (2000×). Note the woven porous appearance of the thick deposit that completely masked the dentinal tubule orifices. (B) The fractured dentin surface (4000×). Note the occluded orifices (arrows) and the material extension into the tubule lumen (curved arrows).*

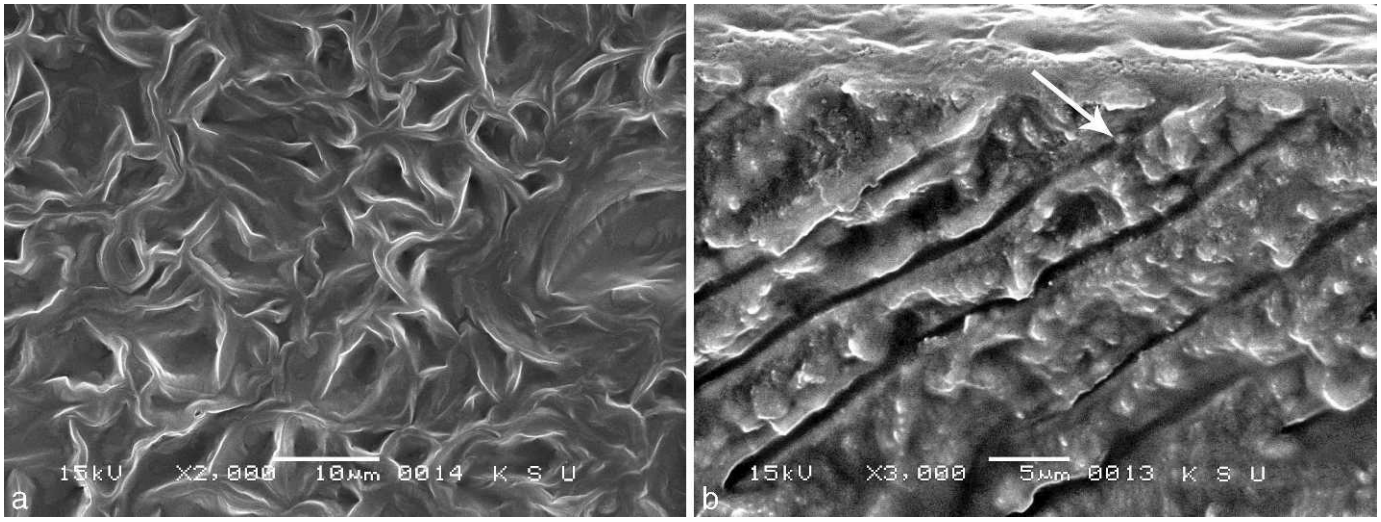

Figure 10. *Scanning electron micrographs showing (A) the dentin surface treated with VivaSens (2000×). Note the complete surface coverage with this resin-based desensitizer. (B) The fractured dentin surface; note the resin-based material extension into the tubules (arrow).*

resin coating that masked the dentinal tubules as well as the whole dentin surface. This micromorphology was observed in the single-application as well as the multiple-application (14-day) treatment samples (Figure 10a). When the treated dentin surfaces were fractured, the surface resin precipitate appeared to be thin (2-5 μm) and intimately coated the dentinal surface. Additionally, this resin material was found to extend into the tubule lumen to some degree (Figure 10b).

For all of the four desensitizing agents, there appeared to be very minor micromorphological differences between the immediate and the 14-day groups. Therefore, the two groups were pooled for the qualitative assessment. The fractured samples were evaluated separately. In the laser-treated groups (Table 3) the majority of the samples fell into categories B and C, while the Gluma samples mainly belonged to category B. For Tenure Quick, Quell, and VivaSens, all of the samples were marked as category D except for the fractured samples, which were marked as category C (Table 4).

DISCUSSION

Interstitial fluid movement within the dentinal tubules is the basis for the transmission of sensations.[27] A possible approach to reducing or eliminating the painful symptoms of dentin hypersensitivity is the interruption of stimuli transmission to the nerve endings of odontoblastic processes by reducing the fluid movement inside the dentinal tubules through the narrowing or occlusion of tubule openings.[30,31] Dentinal tubules can be sealed on the dentin surface, occluded within their orifices or in the subsurface dentin within their tubules. It can be assumed that intradentinal closure or seal is the most promising approach with regard to long-term success.[35]

The dentin surface of all of the samples was prepared by etching with 0.5 M EDTA prior to treatment. This step was done to ensure that the prepared dentin surface was free of any smear layer or smear plugs that might be confused with treatment materials. Furthermore, sensitive dentin has been shown to have wide patent tubules,[3] so the etching process aimed at simulating the open tubules of the sensitive dentin. If etching was not done, the dentin surface would be covered with debris and smear layer from the sample preparation steps; this layer has been shown[30] to reduce the permeability of dentin. Therefore, fluid movement within the dentinal tubules, a prerequisite for sensitivity according to the hydrodynamic theory, would be absent.[33]

The professionally applied desensitizing treatments used in the present study were selected according to their different mechanisms of action, namely the thermal effect of laser energy, the protein precipitation effect of Gluma, the tubule occlusion by ion precipitation produced by Tenure Quik and Quell, and the sealing effect of the resin-based VivaSens desensitizing varnish. These different treatments ultimately produced the desired treatment goal, which was the occlusion of the exposed dentinal tubules. Nd:YAG laser–irradiated samples in all subgroups showed considerable

Table 3: *Micromorphological Ranking of the Nd:YAG Laser Group According to the Qualitative Assessment Categories*[a]

Treatment	Category				
	A	B	C	D	N
Laser step-up	0	3 samples (75)	1 sample (25)	0	4
Laser twice step-up	0		All samples (100)	0	4
Laser one-minute (0.8 W)	0	3 samples (75)	1 sample (25)	0	4
Laser two-minute (0.8 W)	1 sample (25)	0	All samples (75)	0	4

[a] *Parenthetical values represent percent. A = The dentinal tubules are partially occluded and dentinal orifices are slightly smaller, with little or no debris. B = The dentinal tubules are mostly occluded; the dentinal surface is devoid of film or precipitate. C = The dentinal tubules were mostly occluded; the dentinal surface is partially covered with film or precipitation or shows some surface alterations. D = All the dentinal tubules are totally occluded and the surface is totally covered with precipitate and/or a resin film. N = number of specimens.*

occlusion of the open dentinal tubules to varying degrees. It was clear that the laser energy had affected and modified the whole dentin surface and not only the dentinal tubules, where melting and re-solidification of the area surrounding the tubules was seen, particularly in group 4, in which the samples had been lased for two minutes. The lased dentin surface appeared smooth, with round-elliptical, bubble-like changes at (and around) the area of the dentinal tubule orifices. This could be attributed to the photo-thermal effect of the laser energy, whereby the high temperatures caused the tubular orifices to melt and swell, leading to this appearance. Such changes indicate excessive heating of dentin and could result in pulpal damage in vital teeth. Our findings are consistent with those of Cox and

Table 4: *Micromorphological Ranking of the Desensitizer Groups: 1 (Gluma®), 2 (Tenure Quick®), 3 (Quell™), and 4 (VivaSens®), According to the Qualitative Assessment Categories*[a]

Treatment	Category				
	A	B	C	D	N
Gluma® immediate and 14 days	0	All samples (100)	0	0	8
Gluma® fractured	All samples (100)	0	0	0	4
Tenure Quick® immediate and 14 days	0	0	0	All samples (100)	8
Tenure Quick® fractured	0	0	All samples (100)	0	4
Quell™ immediate and 14 days	0	0	0	All samples (100)	8
Quell™ fractured	0	0	All samples (100)	0	4
VivaSens® immediate and 14 days	0	0	0	All samples (100)	8
VivaSens® fractured	0	0	All samples (100)	0	4

[a] *Parenthetical values represent percent. A = The dentinal tubules are partially occluded and dentinal orifices are slightly smaller, with little or no debris. B = The dentinal tubules are mostly occluded; the dentinal surface is devoid of film or precipitate. C = The dentinal tubules were mostly occluded; the dentinal surface is partially covered with film or precipitation or shows some surface alterations. D = All the dentinal tubules are totally occluded and the surface is totally covered with precipitate and/or a resin film. N = number of specimens.*

others,[14] as they observed melting and crazing on the dentin surface, slight debris formation, and modification of dentin tubule structure where the tubule periphery had melted.

The mechanism of the Nd:YAG laser's effect on dentin is thermal energy absorption,[8] leading to laser-induced occlusion or narrowing of dentinal tubules.[7] Whitters and others[34]suggested direct nerve analgesia as a possible mechanism; they conducted a clinical trial using an electric pulp tester to measure the extent and duration of any analgesic effect induced by pulsed Nd:YAG laser treatment. A statistically significant increase in pain thresholds was observed in the mean responses measured five minutes after laser treatment with 113-mJ pulses at 15 pulses (pps) for three minutes. However, the pain thresholds returned to baseline values after 60 minutes. On the other hand, Funato and others[35] suggested thermally mediated effects on microcirculation. They observed some vascular changes shortly after the Nd:YAG laser was applied; these changes included vascular shrinkage, degeneration, coagulation, and stasis associated with irradiation. Their results indicate that the effects of Nd:YAG laser irradiation are primarily thermal, that this laser irradiation has a good hemostatic and coagulation ability, and that some of the changes are nerve-mediated by low-energy irradiation.

Samples treated with Gluma showed some closed tubules, but the majority were open. Similar results were obtained by Kolker and others.[25] Gluma desensitizer contains glutaraldehyde (GA) and 2-hydroxyethyl-methacrylate (HEMA). HEMA, which is well known for its water solubility, may act as a carrier/wetting agent to GA and may thus promote deep penetration of the GA component into the tubules. GA is a biological fixative and effective disinfectant, which upon reacting with the proteins in the dentinal fluid induces a precipitation and thus a partial or total occlusion of dentinal tubules. It kills bacteria and coagulates the plasma proteins within the dentinal fluids, forming a coagulation plug. Bergenholtz and others[36] showed a complete cessation of the outward flux of serum albumin in monkey dentin treated with Gluma *in vivo*. Clinically, Dondi and Melferrari[37] found that Gluma desensitizer showed a statistically significant reduction in sensitivity between baseline and postoperative pain scores and between the postoperative and the one-week responses. The sensitivity scores were not different between one week and six months.

Quantitative assessment of the first two groups (laser and Gluma) revealed some statistically significant differences in terms of tubule occlusion. In general, it could be suggested that the main effect of the two treatments (laser and Gluma) was almost the same, although the mechanism of action was totally different. The laser two-minute group produced a higher percentage of occluded tubules than did the one-minute group, with evident dentin surface changes, such as melting and bubble-like appearance of the dentinal tubules peripheries, which are favorable effects in terms of dentin desensitization. Clearly the time is a key factor in these differences: the longer the exposure of the dentin surface to the Nd:YAG laser energy, the more profound the resulting morphological changes. These morphological changes include closure of a larger number of open dentinal tubules, which is the treatment aim. Therefore, more effective dentin desensitization will presumably take place. However, the thermal effect of the relatively long exposure of the dental pulp to the laser energy must be considered. The minimum energy power that could achieve the desired treatment effect must be the ultimate goal in treating dentin hypersensitivity.

Tenure Quick, one of the tested desensitizers, totally covered the dentin surface with a solid, uniformly thick resin layer that was slightly rough and that had numerous crystal-shaped projections. In addition, the dentinal tubules were found to be plugged below the surface in the fractured samples. Presumably, the oxalate from aluminum oxalate, the active ingredient of this desensitizer, reacts with the calcium ions in the dentinal tubules and forms calcium oxalate crystals that precipitate and block the tubule. It is also possible that the thickness and the density of the precipitate increase with the number of applications. Gillam and others[10] showed very similar SEM results and suggested that the tubules are probably occluded with the methacrylate carrier.

Quell desensitizer, an ACP agent, is based on the idea of the physiologic process of sclerotic dentin. Since the occlusive material is apatitic mineral approximating the chemical formula of the main inorganic mineral dentin content [hydroxyapatite $(Ca_3 (PO_4)_2)_3 Ca (OH)_2$], it provides natural occlusion of dentinal tubules and thus reduces the permeability and sensitivity. It was suggested that this blocking of the tubules is not merely mechanical but also occurs as a result of a chemical interaction between calcium and phosphate ions, increasing the mineral contents of dentin and forming tri-calcium phosphate crystals, which obliterate the dentinal tubules.

Complete dentinal surface coverage was observed in the samples treated with VivaSens. As the resin liquid varnish sealed the dentinal surface, other constituents may have occluded the dentinal tubules. According to the manufacturer this occurs as a result of precipitation of calcium ions and proteins in the dentinal fluid. Potassium fluoride is one of the constituents of this material, which may form fluorapatite or calcium fluoride, in addition to the depolarizing effect of blocking the nerve conduction of potassium ions, which is another possible mechanism. Preparations containing potassium salts were reported[38-41] to be clinically effective in reducing dentin hypersensitivity. Those different mechanisms were thought to work synergistically to alleviate the dentin sensitivity. However, there are no studies to date regarding the long-term effectiveness of this material.

The relationship between surface and intratubular precipitation with sensitivity is not a simple one. It is not just the amount of precipitation that is important, it is also the quality of deposits, their density, degree of porosity, depth of penetration into tubules, and how well they are bound to the dentin surface. All of the materials tested in this study showed some loss of surface precipitation during preparation for fracturing, which indicates that the surface deposits are not firmly bound to the dentinal surface; given the dynamic nature of the oral environment and the local action of brushing, chewing, and saliva, the longevity of their protection is questionable. Further studies are needed to verify this assumption. The determining factor for the effectiveness of all tested treatments is how long the treatment effect will last, which can be known only through the conducting of long-term *in vivo* studies. As the laser-induced changes altered the dentin surface characteristic and morphology, it could be assumed that the laser may have a longer lasting effect than is noted on similar surfaces treated with the different desensitizing agents. The decision to use one treatment over the other should be based on the severity of individual clinical conditions, as each dentin hypersensitivity case has its own etiology and consequently requires different management. Simple approaches to manage hypersensitivity (such as dietary modifications, occlusal adjustments, the use of over-the-counter desensitizing dentifrice, etc) must not be underestimated. Desensitizing agents are inexpensive and easily applied, with the possibility of multiple reapplications. The initial cost of the laser machine is high, but it is cost effective in the long term.

The qualitative and quantitative results of our study can only form the basis of further research. Primarily, the pulp response to the treatments used should be investigated. Both quantitative and functional studies are required in order to determine the effects of these agents on dentin permeability (fluid flow). Clinical studies are needed as well to determine the effectiveness of these agents over time in terms of reducing the pain arising from dentin hypersensitivity. It is also necessary to simulate intraoral conditions, including brushing, acidic challenges, and other conditions.

CONCLUSIONS

Within the limitations of this *in vitro* study, we conclude the following:

- Based on the principle of tubule occlusion for dentin hypersensitivity treatment, all of the investigated treatments have promising desensitizing potential based on their various mechanisms of action. However, their long-term effectiveness must be determined through future clinical studies.
- Within the parameters used in this study, Nd:YAG laser irradiation on dentin resulted in occlusion or narrowing of the open dentinal tubules to various degrees.
- Thick precipitates were produced by Tenure Quick®, Quell™ desensitizer, and VivaSens®, covering the entire dentin surface and completely masking the dentinal tubule orifices. Gluma® desensitizer produced narrowing and occlusion of tubule lumen to some extent, without evident surface precipitate.

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Effects of Potassium Oxalate on Knoop Hardness of Etch-and-Rinse Adhesives

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Clinical Relevance

Application of potassium oxalate to acid-etched dentin may interfere with the properties of adhesives that are subsequently applied to dentin.

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SUMMARY

The objective of this study was to determine whether the hardness of etch-and-rinse adhesives may be affected by the pretreatment of acid-etched dentin with potassium oxalate desensitizer. Unerupted human third molars were cut into crown segments by removing the occlusal enamel and roots. The pulp chamber of these crown segments was connected to a syringe barrel filled with phosphate-buffered saline so that the moisture of dentin was maintained during the bonding procedures. Three etch-and-rinse adhesives—two two-step systems (Adper Single Bond 2 [SB], One-Step [OS]) and one three-step system (Adper Scotchbond Multi-Purpose [MP])—were applied to acid-etched dentin that had been treated (experimental groups) or not (control groups) with potassium oxalate (BisBlock). The Knoop hardness (KHN) of adhesives was taken at different sites of the outer surface of the adhesive-bonded dentin. The KHN of the three tested adhesives applied to acid-etched dentin

treated with potassium oxalate was significantly lower than that exhibited by the respective controls (not treated with oxalate; $p<0.05$). Regardless of the adhesive, the treatment with potassium oxalate reduced the adhesives' KHN ($p<0.05$), with the OS system exhibiting the lowest KHN compared with the MP and SB systems.

INTRODUCTION

In contemporary dental adhesives, high concentrations of relatively hydrophilic methacrylate monomers (ie, HEMA, BPDM, PENTA) are blended with relatively hydrophobic monomers (ie, Bis-GMA, UDMA) and solvents (ie, ethanol, acetone) to enhance bonding to intrinsically water-wet dentin. The presence of hydrophilic monomers and volatile solvents improves the wetting performance of dental adhesives when applied to acid-etched dentin that is intentionally saturated with water. Volatile solvents facilitate the displacement of water from the acid-etched dentin matrix,[1] ensuring better penetration of resin monomers into the micro- and nanoporosities left between the collagen fibrils,[2] which in turn improve the microretention between the restorative material and tooth substrates.[3,4]

Nevertheless, while the water-wet bonding technique may facilitate the infiltration of hydrophilic resin monomers into demineralized dentin, the presence of residual solvent/water before the photoactivation of adhesives has been thought to be responsible for producing areas of incomplete monomer conversion,[5,6] which correspond to the porosities that are revealed by silver deposition in nanoleakage studies.[7] These porous interfaces/polymers are prone to permeation by water[7] and, to a certain extent, by small solutes.[8,9] Polymers containing a mixture of hydrophilic and/or ionic domains become swollen due to water sorption,[10] allowing fluid transport in and out across the cross-linked polymer network.[6,7] The hydrophilic adhesives, therefore, behave as permeable membranes[11] that cannot achieve the requirement for perfect sealing of dentin.[12,13]

During bonding procedures, most of the water that is trapped within the adhesive layer or accumulated on its surface originates from the underlying hydrated dentin.[14] Studies have suggested that even the solvent evaporation procedures (ie, air blast) or the hypertonicity of solvated adhesive may be responsible for creating an osmotic gradient that induces outward water movement from the underlying hydrated dentin into the adhesive.[6,12]

The application of oxalate desensitizers to dentin prior to the bonding procedures has been considered as an alternative to block the transit of fluid across the resin-bonded interface and adhesive layer.[15,16] Oxalate solutions or gels react with ionized calcium in dentin to form insoluble crystals of calcium oxalate.[17,18] Because of their ability to occlude the dentinal tubules, oxalate-based desensitizers are considered potent agents in the treatment of dentin sensitivity.[18] The calcium oxalate crystals formed into the dentinal tubules were shown to reduce the fluid conductance of dentin,[19,20] reducing the pain sensation.[21] As a side effect, the obstruction of dentinal tubules with oxalate crystals may help clinicians gain better control of the moisture that is present on the surface of acid-etched dentin during bonding procedures. In theory, the presence of oxalate crystals reduces the free fluid conductance of dentin, creating environmental conditions for adhesives to polymerize more suitably, without any or limited presence of water.

When applied to acid-etched dentin, the calcium oxalate crystals are formed beyond the acid-etched surface, and in theory, they should not interfere with the subsequent bonding procedures.[15] However, the data on the effect of oxalate desensitizers on the bonding performance of adhesives are scant and unclear. While the oxalates can constitute an alternative to prolong the longevity of resin-dentin bonds, we have recently observed that the application of potassium oxalate to the acid-etched dentin affected the baseline bond strength of two- and three-step etch-and-rinse adhesive systems.[22] We speculated that even though the oxalate solution has been thoroughly rinsed before the application of adhesives to dentin, residual oxalic acid or any other of its by-products (ie, thickening agent) could have remained on the dentin surface, interfering with the proper polymerization of the adhesives and, consequently, compromising their bonding performance.

The aim of this study was to determine whether the polymerization of adhesives can be affected by the treatment of acid-etched dentin with a potassium oxalate desensitizer. Since hardness measurements have been accepted as a good predictor of the degree of polymerization of dental resins,[23-25] we tested the hardness of three etch-and-rinse adhesives after their application to acid-etched dentin that was treated or not with potassium oxalate gel. The hypothesis of this study was that the application of potassium oxalate to acid-etched dentin interferes with the hardness of etch-and-rinse dental adhesives.

MATERIALS AND METHODS

Tooth Preparation

Thirty noncarious human third molars extracted for orthodontic reasons were collected after patients' informed consent had been obtained under a protocol reviewed and approved by the Ethics Committee of the University of Campinas. Teeth were stored in saline containing 0.02% sodium azide and used within no longer than six months after extraction.

Crown segments were prepared by removing the occlusal enamel and root of these teeth using a slow-speed diamond saw under water cooling (Labcut 1010, Extec Corp, Enfield, CT, USA). The pulp tissue was carefully removed with a pair of small forceps. Care was taken to avoid touching the pulp chamber walls in order not to crush the predentine toward the dentinal tubules, which could alter the final permeability of dentin (D. Pashley, personal communication). The dentin surface was further abraded with 600-grit silicon carbide paper, until a remaining dentin thickness of 1.5 ± 0.2 mm was achieved from the ground surface to the highest pulp horn. The resulting crown segments were glued to Plexiglass slabs (1.8×1.8×0.7 cm) using viscous cyanoacrylate (Zapit, Dental Ventures of American, Corona, CA, USA), which also covered the entire peripheral cementum. Each Plexiglass slab was penetrated by a short length of 18-gauge stainless-steel tubing, permitting the pulp chamber to be filled with phosphate-buffered saline (pH 7) supplemented with 0.02% sodium azide to keep dentin hydrated during bonding procedures.

Bonding Procedure

The exposed dentin surfaces were polished with a 600-grit SiC paper during 30 seconds and then were acid-etched with 35% phosphoric acid gel (3M ESPE, St Paul, MN, USA) for 15 seconds and rinsed thoroughly with distilled water for 30 seconds. The specimens were divided into two groups: 1) control: the bonding procedures were performed as recommended by manufacturers; 2) experimental: the bonding procedures were performed after the treatment of acid-etched dentin with potassium oxalate. For the experimental group, the potassium oxalate gel, BisBlock (BISCO Inc, Schaumburg, IL, USA), was applied on the surface for 30 seconds and rinsed off with distilled water for 60 seconds. The enamel margins were re-etched for 15 seconds and rinsed thoroughly with water as recommended by the manufacturer (BisBlock, Technical Profile).

Three etch-and-rinse adhesive systems were selected for this study: the two-step systems Adper Single Bond ([SB] 3M ESPE) and One-Step ([OS] BISCO) and the three-step system Adper Scotchbond Multi-Purpose ([MP] 3M ESPE). In principle, these adhesives were applied to the acid-etched dentin surfaces of control and experimental groups while dentin was visibly moist with water as recommended by manufacturers. Nevertheless, this condition created such a soft bonded surface that the hardness could not be recorded even after storing the specimens in dry conditions for 48 hours after the adhesives' polymerization. Thus, adhesives were applied vigorously to the acid-etched dentin of control and experimental groups after the dentin surface was air dried for 30 seconds with oil-free compressed air.[26] This dry bonding technique ensured that the only possible source of water that could potentially interfere with adhesives hardness was that present in the pulp chamber.

Surfaces were checked to ensure complete covering with adhesives, and a glass coverslip was placed on the top of the adhesive to create a flat surface, avoiding excessive contact with the atmospheric oxygen during light activation.[27] The adhesives were light cured for 20 seconds using a halogen-tungsten unit (Degulux, DEGUSSA HÜLS, Frankfurt, Germany) operated at 500 mW/cm^2. Once polymerized, the specimens were stored in dry conditions at 37°C until the hardness measurement was taken.

Hardness Measurement

Twenty-four hours after the bonding procedures were completed, the specimens' hardness was determined with a Shimadzu HMV-2 hardness tester (Shimadzu Corporation, Kyoto, Japan), equipped with a Knoop indenter at 25 g of load and 6 seconds of dwell time. Six indentations were performed on the top of the adhesive-bonded dentin surfaces, over the sites that correspond to the pulp horns, where the pulp chamber wetness could impose a challenge to dental adhesive systems. At least three indentations per crown segment were performed in bonded enamel, which represents a condition in which the adhesives hardness should not be affected by the surface wetness (negative control).

The Knoop hardness was determined by examining the surface with an optical microscope (40×) and expressed as the Knoop hardness number (KHN).

Statistical Analysis

The KHN determined for control and experimental groups was analyzed by two-way analysis of variance tests, having as main factors the adhesives (ie, SB,

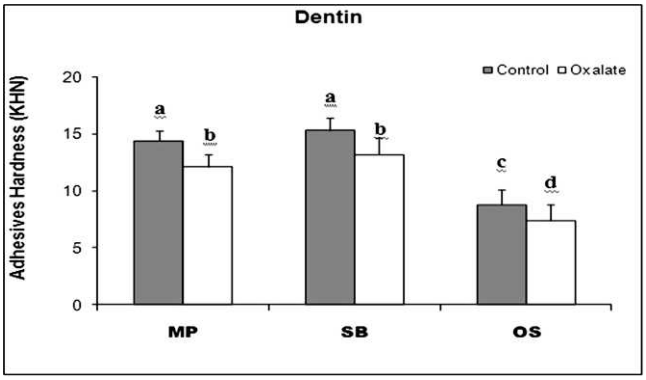

Figure 1. *The mean KHN and standard deviation (in KHN) of adhesives applied to dentin (n=60).* ***MP*** *= Adper Scotchbond Multi-purpose system;* ***SB*** *= Adper Single Bond 2 system;* ***OS*** *= One-step system. The height of the bars is KHN mean; half-brackets indicate plus one standard deviation. Groups identified with the same case letters did not differ statistically (p>0.05).*

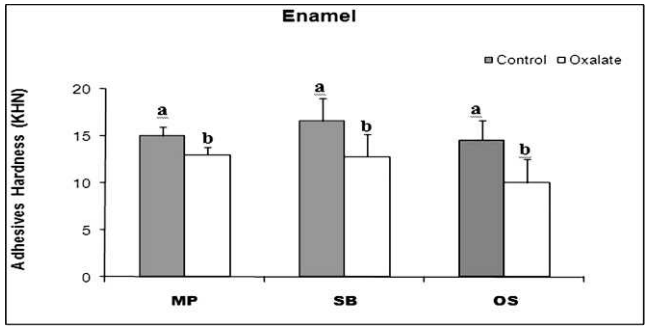

Figure 2. *The mean KHN and standard deviation (in KHN) of adhesives applied to enamel substrate (n=60).* ***MP*** *= Adper Scotchbond Multi-purpose system;* ***SB*** *= Adper Single Bond 2 system;* ***OS*** *= One-step system. The height of the bars is KHN mean; half-brackets indicate plus one standard deviation. Groups identified with the same case letters did not differ statistically (p>0.05).*

OS, and MP) and the substrate treatments (ie, control vs oxalate treated), with the data derived from dentin being analyzed separately from those derived from enamel. Post hoc multiple comparisons were performed using Tukey's tests. Statistical significance was preset at $\alpha=0.05$.

RESULTS

The mean KHN of the adhesive-bonded dentin and adhesive-bonded enamel are seen Figures 1 and 2, respectively. The treatment of dentin or enamel with potassium oxalate was shown to affect significantly the KHN of the three adhesives when compared with respective controls ($p<0.05$; Figures 1 and 2).

For both tested substrates (ie, dentin and enamel), the OS system exhibited the lowest KHN when compared with the SB and MP systems ($p<0.05$), regardless of the surface treatment (control or oxalate treated). The mean KHN for OS adhesive applied to dentin varied between 8.6 (control) and 7.6 (oxalate-treated dentin). For the MP adhesive system, the mean KHN tested on dentin ranged from 14.2 (control) to 12.4 (oxalate-treated dentin). These values did not differ significantly ($p>0.05$) from those observed for the SB adhesive (for comparisons between correspondent groups, ie, MP control vs SB control; MP experimental vs SB experimental), for which the mean KHN varied between 15.0 (control) and 13.2 (oxalate-treated dentin).

For enamel substrate, the differences in the KHN were significant only for the factor substrate treatment ($p<0.05$), while statistical significance for the main factor adhesives and the interaction between the two main factors (adhesives × substrate treatment) was not observed ($p>0.05$; Figure 2).

DISCUSSION

All tested etch-and-rinse adhesives (OS, SB, and MP) had their Knoop hardness significantly compromised, which led us to accept the anticipated hypothesis.

Several studies have found a positive correlation between hardness and degree of conversion.[23-25] While this may not be a full consensus,[28,29] as hardness may reflect the degree of cross-linking between polymer chains,[30] it has been conveniently used to compare the extent of polymerization exhibited by different dental resins under numerous testing conditions (ie, varying the light-curing time, the amount of energy delivered during photoactivation, the type of light source, the distance between the light-curing unit and the sample, etc).[23,29,30] The density and distribution of cross-links between polymer chains[31,32] play an important role in the final cohesion of polymers.[33] Polymer networks with homogeneous packing density (ie, restricted free volume and high level of polymer's chain cross-linking) tend to exhibit higher mechanical properties.[34] According to Rueggeberg and Craig,[27] hardness is sensitive in detecting small changes in polymer cross-linking so that reductions in KHN may suggest the existence of a less densely cross-linked polymer.[30] If this assumption was extrapolated to explain the present results, we could presume that the application of potassium oxalate interfered with adhesives' polymerization and mechanical properties by favoring the formation of poorly cross-linked adhesives.

Previous studies that evaluated the effectiveness of oxalate desensitizers to reduce the permeability of the resin-bonded dentin also indicated that these oxalates may have a varying effect on the bond strength of resin-bonded dentin,[15,16,35] depending on with which adhesive they were combined.[35] Yiu and coworkers[35] showed that fluoride ions released from fluoride-containing adhesives with relatively low pH may potentially interact with calcium-oxalate crystals to form spherical loosely bound calcium fluoride crystals that could interfere with the resin monomers' infiltration/polymerization, thereby compromising the hybrid layer formation. According to that study,[35] the pH of the adhesives SB, OS, and MP would not be low enough to cause the solubility of calcium oxalates. For this reason, we decided to test adhesives with low fluoride content and relatively high pH (3.3 for MP, 3.6 for SB, and for 4.5 for OS; 3M ESPE, Technical Product Profile, respectively). We believe, therefore, that the low values of hardness observed for adhesives applied on oxalate-treated dentin were not related to their pH and/or fluoride content.

In principle, it might be speculated that despite the oxalate solution having been thoroughly rinsed before the adhesives' application, residual oxalic acid may have remained to react with calcium, causing crystal precipitation on the dentin surface, which could compromise the adhesives' hardness. However, since the dentin surface was probably deprived of calcium phosphate due to its previous demineralization with phosphoric acid,[36] we are induced to consider that the decrease in the adhesives' hardness was probably not caused by the crystals' precipitation. We also do not believe that instantaneous dissolution of calcium oxalate crystals present in the dentinal tubules may potentially provide free calcium for reacting with residual oxalic acid to cause precipitation of calcium oxalate on the acid-etched dentin surface. Indeed, it seems more likely that other by-products of BisBlock had not been completely removed from the surface after the rinsing, thereby interfering with the proper polymerization of adhesives. This speculation may also explain the decrease in KHN observed for enamel specimens that were acid etched with phosphoric acid after potassium oxalate application (Figure 2) as recommended by the manufacturer.

An unexpected finding of this study was that the three-step adhesive (ie, MP) did not show a higher KHN value when compared with SB, a two-step adhesive. Supposedly, the inclusion of solvent and hydrophilic components in two-step etch-and-rinse adhesives should make these materials softer than those nonsolvated systems.[5,37] This is because residual solvent, which cannot be completely eliminated from the adhesive before light curing,[38] may plasticize the polymer network and reduce its mechanical strength. Thus, the adhesive MP, which requires a separate application of a relatively hydrophobic, nonsolvated resin over the acid-etched primed dentin, was supposed to exhibit the highest KHN values. However, by assuming that a BisBlock by-product has remained in tested specimens, one may speculate that these by-products could have also contaminated the bonding agent of MP adhesive as this was partially mixed with the unpolymerized primer solution that was set on the oxalate-treated dentin.

Although the results of the present study showed that potassium oxalate applied on acid-etched dentin affected the baseline hardness of the tested adhesives, a parallel study that we recently concluded showed that potassium oxalate played an important role in decelerating the long-term degradation of the resin-dentin bonds created using the same adhesives.[22] Most likely, the presence of calcium oxalate crystals partially blocking the fluid transudation across the dentinal tubules may have prevented the adhesives to prematurely absorb water, decelerating their mechanical disruption by the plasticizing effects of water.

The use of potassium oxalate on acid-etched dentin could also be useful in the ethanol wet-bonding technique.[39] The objective of this technique is to use more hydrophobic resin blends for dentin bonding to reduce adhesive permeability and dentin-bond degradation. As hydrophobic monomers do not bond well to the water-saturated dentin, ethanol is used to replace rinse water from acid-etched matrices.[40,41] However, as the ethanol wet-bonding protocol was found to be very technique sensitive in the presence of water, the dentinal tubules could be patently blocked with calcium oxalate crystals to prevent fluid contamination during the application of hydrophobic adhesives.[42]

Recently, studies have shown that infiltration of relatively hydrophobic monomers in ethanol-filled interfibrillar spaces produced similar or higher initial bond strengths to dentin in comparison to those achieved using the wet-bonding technique.[40,41] If the concept of ethanol wet bonding with hydrophobic monomers was proven to be effective in prolonging the durability of resin-dentin bonds, the application of oxalates to acid-etched dentin saturated with ethanol might be a way to avoid the contamination of hydrophobic monomers with den-

tinal water, thereby favoring monomer conversion. However, before the clinical implementation of this approach, additional and conclusive studies are necessary to access the influence of potassium oxalate gels on the performance of hydrophobic and hydrophilic adhesives applied to ethanol-saturated dentin.

CONCLUSIONS

Although potassium oxalate has previously shown to produce resin-dentin bonds that are more stable over time, it may adversely affect the baseline hardness of etch-and-rinse adhesives.

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Effect of Caries Infiltration Technique and Fluoride Therapy on Microhardness of Enamel Carious Lesions

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Clinical Relevance

An increase in microhardness of demineralized enamel after resin infiltration and acid resistance after exposure to a second demineralizing challenge can show the efficacy of the technique in controlling white spot lesions.

SUMMARY

Enamel white spot subsurface lesions compromise esthetics and precede cavitation; therefore, they must be halted. The aim of this study was to evaluate the effect of a caries infiltration technique and fluoride therapy on the microhardness of enamel carious lesions. Subsurface carious lesions were produced in 60 bovine specimens with polished enamel surfaces. The specimens were divided into four groups (n=15), according to the treatment used: CON, control—immersion in artificial saliva; DF, daily 0.05% fluoride solution; WF, weekly 2% fluoride gel; and IC, resin infiltration (Icon). The specimens were kept in artificial saliva and evaluated for microhardness at five points: baseline, after caries production, after four and eight weeks of treatment, and a final evaluation after being submitted to a new acid challenge. The repeated-measures analysis of variance showed significant differences according to the type of treatment (TREAT; p=0.001) and time of evaluation (EV; p=0.001). The results of the Tukey test were TREAT: CON = 45.18 (±29.17)a, DF = 107.75 (±67.38)b, WF = 83.25 (±51.17)c, and IC = 160.83 (±91.11)d. Analysis of correlation between the TREAT and EV factors showed no significant differences for DF (138.63 ± 38.94) and IC (160.99 ± 46.13) after the new acid challenge. The microhardness results in decreasing order after eight weeks were IC > DF > WF > CON. It was concluded that the microhardness of carious lesions increased with the infiltration of resin, while the final microhardness after a new acid challenge was similar for DF and IC.

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INTRODUCTION

Dental caries is defined as the destruction of tooth tissues by acid that is generated as a by-product of bacterial metabolism in dental plaque.[1] The first clinical sign of enamel caries is a white spot lesion, which precedes cavitation. Therefore, the early diagnosis and treatment of white spot lesions is extremely important. White spot lesions are considered reversible if they are detected early.[2]

Great attention has been devoted to noninvasive treatment of early proximal carious lesions, with the maximum preservation of tooth structure,[3] with no need of restorations.

As a noninvasive treatment, the use of topical fluoride associated with plaque removal is indicated to promote lesion remineralization.[4] Remineralization is the natural repair process for noncavitated lesions and relies on calcium and phosphate ions, assisted by fluoride, to rebuild a new surface on existing crystal remnants in subsurface lesions remaining after demineralization.[5] Fluoride ions incorporate into remineralizing enamel/dentin, changing carbonated apatite to a fluoroapatite-like form that is more acid tolerant and makes the hard tissues more acid resistant.[6]

Fluoride plays a key role in the prevention and control of dental caries.[7] However, this approach is not always successful, as it requires good compliance of the patient, with a change of harmful habits, with many of the patients abandoning the treatment before completion.[8]

Sealants have been used therapeutically on noncavitated occlusal caries as an attempt to reduce lesion progression.[9,10] In addition, these materials have been applied to interproximal smooth surfaces after temporary tooth separation to act as diffusion barriers.[3,11]

Enamel carious lesions are characterized by mineral loss in the body of the lesion, whereas the surface remains comparably highly mineralized.[2,12] The pores within the body of enamel caries provide diffusion pathways for acids and dissolved minerals. Therefore, an alternative approach for superficial sealing might be to arrest carious lesions by infiltration of these pores with light-curing resins, creating a diffusion barrier within the lesion without establishing any material on the enamel surface.[13]

Based on the available laboratory and clinical studies, it seems convincing that resin infiltration of enamel lesions should reduce (or even stop) the progress of white spot lesions.[14] This technique is considered microinvasive and might bridge the gap between noninvasive and minimally invasive treatment of initial dental caries, postponing as long as possible the need for a restoration.[15]

Caries resin infiltration represents a new concept in dentistry and therefore needs to be better investigated. The aim of this current study was to evaluate the resin infiltration technique and remineralization of enamel caries with a fluoride solution or gel on the microhardness of enamel caries and to investigate the resistance of these treatments when subjected to a new acid challenge. The null hypotheses tested were 1) the treatments tested did not alter the microhardness of the initial enamel carious lesion and 2) demineralization subsequent to treatments did not influence the microhardness of treated carious lesions.

METHODS AND MATERIALS

Sample Preparation

The methods described by Wiegand and others[16] were used to prepare the specimens. Thirty extracted, nondamaged, intact bovine incisors were stored in 0.1% thymol solution at room temperature until required. From each crown, two enamel-dentin specimens, 3 mm in diameter and 2.2-mm thick, were prepared from the labial surface using a trephine mill (Dentoflex, São Paulo, Brazil).

The specimens were positioned in a silicon mold with a cavity 6 mm in diameter and 2 mm deep. On the bottom of the mold, there was a second level cavity 3 mm in diameter and 0.1 mm deep. The specimens were positioned inside the internal cavity with the enamel surface facing the bottom of the mold. The mold was filled with acrylic resin (Extec Fast Cure Acrylic, Extec Corp, Enfield, CT, USA). The specimens were attached to a metal holder, and 0.1 mm of enamel was removed by polishing with sequential aluminum oxide abrasive papers (1200-, 2400-, and 4000-grit; FEPA-P, Struers, Ballerup, Denmark) in a polishing device (DP-10, Panambra Industrial e Técnica SA, São Paulo, SP, Brazil) for 20 seconds each. The dentin side of the specimens was abraded with 1200-grit abrasive paper, removing 0.1 mm of dentin and resulting in specimens with 1 mm of enamel and 1 mm of dentin. The prepared specimens were examined under a stereomicroscope to ensure the absence of cracks or other surface defects. After preparation, the specimens were stored in 0.1% thymol solution to avoid dehydration.

The microhardness determination was performed with a microhardness tester (FM-700, Future-Tech,

Tokyo, Japan) fitted with a 50-g load, which was used to make indentations on the enamel surface. The loaded diamond was allowed to sink and rest on the enamel surface for 10 seconds, and the Vickers hardness number (VHN) was determined. Three indentations, 100 μm apart, were performed at the center of each specimen and were averaged. The mean VHN value of each specimen was used for stratified allocation of all samples among the various experimental groups.

Sample Demineralization

Following the proposal by Queiroz and others,[17] artificial enamel subsurface lesions were created by individually immersing and storing the specimens in a buffer solution. The demineralizing solution was composed of 50 mM acetate buffer solution containing 1.28 mM $Ca(NO_3)_2 \cdot 4H_2O$, 0.74 mM $NaH_2PO_4 \cdot 2H_2O$, and 0.03 ppm F at a pH of 5.0 for 16 hours. The specimens were immersed separately in an unstirred solution at 37°C. The total volume of solution used was calculated using 2 mL/mm^2 of the enamel area.

Artificial saliva was prepared according to the formulation of Gohring and others[18] and consisted of hydrogen carbonate (22.1 mmol/L), potassium (16.1 mmol/L), sodium (14.5 mmol/L), hydrogen phosphate (2.6 mmol/L), boric acid (0.8 mmol/L), calcium (0.7 mmol/L), thiocyanate (0.2 mmol/L), and magnesium (0.2 mmol/L). The pH was between 7.4 and 7.8.

After this treatment, a new microhardness measurement was performed and baseline values were obtained. The specimens were then divided into four groups (n=15), according to the caries treatment employed:

CON (Control)—Specimens were stored in 5 mL of artificial saliva for eight weeks, which was changed every day.

DF (0.05% Fluoride Solution)—Specimens were immersed daily for 1 minute in 1 mL of 0.05% NaF solution for eight weeks. The fluoride solution was manipulated in the laboratory. After the daily fluoride immersion, the specimens were rinsed with deionized water and stored in artificial saliva.

WF (2% Neutral Fluoride Gel)—One milliliter of 2% NaF neutral gel (SS White Artigos Dentários Ltda, Rio de Janeiro, RJ, Brazil) was applied weekly for one minute on the surface of the specimens for eight weeks. After the gel application, the specimens were rinsed with deionized water and stored in artificial saliva.

IC (Resin Infiltration)—Specimens were resin infiltrated (Icon, DMG, Hamburg, Germany) and stored in artificial saliva for eight weeks. The infiltration procedure was performed according to the manufacturer's instructions:

- Icon-Etch was applied for two minutes.
- Specimens were water rinsed and air dried for 30 seconds.
- Icon-Dry was applied for 30 seconds and air dried.
- Icon-Infiltrant was applied two times, the first time for three minutes and the second time for one minute. Both applications were light cured for 40 seconds;
- Specimens were polished with aluminum oxide abrasive papers (4000 grit; FEPA-P, Struers) for 20 seconds.

The specimens of all groups were reevaluated for microhardness at four and eight weeks after the beginning of the treatments. After these periods, the samples were immersed again in the previously described demineralizing solution to evaluate the acid resistance of the treated surfaces and were submitted to a microhardness final evaluation. The residuals from the statistical analyses were examined to check for departures from normality and variance heterogeneity. No violations of the assumptions were found. The data were statistically analyzed, using repeated-measures analysis of variance (RM ANOVA) and Tukey test. Analyses were performed with statistical software STATISTICA 10 (Stat Soft Inc, Tulsa, OK, USA).

RESULTS

Evaluation with RM ANOVA (time as the repeated variable) revealed significant differences for the factors of treatment, time, and interaction (p=0.001). The Tukey test was then applied for the treatment factor and showed that group IC exhibited the highest microhardness means, followed by groups DF, WF, and CON, respectively (Table 1).

Evaluation with the Tukey test for the time factor showed significant differences for all periods of time evaluated. Baseline values (after white spot lesion formation) presented the lowest VHN means, followed by values measured after the new acid challenge (Ac; when specimens were immersed again in the demineralizing solution). Means obtained after four weeks presented intermediate values, and after eight weeks of treatment, the specimens showed the highest microhardness means (Table 2).

Figure 1 shows the results of the Tukey test for the interaction between the treatment and time factors. The specimens stored in saliva for four weeks did not

Table 1: *Means and Standard Deviation (SD) Data for the Tested Groups and Results of Tukey's Test for Treatment Factor.*

Treatment	Mean	SD	Homogen Groups[a]			
IC	160.83	91.11	A			
DF	107.75	67.38		B		
WF	83.25	51.17			C	
CON	45.18	29.17				D

[a] *Different letters in the same column indicate significant difference.*

show significant differences in the microhardness mean when compared with the baseline groups (values obtained after white spot formation), but after 8 weeks, a significant increase in microhardness mean was observed.

Specimens exposed to daily 0.05% fluoride solution after eight weeks (WF-8W) exhibited significantly higher microhardness means than the specimens exposed to weekly 2% fluoride gel (DF-8W). The results of microhardness after the new acid challenge were significantly higher for the resin-infiltrated (IC-New Ac) and fluoride solution groups (DF-New Ac) when compared with the fluoride gel (WF-New Ac) and control groups (CON-New Ac). The highest microhardness means were obtained for the resin-infiltrated groups and exposed to artificial saliva for four and eight weeks (IC-4W and IC-8W).

Figure 2 presents the performance of all tested groups, at different periods of time, in which an increase of the microhardness means can be observed for all treatments tested, especially for the resin-infiltrated groups after four and eight weeks. The illustration also presents the similar performance of the IC and DF groups after they were exposed to a new demineralizing solution.

Table 2: *Means and Standard Deviation (SD) Data for the Tested Groups and Results of Tukey's Test for Time Factor.*

Time	Mean	SD	Homogen Groups[a]			
Baseline	19.67	8.43	A			
New acid challenge	108.86	58.28		B		
After four weeks	127.42	78.26			C	
After eight weeks	141.06	69.02				D

[a] *Different letters in the same column indicate significant difference.*

DISCUSSION

The first null hypothesis of this current study was rejected, as all of the treatments tested increased the microhardness of the initial enamel carious lesion. Remineralization of noncavitated lesions has been reported for more than one century, when demineralized enamel was observed to harden in the presence of saliva.[19] In fact, patients with diminished salivary flow show an increased caries incidence.[20] Saliva can act on the acids themselves (via buffering or neutralization), on the bacteria (via inhibition of the metabolic process involved in acid production), and on the enamel (by maintaining chemical supersaturation in the adjacent plaque fluid).[14] In this present study, the remineralization potential of saliva was observed, as demineralized specimens immersed in artificial saliva showed increased microhardness values. Nevertheless, this remineralizing action was too small when compared with the remineralizing potential obtained when fluoride and resin-infiltration treatments for caries were used.

In the presence of fluoride, the remineralizing effect of saliva has been shown to be enhanced.[21] Fluoride would make demineralization more difficult and remineralization would be favored. This is essentially the basis for the effect of fluorides on dental tissues in reducing dental caries.[1]

The remineralization action of highly concentrated fluorides, such as those found in oral rinses, was observed in previous studies that showed the prevention of incipient caries progression.[22] In the present study, the remineralization action of fluoride in white spot lesions was also observed, but the 0.05% fluoride solution daily applied was considered more effective than the 2% fluoride gel applied weekly (ie, the frequency of application was more important than its concentration). The mainstay in caries prevention and remineralization is frequent exposure to low levels of fluoride.[23] Higher fluoride concentrations can cause rapid mineral precipitation on the enamel surface and obturation of the surface enamel pores that communicate with the underlying demineralized lesion. This process can further limit remineralization of the subsurface demineralized enamel.[24]

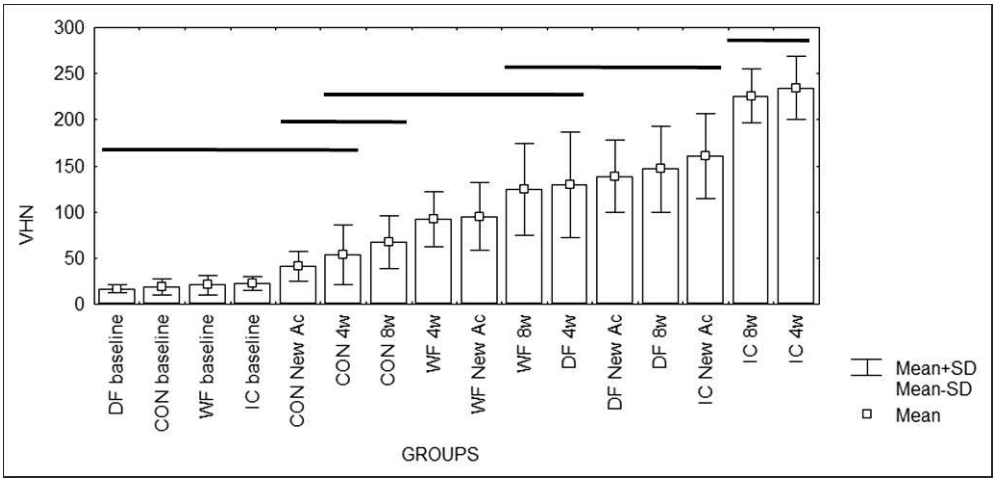

Figure 1. *Graph of microhardness means for the tested groups in different times and results of the Tukey test (horizontal bars) for the interaction between treatment and time factors.*

After the new acid challenge, the groups treated with fluoride exhibited no significant reduction in microhardness values compared with the groups treated for eight weeks (Figure 1). The remineralized enamel surface is different from the original in its composition and structure and is more resistant to demineralization than sound enamel.[25] The applied fluoride incorporates into enamel crystals, thereby forming a fluoroapatite-like mineral that improves the ability of the enamel to resist further acid challenge.[23,26]

The caries infiltration technique is an innovative approach investigated in previous studies, showing good results in hampering the progression of enamel caries.[12,27-30]

The infiltration technique, in contrast to the application of sealants, in which the diffusion barrier remains on the enamel surface, creates a diffusion barrier inside the enamel lesion and possibly strengthens the demineralized enamel structure with the resin matrix, preventing cavitation.[13] In the present study, the infiltration technique showed significantly higher microhardness means than all other tested groups (Table 1). This reflects the ability of the low-viscosity resin to fill the spaces between the remaining crystals of the porous lesion and

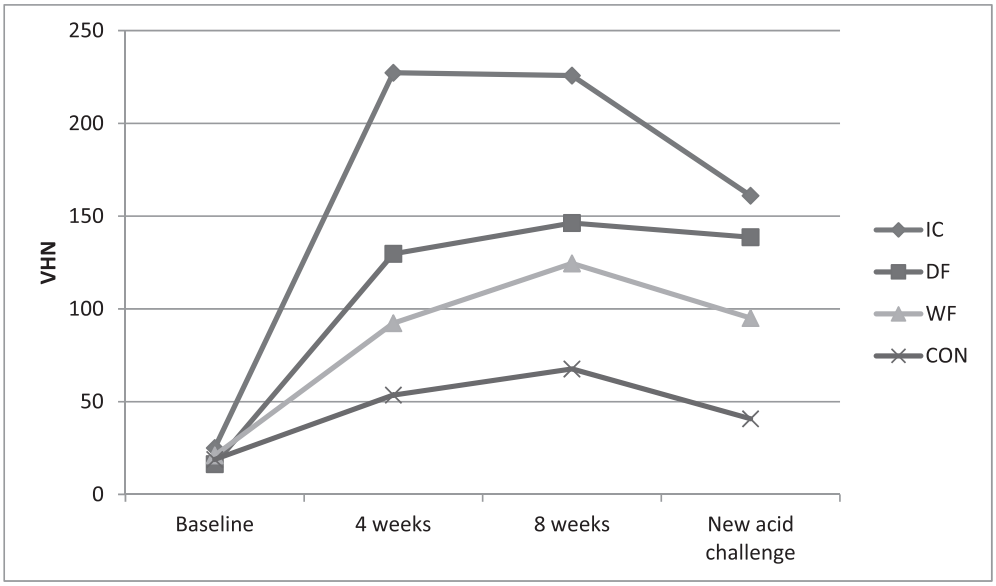

Figure 2. *Graph of microhardness values for the tested groups in different times.*

reharden the demineralized tissue, improving its mechanical strength.

Previous studies showed that although the resin-infiltration technique results in a significant reduction in lesion progression under demineralizing conditions, some demineralization can still occur after Icon treatment.[12,15,28] This result was also observed in the present study, in which the artificial lesions infiltrated with Icon presented lower microhardness means after the new acid challenge. This could be due to the partial dissolution of the remaining mineral in the lesion body that was not completely embedded within the resin matrix or by the resin shrinkage during light curing, which results in leakage and consequently reduction of acid resistance.[13] Nevertheless, despite this reduction in microhardness after the demineralization process, the resin-infiltrated group exhibited microhardness means similar to those obtained by the daily fluoride-treated group after the new acid challenge (Figure 2).

This result shows that resin infiltration is a promising technique to treat enamel caries, but the oral hygiene and diet education methods should be instituted for high-risk caries patients, and perhaps a new infiltration of resin should be conducted. It was shown that a repeated application of resin can reduce the leakage of acids in the lesion body.[31]

Although all tested treatments were capable of enhancing the microhardness of demineralized enamel, it should be emphasized that any strategy to reduce the progression of carious lesions should be based on the control of caries as a biofilm-dependent disease, and it should be controlled with tooth brushing by means of oral hygiene education and dietary control.[32]

It has to be considered that an artificial bovine enamel lesion model was used, but this limits the external validity of the study because, under clinical situations, the lesions to be resin infiltrated are deeper.[15] More studies are needed to confirm the efficacy of resin infiltration technique in clinical conditions.

CONCLUSION

It was concluded that the microhardness of initial enamel carious lesions increased significantly with the resin-infiltration technique and the final microhardness after a new acid challenge was similar in the specimens infiltrated with resin and treated with 0.05% daily fluoride solution.

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Effect of Temperature on the Degree of Conversion and Working Time of Dual-Cured Resin Cements Exposed to Different Curing Conditions

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Clinical Relevance

A high degree of conversion can be achieved when dual-cured resin cements are used with increased temperature even when the curing light is compromised by the presence of ceramic restorations. However, caution is recommended before the clinician decides to warm up the resin cement, as this procedure may compromise the working time, depending on the temperature and product.

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SUMMARY

Objectives: This study evaluated the degree of conversion (DC) and working time (WT) of two commercial, dual-cured resin cements polymerized at varying temperatures and under different curing-light accessible conditions, using Fourier transformed infrared analysis (FTIR).

Materials and Methods: Calibra (Cal; Dentsply Caulk) and Variolink II (Ivoclar Vivadent) were tested at 25°C or preheated to 37°C or 50°C and applied to a similar-temperature surface of a horizontal attenuated-total-reflectance unit (ATR) attached to an infrared spectrometer. The products were polymerized using one of four conditions: direct light expo-

sure only (600 mW/cm^2) through a glass slide or through a 1.5- or 3.0-mm-thick ceramic disc (A2 shade, IPS e.max, Ivoclar Vivadent) or allowed to self-cure in the absence of light curing. FTIR spectra were recorded for 20 min (1 spectrum/s, 16 scans/spectrum, resolution 4 cm^{-1}) immediately after application to the ATR. DC was calculated using standard techniques of observing changes in aliphatic-to-aromatic peak ratios precuring and 20-min postcuring as well as during each 1-second interval. Time-based monomer conversion analysis was used to determine WT at each temperature. DC and WT data (n=6) were analyzed by two-way analysis of variance and Tukey *post hoc* test ($p=0.05$).

Results: Higher temperatures increased DC regardless of curing mode and product. For Calibra, only the 3-mm-thick ceramic group showed lower DC than the other groups at 25°C ($p=0.01830$), while no significant difference was observed among groups at 37°C and 50°C. For Variolink, the 3-mm-thick ceramic group showed lower DC than the 1-mm-thick group only at 25°C, while the self-cure group showed lower DC than the others at all temperatures ($p=0.00001$). WT decreased with increasing temperature: at 37°C near 70% reduction and at 50°C near 90% for both products, with WT reduction reaching clinically inappropriate times in some cases ($p=0.00001$).

Conclusion: Elevated temperature during polymerization of dual-cured cements increased DC. WT was reduced with elevated temperature, but the extent of reduction might not be clinically acceptable.

INTRODUCTION

Dual-cured resin cements were developed to achieve optimal polymerization, even when the curing light is attenuated or blocked by the presence of an indirect restoration. For this purpose, polymerization of these products can be initiated by three activation modes: light-cure, self-cure or the association of both (dual cure).[1,2] Previous work indicates that the self-cure mode is less effective than the dual-cure mode since these cements produce lower degrees of conversion (DC) when access to the curing light is attenuated or blocked by the presence of an indirect restoration rather than when they are directly exposed.[3-5] In these detrimental clinical conditions, dual-cured resin cements may present lower hardness, higher solubility, lower flexural and compressive strengths, and lower bond strength values to dentin when compared to resin cements that are directly light cured.[6-13] Compromised light delivery to dual-cure cements is a common clinical reality, as only 10% to 15% of light remains after passing through a 2-mm-thick indirect restoration, with shade varying from A2 to A4.[3,9,14]

One possible explanation for the lower effectiveness of self-curing components in dual-cured resin cements is related to the slow rate of polymerization activation and subsequent propagation of radicals in comparison to a directly photoactivated product.[15] After reaching a maximal rate of cure, this slow self-polymerization reaction passes through a phase called autodeceleration caused by a decrease in monomer and free radical mobility, resulting from the increase in network formation and resultant elevation in viscosity.[16] The viscosity of self-curing reactions impacts polymerization kinetics to a greater extent than those that are photocured only.[17] Another factor responsible for the poorer performance of self-curing components is related to the low amount of benzoyl peroxide able to be incorporated into these materials as well as the need to include inhibitors to prolong product shelf-life and provide a clinically realistic working time (WT), ranging from 2 to 5 minutes. For these reasons, the composition of dual-curing products still needs to provide the clinician with a reasonable WT, typically from 3 to 5 minutes.

The polymerization kinetics and mechanical properties of polymers are affected by temperature.[18-20] Increased resin temperature prior to and during polymerization leads to higher DC values for light-cured resin composites.[21] An increase in temperature during polymerization promotes free radical and monomer mobility, leading to higher polymerization rates and elevated DC.[21,22] As a consequence, preheated resin composites may reach similar DC as those exposed to room temperature, establishing a clinical advantage of this procedure by using shorter exposure durations.[23,24]

Following this line of thought, increasing the temperature of dual-cured resin cements during the mixing procedure may be a valuable option to promote optimal polymerization when resin cements rely mostly on the self-curing mode. However, the optimum precementation temperature for a specific material may result in clinically unacceptable WT. Only a few studies have evaluated the effects of increased temperature on marginal adaptation and bond strength of indirect restorations to tooth with dual-cure resin cements.[25,26] Higher bond strength

Table 1: *Brand, Composition, and Batch Number of the Dual-Cured Resin Cementing Systems*

Product (Manufacturer)	Composition (Batch Number)
Calibra regular viscosity shade medium (Dentsply Caulk)	Base paste: barium boron fluoroalumino silicate glass, Bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide; other colorants are inorganic iron oxides.
	Catalyst paste: barium boron, fluoroalumino silicate glass, Bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, benzoyl peroxide (base: 081105; catalyst: 0812011)
Variolink II low viscosity (Ivoclar Vivadent/Schaan, Liechtenstein)	Paste of dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments, Bis-GMA, TEGDMA, urethane dimethacrylate, benzoyl peroxide (base: L46354; catalyst: L36656)

Abbreviations: TEGDMA, triethylene glycol dimethacrylate; bis-GMA, bisphenol A diglycidyl ether methacrylate.

results obtained by the association between elevated DC and improved marginal adaptation have been attributed to the lower viscosity observed in resin cements at higher temperatures.[25,26] However, no information regarding monomer conversion and WT of such cements when polymerized at higher temperatures is available.

The purposes of this study were to evaluate the effects of increased temperature prior to and during polymerization of commercial dual-curing resin cements on their DC, time-based conversion profiles, and WT. In addition, the influence of different polymerization activation modes correlated with varied light attenuation scenarios was tested. The first research hypothesis anticipated that dual-cured resin cements polymerized at either 37°C or 50°C will demonstrate higher DC than the same products polymerized at room temperature. This result is expected even when the curing light is attenuated by the presence of indirect ceramic restorations with varying thickness or when no curing light is available; the material is allowed to polymerize totally in the self-cure mode. The second hypothesis expected to find a significant decrease in WT when the temperature during polymerization is increased to either 37°C or 50°C over that seen at room temperature (25°C).

MATERIALS AND METHODS

Specimen Preparation

The specific dual-cure resin cement products selected for testing (Table 1) were chosen for their differing ability to polymerize according to activation mode, as previously reported:[27,28] a product demonstrating better self-cure and light cure (Calibra Regular, Dentsply Caulk, Milford, DE, USA) and a product producing poor self-cure (Variolink II low viscosity, Ivoclar Vivadent, Schaan, Liechtenstein). Ceramic discs of varying thickness were selected to model indirect restorative materials that offer different levels of light transmission to an underlying dual-cured resin cement: 2-cm diameter by either 1.5- or 3.0-mm thickness (IPS e.max, A2 shade, Ivoclar Vivadent).

In order to provide elevated resin temperatures during polymerization (37°C or 50°C), the base and catalyst pastes were equally dispensed on a glass plate that had been placed on a heated stirrer surface (103, Cientec, Piracicaba, São Paulo, Brazil) set at 37 ± 1°C or 50 ± 1°C. Resin and glass plate temperatures were constantly measured with a K-type thermocouple (SmartMether, Novus, Porto Alegre, RS, Brazil) to ensure that the paste components reached the desired temperature for each experimental group prior to their mixture. Once the proper temperature value had been reached, the cement components were hand mixed using a metal spatula and were then applied to the horizontal diamond element of an attenuated total reflectance (ATR) unit attachment (Golden Gate, Specac, Woodstock, GA, USA) in the optical bench of a Fourier transform infrared spectrometer (Tensor Series, Bruker Optik GmbH, Ettlingen, Germany). Prior to resin placement, adhesive tape (3M, St. Paul, MN, USA) was placed around the diamond surface to act as a spacer, ensuring standard thickness for all specimens (100–120 μm). For the preheated groups, the diamond surface temperature was elevated to 37°C or 50°C using a custom-made heating device, with the surface temperature constantly monitored

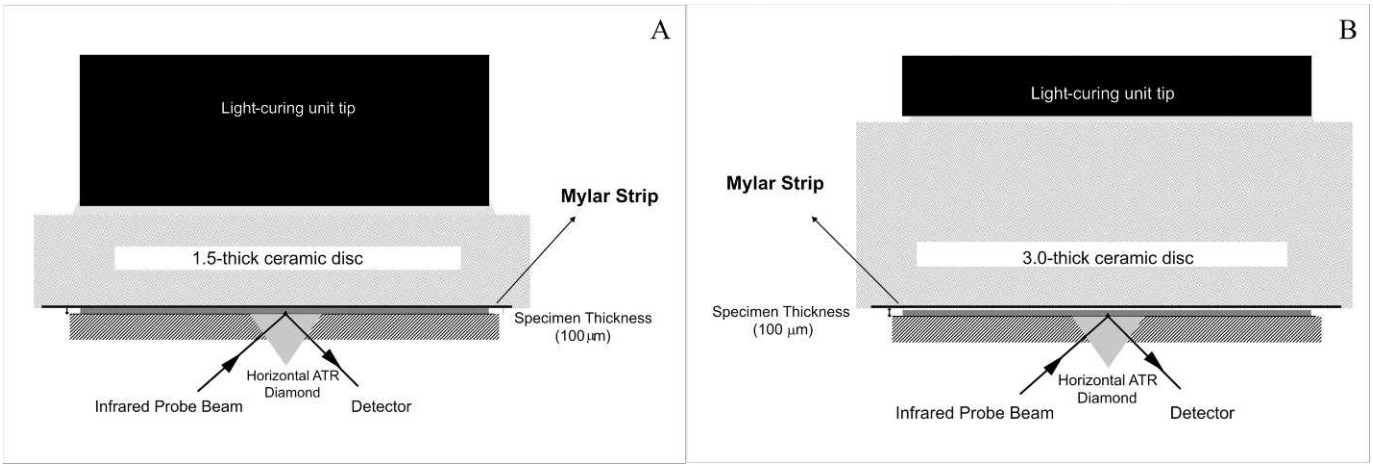

Figure 1. *Illustrative diagram demonstrating the interaction between the infrared beam and the specimen, as well as the position of the 1.5- (A) or 3.0-mm (B) thick ceramic disc and the light-curing unit tip.*

using the K-type thermocouple (SmartMether) during resin cement polymerization. For groups of resin cements polymerized at room temperature, all procedures described above were performed at 25°C.

The deposited resin cement was covered with a Mylar strip and polymerized using one of four different curing modes: direct light exposure, exposure to attenuated light by the presence of either 1.5- or 3.0-mm thick overlying ceramic discs, and total absence of curing light (self-curing mode). Specimens were exposed directly to light activation (manufacturer-recommended conditions of 20 seconds for Calibra and 40 seconds for Variolink II) from a light source emitting 600 mW/cm^2 (Optilux 501, Demetron Kerr, Danbury, CT, USA). The specimens were exposed without any overlying restorative material (direct light exposure [DLE], control group). The emitting end of the light guide was placed directly against a 2-mm-thick glass slide, positioned directly over top of the Mylar-covered resin specimen. When exposing cements to light using different thickness of overlying ceramic discs, the discs were placed directly between the Mylar sheet and the emitting end of the light guide (Figure 1). In addition, specimens were also allowed to polymerize in the total absence of the curing light by merely placing the 2-mm-thick glass slide over the Mylar and not supplying any photoactivating light (self-cure).

Degree of Conversion

Infrared spectra were collected between 1680 and 1500 cm^{-1} at a rate of one spectrum per second (16 scans/spectrum) at 4 cm^{-1} resolution. Data were counted from the moment the infrared scan demonstrated that the resin was stabilized on the ATR surface and any overlying objects had been placed. Spectra were recorded continuously during each 1-second interval for 20 minutes. Six replications were made for each test condition (n=6) based on previous studies using the same methodology.[3,27-29] Monomer conversion was calculated using standard methods that evaluated changes in the ratios of aliphatic-to-aromatic C=C absorption peaks (1636 cm^{-1}/1608 cm^{-1}) in the uncured and cured states obtained from the infrared spectra.[30,31] Prior to determining conversion, calibration graphs were made relating the absorbance ratios of known molar concentrations of aliphatic and aromatic C=C to their respective absorbance height ratios. Conversion values among all curing modes were compared statistically within each product only at 20 minutes from the time the resin cement was stabilized on the ATR surface. All polymerized specimens were carefully removed from the ATR plate and measured for thickness to the nearest 0.01 mm using a digital micrometer (Series 406, Mitutoyo America Corp, Aurora, IL, USA) to ensure similar thickness among all specimens.

Working Time

For purposes of the study, WT was defined as the moment when infrared spectra first indicated evidence that conversion values rose above that of the zero-value baseline. Thus, individual analysis of polymerization kinetic graphs from each specimen was performed to determine the time (in seconds) elapsed between the first Fourier transformed infrared analysis spectrum and the moment when polymerization started (Figure 2).

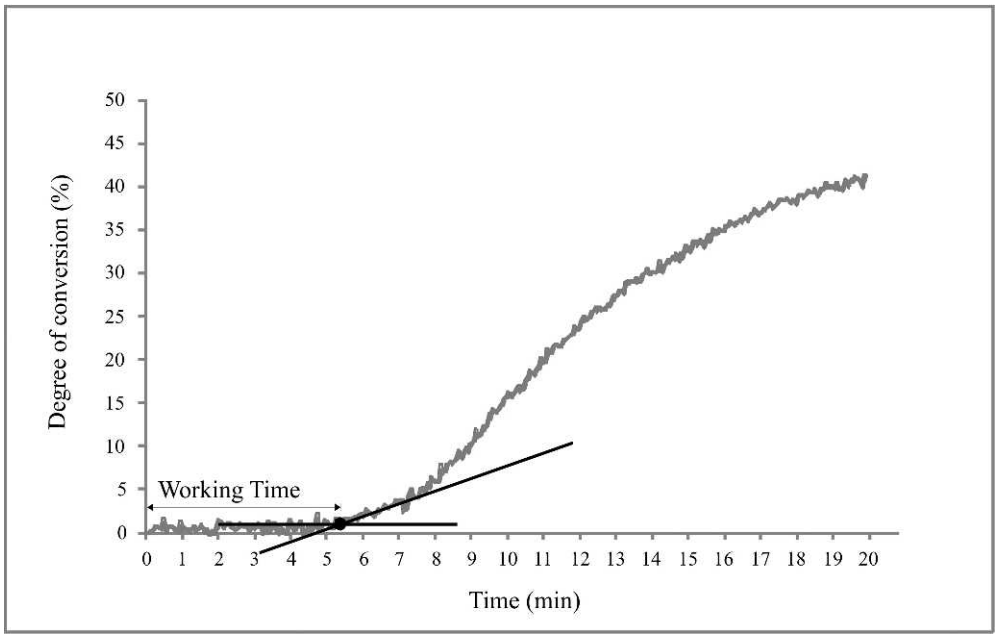

Figure 2. *Illustrative polymerization kinetics of Variolink II polymerized by the self-curing mode exhibiting the time elapsed before the beginning of polymerization.*

Statistical Analyses

Degree of conversion data was evaluated within each dual-cure resin product using a two-way analysis of variance (ANOVA; factors: temperature [three levels] and curing condition [four levels]) followed by Tukey *post hoc* test. Direct comparison of conversion values between products was not made, because the result would not have had any meaning; only when resins have the same chemical formulation can such comparisons be made. The results of WT were pooled for the factor of curing mode and were subjected to two-way ANOVA (factors: temperature [three levels] and product [two levels]) followed by Tukey *post hoc* test. All testing was performed at a preset alpha of 0.05 using personal statistical software (SAS 8.0 for Windows, SAS Institute Inc, Cary, **NC,** USA). Post hoc power analysis was performed for the statistical analysis of DC and WT data using G*3 Power statistical software.[32]

RESULTS

Degree of Conversion

For the number of specimens used (n=6), the study was adequately powered for both factors, temperature and curing mode (over 95%; α=0.05). For both products, the two-way ANOVA indicated that the interaction between curing mode and temperature was a significant factor in affecting DC for both Calibra (p=0.01830) and Variolink II (p=0.00001). Table 2 presents the DC of dual-cure cements exposed using the four curing conditions at three temperatures. Temperature increase resulted in significantly higher DC regardless of curing mode and product. The effect of different curing conditions on DC varied according to the temperature for both cements. For Calibra at 25°C, a lower DC was observed only when the thick ceramic disc was used in comparison to the DC of groups with other modes of attenuation, while no significant difference in DC was noted among curing conditions at either 37°C or 50°C. At 25°C, DC of the DLE and thin ceramic groups of Variolink II were the highest and not significantly different. However, use of the thick ceramic disc resulted in lower DC, and the lowest value was seen in the self-cure group. At 37°C, the DLE group showed the highest DC, while curing this material under either thickness of ceramic demonstrated less conversion. Again, the lowest conversion was seen using the self-cure condition. At 50°C, no significant difference in DC was noted among groups utilizing any form of light curing, while DC of the self-cure group was significantly lower.

Figures 3 and 4 show the effects of temperature on the time-based conversion profiles of Calibra and Variolink II, respectively, including comparison to the 20-minute DC value from DLE group at 25°C (dashed line) as control. At 25°C (Figure 3A), time-based conversion changes measured through the thin and thick ceramic discs and the self-cure group

Table 2: *Degree-of-conversion means (Standard Deviation) of Calibra and Variolink II Exposed to Three Temperatures and Four Curing Modes*[a]

Resin Cement	Temperature	Direct Light Exposure	Thin Ceramic Disc	Thick Ceramic Disc	Self-Cure
Calibra	25°C	55.9 (1.4) Aa	56.0 (1.9) Aa	52.9 (1.6) Ab	56.0 (1.2) Aa
	37°C	63.2 (0.9) Ba	62.4 (0.7) Ba	62.9 (1.1) Ba	61.7 (1.0) Ba
	50°C	67.3 (2.3) Ca	67.7 (1.3) Ca	67.2 (2.6) Ca	66.0 (1.6) Ca
Variolink II	25°C	62.3 (0.7) Aa	59.9 (2.4) Aa	54.5 (3.2) Ab	41.7 (1.0) Ac
	37°C	67.3 (1.4) Ba	64.1 (1.8) Bb	63.6 (1.3) Bb	56.8 (1.3) Bc
	50°C	72.6 (1.1) Ca	72.4 (1.3) Ca	70.3 (1.3) Ca	65.6 (1.0) Cb

[a] *Significant differences are indicated by different letters (uppercase letters within column; lowercase letter within row) according to Tukey post hoc test at a preset alpha of 5%. No comparisons were made between products.*

of Calibra exhibited DC values as high as those observed in the DLE groups at 20 minutes. The rate of conversion observed when directly exposing the cement (DLE) was very high at first, until the light-curing unit shut off, and then a slow, continual increase was noted. When exposing the cement through the thin ceramic disc, the curing rate was less than the DLE mode but more rapid than when the thicker ceramic disc was interposed. The slowest initial curing rate was noted for the self-cure-only mode. Interestingly, the basic profile of time-based conversion for light curing through the ceramic discs appeared very much like that of the self-cure-only group, indicating that the overall polymerization reaction when curing through the ceramic discs was related to the self-cure reaction. At 37°C (Figure 3B)

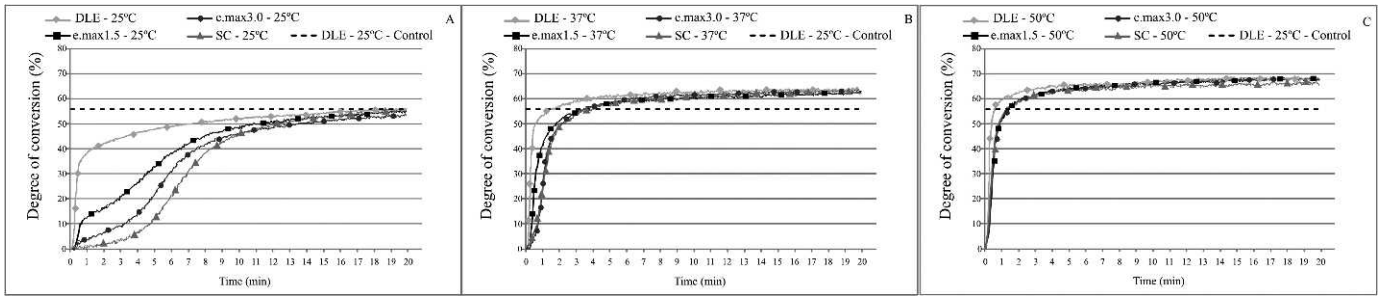

Figure 3. *Polymerization kinetics of Calibra at a 25°C (A), 37°C (B), and 50°C (C) exposed to four curing conditions. The dashed line represents the degree of conversion after light activation through a glass slide at 25°C. Thin ceramic disc: e.max1.5; thick ceramic disc: e.max3.0.*

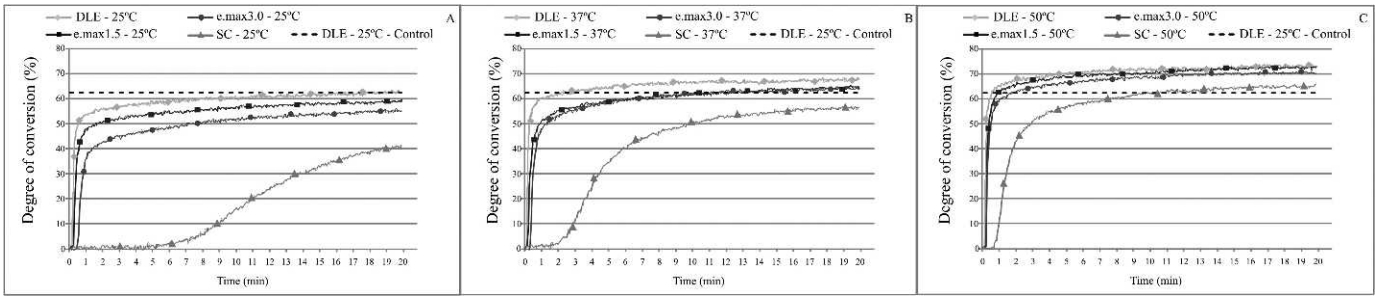

Figure 4. *Polymerization kinetics of Variolink II at a 25°C (A), 37°C (B), and 50°C (C) exposed to four curing conditions. The dashed line represents the degree of conversion after light activation through a glass slide at 25°C. Thin ceramic disc: e.max1.5; thick ceramic disc: e.max3.0.*

and 50°C (Figure 3C), the time-based conversion profiles appeared much more like that of the DLE group when exposing through the different thicknesses of ceramic discs as well as when the material was allowed to self-cure.

On the other hand, the conversion profile of Variolink II (Figure 4) was seen to depend mostly on the ability of the curing light to reach the resin cement layer in order to provide similar DC to that observed in the DLE group at 25°C. At that temperature (Figure 4A), it can be seen that the time-based conversion profiles, when shining curing light through either thickness of ceramic disc, appear much more like that of the group receiving DLE. The self-cure reaction appears to be very slow and does not reach the conversion levels after 20 minutes that the other groups did. At 37°C (Figure 4B), little difference is seen in the conversion profiles when using either thickness of ceramic. The rate of conversion increase with time for the self-cure mode increased over that seen at 25°C but is still less than other conversion values after 20 minutes. At 50°C, there is very little distinction in conversion profiles of the DLE group, and those polymerized through either thickness of ceramic. Conversion values for the self-cure group appear to be much greater than when at previous temperatures and finally approach DC values seen for the DLE group when polymerized at 25°C. As seen with specimens from Calibra, those from Variolink II polymerized at 50°C are well above the DC value of the respective DLE group at 20 minutes.

Working Time

For statistical analysis of WT data, the study was adequately powered for both factors, temperature and product (over 99%; $\alpha=0.05$). The WT values of both resin cements in the self-cure mode at different temperatures are displayed in Table 3. The ANOVA indicated that the interaction between both temperature and product ($p=0.00001$) significantly influenced WT. A significant decrease (73%) in the WT of Calibra was seen when the temperature increased to 37°C from 25°C, while no significant difference was noted increasing polymerization temperature from 37°C to 50°C, although the WT was noted to decrease by 90%. For Variolink II, significant decreases of 69% and 88% were observed in WT when resin cement temperature increased to 37°C and 50°C, respectively, compared to that at 25°C. Variolink II exhibited longer WT than Calibra regardless of temperature.

Table 3: *Working Time in Seconds (Standard Deviation) of Calibra and Variolink II Polymerized at Three Temperatures*[a]

	Calibra	Variolink II
25°C	83.4 (7.4) Ab	311.4 (80.7) Aa
37°C	22.5 (10.0) Bb	95.3 (31.0) Ba
50°C	10.1 (4.2) Bb	36.7 (5.3) Ca

[a] *Significant differences are indicated by different letters (uppercase letters within column; lowercase letter within row) according to Tukey post hoc test at a preset alpha of 5%.*

DISCUSSION

The research findings validated the first research hypothesis, which anticipated that dual-cured resin cements polymerized at either 37°C or 50°C will demonstrate higher DC than the same products polymerized at room temperature, even when the curing light is attenuated by the presence of indirect ceramic restorations with varying thickness or even when no curing light is available. Data seen in Table 2 provide strong evidence to support these statements. In contrast to light-cured-only resin composites, dual-cured resin cements contain not only photoinitiators but also self-curing components, such as benzoyl peroxide. Since the degradation rate of benzoyl peroxide into radicals increases with increased temperature,[33] radicals are created more rapidly when heated. Thus, the increase in DC observed in both resin cements to values as high as those observed in the DLE groups at 25°C could also be attributed to the higher amount of these radicals being created in the early stages of polymerization. These increases occurred even in conditions simulating clinically unfavorable environments, such as when a thick indirect restoration is interposed between the curing unit tip and the underlying resin cement layer.

Despite the well-reported severe attenuation in light intensity caused by the presence of indirect restorations between the resin cement layer and curing unit tip,[3,14] both resin cements exposed through 1.5 mm of overlaying ceramic at 25°C showed DC values as high as those observed in DLE groups after 20 minutes. These results are in agreement with those reported by others[3,14] who demonstrated that use of indirect restorations with thickness lower than 2 mm does not compromise DC of selected dual-cured resin cements. Based on the analysis of time-based conversion of Calibra at 25°C

(Figure 3A), the importance of self-curing components to compensate for lower light levels reaching the resin cement was evident, as a significant increase in conversion value with time was noted after light exposure in both the 1.5- and the 3.0-mm ceramic disc thickness groups. On the other hand, the light-curing components in Variolink II responsible for compensating for light attenuation were seen to work more effectively than in Calibra. For example, at 25°C, the conversion-based profile using a 1.5-mm-thick ceramic disc produced nearly the same result as the unattenuated DLE group.

A unique aspect of the current study was the inclusion of clinically relevant methods to attenuate the light-curing unit from providing radiant energy as an initiator for the photoactivated aspect of the dual-cure resins tested. Thus, a range of light availability was devised from no attenuation (DLE), through two different thicknesses of a commonly used ceramic material (1.5 or 3.0 mm in height), or the products were allowed to polymerize totally in the dark (self-cure only). Differences in the findings emphasize how commercial products vary in their ability to achieve high levels of conversion using either mode of polymerization activation. Calibra appears to be activated mostly by its self-curing components (Figure 3A), while Variolink II seems to be most effectively cured when receiving light (Figure 4A). Thus, one cannot make generalized statements about the overall ability of dual-cure cements to perform well in either type of curing mode. The differences in abilities of the different curing modes was somewhat overcome when resins were preheated compared to their use at room temperature. However, even when polymerization occurred at 50°C, the self-cure reaction of Variolink II still lagged behind the other groups.

It should be noted that the temperature spans tested are clinically relevant. These products are designed to be proportioned and mixed at room temperature and then placed against a freshly prepared tooth surface. Although many researchers feel that the prepared tooth surface is at body temperature (37°C), as tested in the current work, instead it is more between 27°C and 30°C.[34] Use of the 50°C temperature was applied in an attempt to see if the shortcomings of differences in light- and self-cure reactions could be overcome with preheating. Use of this temperature is well within those applied clinically, as composite is preheated and placed at temperatures well above this value: 68°C (Calset Composite Heater, AdDent Inc, Danbury, CT, USA). Interestingly, reduced exposure times are advocated with use of the preheating device, as previous work has shown that elevated temperatures cause enhanced conversion levels when using less-than-recommended exposure times.[24] It is thought that the increased monomer and radical mobility conferred by lower system viscosity from the heated material is responsible for these more rapid curing and higher converted systems.[24]

In the current study, the effects of temperature were also evaluated with respect to the clinically relevant aspect of WT. This parameter is of importance because, if WT is reduced to the point the material becomes impossible to handle because it sets so rapidly, then little benefit would be gained by enhancing conversion as a result of preheating.

The second research hypothesis anticipated that a significant decrease in WT would occur when the temperature during polymerization was increased to either 37°C or 50°C over that seen at room temperature (25°C) for either product and was only partially upheld by the data. Table 3 indicates that the WT of each product decreased with increasing polymerization temperature; however, there was no significant difference between WT values for Calibra between 37°C and 50°C. At 25°C, Calibra exhibited a WT of approximately 83 $\pm$7 seconds, while the value of Variolink II was much longer and much more variable at 311 $\pm$81 seconds. This difference might be attributed to the formulation between the products: concentration of polymerization inhibitors and benzoyl peroxide. According to the material safety data sheet information, Calibra contains approximately 2% benzoyl peroxide, while Variolink II has approximately half that amount (1%). For this reason alone, the better self-curing capability of Calibra over Variolink II may be explained.

Other studies have found that elevated resin temperatures may reduce WT,[25,26,35] even to intervals that are not clinically useful. In the current study, reduction in WT of Calibra from 83 seconds at 25°C to 23 seconds at 37°C and then to only 10 seconds at 50°C might compromise the seating of indirect restorations and the success of the restorative procedure as a consequence. Based on this result, it seems that preheating of resin cements with higher content of self-curing components should be avoided. Significant reduction in WT was also observed for Variolink II: from 311 seconds at 25°C to 95 seconds at 37°C and 37 seconds at 50°C. Although use of Variolink II at 50°C led to a clinically unfavorable WT of 37 seconds, the use of this resin cement at a temperature close to that of the oral cavity (37°C) led to a WT of 95 seconds, falling within the time range

considered by manufacturers as acceptable for clinicians to seat the indirect restoration.

Future studies simulating the clinical condition are required to evaluate the effects of preheating dual-cure cement temperature on mechanical properties and bond strength. In addition, studies need to be performed to measure the actual temperature value of preheated dual-cure cements when they are placed against freshly prepared teeth *in vivo*. With the teeth being much cooler than the heated cements, a dramatic decrease in cement temperature may result, negating the anticipated enhanced properties of increased conversion and decreased WT. Only selected dual-cure resin products were tested in the current work, and thus the results do not present the effects of the wide range of composition variation among these products. However, the two products selected, showing clear distinctions in their differing ability to utilize either the light- or the self-cure polymerization reactions, do clearly indicate the need for clinicians to be aware of differences among the commercial products available.

CONCLUSION

Within the limitations imposed by the current study, the following conclusions may be made:

1. Preheating dual-cured resin cements results in elevated monomer conversion values, even in a very inaccessible light-curing situation.
2. Increased resin cement temperature compensated for the lower radiant energy delivered from the light-curing unit caused by the presence of indirect restorations with varying thicknesses.
3. The WTs of dual-cure resins can be significantly decreased as a result of preheating, often leading to extremely rapid reactions, that would make clinical use of such a heated product inadvisable.

Acknowledgements

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Conflict of Interest Declaration

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

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Effect of Light Activation on Resin-modified Glass Ionomer Shear Bond Strength

NC Lawson • D Cakir • P Beck
L Ramp • JO Burgess

Clinical Relevance

All light-cured resin modified glass ionomers should be light polymerized after placement. This will greatly increase the bond strength of the material and should improve the longevity of the restoration.

SUMMARY

Objective: Recent studies confirmed that resin-modified glass ionomers (RMGIs) set on the basis of two competing mechanisms, an acid-base reaction and a light-activated resin polymerization. This study evaluated the effect of the setting mechanism on bond strength by measuring the shear bond strength of three RMGIs to dentin with and without light activation.

Methods: Sixty human molars were ground to midcoronal dentin and randomly divided into six even groups: 1) Ketac Nano (KN), 2) KN without light cure (woLC), 3) Fuji Filling LC (FF), 4) FF woLC, 5) Fuji II LC (FII), and 6) FII woLC. The dentin surfaces of the specimens were conditioned/primed according to the manufacturers' instructions. A 1.54-mm diameter plastic tube was filled with RMGI material and affixed to the dentin surface. Groups 1, 3, and 5 were light cured for 20 seconds, and groups 2, 4, and 6 were immediately placed in a damp dark box with no light curing at 37°C for 24 hours. Shear bond strength testing was performed in an Instron device at 1 mm/min. Data were analyzed with a one-way analysis of variance (ANOVA) and Tukey/Kramer test (α=0.05).

Results: Mean ± standard deviation shear bond strength values (MPa) are: 7.1 ± 4.2 (KN), 11.7 ± 3.9 (FF), 10.2 ± 3.2 (FF woLC), 12.5 ± 5.1 (FII), and 0.3 ± 0.4 (FII woLC). Two

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KN, all KN woLC, and seven FII woLC specimens debonded before testing. Tukey/Kramer analysis revealed no significant differences in bond strength between the three light-cured RMGIs. KN and FII showed significantly lower bond strength without light cure, but no significant difference was observed between FF and FF woLC.

Conclusions: The results of this study strongly suggest that light activation is necessary to obtain optimal bond strength between RMGI and dentin. FF may contain components that chemically activate resin polymerization. Clinically, KN and FII need to be light cured after placement of these RMGIs.

INTRODUCTION

Resin-modified glass ionomers (RMGIs) were introduced as a hybrid between conventional resin composites and glass ionomers.[1] Because these materials do not have the strength or wear resistance of conventional composites,[2] their use as posterior occlusal load bearing restoratives is questionable.[3-6] Resin-modified glass ionomers have poor retention as pit and fissure sealants[7] but excellent longevity as cervical restorations[8] and as liners and bases.[9] In these applications, it is critical for the RMGI to develop an effective bond to tooth structure.

Traditional glass ionomers bond to dentin by an ionic bond with hydroxyapatite, and conventional composite materials bond to dentin through micromechanical interlocking with collagen fibrils and dentinal tubules. RMGIs contain components of glass ionomers (fluoro-aluminosilicate glasses and polyacrylic acid) as well as resin composites (photo or chemical initiators and methacrylate monomers).[10] Due to their hybrid nature, RMGIs bond to dentin through both an ionic bond between polyacrylic acid and hydroxyapatite and mechanical interlocking with collagen and the resin monomer.[11] The initiation of this bond can be attributed to the various methods of polymerization of the RMGIs.

These materials polymerize by up to three mechanisms: 1) an acid-base reaction between the polyacrylic acid and the fluoro-aluminosilicate glass, 2) a photo-initiated free-radical reaction between methacrylate monomers, and 3) a chemically-initiated reaction between methacrylate monomers remaining after photo-initiation.[12] Recent studies have shown the acid-base and photo-initiated free-radical reactions inhibit each other, with photopolymerization of RMGIs reducing the acid-base reaction.[10,13] Another recent study concluded that the acid-base induced chemical interaction between RMGI and dentin is the main mechanism of bonding with RMGI.[14] Therefore, it is hypothesized that RMGIs allowed to polymerize without light activation will develop bond strength through the ionic bonding between RMGIs and dentin and should therefore have an equivalent or higher bond strength than light-cured RMGIs.

To test the hypothesis, the bond strength of RMGI to dentin with and without light polymerization was compared. It was assumed that bond strength to dentin with uncured RMGI would be attributed to an acid-base reaction. The null hypothesis was that there would be no difference between the light-cured and uncured groups.

MATERIALS AND METHODS

Sixty freshly extracted human molars were collected from the University of Alabama at Birmingham School of Dentistry. The occlusal surfaces of the teeth were ground to expose midcoronal dentin with a grinding wheel (Model 108; Wehmer Co, Addison, IL, USA). Specimens were polished with 600-grit SiC paper under water. The teeth were evenly and randomly divided into six groups: 1) Ketac Nano (KN), 2) KN without light cure (woLC), 3) Fuji Filling LC (FF), 4) FF woLC, 5) Fuji II LC (FII), and 6) FII woLC.

Immediately following polishing, all groups (n=10) were conditioned and primed according to the manufacturers' instructions (Table 1). A transparent rubber tube (1.54 mm inner diameter and 4 mm length) was filled to a depth of 2 mm with the corresponding RMGI and pressed to the prepared surface of each sample. Groups 1, 3, and 5 were then light polymerized with an Elipar S10 LED curing light (813 mW/cm^2, 3M ESPE, Seefeld, Germany) from above and both sides for 20 seconds and placed in a damp, dark box. Groups 2, 4, and 6 were placed in the damp dark box within one minute after affixing the rubber tube without light curing. The box was then placed in a 37°C incubator (Queue; ThermoElectron, Waltham, MA, USA) for 24 hours.

The specimens were removed from storage and dried with absorbent wipes. The rubber tube was cut away to reveal a 1.54-mm diameter RMGI cylinder bonded to each specimen. The specimens were loaded into a custom fixture in a universal testing device (Instron, model 4411, NVLAP, Canton, MA, USA) that secured the teeth with the bonded RMGI from three sides. A blade attached to the upper member of the Instron was used to fracture the

Table 1: *Products Used in This Study*

Material	LOT	Composition	Directions
Ketac Nano – A3	20090515	Silane-treated glass (40%), Silane-treated zirconia (20%), PEGDMA (5%), silane-treated silica (5%), HEMA (1%-15%), Glass powder (<5%), Bis-GMA (<5%), TEGDMA (<1%)	—
Ketac Nano GI Primer	7AC	Water (40%), HEMA (35%), copolymer of acrylic and itaconic acids (10%)	Rinse and dry tooth, apply primer for 15 s, dry for 10 s, and light cure for 10 s
GC Fuji Filling LC – A2	0905011	Paste A: alumino-silicate glass (75%), HEMA (10%), UDMA (2%); paste B: water (20%), polyacrylic acid (20%), UDMA (12%), silica (10%)	—
GC Self Conditioner	0906021	Ethanol (28%), water (30%), HEMA (20%), 4-methacryloxyethyl trimellitate anhydride (5%)	Rinse and dry tooth, apply conditioner for 10 s, dry for 5 s
GC Fuji II LC Capsules – A2	0804255	Powder: alumino-silicate glass (75%); liquid: polyacrylic acid (20%), HEMA (35%), propriety ingredient (5%), 2,2,4 trimethyl hexamethylene dicarbonate (5%), TEGDMA (4%)	—
GC Cavity Conditioner	0604191	Polyacrylic acid (20%), water (77%), aluminum chloride hydrate (3%), blue food additive (<0.1%)	Rinse and dry tooth, apply conditioner for 10 s, rinse and blot dry

Abbreviations: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, hydroxy ethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

specimens at a crosshead speed of 1 mm/min. The maximum load required to separate the RMGI cylinder from dentin was recorded. Shear bond strength (SBS) was determined: maximum load/surface area of composite post. The fracture surfaces of the specimens were examined under light microscopy at 100× magnification (VHX-600, Keyence Co, Osaka, Japan) to determine the mode of failure.

The normal quantile plot of shear bond strength by treatment was examined to determine acceptable normality for the data. Differences between treatment groups were analyzed statistically by analysis of variance (ANOVA) (α=0.05). Groups were compared to the mean using a Tukey *post-hoc* test (α=0.05).

RESULTS

Table 2 shows the mean SBS and standard deviations of all groups tested. Two KN, all KN woLC, and seven FII woLC specimens debonded before testing. For these groups, the specimens with premature failures were given a value of zero and included in the statistical analyses. A one-way ANOVA revealed significant differences among testing groups ($p<0.05$). A Tukey/Kramer analysis revealed no significant differences in bond strength between the three light-cured RMGIs (Ketac Nano, Fuji II LC, and Fuji Filling). KN was assumed to be greater than KN woLC because the bond with KN woLC was too weak to survive testing. FII was significantly

Table 2: *Mean Shear Bond Strength (MPa) and Standard Deviation of the Resin-modified Glass Ionomers Tested (N=10)*

Group	Mean ± SD
1 Ketac Nano (KN)	7.07 ± 4.21
2 Ketac Nano without light cure (KN woLC),	0
3 Fuji Filling LC (FF)	11.66 ± 3.91
4 Fuji Filling LC without light cure (FF woLC)	10.18 ± 3.23
5 Fuji II LC (FII)	12.46 ± 5.06
6 Fuji II LC without light cure (FII woLC)	0.26 ± 0.45

Table 3: *Mode of Failure of Each Group*

Group	Adhesive	Mix	Cohesive
1 Ketac Nano (KN)	4	5	1
2 Ketac Nano without light cure (KN woLC),	8	2	—
3 Fuji Filling LC (FF)	7	3	—
4 Fuji Filling LC without light cure (FF woLC)	8	2	—
5 Fuji II LC (FII)	4	3	3
6 Fuji II LC without light cure (FII woLC)	8	2	—

greater than FII woLC ($p<0.05$), but no significant difference was observed between FF and FF woLC. Fracture modes of failure are presented in Table 3. Uncured specimens most frequently fractured by adhesive failure, while some light-cured specimens failed cohesively. The ratios of adhesive:mixed:cohesive fractures for cured KN and FF, respectively, were 4:5:1 and 4:3:3, and uncured KN and FF were 8:2:0 for both. The same ratio for cured and uncured FII samples were 7:3:0 and 8:2:0, respectively.

DISCUSSION

Light-cured RMGIs develop bond strength through the fast-acting light-initiated reaction of resin monomers as well as an acid-base reaction and a chemically-initiated free-radical reaction. Non–light-cured RMGIs polymerize through only the acid-base and chemical-initiated free-radical reactions. Our study reports low bond strength of non–light-cured RMGIs (excluding FF) and relatively high bond strength of cured RMGIs. Therefore, our results suggest that most RMGIs do not develop adequate bond strength without light-activated resin polymerization, rejecting the null hypothesis.

The non–light-polymerized Fuji Filling LC was the only non–light-cured material to produce the same SBS as the light-cured specimens. The manufacturer of this material indicated that a chemical initiator is included in FF but not FII, and it is possible that the chemical initiator activated the free radical resin polymerization in FF. The manufacturer of KN indicated that a chemical initiator is not included in their material. Therefore, the KN woLC and FII woLC groups polymerized only through an acid-base reaction.

The contribution of the acid-base and free radical reactions on the mechanism of bonding of RMGIs has not been completely elucidated, and both mechanisms of bonding have been reported in dental literature. Mitra and others[15] and Coutinho and others[16] analyzed the reaction between methacrylated copolyalkenoic acid (a key component of RMGIs) and pure hydroxyapatite (HAP) crystals. Fourier transform infra-red spectroscopy and X-ray photoelectron spectroscopy analyses indicated an ionic bond between the methacrylated polyalkenoic acid and HAP, suggesting an acid-base reaction. On the other hand, scanning electron microscopy analysis of interfacial microstructure between RMGIs and dentin, performed by Coutinho and others,[16] showed a submicron hybrid layer of RMGI and an analysis by Mitra and others[15] showed tag-like structures of RMGI penetrating dentin. Carvalho and others[17] describe a similar phenomenon terming it a demineralized, resin-infiltrated zone—a characteristic of free-radical polymerization based bonding.

A study by Cardoso and others[14] measured the bond strength of RMGIs bonded to fractured dentin with and without the use of a conditioner. Micrographs revealed the presence of a hybrid layer in the conditioned dentin specimens but not in the unconditioned specimens. However, no difference in bond strength was measured between the conditioned and unconditioned dentin groups. Based on these observations, the authors suggested that the mechanical interlocking provided by a hybrid layer is not the primary mechanism of bonding with RMGIs. The authors later acknowledged the possibility of a nanometer-scaled hybrid layer created by the mild self-etching properties of the polyalkenoic acid present in the RMGI. In summary, the effect of either bonding mechanism towards the adhesion of RMGIs to dentin is still unclear.

Traits of both ionic (acid-base) and resin infiltration (free-radical) bonding are reported in current literature; however, this study suggests resin infiltration creates the strength of the bond of RMGI to dentin. *In vitro* testing has shown that the SBS of glass ionomer materials is weaker than that of resin composites. Additionally, previous studies have reported that RMGIs have SBS values greater than glass ionomer materials but less than composites.[18,19] Therefore, it would be expected that conditions that enable a RMGI to behave like a

resin composite material, such as light polymerization, would favor higher SBS values.

The failure analysis revealed that more of the light-cured specimens of FII and KN failed by cohesive and mixed failures. This result further indicates that a stronger bond was formed between light-cured RMGI and dentin than uncured RMGI and dentin. The failure modes of cured and uncured FF were nearly identical, most likely due to the chemical-initiated free radical polymerization of the uncured FF.

All RMGI groups in this study were applied preceding a coat of conditioner or primer as indicated by the manufacturer. FF and FII both use a conditioner. Conditioners can remove the smear layer and partially demineralize dentin,[20] and the use of a conditioner can significantly improve bond strength.[21,22] KN uses a light-cured primer. The requirement to light polymerize this primer indicates that it contains a photo-initiated resin component—similar to an adhesive. Pereira and others[23] concluded that the SBS of RMGI to dentin improved with the use of an adhesive, a technique that facilitates resin bonding. The layer of adhesive, however, decreases the fluoride release from these materials.[24-25] The results of Pereira and others reinforce the finding that light-activated resin polymerization provides the strength of the bond between RMGI and dentin.

A limitation of this study is that only three RMGIs were examined. Varying chemistries of other RMGIs may produce different results. Future studies could compare the SBS of additional RMGIs.

CONCLUSIONS

KN and FII have increased bond strength after light polymerization. The results suggest that light activation is needed to obtain optimal bond strength to dentin with some RMGIs. Clinically, KN and FII should be light cured after placement to achieve optimum bond strength.

Conflict of Interest Declaration

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

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Influence of Matrix Metalloproteinase Synthetic Inhibitors on Dentin Microtensile Bond Strength of Resin Cements

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Clinical Relevance

Application of metalloproteinase synthetic inhibitors such as 2% chlorhexidine does not compromise immediate dentin bond strength of self-adhesive resin cements. The application of 24% ethylenediamine tetra-acetic acid (EDTA) gel increases dentin bond strength when self-adhesive resin cements containing methacrylated phosphoric acid ester monomers are used. Since EDTA is also considered a synthetic matrix metalloproteinase inhibitor, 24% EDTA gel might be a simple alternative to be used in conjunction with self-adhesive resin cements to improve their clinical performance.

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SUMMARY

This study evaluated the effect of dentin pretreatment with 2% chlorhexidine (CHX) or 24% ethylenediamine tetra-acetic acid gel (EDTA) on the dentin microtensile bond strength (μTBS) of resin cements. Composite blocks were luted to superficial noncarious human dentin (n=10) using two resin cements (RelyX ARC [ARC] and RelyX U100 [U100]) and three dentin pretreatments (without pretreatment–control, CHX, and EDTA). CHX was applied for 60 seconds on the acid-etched

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dentin in the ARC/CHX group, and for the same time on smear layer-covered dentin in the U100/CHX group. EDTA was applied for 45 seconds on smear-covered dentin in the U100/EDTA group, and it replaced phosphoric acid conditioning in the ARC/EDTA group for 60 seconds. After storage in water for 24 hours, specimens were prepared for microtensile bond strength testing. The results were submitted to two-way analysis of variance (ANOVA) followed by Tukey test. ARC produced significantly higher μTBS ($p<0.05$) compared to the U100, except when EDTA was used. For ARC, no pretreatment and CHX produced higher μTBS than EDTA. For U100, EDTA produced higher μTBS; no statistical difference occurred between CHX pretreatment and when no pretreatment was performed. While CHX did not affect immediate dentin bond strength of both cements, EDTA improved bond strength of U100, but it reduced dentin bond strength of ARC.

INTRODUCTION

The clinical success of indirect restorative procedures depends in part on the cementation technique used to create a stable link between the restoration and the different tooth structures. Resin cements have been widely used for this purpose, including fixation of inlays, onlays, crowns, posts, and veneers because of their enhanced mechanical properties, ease of handling, and good esthetic qualities. In this context, self-adhesive cements were recently introduced to simplify clinical practice as dentin acid-etching is not necessary. Currently, it is well accepted that a strong and stable union between the resin material and tooth substrate is highly important in determining the durability of the tooth-restoration complex.[1]

Despite many significant improvements in the past years, the bonded interface still remains the weakest area of the restorative complex.[2] Degradation of resin-dentin bonds, due to hydrolysis of the collagen fibrils, occurs over time[3-5] involving the participation of endogenous matrix metalloproteinases (MMP), which become entrapped within the dentin substrate during tooth development. When dentin is etched using phosphoric acid, latent MMP is denatured as rapidly as more proteases are exposed during dentin demineralization.[6] As a result, collagen fibrils that are not completely protected by resin monomers during dentin hybridization become highly susceptible to hydrolytic degradation over time, reducing bond strength. These gelatinolytic/collagenolytic enzymes can be produced by odontoblasts when a low pH environment is created along the dentin substrate.[7] Mild acids are known to activate MMP.[6] Low initial pHs are produced when resin adhesive materials composed of acidic monomers are coupled to dentin. An increase in MMP expression by the dentin pulp-complex,[7] as well as increased collagenolytic activity[8] to near-maximum levels,[6] can be expected when self-adhesive cements are bonded to dentin, similar to adhesive procedures involving self-etching adhesives, because residual unpolymerized acidic monomers might continue to etch the dentin substrate and activate MMP.[9] These events may contribute to resin-dentin bond degradation over time[6-8] when low pH adhesive resins such as self-adhesive resin cements are bonded to dentin. Even though most adhesive resin materials fulfill the necessity of a strong bond immediately after dentin coupling, bonding effectiveness naturally drops over time.[10-13]

Many approaches have been tested to increase the bonded interface durability by overcoming this self-degradation process. The use of matrix metalloproteinase synthetic inhibitors, such as chlorhexidine (CHX)[14-17] and ethylenediamine tetra-acetic acid (EDTA),[5,18] is a valid alternative as an attempt to prolong the resin-dentin bonding stability.[2,16] Nevertheless, little information involving self-adhesive resin cements and synthetic MMP inhibitors can be found. While CHX seems to impair self-adhesive resin cement bond strength to dentin,[19] EDTA does not affect dentin microtensile bond strength of self-adhesive resin cements.[20]

Dentin pretreatment with MMP synthetic inhibitors would benefit the dentin bond of self-adhesive resin cements over the course of time, as long as dentin bond strength is not immediately impaired. The purpose of this study was to assess the influence of a higher concentration of EDTA and 2% chlorhexidine digluconate, on the dentin microtensile bond strength of a self-adhesive resin cement and a conventional dual-cure resin cement. The hypotheses to be tested were: 1) 2% CHX has no influence on the immediate dentin bond strength of both a self-adhesive resin cement and a conventional resin cement; 2) 24% EDTA gel increases self-adhesive resin cement dentin bond strength and; 3) substitution of phosphoric acid etching with 24% EDTA gel does not affect dentin bond strength of conventional resin cements.

MATERIALS AND METHODS

Tooth Preparation

Sixty recently extracted human noncarious lower third molars stored at 4°C in saline solution with 0.2% sodium azide for up to one month were obtained after patient informed consent under a protocol analyzed and approved by the Ethical Committee of the Federal University of Uberlândia, Brazil. After disinfection and removal of soft tissues, a flat coronal dentin surface, perpendicular to the tooth's longitudinal axis, was ground flat using 180-grit SiC paper (Norton, Saint-Gobain Abrasives, Garulhos, SP, Brazil) and standardized with 600-grit SiC paper (Norton, Saint-Gobain Abrasives) for one minute under water cooling.

Cylindrical composite blocks were prepared using a nanofilled light-activated resin composite (Filtek Supreme Z-350, 3M ESPE, St Paul, MN, USA). Incremental layers measuring no more than 2 mm in thickness of composite were placed into a Teflon mold (5 mm in thickness and 10 mm in diameter) and individually light-cured using a quartz-tungsten halogen (QTH) unit (3M Curing Light, 3M ESPE) with irradiance of 550 mW/cm^2. To improve double-bond conversion, the cylindrical blocks were heat treated at 110°C for five minutes inside an inlay composite chamber (Fotoceram, Goiânia, GO, Brazil). One side of the composite block was abraded with 600-grit SiC paper (Norton, Saint-Gobain Abrasives) under water cooling to create a flat surface with standardized roughness and air-abraded with 50-μm aluminum oxide particles (Bioart, São Carlos, SP, Brazil) for 10 seconds, at four bars pressure and 10 mm away from the composite surface. The composite blocks were ultrasonically cleaned in distilled water for 10 minutes, rinsed with running water, completely air dried, treated with a prehydrolyzed silane solution (Prosil, FGM, Joinville, SC, Brazil), and blow dried before bonding.

Luting Procedures

Two resin cements were used: one self-adhesive luting cement that requires no substrate pretreatment (RelyX U100, 3M-ESPE) and one conventional dual-cured cement requiring previous dentin etching and application of an adhesive system prior to the luting procedure (RelyX ARC, 3M-ESPE) (Table 1). Teeth were randomly assigned to six groups (n=10): 1) ARC, 2) ARC/CHX, 3) ARC/EDTA, 4) U100, 5) U100/CHX, and 6) U100/EDTA. The groups were divided according to the following factors: resin cement (ARC or U100) and dentin pretreatment (CHX, EDTA or distilled water). Control groups were treated with distilled water instead of CHX or EDTA before dentin hybridization or luting with the self-adhesive cement. In all groups, moisture control was performed with sterilized lint-free 1-cm diameter absorbent papers (Mellita Clássico, Celupa Industrial Cellulose e Papel Guaíba LTDA, RS, Brazil) before and after dentin pretreatments: absorbent papers were gently placed on top of the flat dentin surface and replaced after five seconds until visible water was no longer absorbed and a dentin surface with a slightly glossy appearance was observed. This step was performed to remove excess moisture while ensuring adequate conditions for bonding of both resin cements. Adhesive procedures were then immediately carried out in a controlled environment with a temperature of 24°C ± 1°C and a relative humidity of 50% ± 5% using the same QTH unit used for the photocuring of the indirect restorations.

In group ARC/CHX, the flat dentin surface was etched with 37% phosphoric acid (Scotchbond Etchant, 3M ESPE) for 15 seconds and rinsed with water for 30 seconds; excess moisture was removed with absorbent paper. CHX pretreatment was performed and consisted of light-pressure circular rubbing movements of 2% chlorhexidine digluconate (Clorhexidina s, FGM) for 60 seconds, using a cavity brush (Cavibrush, FGM). Excess moisture was removed once again with absorbent paper, and one coat of primer (Adper Scotchbond Multi-Purpose, 3M ESPE) was applied actively for 10 seconds and gently blow-dried; this was followed by active application of one coat of adhesive (Adper Scotchbond Multi-Purpose, 3M ESPE) for 10 seconds and then light-activation for 10 seconds. The indirect restoration was then luted with RelyX ARC. In group U100/CHX, moisture control was performed and CHX was applied on the smear layer-covered dentin for 60 seconds with a cavity brush. Excess moisture was removed with absorbent paper and the indirect restoration was luted with RelyX U100.

For group U100/EDTA, moisture control was performed, EDTA 24% gel (E.D.T.A. Gel, Biodinâmica, Ibiporã, PR, Brazil) was carefully applied for 30 seconds on the smear layer-covered dentin with circular rubbing movements using a cavity brush and rinsed for 30 seconds. Excess moisture was removed and the indirect restoration was luted with RelyX U100. In group ARC/EDTA, moisture control was performed, EDTA was applied for 60 seconds with circular rubbing movements replacing the phosphoric acid-etching step and rinsed for 30 seconds. Excess moisture was removed and the

Table 1: *Material Brand Name, Composition, and Manufacturer*

Brand Name	Composition	Manufacturer
Scotchbond Etchant	37% phosphoric acid	3M ESPE Dental Products, St Paul, MN, USA
Adper Scotchbond	Primer: HEMA, polyalkenoic acid methacrylate copolymer Adhesive: Bis-GMA, HEMA, photo-initiators	3M ESPE Dental Products, St Paul, MN, USA
RelyX U100	Phosphoric acid methacrylates, dimethacrylates, inorganic fillers (72 wt %), fumed silica, initiators	3M ESPE Dental Products, St Paul, MN, USA
RelyX ARC	TEGDMA, Bis-GMA, zirconia/silica filler (67.5 wt%, initiators	3M ESPE Dental Products, St Paul, MN, USA
Filtek Z-350	Bis-GMA, UDMA, TEGDMA, ethyl methacrylates, inorganic fillers, photo-initiators	3M ESPE Dental Products, St Paul, MN, USA
E.D.T.A. Gel	24% EDTA	Biodinâmica, Ibiporã, PR, Brazil
Clorhexidina s	2% Chlorhexidine	FGM, Joinville, SC, Brazil

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

dentin hybridization and luting procedures were done as described for the ARC/CHX group.

In the control groups (ARC and U100), no dentin pretreatment with MMP synthetic inhibitors was performed: distilled water was applied to dentin to serve as a control treatment. Since both CHX and EDTA have water in their chemical compositions, we found it reasonable to submit control groups to this moisture factor to keep the experiment in a more controlled condition. An additional removal of excess water happened, returning the dentin to the same initial condition. In the ARC group, the same bonding procedures were carried out as in the ARC/CHX group except that CHX application was also replaced with distilled water for 60 seconds. Moisture control with absorbent paper, adhesive system application and luting with RelyX ARC were the same. In the U100 group, moisture control was performed, distilled water was applied on the smear layer-covered dentin for 60 seconds, moisture control was performed again, and the indirect restoration was luted with RelyX U100.

In all groups, following the application of the respective resin cement on the dentin surface, the composite resin blocks were placed on top of the flat dentin surfaces and received a constant seating pressure of 3 kg for three minutes[19] after which excess cement was removed and then light-activation was performed from four different directions for 40 seconds along the cement interface.

Microtensile Bond Test

After storage in distilled water at 37°C for 24 hours, the bonded teeth were sectioned (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL, USA) occlusogingivally into serial slabs and further into 0.9 mm × 0.9 mm composite-dentin sticks. Ten restored teeth were used in each group. Only central sticks were selected as peripheral sticks may not have had the same dentin thickness. Six central sticks were individually attached to a metallic grip with a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA) and submitted to the microtensile bond test on a mechanical testing machine (DL2000, EMIC, São José dos Pinhais, PR, Brazil) at crosshead speed of 0.5 mm/min until failure using a Geraldeli device. The bond strength value for each tooth was determined by the microtensile bond strength average values including the six sticks from each tooth. After testing, the specimens were carefully removed from the grips with a scalpel blade, and the cross-sectional area at the site of the fracture was measured to the nearest 0.001 mm using a digital micrometer (Digimatic

Micrometer, Mitutoyo, Japan). Microtensile bond strength test values were expressed in MPa, and the data were submitted to two-way analysis of variance (ANOVA) followed by Tukey test. Statistical significance was set in advance at $\alpha=0.05$, considering the tooth as the statistical unit. Sticks with pretest failures were recorded as null bond strengths, and those values were included in the statistical analysis.

SEM Failure Mode Analysis

After bond strength testing, fractured sticks were mounted on aluminum stubs, gold-sputtered under high-vacuum (MED 010, Balzers Union, Balzers, Liechtenstein) and analyzed using a scanning electron microscope (LEO 435 VP; LEO Electron Microscopy Ltd, Cambridge, UK). The work distances ranged between 18 and 22 mm, according to specimen height. Each specimen was classified according to the predominant remaining structure upon the dentin surface following the described failure mode classification:[19] 1: adhesive failure along the cement/dentin interface, 2: adhesive failure along the cement/composite interface, 3: cohesive failure within resin cement, 4: mixed failure of 1 and 3, and 5: mixed failure of 2 and 3.

RESULTS

ANOVA two-way analysis of variance revealed that resin cement ($p<0.001$), as well as the interaction between dentin pretreatment and resin cement ($p<0.001$), had significant effects on the microtensile bond strengths. RelyX ARC produced significantly higher microtensile values ($p<0.05$) when compared to the groups luted with RelyX U100, except when EDTA was used (Table 2). CHX pretreatment did not have significant effects on the dentin microtensile bond strengths ($p<0.05$) regardless of the resin cement used. In contrast to the distilled water and CHX pretreatment, dentin pretreatment with 24% EDTA gel significantly reduced dentin microtensile values ($p<0.05$) when Relyx ARC was used, and it increased bond strength ($p<0.05$) when dentin was luted with RelyX U100.

Cohesive failures within the resin composite or dentin substrate were not observed in any of the groups. Representative failure modes for RelyX ARC can be observed in Figure 1. In group ARC, the prevalent type of failure was adhesive along the cement/composite interface (2), followed by a mixed pattern of adhesive failure along the cement/composite interface and cohesive failure along resin cement (5), which were the same predominant failures observed in group ARC/CHX (Table 2). Fractured resin tags tightly occluding the enlarged dentinal tubule entrances after H_3PO_4-etching were observed when adhesive failure occurred along the cement/dentin interface (Figure 1F). The group ARC/EDTA presented predominately a mixed pattern of adhesive failure along the cement/dentin interface and cohesive failure along resin cement (4), followed by adhesive failures of the cement/dentin interface (1). Sparse enlarged tubule entrances were evident when adhesive failure along cement dentin interface occurred.

Table 2: *Overall Means and Standard Deviations of Microtensile Bond Strength Values for All Groups (MPa ± SD) and Fracture Modes. The Tooth Was Considered the Statistic Unit (n=10)*[a]

Pretreatment	RelyX ARC	RelyX U100
Distilled water	42.72^{aA} ± 4.3 (1/21/15/3/20)	12.98^{bB} ± 3.4 (6/1/26/22/2) N_0=3
Chlorhexidine	39.19^{aA} ± 7.7 (3/24/10/2/21)	11.56^{bB} ± 2.6 (10/1/14/29/1) N_0=5
EDTA	22.78^{bA} ± 5.6 (8/14/6/13/19)	18.61^{aA} ± 4.6 (3/4/30/17/4) N_0=2

Abbreviation: EDTA, ethylenediamine tetra-acetic acid.
[a] Values with different lowercase letters indicate significant difference according to Tukey test (p<0.05) when analyzed per column. Different capital letters indicate significant difference according to Tukey test (p<0.05) when analyzed per row. Numbers in parentheses are the number of specimens classified into five fracture modes in sequence (1/2/3/4/5): 1) adhesive failure along the cement/dentine interface; 2) adhesive failure along the cement/composite interface, 3) cohesive failure within resin cement, 4) mixed failure of 1 and 3; and 5) mixed failure of 2 and 3 (N_0=the number of premature failures).

Representative failure modes for RelyX U100 can be observed in Figure 2. In group U100, the prevalent mode of fracture occurred cohesively within the resin cement (3), which also was observed in group U100/EDTA. When CHX pretreatment was performed, RelyX U100 presented mainly a mixed pattern between adhesive failure in the cement/dentin interface and cohesive failure along resin cement (4), followed by cohesive failure of resin cement (3). When adhesive failures along the cement/dentin interface occurred for RelyX U100, no open dentinal tubules were evident irrespective of the dentin pretreatment. When CHX pretreatment was performed, a high number of bubbles could be observed when adhesive failure along the dentin/cement interface occurred (Figure 2F).

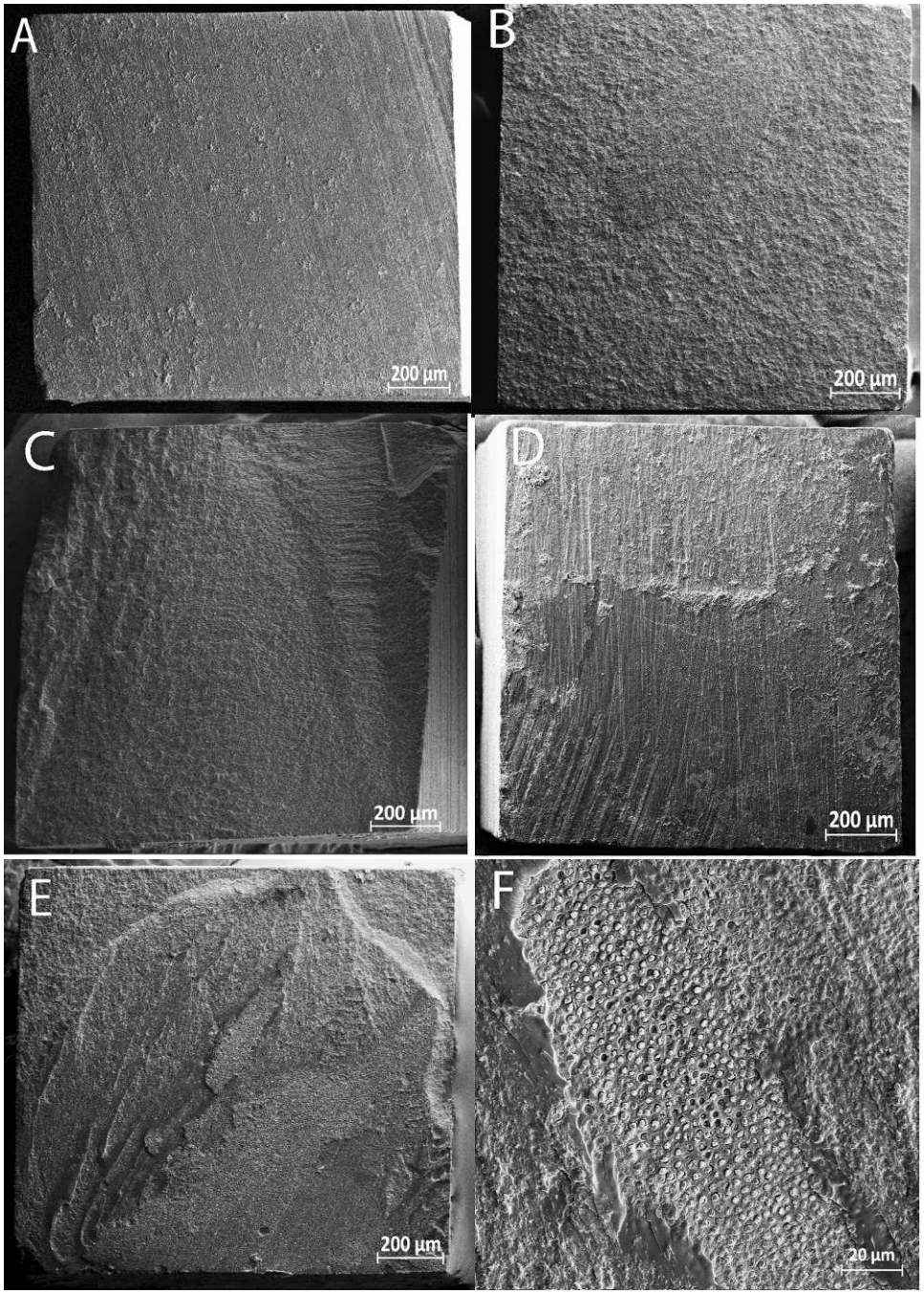

Figure 1. *Representative scanning electron micrographs for RelyX ARC failure modes. Dentin sides of fractured sticks are shown. (A): Adhesive failure along the cement/dentin interface (1). (B): Adhesive failure along the cement/composite interface (2). (C): Cohesive failure within resin cement (3). (D): Mixed failure of 1 and 3 (4). (E): Mixed failure of 2 and 3 (5). (F): Higher magnification showing a mixed failure at the base of the hybrid layer and cohesively along resin cement, with fractured resin tags tightly occluding the enlarged dentinal tubule entrances after H_3PO_4-etching.*

DISCUSSION

Since 2% CHX application produced no negative effects on the dentin microtensile bond strength of either the conventional or the self-adhesive resin cement, the first hypothesis is accepted. Conventional resin cement bonded to phosphoric acid-etched dentin exhibited higher microtensile values than self-adhesive resin cement, irrespective of CHX application, which can be explained by the differences in the bonding mechanism of both cements. Self-adhesive resin cements are composed of acidic monomers that

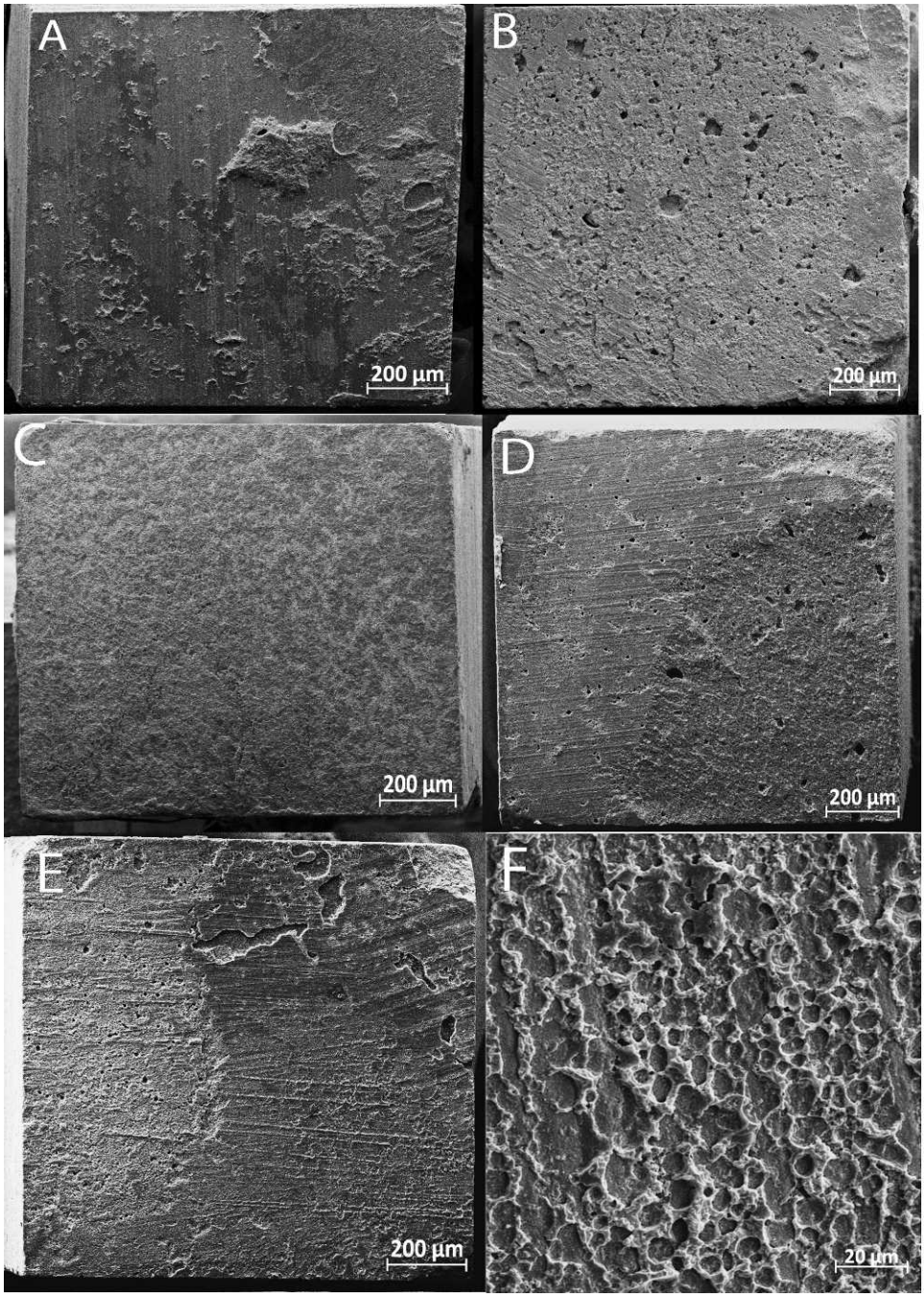

Figure 2. *Representative scanning electron micrographs for RelyX U100 failure modes. Dentin sides of fractured sticks are shown. (A): Adhesive failure along the cement/dentin interface (1). (B): Adhesive failure along the cement/composite interface (2). (C): Cohesive failure within resin cement (3). (D): Mixed failure of 1 and 3 (4). (E): Mixed failure of 2 and 3 (5). (F): Higher magnification of adhesive failures along dentin/cement interface when dentin was pretreated with chlorhexidine with formation of a high number of bubbles. Notice the absence of open dentinal tubules, which was observed in all specimens luted with RelyX U100 irrespective of dentin pretreatment.*

simultaneously demineralize and infiltrate the tooth substrate, resulting in micromechanical retention. This single step resin cement complies with the demand for simplification of luting procedures. Although this bonding strategy makes clinical practice easier, the absence of a dentin conditioning step creates a limited decalcified substrate and decreases resin monomer diffusion into dentin.[21] Even with a low initial pH of 2.1,[22] nearly no demineralization of the dentin surface below the smear layer is noticed

after cement setting.[23,24] The relatively high viscosity of resin cement[25] associated with a low demineralizing capacity[24] contributes to low monomer infiltration into dentin, reducing micromechanical retention. Nevertheless, RelyX U100 has a dual-set polymerization reaction, a dual-cured redox-reaction for polymerization of the resinous phase, and an acid-base reaction. In this bonding mechanism, the calcium atoms present in the dentin hydroxyapatite act as electron acceptors promoting chemical union between the acidic resin monomers and the hard dental tissues,[26] resulting in the formation of calcium phosphates.[24] Such bonds do not exhibit a high bonding energy. As a consequence, low dentin microtensile values were obtained for the groups luted with RelyX U100. The use of CHX along with total etch adhesive resins does not impair their ability to bond to dentin.[14,16,27-31] This finding is in agreement with the present study where a bisphenol A-glycidyl methylmethacrylate (Bis-GMA)/hydroxyethyl methacrylate (HEMA) three-step etch and rinse adhesive system was used along with a conventional dual resin cement.

The priming step was performed with a HEMA-based primer that does not debond CHX molecules from the dentin substrate[32] allowing the CHX molecules to remain trapped within the base of the hybrid layer. In addition, a total-etch hybridization process involving twostep adhesive resins forms a superficial hydrophobic layer previous to the application of the resin cement. This layer acts as a barrier decreasing the possibility of CHX molecules located in the dentin substrate to chemically interact with methacrylate monomers present in the resin cement; any interference in the bonding process of conventional resin cements would be minimized. As a consequence, chlorhexidine application did not alter failure modes when dentin was luted with RelyX ARC. Similarly, CHX had no influence on the immediate dentin bond strength of tested self-adhesive resin cement. This finding is not in agreement with a previous study[19] where moisture removal was not properly performed and lower dentin bond strength values were obtained when CHX was used prior to the application of a self-adhesive resin cement with the same composition as RelyX U100. Water has a critical role in the effectiveness of self-adhesive resin cement bonding. It is generated during neutralization of functional groups modified by phosphoric acid and reused to react with acidic functional groups and ion-releasing basic filling bodies. However, when excess water is present, the polymerization reaction might be affected because the accumulation of oversaturated water droplets in the microvoids within the polymer network might decrease the cohesive strength of the self-adhesive resin cement. Since adequate moisture removal was performed, dentin pretreatment with CHX did not affect RelyX U100 bond strength. The prevalent mode of fracture for RelyX U100 occurred cohesively within the resin cement, which is in agreement with other studies.[19,20] When chlorhexidine was used, an increased number of mixed failures involving adhesive failure in the cement/dentin interface and cohesive failure along the resin cement occurred. In addition, resin cement remnants on the dentin side of fractured sticks presented a consistent bubbly pattern (Figure 2), which might suggest a possible interaction between RelyX U100 and CHX. Regardless, such interaction did not impair the immediate dentin bond strength of the resin cement. Further studies are needed to support the use of CHX as a widespread dentin pretreatment along with self-adhesive resin cements.

EDTA is a MMP synthetic inhibitor[18] that has been widely studied as a dentin-etching agent.[29,33-37] At neutral pH,[38] it is considered a mild chelating agent that produces different effects on dentin depending on its concentration and time of exposure.[39] EDTA dissolves dentinal mineral phase without shifting dentin proteins,[40] which avoids major alterations of the collagen fibrillar structure, conferring stability to the organic-matrix.[33,38,41] The extent of dentin demineralization is reduced when compared with phosphoric acid etching; hydroxyapatite is selectively removed, promoting partial removal of the smear layer. Maintenance of about 30% of the smear plugs and no morphologic alteration of the dentin surface are observed following application of 17% EDTA for 60 seconds.[37] The self-limited chelation reaction between EDTA and calcium present on the dentin substrate occurs in a ratio of 1:1, creating a stable EDTA-Ca compound responsible for dentin demineralization.[42] When dentin is etched with 30%-40% phosphoric acid, the smear layer is completely removed forming a layer of mineral-depleted collagen fibrils. Inadequate monomer infiltration of such compact collagen mesh impairs the dentin bond strength of self-adhesive resin cement to phosphoric acid-etched dentin.[43] Therefore, partial removal of the smear layer with EDTA solution was tested in a previous study, but no improvement in the self-adhesive resin cement-dentin bond occurred.[20] In the present study, an even higher EDTA concentration was tested to combine MMP synthetic inhibition properties with more pronounced smear layer removal[44] in an

attempt to promote better interaction between resin cement and underlying dentin without compromising bond strength. As a consequence, dentin pretreatment with 24% EDTA gel significantly increased the self-adhesive resin cement bond to dentin, so the second hypothesis must be accepted. Statistically similar bond strengths were obtained between RelyX ARC and RelyX U100 when EDTA gel was used. The 24% EDTA gel improved the RelyX U100 dentin bond, while it decreased RelyX ARC bond strength. Nevertheless, RelyX ARC conventionally bonded to dentin still exhibited superior dentin bond strengths. In the ARC/EDTA group, an increase in mixed failures along the cement/dentin interface and resin cement occurred when compared to the ARC group. In this manner, the EDTA-treated dentin surface acted as a weak link to the bonded restoration. Sparse enlarged dentinal tubule entrances were evident when adhesive failures occurred at the dentin/cement interface, suggesting that adhesive system penetration into dentin was hampered. The adhesive system inability to properly infiltrate dentin certainly contributed to bond strength reduction when dentin was treated with EDTA. No differences in failure modes occurred between EDTA-treated and nontreated dentin when RelyX U100 was used. In specimens with adhesive failures at the dentin/cement interface, incomplete removal of the smear layer was observed when EDTA was used. Despite the use of a demineralizing agent, no open dentinal tubules and resin tag occlusion of dentinal tubule entrances were evident demonstrating low capacity of RelyX U100 to demineralize and infiltrate dentin.

From a clinical perspective, it remains to be proved if EDTA pretreatment will be able to effectively prevent resin-dentin bond degradation once it does not impair immediate self-adhesive dentin bonding. Consequently, longer term studies would be advisable. Dentin demineralization by acidic monomers may release some sequestered growth factors, which could, along with unpolymerized acidic monomers, diffuse through dentinal tubules stimulating the expression of MMP by odontoblasts,[7] possibly contributing to bond degradation. For these reasons, dentin pretreatment with MMP inhibitors might play an important role preserving the self-adhesive resin cement dentin bond. Regarding EDTA pretreatment and conventional resin cements, the third hypothesis was rejected because replacement of phosphoric acid etching for 15 seconds with 24% EDTA gel for 60 seconds reduced dentin bond strength of the conventional resin cement tested. Because one of the most important factors determining the bonding effectiveness of conventional resin cements is the adhesive system,[1] impairment of its capability to diffuse into substrate affects dentin bond quality. While self-etching adhesives seem to benefit from additional dentin pretreatment with mild chelating etchants,[34] conflicting results are found in the literature regarding total-etch adhesive systems when EDTA is used in place of phosphoric acid etching. Even though different application times (which may vary from 30 to 240 seconds) of EDTA 24% gel do not affect dentin shear bond strengths,[38] EDTA can improve,[45] impair,[34] or produce similar dentin bond strength values[29] to phosphoric acid etching depending on EDTA concentration and on the adhesive system used.[45] Lower dentin microtensile bond strength values were obtained when 24% EDTA gel replaced phosphoric acid etching prior to RelyX ARC luting. Since EDTA is a mild etchant, it may not sufficiently demineralize dentin as happens when lower pH etching agents such as phosphoric acid are used In addition, the chemical composition of adhesive systems is a determining factor influencing the ability to bond to EDTA etched dentin.[46] When acidic monomers capable of dentin demineralization, such as PENTA, which has a functional phosphoric acid group, are bonded to EDTA etched dentin, higher dentin bond strengths can be obtained.[45] However, the water-based adhesive system composed by HEMA, Bis-GMA, and polyalkenoic acid copolymers was not able to produce comparable bond strength values when dentin was etched conventionally with phosphoric acid or 24% EDTA gel for 60 seconds. Another relevant aspect that must be considered is EDTA delivery form. Even at a higher concentration, a 24% EDTA gel might not be able to etch dentin in the same manner as EDTA in aqueous solution due to its lower wetting capacity. This might explain why a lower concentration of EDTA produced equivalent microtensile bond strength to phosphoric acid etching when a one-step total etch adhesive with similar monomer composition was bonded to EDTA etched dentin.[29]

CONCLUSIONS

Based on the results and taking into consideration the limitations of this *in vitro* study, the following conclusions were drawn:

1. Application of 2% CHX does not compromise immediate microtensile dentin bond strength of self-adhesive resin cements.

2. Conventional resin cements bonded to phosphoric acid etched dentin produce higher bond strength compared to self-adhesive resin cements.
3. A 24% EDTA gel is a useful alternative to increase dentin bond strength of self-adhesive resin cements.
4. Dentin pretreatment with 24% EDTA gel for 60 seconds does not substitute for conventional phosphoric acid etching when resin cements are used along with HEMA/Bis-GMA adhesive systems.

Conflict of Interest Declaration

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

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Reliability of Fiber Post Bonding to Root Canal Dentin After Simulated Clinical Function *In Vitro*

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Clinical Relevance

The present data generated *in vitro* suggests that retention of fiber posts may be reduced after clinical function and that the adhesive interface inside the root canal undergoes certain degradation processes. Therefore, endodontically treated teeth restored using fiber posts may benefit from additional reinforcement via coronal restoration using adequate ferrules and/or adhesive techniques.

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SUMMARY

The aim of this study was to investigate the effect of thermomechanical loading (TML) on the bond strength of fiber posts luted with three different resin cements.

Sixty-six extracted human anterior teeth were endodontically treated and restored with fiber posts (RelyX Fiber Posts, 3M ESPE) using three commercially available resin cements and three corresponding core build-up materials (n=22 each): Panavia F 2.0/Clearfil DC Core Automix (Kuraray), Variolink II/Multicore Flow (Ivoclar Vivadent), and RelyX Unicem/ Filtek Z250 (3M ESPE). Twelve specimens of each group received all-ceramic crowns and were subjected to TML. The other 10 specimens were stored in saline solution for 24 hours. The roots were sectioned and bond strength was measured using a push-out test. Adhesive interfaces of two specimens of each group subjected to TML were analyzed using field emission scanning electron microscopy (FESEM).

Bond strengths of fiber posts were significantly affected by the type of resin cement ($p<0.0005$) and TML ($p<0.0005$; two-way analysis of variance). TML significantly reduced bond strengths for all materials ((6.0 (6.2) MPa)) compared with initial bond strengths ((14.9 (10.4) MPa)). RelyX Unicem resulted in significantly higher bond strengths before ((18.3 (10.3) MPa)) and after TML ((9.8 (7.5) MPa)) compared with the other materials ($p<0.0005$; Tukey HSD). Using FESEM, Variolink II and Panavia F demonstrated a hybrid layer partly detached from the underlying resin cement, whereas no hybrid layer was observed for RelyX Unicem.

The decrease in bond strength after TML suggests that retention of fiber posts may be reduced after clinical function. Therefore, endodontically treated teeth that are restored using fiber posts may benefit from additional reinforcement via coronal restorations using adequate ferrules and/or adhesive techniques.

INTRODUCTION

The restoration of endodontically treated teeth using adhesively luted fiber-reinforced composite (FRC) posts is routinely performed in dental practice. Although clinical studies revealed promising results for using fiber posts,[1,2] bonding to root canal dentin is still a challenge because of limited access and visibility, reduced number of dentinal tubules in the apical third of the root, and deposition of cementum and secondary dentin.[3] In addition, the configuration factor, or C-factor, inside the root canal has been shown to be extremely high,[4] and the polymerization of light- or dual-curing resin cements might be hampered due to the varying degree of the light-transmitting ability of translucent fiber posts.[5] The large variety of products on the market for luting fiber posts, along with the intrinsic difficulties of bonding inside the root canal, complicate the selection of a luting strategy that may provide long-lasting bonding to root dentin. Consequently, it is important to investigate the stability of the adhesive interface of fiber posts inside the root canal to gain information about the long-term performance of adhesively luted FRC posts.

Self-adhesive resin cements are easy to handle and provide a time-saving procedure as no etching and bonding steps are required. *In vitro* investigations on the bonding behavior of the self-adhesive resin cement RelyX Unicem (3M ESPE, Seefeld, Germany) have resulted in contradictory findings.[6–11] Another approach is the use of a separate self-etching or an etch-and-rinse adhesive prior to inserting the resin cement inside the root canal. It has been speculated that the use of phosphoric acid inside the root canal might be advantageous with respect to dissolving the thick smear layer.[12] On the other hand, a universal occurrence of interfacial gaps inside the root canal along the hybrid layer surface of etch-and-rinse adhesives was observed using scanning electron microscopy (SEM).[13]

Little is known about the long-term bonding behavior of various luting agents based on different bonding strategies inside the root canal.[14–16] One study indicated an increase of debonding at the tooth restoration margins of various post and core restorations due to mechanical loading[17]; these findings were corroborated by another investigation that revealed a significant reduction of bond integrity after three months of water storage inside the root canal.[18] Conversely, no effects of mechanical cycling on bond strength of fiber posts to root dentin could be detected, although this study revealed effects of mechanical cycling on bond strength of zirconia posts, indicating that post rigidity might influence the effects of mechanical loading.[15] Consequently, the aim of the present study was to investigate the effect of thermomechanical loading (TML) on bond strength of fiber posts luted with a self-adhesive resin cement, a one-step self-etching adhesive, and a two-step etch-and-rinse system. The null hypothesis to be tested was that bond strengths of fiber posts are not affected by TML or type of luting cement system.

METHODS AND MATERIALS

Specimen Preparation

The crowns of 66 extracted human upper central anterior teeth were sectioned at the proximal cementoenamel junction using a diamond blade under constant water cooling. Root canal preparation was performed at a working length of −1 mm from the apical foramen using FlexMaster rotary instruments (VDW, Munich, Germany) with a crown-down technique. Apical enlargement was performed to size 0.02/50, and the teeth were filled by means of cold lateral condensation using gutta-percha points (VDW) and AH Plus (Dentsply DeTrey, Konstanz, Germany) as a sealer and stored in water for 24 hours.

The specimens were randomly divided into three groups of 22 teeth each. The root canals were enlarged with a slow-speed drill provided by the manufacturer of the selected post system (RelyX Fiber Post Size 2, 3M ESPE). The depth of the post

Table 1: *Composition of Materials Used*

Luting Agent (Lot No.)	Bonding Agent (Lot No.)	Core Build-up Material/ Adhesive (Lot No.)	Manufacturer	Composition of Composite Resins	Composition of Primers	Composition of Core Build-up/ Adhesive	Irrigation After Post Space Preparation	Application of Resin Cement
Panavia F 2.0 (41173)	Ed Primer (41173)	Clearfil DC Core Automix (039AA) Ed Primer (41173)	Kuraray, Osaka, Japan	Barium glass powder, sodium fluoride, dimethacrylate, 10-MDP, silica, benzoyl peroxide, amine, sodium aromatic sulfinate	10-MDP, HEMA, N-methacryl 5-aminosalcylic, sodium benzene sulfinate, N,N′diethanol p-toluidine, water	Silanated glass and silica, Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, dl-camphorquinone, benzoylperoxide	CHX 0.2%	Cement onto the post surface and into the orifice of the canal
Variolink II (base: K09191, catalyst: K0511)	Excite DSC (H9851)	Multicore Flow (I22087) AdheSE (K0319)	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, UDMA, TEGDMA, ytterbium trifluoride, barium glass, silica	HEMA, Bis-GMA, dimethacrylate, phosphonic acid acrylate, silica, ethanol, catalysts, stabilizers	**Multicore:** dimethacrylate, barium glass, fillers, Ba-Al-fluorosilicate glass, silicon dioxide, ytterbium trifluoride, catalysts, stabilizer, pigments **AdheSE:** phosphonic acid acrylate, Bis-acrylamide, water, initiators, stabilizer, dimetha-crylates, HEMA, silicon dioxide, initiators, stabilizer	CHX 0.2%	Cement onto the post surface and into the orifice of the canal
RelyX Unicem Aplicap (290958)		Filtek Z250 (7XN) Adper Prompt L Pop (292918)	3M ESPE, Seefeld, Germany	Glass powder, calcium hydroxide, methacrylated phosphoric ester, dimethacrylate, initiators	No primer	**Filtek Z250:** zirconia and silica fillers, Bis-GMA, UDMA, Bis-EMA **Adper Prompt LPop:** Methacrylic phosphates, Bis-GMA, photoinitiator camphor-quinone, water, HEMA, polyalkenoic acid polymer	NaOCl 1%, H_2O	Cement was applied into the canal using the provided elongation tip

Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogenphosphate; BIS-EMA, ethoxylated bisphenol A glycol dimethacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; CHX, chlorhexidine digluconate; HEMA, 2-hydroxyethyl methacrylate; NaOCl, sodium hypochlorite; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

space preparation was 8 mm. Irrigation was performed after post space preparation according to the manufacturer's recommendations (Table 1).

RelyX Fiber Posts (3M ESPE) were tried in and inserted with one of the following three different resin cements according to the manufacturer's instructions: 1) Panavia F 2.0 (Kuraray, Osaka, Japan), 2) Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein), and 3) RelyX Unicem (3M ESPE). The posts were inserted into the canal, excess was removed, and light curing was performed using an LED curing unit (1200 mW/cm^2; Elipar Freelight 2, 3M ESPE) according to the manufacturer's recommendations. Light intensity of the light-curing unit was checked prior to use (LED radiometer, Demetron, Kerr, Orange, CA, USA). The core build-ups were conducted using the corresponding core build-up materials listed in Table 1. Twelve teeth of each group received preparations for all-ceramic crowns, including a circumferential 1.2-mm shoulder and a ferrule of 2 mm. With the help of silicone impressions of the original crowns, 36 lithium-disilicate ceramic crowns were fabricated (IPS e.max Press, Ivoclar Vivadent) and adhesively luted using the same materials used for post luting. Light curing was performed for 40 seconds from each surface (Elipar Freelight 2, 3M ESPE). After seven days of storage in distilled water at 37°C, the specimens were subjected to thermomechanical fatigue including 5000 thermal cycles (5°C/55°C, two minutes each cycle) and 1.2×10^6 mastication cycles at an angle of 135°. A force of 50 N was applied 3 mm below the incisal edge on the palatal surface of the crown (EGO chewing simulator, EGO Kältetechnik GmbH, Regensburg, Germany). After thermomechanical fatigue, the specimens were stored again for 21 days in distilled water at 37°C. The other 10 teeth of each group were stored in 100% humidity without crowns for 24 hours to allow for complete polymerization.

Push-Out Testing

The roots were sectioned perpendicular to the long axis of the root into four slices (thickness of 1 mm) using a band saw (Exakt Apparatebau, Norderstedt, Germany), and for 10 specimens per group, micro push-out testing was performed (Universal testing machine, Zwick, Roell, Ulm, Germany) at a crosshead speed of 0.5 mm/min. With regard to the tapered design of the post, three different sizes of punch pins as well as three different openings were used for the push-out testing.[6] The maximum stress was calculated from the recorded peak load divided by the computed surface. To calculate the exact bonding surface, the tapered design of the posts with regard to the respective part of the post was considered. Therefore, each specimen was measured with a micrometer screw (Mitutoyo Messgeräte GmbH, Neuss, Germany), and the bonding surface was calculated using the formula of a conical frustrum: $\pi(R_1 + R_2)\sqrt{(R_1 + {}^{11}R_2)^{22}2 + h^2}$. After the push-out test, each specimen was observed using a stereomicroscope (DV 4, Zeiss, Jena, Germany) at 40× magnification to determine the failure mode. The specimens were divided into four groups according to the failure modes: 1) adhesive failures between dentin and cement, 2) adhesive failures between post and cement, 3) mixed failures, and 4) cohesive failures inside the post.

SEM Analysis

The slices of two sectioned specimens per group that were subjected to TML were fixated in 2.5% glutaraldehyde at pH 7.4 for 12 hours at 4°C,[19] dehydrated in ascending concentrations of ethanol, and polished. Subsequently, the interfaces were treated with 6 N hydrochloric acid for 30 seconds followed by a 10-minute immersion in 2.5% sodium hypochlorite and coated with Au-Pd (DV 502A Vacuum Evaporator), and secondary images were obtained at 5 kV using a field emission scanning electron microscope (FESEM; S-4700, Hitachi High Technologies America Inc, Pleasanton, CA, USA).

Statistical Analysis

Statistical analysis was performed using SPSS version 16.0 software (SPSS, Chicago, IL, USA). The alpha (Type I) error level was set to 0.05. To achieve normality in the study groups, bond strengths were subjected to a suitable Box-Cox transformation before analysis. Then the effects of resin cement and TML on bond strength were analyzed using two-way analysis of variance (ANOVA) and Tukey HSD post hoc test. The effects of the materials on the failure modes were investigated using Pearson's chi-square test.

RESULTS

Bond strengths of fiber posts were significantly affected by the type of resin cement ($p<0.0005$) and TML ($p<0.0005$; two-way ANOVA). TML significantly reduced bond strengths for all materials ((6.0 (6.2) MPa)) compared with initial bond strength values ((14.9 (10.4) MPa)). RelyX Unicem revealed significantly higher bond strengths before ((18.3 (10.3) MPa)) and after TML ((9.8 (7.5) MPa)) compared with Panavia F before ((13.2 (9.5) MPa))

Table 2: *Analyses of the Failure Modes (in Percentages) in Each Group*[a]

Resin Cement	Thermomechanical Loading	Failure Mode, %					*p* Value
		I Adhesive Cement Dentin	II Adhesive Post Cement	III Mixed	IV Cohesive Post	Not to Assess	
RelyX Unicem	Initial	20	78	2	0	0	$p<0.0005$
	TML	58.5	19.5	19.5	0	2.5	
Panavia F 2.0	Initial	28	68	4	0	0	$p=0.005$
	TML	43.9	36.6	19.5	0	0	
Variolink II	Initial	72	20	8	0	0	p=0.148
	TML	65.1	25,6	9.3	0	0	

[a] *$p<0.05$ indicates significant effects of thermomechanical loading (TML) on the failure modes (Pearson*).

and after TML ((3.5 (2.8) MPa)). Rely X Unicem also resulted in significantly higher bond strengths than Variolink II before ((13.2 (10.6) MPa)) and after TML ((4.8 (5.5) MPa)); ($p<0.0005$; Tukey HSD).

Analyses of failure modes with respect to the resin cement and TML are presented in Table 2. TML significantly affected the failure modes of the materials RelyX Unicem ($p<0.0005$) and Panavia F 2.0 ($p=0.005$) but not the failure modes of Variolink II ($p=0.148$; Pearson's chi-square test).

Representative images for Variolink revealed a distinctive hybrid layer and numerous resin tags (Figure 1a). Higher magnification revealed a detached hybrid layer from the underlying resin cement (Figure 1b). For the self-etching primer, ED Primer/Panavia F 2.0, a narrow hybrid layer was observed (Figure 1c,d); however, parts of the hybrid layer were also detached from the underlying resin cement. The self-adhesive resin cement RelyX Unicem showed no hybrid layer formation under the magnification used in this study, and scarce penetration into dentinal tubules could be observed (Figure 1e,f).

DISCUSSION

The null hypothesis of the present study had to be rejected since the resin cement and TML significantly affected bond strengths of fiber posts inside the root canal.

The resin cements of the present study were selected according to different bonding strategies and were all dual-cure resin cements. Light polymerization of these cements leads to a higher conversion rate,[20] and this might affect their physical-mechanical properties.[21] However, a decrease in light intensity from the coronal to the apical aspect of translucent fiber posts has been reported, and this could affect the curing efficacy of resin composites in the depth of the root canal.[5] Consequently, the bond strengths of the luting agents used in the present study could have been affected by a possible insufficient degree of conversion of the luting agents. However, this assumption could not be supported by the results of the failure mode analysis, for which a low percentage of cohesive cement failures was observed. Consequently, this aspect should be evaluated in further studies.

The thin push-out test is considered as a valid method to analyze the bond strengths of fiber posts to root canal dentin.[22] Nevertheless, the exposure of the fiber post to the dislodging forces during the push-out test cannot be compared with functional forces during clinical service.[10] Moreover, the sectioning process, especially of the teeth subjected to TML, may induce artifacts that could influence the test results. In addition, remnants of root canal obturation materials left inside the root canal and accessory canals[23] could affect bond strengths of

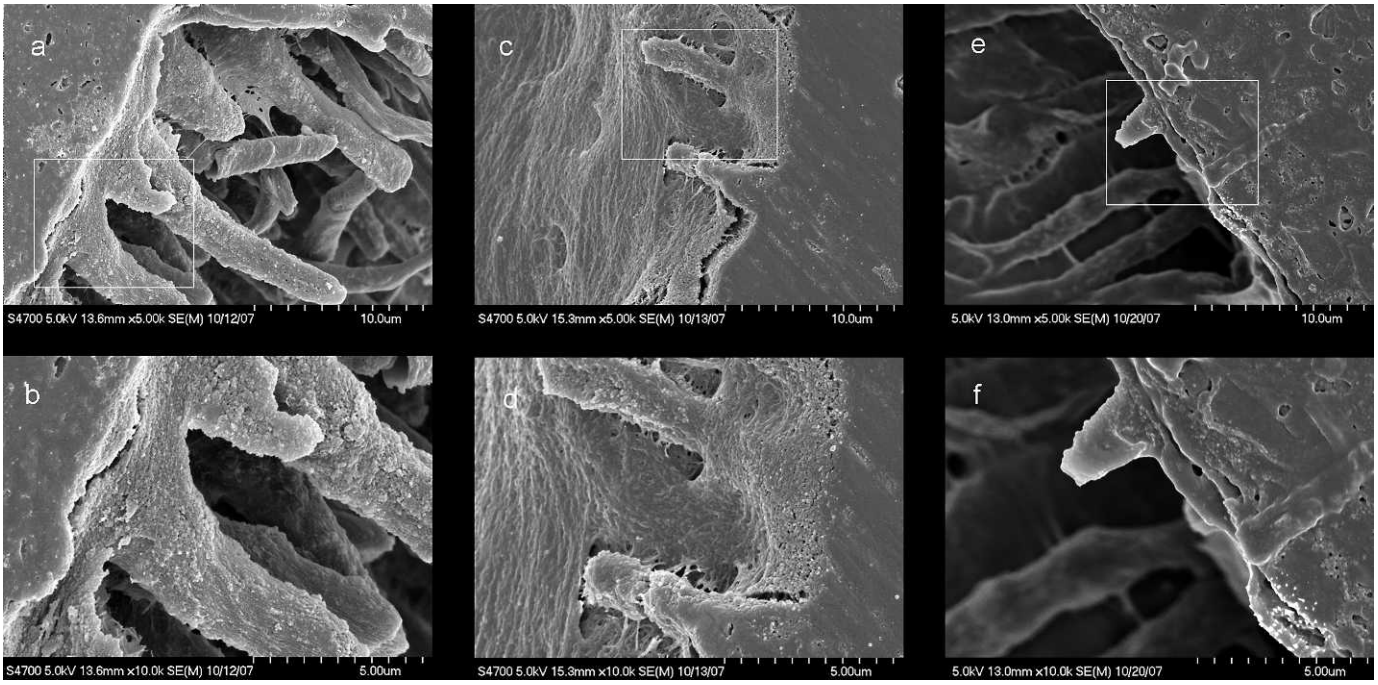

Figure 1. *(a): Representative images for Variolink revealed a distinctive hybrid layer and numerous resin tags. (b): Higher magnification depicted a detached hybrid layer from the underlying resin cement. (c, d): For the self-etching primer, ED Primer/Panavia F 2.0, a narrow hybrid layer could be detected; parts of the hybrid layer were also detached from the underlying resin cement. (e, f): The self-adhesive resin cement RelyX Unicem showed no hybrid layer formation with the magnification used in this study, and scarce penetration into dentinal tubules could be observed.*

fiber posts inside the canal. This would explain, at least partially, the high standard deviation that was observed in some groups.

The aim of the present study was to simulate and accelerate aging *in vitro* in a clinically relevant manner to assess the long-term behavior of adhesively luted fiber posts. Consequently, the specimens subjected to TML were restored using all-ceramic crowns to ensure a clinically relevant loading to mimic the clinical situation as closely as possible. In the control group, the initial push-out bond strength was measured after 24 hours. This would not allow enough time for the fabrication of crowns in this group. However, there might be a possibility of either mechanical disruption during preparation or stresses from crown cementation affecting the cement-post interface. These possible effects cannot be detected in the design used for this study.

A significant decrease was observed in bond strength after TML of all samples irrespective of the resin cement used. Changes in the bonded interface *in vivo* may be caused by flexure of the restored tooth under occlusal stresses. Because of water sorption at the resin-dentin interface, fluid movement may occur at the junction of adhesive resin, hybrid layer, and dentin during loading of the restoration. This could result in mechanical and chemical degradation of the cured resin.[24] The degree of degradation has been shown to be related to the chemical composition of the monomers, the degree of conversion, and the degree of cross-linking in the polymerized matrix.[25] Moreover, degradation of dentin-adhesive interfaces might be caused by discrepancies between the depth of acid demineralization and that of hydrophilic monomer infiltration. This may result in unprotected collagen fibrils at the bottom of the hybrid layer that can be hydrolyzed by the free water entrapped around the collagen fibrils.[26] It has also been demonstrated that the bottom of the hybrid layer formed by self-etching adhesives contains unprotected collagen fibrils.[27] The activity of bacteria-produced collagenases, as well as the activation of host-derived matrix metalloproteinases, has been shown to contribute to the degradation of the adhesive interface.[28,29] This could compromise the long-term bonding behavior of all investigated systems after TML.

A recent study also revealed a significant decrease in bond strength after thermocycling (40,000 cycles, representing approximately four years of functional service[30]) for the self-adhesive resin cement RelyX Unicem and the self-etch adhesive system Ed Primer/Panavia F 2.0, but not for the etch-and-rinse strategy XP Bond/CoreXFlow.[31] The initiator-cata-

lyst system in the self-cure activator of XP Bond might promote adhesion of compatible dual-curable resin-based luting agents to the adhesive layer and accelerate their polymerization.[32] However, RelyX Unicem also revealed significantly higher bond strength values after thermocycling compared with Panavia F 2.0 as observed in the present study.[31]

The results of the present study corroborate those of a previous investigation that demonstrated distinctive nanoleakage patterns for the etch-and-rinse system Excite DSC/Variolink II and the self-etch adhesive Ed Primer/Panavia F 2.0 up to 0.8 mm inside the root canal after TML, whereas the self-adhesive resin cement RelyX Unicem was able to prevent distinctive leakage at this penetration depth.[33] In that study, none of the luting systems was able to hermetically seal the root canal if leakage occurred around the margins of the coronal restoration, which correlates well with the observed decrease in bond strengths for all materials in the present study. Conversely, Bottino and others reported no effect on the push-out bond strength of adhesively luted FRC posts after 2,000,000 cycles of mechanical loading compared with an unloaded control group.[15] This loading protocol was conducted without thermocycling, which is known to challenge the adhesive interface both chemically (hot water accelerates hydrolysis and elution of the interface components) and mechanically (repetitive contraction and expansion stresses), besides enhancing the effect of temperature and water-mediated aging phenomena.[34]

In the present study, the self-adhesive resin cement RelyX Unicem showed higher bond strengths compared with the other materials investigated. The results are in the same line as previous investigations.[6,7,35] Another study also found significantly higher push-out bond strengths of RelyX Unicem compared with Panavia F 2.0.[10] In contrast, other studies demonstrated either a similar bond strength for the self-adhesive resin cement compared with a self-etch approach or an etch-and rinse approach[11,31] or even lower bond strength values for the self-adhesive resin cement compared with a self-etch approach or an etch-and-rinse adhesive system.[8] In the last mentioned study, RelyX Unicem was used in the self-cure mode. The low degree of conversion of this cement in the self-cure mode[36] may have contributed to the lower bond strength values.

The bonding mechanism of RelyX Unicem differs from that of self-etching adhesives since no distinct demineralization and hybridization were observed upon Transmission Electron Microscopy morphological interface examination.[8] In addition, RelyX Unicem resulted in a shallow demineralization despite its low initial pH.[37] This is in agreement with the present FESEM analysis since no hybrid layer formation was observed under the magnification used and scarce penetration of the cement into dentinal tubules was observed (Figure 1e,f). The use of the self-adhesive resin cement RelyX Unicem with the RelyX Fiber Post could have contributed to the favorable bond strength of this cement in the present study since the manufacturer claims both chemical compatibility and strong micromechanical interlocking of this post-cement system. Despite the high bond strength of RelyX Unicem in the present study, the predominant failure mode of this cement was adhesive between post and cement before TML, indicating that the weak part of this interface was still between post and cement. For the luting agents RelyX Unicem and Panavia F 2.0, the predominant failure mode changed significantly after TML from adhesive between post and cement into adhesive between dentin and cement. This change corroborates the respective reduction in bond strengths and the potential degradation of the resin-dentin interface.

All materials were applied into the root canal according to the manufacturers' recommendations. RelyX Unicem was the only material that was inserted using a flexible root canal–shaped application tip, whereas all other materials were applied onto the post surface as well as into the orifice of the canal prior to post insertion. Although the direct application of resin cements with a syringe has been shown not to affect the bond strengths to root dentin of etch-and-rinse resin cements,[38] it could have contributed to the favorable results of RelyX Unicem, since the use of this application aid reduced the number of imperfections within the self-adhesive cement interface compared with the conventional application technique.[39]

The self-etching ED Primer of Panavia F 2.0 is a one-step adhesive. These adhesives have been described as behaving like semipermeable membranes after polymerization because of their higher concentration of hydrophilic monomers and the lack of the subsequent application of a more hydrophobic resin coating.[40] Water sorption by hydrophilic resin monomers within resin-dentin interfaces could contribute to their degradation over time[26] and affect the bond durability of one-step self-etching adhesives. Hydrophilicity and hydrolytic stability are antagonistic properties,[41] and this could explain the reduction of bond strength after TML for this cement

despite the ability of functional monomers, ie, 10-methacryloxydecyl dihydrogen phosphate, present in ED Primer that may interact with hydroxyapatite to form calcium salts and contribute to a stable bond.[42]

The strong etching effect of phosphoric acid totally removes mineral crystals and exposes the collagen fibers.[43] The subsequently applied adhesive must infiltrate around these fibers. Porosities within an incompletely infiltrated hybrid layer, any discrepancy between the etching depth and the following resin infiltration depth, as well as technique sensitivity could have contributed to the low bond strength values of Excite DSC/Variolink II in the present study after TML. Nevertheless, this was the only etch-and-rinse cement evaluated in this study; thus, the current findings should not be generalized to all etch-and-rinse cements.

CONCLUSIONS

The decrease in bond strength after TML suggests that retention of fiber posts inside the root canal may be reduced after clinical function. Therefore, endodontically treated teeth that are restored using fiber posts may benefit from additional reinforcement via coronal restorations using adequate ferrules and/or adhesive techniques.

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Contrast Ratio and Masking Ability of Three Ceramic Veneering Materials

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Clinical Relevance

Masking severely discolored dentitions is one of the indications for the use of ceramic veneers. IPS e.max Press and Vita VM7 had significantly higher contrast ratios and masking abilities than Nobel Rondo Press Alumina: Solo. However, none of the materials tested was able to completely mask the black background.

SUMMARY

Statement of the Problem: Porcelain veneer materials are translucent and are therefore affected by their thickness as well as the color of the underlying substructure, which limits their masking ability and compromises the esthetic result in heavily stained teeth.

Purpose: The purpose of this study was to compare the contrast ratio (CR) and masking ability of three different veneering ceramics with two thicknesses by measuring the color differences over white and black backgrounds. Correlations between CR and masking ability of these veneering ceramics were evaluated.

Methods and Materials: A total of 30 disc-shaped specimens (12 mm diameter × 1.0 mm or 1.5 mm) were fabricated in shade A2 from three types of all-ceramic systems: IPS e.max Press (IPSe; Ivoclar Vivadent, Schaan, Liechtensein), Vita VM7 (VM7; VITA Zahnfabrik, Bad Säckingen, Germany), and Nobel Rondo Press Alumina: Solo (NRPA; Nobel Biocare, Zürich-Flughafen, Switzerland). The CR, defined as the ratio of illuminance (Y) of the test material when placed on the black background (Yb) to the illuminance of the same material when placed over a white background (Yw), was determined (CR=Yb/Yw). The color (CIE L*a*b*) and Y of each specimen were measured over standard white and black tiles using a spectrophotometer (ColorEye 7000 A, Model C6, GretagMacbeth, New Windsor, NY, USA). Masking abilities of the specimens were determined by measuring the color difference (ΔE) over white and black backgrounds. Both CR and ΔE data were analyzed using two-way analysis of variance (ANOVA). One-way ANOVA was used to compare the mean values of CR across the three materials followed by the Duncan multiple comparison test. The correlations between CR and ΔE were determined by comparing R^2 values obtained from a linear regression analysis. A Student *t*-test for inde-

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pendent samples was used to compare the mean contrast ratio and ΔE values for the two thicknesses.

Results: CR values of NRPA were significantly less than those of IPSe and VM7, and the CR of IPSe was higher than that of VM7. Furthermore, CR increased as the thickness of the discs increased to 1.5 mm for all three materials. Mean ΔE values were significantly higher with 1.0-mm-thick discs than with 1.5-mm discs. Among the three materials it was observed that NRPA had the highest ΔE when compared with IPSe or VM7, whereas the ΔE of the latter two were not significantly different from one another. There was a strong linear correlation between CR and masking ability.

Conclusion: CR and masking ability are affected by the type as well as the thickness of the ceramic used. IPSe and VM7 are similar in their masking abilities, whereas NRPA had the lowest masking ability. NRPA was the most translucent, followed by VM7; IPSe was the most opaque. None of the materials tested was able to completely mask the black background. It is therefore recommended that the type of ceramic should be chosen according to each clinical situation.

INTRODUCTION

Over the past decade ceramic restorations have become increasingly popular despite some of their shortcomings, including brittleness, catastrophic failure, and wearing of opposing teeth. Their popularity is attributed to their superior esthetic properties, biocompatibility, and longevity.[1–3] Porcelain veneers are perceived to be one of the most conservative means of restoring unesthetic anterior teeth. Their indications include discoloration, tetracycline staining, fluorosis, diastema closure, and malformed and malpositioned teeth.[4–7] Veneers go as far back as the late 1930s, where they were temporarily used by actors during filming; at that time adhesive systems did not exist, and therefore long-term retention was not possible.[8] Adhesive retention of veneers was later demonstrated by Calamia, Simonsen, and Horn in the early 1980s by using hydrofluoric acid etching in combination with silane coupling agents.[5,6,9]

The final color of the veneer depends on three chief elements and their interaction with one another. These elements are the color of the tooth/substructure, the thickness and type of ceramic material used, and the resin cement selected. Combining the three is the means by which an optimal esthetic outcome can be realized.[10–13]

The evidence within the literature indicates that masking of heavy stains such as fluorosis and tetracycline discoloration requires the use of opaquers, opaque luting cements, or tints.[14–19] Another approach is the use of opaque cores, including alumina or zirconia substructure.[15,19–21] When that is done, the resulting veneer becomes opaque and lifeless and esthetics is compromised.[15,20]

The other possible solution to the problem of heavily stained teeth is to increase the thickness of the veneer from the standard 0.75-mm thickness to 1.0–1.5 mm because it appears that the thickness of the material influences its opacity, which in turn may increase its masking ability. Further reduction of the labial and interproximal surfaces may provide additional space for the veneer, which allows more leeway for color correction,[17,20,22] but this might result in dentine exposure and consequent sensitivity after cementation. However, it was observed that within two weeks sensitivity disappeared.[17]

Contrast ratio (CR) is one of the methods used to compare opacity of all ceramic systems. Differential colorimetric assessment of the ceramic materials on white and black backgrounds may be used to measure the relative opacity of dental porcelain. CR can be computed from the Y*xy* color space system measurements as the ratio of reflectance (Yb/Yw) when the specimen is placed on a black tile (Yb) relative to that obtained when the specimen is placed on a white tile (Yw). The values of the hue (x), chroma (y), and luminous intensity (Y) can be obtained from spectrophotometric measurements as well as colorimetric measurements.[23–25]

Chu and others[25] compared the masking ability and CRs of 0.7-mm-thick ceramic veneers and found that Vitadur Alpha had the lowest CR and poorest masking ability as compared with Procera and Empress II. In addition, they concluded that the use of both Procera and Empress II as veneering materials may be limited when heavily discolored teeth are involved because they were not fully capable of masking a black background.

The effect of porcelain opacity on the final shade of 0.7-mm-thick veneers cemented to dark substrates was evaluated by Davis and others.[26,27] Their results showed that the veneers provided a masking effect; however, the resultant color of the veneer-substrate system will not be that of the porcelain or the substrate. Furthermore, the translucency of the

porcelain is as influential to the final result as the color of the substrate. Similar results were reached by Yaman and others[28] when researching the effect of adding opaque porcelain on the final color of porcelain veneers.

Vichi and others[29] studied the effect of different opaque posts on the masking ability of IPS Empress leucite-reinforced ceramic of various thickness (1.0, 1.5, or 2.0 mm) and noted that the ceramic restoration was not affected by the different substructures when its thickness was 2.0 mm.

CRs of six different core ceramics (ie, IPS Empress, IPS Empress II, In-Ceram Alumina, In-Ceram Spinell, In-Ceram Zirconia, and Procera All-Ceram) were compared by Heffernan and others (2002). The authors ranked these materials' CRs in order of decreasing translucency as follows: In-Ceram Spinell > Empress, Procera, Empress II > In-Ceram Alumina > In-Ceram Zirconia.[24] In part II of that study, the authors compared the translucency of these ceramic systems when veneered with their respective porcelains and after glazing. Significant differences in CR were found among these ceramic systems, and a range of translucency was established for the veneered all-ceramic systems. Such variety may ultimately affect the ability of the ceramics to match the natural tooth. In addition, the glazing cycle resulted in decreased opacity for all materials tested, with the exception of the opaque In-Ceram Zirconia and metal-ceramic specimens.[30]

The purpose of the present study was to compare the CR (opacity) and masking ability of three different veneering ceramics: IPS e.max Press (IPSe; Ivoclar Vivadent, Schaan, Liechtensein), Vita VM7 (VM7; VITA Zahnfabrik, Bad Säckingen, Germany), and Nobel Rondo Press Alumina: Solo (NRPA; Nobel Biocare, Zürich-Flughafen, Switzerland) with two thicknesses (1.0 and 1.5 mm) by measuring the color differences over white and black backgrounds. In addition, this study tested the correlation between the CR and the masking ability of these veneering ceramics.

The null hypothesis of this study was that there would be no significant difference in the masking ability and CR of the ceramics tested and between the different thicknesses of these materials. In addition, there would be no correlation between the CR and the masking ability of the three materials.

MATERIALS AND METHODS

A total of 30 disc-shaped specimens were fabricated in shade A2 from three types of all-ceramic systems: IPSe (Ivoclar Vivadent), VM7 (VITA Zahnfabrik), and NRPA (Nobel Rondo). The discs were 12.0 mm in diameter and 1.0 mm or 1.5 mm thick. All discs were constructed using stainless steel molds that were custom made to meet the desired diameter and thickness (Figure 2).

IPSe offers lithium disilicate glass ceramic ingots for the press technique. These ingots have been developed on the basis of a lithium silicate glass ceramic. Five 1.0 mm- and five 1.5-mm-thick discs were fabricated as recommended by the manufacturer using lost wax and heat-pressed techniques. One firing cycle at 700°C was accomplished in a calibrated furnace (EP 600, Ivoclar Vivadent). Later, the discs were immersed in IPSe Invex liquid (<1% hydrofluoric acid, Ivoclar Vivadent) and cleaned in an ultrasonic cleaner (NEY, Dentsply International, York, PA, USA), then subjected to airborne-particle abrasion using 50 μm aluminum oxide powder at 2 bar pressure (BEGO, ZiroDent Dentalhandel GbR, Cologne, Germany). A staining technique was used for the IPSe. Finally, etching of the discs was done using IPS etching gel for 20 seconds.

A layering technique was used in the fabrication of the 1.0- and 1.5-mm-thick VM7 discs, where every 2.1 g of powder were mixed with 1 mL of liquid. One dentin and two enamel ceramic layers were fired at 200°C-910°C in three firing cycles. The discs were etched with 10% hydrofluoric acid for 90 seconds.

NRPA was provided as pellets or ingots with 1 cm diameter and 9 mm thickness. Discs of 1.0- and 1.5-mm thickness were fabricated using lost wax and pressing techniques with two firing cycles at 890°C-900°C (EP 600, Ivoclar Vivadent). Etching with 5%

Figure 1. *Ceramic disk 1.5 mm thick and 12 mm in diameter.*

Figure 2. *Custom made stainless steel mold.*

hydrofluoric acid for four minutes was done to produce a rough surface for adhesive bonding.

All laboratory procedures were carried out to duplicate regular laboratory procedures of finishing, glazing, and etching. The specimens were finished using 400-grit waterproof silicon carbide abrasive papers under running water until the desired thickness was confirmed with a digital caliper (Model 193-111, Mitutoyo Mfg Co, Kawasaki, Japan). Afterward all specimens were cleaned in an ultrasonic bath (Ultrasound Vita-Sonic II, Vita Zahnfabrik, Germany) for five minutes and dried before spectrophotometric measurements were taken.

For each of the ceramic discs, three measurements were made in three different locations around the center of the disc. Therefore, the number of measurements used for statistical analysis was 15 (n=15) for each thickness investigated.

A spectrophotometer (ColorEye 7000 A, Model C6, GretagMacbeth, New Windsor, NY, USA) was used with an aperture of 0.12 × 0.31 inches, alongside ProPallette Gold Color Matching software version 3.1 (GretagMacbeth). This instrument measures the spectral reflectance of a color and converts it into a tristimulus value; it has a spectral range of 360 to 750 nm. The spectrophotometer CIE L*a*b* output is based on D65 illuminant. In CIE L*a*b* colorimetry, the color of an object is defined in a three-dimensional color space expressed in three coordinates: L* represents brightness (white-black), a* is for redness-greenness, and b* is for yellowness-blueness. The illuminance (Y) and color (CIE L*a*b*) of each specimen were measured over standard white and black tiles. The average values of the three measurements were taken. The instrument was calibrated using the white ceramic calibration tile and the zero calibration standard (black). As recommended by the manufacturer, calibration was done before measurements of each group (n=15) were taken or every eight hours. The Y value in Y*xy* color space represents the illuminance, where x is the value of hue and y is the value of chroma. The opacity of the specimen in terms of CR (CR=Yb/Yw) is defined as the ratio of illuminance of the test material when it is placed on the black background (Yb) to the illuminance of the same material when it is placed over a white background (Yw). Masking abilities of the specimens were determined by measuring the ΔE over white and black backgrounds.

The following equation was used:

$$\Delta E^* = [(L_1^* - L_0^*)^2 + (a_1^* - a_0^*)^2 + (b_1^* - b_0^*)^2]^{1/2}$$

L_1^*, a_1^*, b_1^* = Color of the specimens over the white background.

L_0^*, a_0^*, b_0^* = Color of the specimens over the black background.

Statistical Analysis

The data were analyzed using statistical software (SPSS, Version 16.0, SPSS Inc, Chicago, IL, USA). Differences among the CRs and masking abilities of the three materials with the two thicknesses were calculated using two-way analysis of variance (ANOVA). One-way ANOVA was used to compare the mean values of CR across the three materials followed by the Duncan multiple comparison test. The correlation between CR and masking ability was determined by comparing R^2 values obtained from a linear regression analysis. A Student t-test for independent samples was used to compare the mean CR and color difference values for the two thicknesses.

RESULTS

Contrast Ratio

Table 1 shows the mean percentages and corresponding standard deviations (SD) of the CR (opacity) of all ceramic specimens with the two different thicknesses as determined by the spectrophotometer. A Student t-test demonstrated that CR increased as the thickness of the discs increased to 1.5 mm for

Table 1. *The Results of Multiple Comparisons Test of Mean Contrast Ratio Percentages among and within Groups and the Student's t-test Comparison of the Mean Contrast Ratio for the Two Thicknesses*

Material	Thickness (mm)		t-test Sig
	1.0	1.5	
IPSe	0.78 (0.28) [a]	0.88 (0.12) [b]	.0001
NRPA	0.63 (0.42) [c]	0.71 (0.31) [d]	.0001
VM7	0.79 (0.09) [e]	0.85 (0.15) [f]	.0001

The mean difference is significant at the .05 level.
Different letters indicate significant differences.

all three materials, which indicates that opacity of these ceramic materials increases as their thicknesses increase.

It was observed that the CRs of NRPA were significantly less than those of IPSe and VM7 while the CR of IPSe was higher than that of VM7. This means NRPA was the least opaque, followed by VM7 while IPSe was the most opaque.

Two-way ANOVA showed that there was a significant material effect, thickness effect, and material-thickness interaction (p=0.0001), thereby rejecting the null hypothesis that stated there would be no significant difference in the CRs of the ceramics tested and between the different thicknesses of these materials (Table 2).

Masking Ability

Table 3 shows the mean values and corresponding SDs of the color parameters and masking ability of all ceramic specimens with the two different thicknesses as determined by the spectrophotometer. The results of the t-test for the two thicknesses are shown as well.

Table 2: *Two-way ANOVA Results for Comparison of Contrast Ratio Percentages*

	Sum of Square	df	Mean Square	F	Sig
Material	4521.235	2	2260.617	335.898	.0001
Thickness	1258.749	1	1258.749	187.033	.0001
Material x Thickness	167.903	2	83.951	12.474	.0001

Table 3: *Mean Values and SDs of ΔL*, Δa*, Δb* and ΔE (Masking Ability) for all Ceramic Specimens Corresponding to the Two Thicknesses*

Material	Color Coordinates	Ceramic thickness (mm)		t-test Sig
		1.0	1.5	
IPSe	ΔL*	5.24 (1.18)	2.4(0.49)	.0001
	Δa*	1.51 (0.11)	1.25 (0.15)	.0001
	Δb*	5.80(0.93)	3.46 (0.66)	.0001
	ΔE	8.03 (1.26)	4.42 (0.81)	.0001
NRPA	ΔL*	9.01 (3.31)	5.41 (1.52)	.0001
	Δa*	0.76 (0.10)	0.65 (0.07)	.002
	Δb*	5.37 (1.25)	3.86 (0.67)	.0001
	ΔE	10.88 (2.15)	6.81 (1.04)	.0001
VM7	ΔL*	4.96 (0.49)	3.65 (0.56)	.0001
	Δa*	1.68 (0.09)	1.61 (0.09)	.035
	Δb*	5.18 (0.36)	4.18 (0.46)	.0001
	ΔE	7.39 (0.5)	5.81 (0.65)	.0001

The mean difference is significant at the .05 level.

Generally, the ΔL*, Δa*, and Δb* values decreased as the thickness of the ceramic discs increased from 1.0 to 1.5 mm. For the IPSe and NRPA, the ΔL*, Δa*, and Δb* values were significantly lower when the thickness increased to 1.5 mm. VM7 demonstrated the same results with regard to the ΔL* and Δb* values, but Δa* did not show such a significant difference. With all the materials tested, it was observed that mean ΔE values were significantly higher in 1.0-mm-thick discs than in 1.5-mm discs. This indicates that the masking ability of the ceramic veneers increased as their thickness increased.

Among the three materials it was found that NRPA had significantly higher ΔL* values and lower

Table 4: *Multiple Comparisons of ΔL*, Δa*, Δb* and ΔE (Masking Ability) Among the Three Materials*

Color Coordinate	Material		Sig
ΔL*	**IPSe**	NRPA	.0001
		VM7	.243
	NRPA	IPSe	.0001
		VM7	.0001
	VM7	IPSe	.243
		NRPA	.0001
Δa*	**IPSe**	NRPA	.0001
		VM7	.0001
	NRPA	IPSe	.0001
		VM7	.0001
	VM7	IPSe	.0001
		NRPA	.0001
Δb*	**IPSe**	NRPA	.951
		VM7	.811
	NRPA	IPSe	.951
		VM7	.764
	VM7	IPSe	.811
		NRPA	.764
ΔE	**IPSe**	NRPA	.0001
		VM7	.227
	NRPA	IPSe	.0001
		VM7	.0001
	VM7	IPSe	.227
		NRPA	.0001

The mean difference is significant at the .05 level.

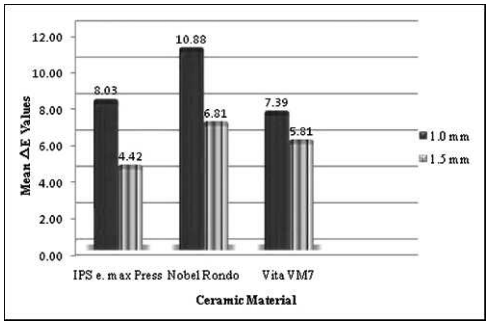

Figure 3. *Graph showing mean ΔE (masking ability) values for all ceramic specimens*

Δa* values (p=0.0001) when compared with IPSe and VM7. Also, VM7 demonstrated significantly higher Δa* values when compared with IPSe (p=0.0001), whereas ΔL* values were not significantly different between IPSe and VM7 (p=0.243). In addition, no significant differences were found in Δb* values among all three materials. It was observed that NRPA had significantly higher ΔE when compared with IPSe (p=0.0001) or VM7, whereas the ΔE of the latter two were not significantly different from one another (p=0.227; Table 4). In other words, the IPSe and VM7 are similar in their masking abilities, whereas NRPA had the lowest masking ability. Figure 3 shows the mean ΔE values of all ceramic specimens.

The results of the two-way ANOVA, presented in Table 5, showed a significant difference in the masking ability of the ceramics tested and between the different thicknesses of these materials; therefore, the null hypothesis was rejected. There was a significant material effect, thickness effect, and material-thickness interaction (p=0.0001).

When the thickness of the veneer was increased by 50% from 1 to 1.5 mm, VM7 exhibited a very small increase in its CR (4.7%) as compared with those

Table 5: *Two-way ANOVA Results for Comparisons of ΔE (Masking Ability) Values*

	Sum of Square	df	Mean Square	F	Sig
Material	120.976	2	60.488	42.22	.0001
Thickness	214.8	1	214.8	149.928	.0001
Material x Thickness	26.204	2	13.102	9.145	.0001

The mean difference is significant at the .05 level.

Table 6: *Percentages of Increase in Contrast Ratio Percentages and Decrease in ΔE Calues for all Ceramic Materials*

Ceramic Material	Percentages of Increase Contrast Ratio	Percentages of Reduction ΔE
IPSe	12.9	45
NRPA	13.9	37
VM7	4.7	21

exhibited by IPSe (12.9%) and NRPA (13.9%). On the other hand, ΔE values for IPSe showed the most significant reduction in ΔE values (45%), followed by NRPA (37%), whereas VM7 showed the least reduction (only 21%; Table 6).

Correlation

From the linear regression analysis it can be observed that there is a strong linear correlation between CR and masking ability (R=–0.80, p<0.0001, R^2=0.644; Figure 4). Furthermore, the Student t-test demonstrated that CR increased as the thickness of the discs increased to 1.5 mm for all three materials, which indicates that opacity of these ceramic materials increases as their thicknesses increase.

DISCUSSION

This *in vitro* study measured the masking ability and CRs of ceramic specimens prepared at different thicknesses. The hypothesis that there was no significant difference in the masking ability and CRs of the ceramics tested and between the different thicknesses of these materials was not supported by the results of this study. In addition, the hypothesis that there would be no correlation between the CR and the masking ability or the thickness of the three materials was also rejected.

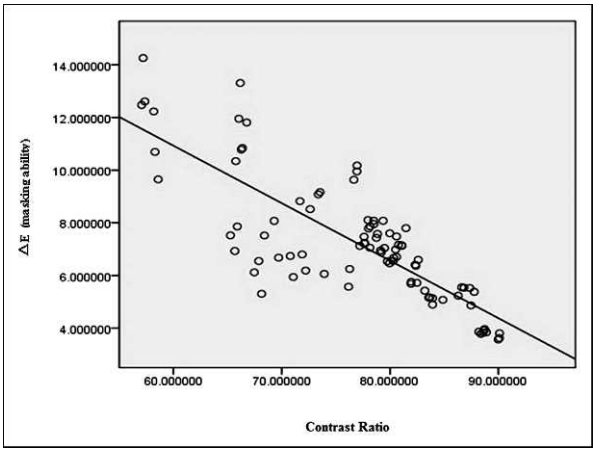

Figure 4. *Regression line for contrast ration (opacity) and ΔE (masking ability).*

The specimens used in this investigation had a thickness of 1.0 or 1.5 mm. It can be observed from the results that the L* a* b* values were affected by the thickness of the ceramic specimens. L* values decreased for all ceramic specimens as their thickness increased, indicating a decrease in brightness. As mentioned in the literature, this may be explained by the fact that more light is absorbed with thicker specimens and less is reflected; hence, lower L* values are recorded. Vichi and others[29] investigated the influence of ceramic and cement thickness on the masking ability of various types of opaque posts and concluded that the thickness of the ceramic was one of the dominant factors affecting the final color of the restoration. Another study demonstrated that the final shade of the porcelain can be influenced by small changes in the thickness of opaque and translucent porcelain layers.[31] The results of the present study are in agreement with two previous studies that have shown that as the ceramic thickness increased, L* values decreased for different types of all ceramic systems including leucite-reinforced ceramic (IPS Empress), a glass-infiltrated ceramic (In-Ceram Spinell), IPS e.max Press and zirconium oxide (DC-Zircon).[32,33]

All ceramic systems exhibited a decrease in a* and b* values with the increase in thickness. However, this decrease was not significant for VM7 with regard to a* values. These results suggest that the redness and yellowness of all specimens decreased as thickness of ceramic specimens increased. These results are not consistent with those of other studies where an increase in a* and b* values was observed with increased thickness of ceramic disks.[31–34] This diversity in results may be attributed to the difference in sample fabrication. The majority of the studies assembled the ceramic samples using a core and veneer combination, varying the thickness of each, whereas this study used only veneer ceramics in the fabrication of the samples.

For NRPA specimens the L* color values were found to be significantly higher and a* color values significantly lower than for IPSe and VM7. This suggests that NRPA is considerably lighter, even more translucent than the other two materials

tested. The a* color values measured for VM7 specimens were greater than those obtained for IPSe and NRPA specimens. These findings indicate that VM7 ceramics have more redness, ie, greater warmth among the three materials evaluated in this study. All materials exhibited the same degree of yellowness because no significant difference was found in b* values. This comes as no surprise given that a shade of A2 was used for all specimens, and thus all ceramic samples will naturally exhibit the same degree of yellowness.

An increase in thickness has resulted in lower ΔE values for all ceramic specimens, which implies an increased masking ability with 1.5-mm-thick discs. These results are in agreement with those reached by Vichi and others[29] where ΔE values decreased as ceramic thickness increased from 1.0 to 1.5 mm. However, the same author has also observed that as ceramic thickness increased to 2.0 mm, no significant difference could be found with regard to the ΔE values. Similarly, Hilgert and others[35] found that increasing thickness of ceramic veneers from 0.4 to 0.7 and 1.0 mm resulted in lower ΔE values. Contrary to the previous studies Shokry and others[32] demonstrated that ΔE values increased as ceramic thickness increased. Ozturk and others[33] concluded that an increase in ceramic thickness, core (1.0 mm) and veneer (0.5, 1, or 1.5 mm), led to an increase in ΔE values for both types of all-ceramic specimens tested (IPS e.max Press and DC-Zirkon). This may be explained by the fact that the latter-mentioned study evaluated the effects of various ceramic thicknesses in combination with repeated firings on the color of ceramics. Repeated firings may have contributed to the significant color changes because several studies have suggested that certain metal oxides are not color stable during firing. Furthermore, color changes of surface colorants after firing have exhibited pigment breakdown at firing temperatures and therefore may affect the final color of ceramics.[36,37]

When the thickness of the veneer was increased by 50% from 1 to 1.5 mm, VM7 exhibited a very small increase in its ratio (4.7%) as compared with those exhibited by IPSe (12.9%) and NRPA (13.9%). This suggests that for the less translucent ceramics, the effect of thickness might not be significant. More translucent ceramics, however, allow more simulation of natural color by varying the thickness level and characterization. As for ΔE values, IPSe showed the most significant reduction in ΔE values (45%), followed by NRPA (37%), whereas VM7 showed the least reduction (only 21%). This indicates that the opacity of this material is innate to its composition and optical properties and less related to its thickness. With IPSe and NRPA, the effect of thickness is more pronounced, with IPSe showing a reduction in ΔE values almost equal to the percentage of increase in thickness. Overall, the results support the belief that feldspathic porcelains like VM7 are less translucent than the ultralow fusing ceramics.[23]

This study has shown that thickness of the veneering ceramic does affect the CR. For all the ceramics tested, the CR had a positive linear relationship with thickness. Antonson and Anusavice[23] evaluated the effect of thickness on the CR of veneering and core ceramics and concluded that CR was reliant on the type as well as the thickness of the material tested. Heffernan and others[24,30] described the effect of core ceramic thickness on its translucency as well as the influence of core plus ceramic veneer thickness on the overall translucency of the specimens and were able to identify a significant range of translucency. The present study confirms that the thickness of ceramics may influence the opacity. In addition, a positive linear relationship between opacity and ability of veneers to mask backgrounds was confirmed, which is in agreement with the results found in previous studies.[25,38]

ΔE values for NRPA specimens were considerably higher than for IPSe and VM7 specimens. Moreover, CR percentages calculated for NRPA were lower than those obtained for IPSe and VM7. These results indicate that masking ability and CRs of IPSe and VM7 were significantly better than those for NRPA. The optical properties of the different constituents in core and veneer materials and the thickness of each material are considered to be the main factors that influence the CR and masking ability of any ceramic system.[25] When a high percentage of alumina is incorporated in the composition of a ceramic, it is believed that it intercepts the incident light more efficiently and subsequently increases the ceramics opacity and masking ability. IPSe is approximately 70% lithium disilicate glass ceramic, with its main component being silicon dioxide (SiO_2). According to the manufacturer additional components of lithium oxide (Li_2O), potassium oxide (K_2O), magnesium oxide (MgO), zinc dioxide (ZnO_2), aluminum trioxide (Al_2O_3), phosphorus pentoxide (P_2O_5) and other oxides are also present within its composition. Due to the use of new technologies and optimized processing parameters, the formation of defects in the bulk of the ingot is avoided. Lithium disilicate, the main crystal phase, consists of needle-like

crystals measuring 3 to 6 μm in length. VM7 is fabricated from glass frits melted in metal oxides, and it is characterized by its enamel-like light refraction and reflection properties. Furthermore, the use of additional fluorescent and opalescent porcelains enables highly individual restorations with a high standard of esthetics to be achieved with these ceramics. NRPA was developed to enable new applications of individual inlays, onlays, and veneers without a supporting framework.

In theory, the masking ability of a porcelain veneer is considered to be perfect when it will have no color change over white and black backgrounds, ie, $\Delta E = 0$. If that is the case, the veneer is "color stable" over white and black backgrounds. There is a controversy in the literature about the ΔE values that are acceptable clinically. Several studies found that ΔE values as low as one unit are visually detectable.[39,40] On the other hand, Ruyter and others[41] reported a threshold for visually acceptable color change to be up to 3.3 units. Another study reported a visual match between a resin composite veneer and a tooth when the mean ΔE was 3.7 units.[42] Chu and others[38] considered $\Delta E \leq 5$ to be the representative value of acceptable color difference for veneers with the corresponding CR percentage to be at 0.91, above which the restoration is capable of masking the background color changes from white to black. The author stated that estimating a threshold CR percentage is helpful in predicting whether a porcelain veneer or crown will be affected by the underlying tooth color. In the present study none of the veneers had a ΔE below 5 or a CR over 0.91. Therefore, it is acceptable to say that the porcelain veneers with 1.0- or 1.5-mm thickness were not completely able to mask the underlying black background, although their masking ability improved with increased thickness. Thus, if the clinical situation requires color masking, thicker, more opaque materials should be used.

It is the responsibility of the clinician to fully understand the limitations of veneers in masking severe discolorations, taking into account that each ceramic system is unique in its optical properties. This will ultimately affect the final esthetics of the restoration; therefore, the type of ceramic needs to be chosen according to each clinical situation.

Finally, further studies are needed to evaluate the clinical implications of these findings. In some clinical situations varying degrees of dark stains need to be masked. Therefore, the interaction between the background color and the thickness of the veneer needs to be examined. A range of different background colors mimicking the stains a clinician may encounter should be used along with different thicknesses of the veneers. Furthermore, different shades of ceramic veneers, other than A2, and luting cements used in the clinic may also be used to accurately imitate the clinical situation. In addition, the effect of daylight and incandescent and fluorescent lights must be considered, because it has been shown that they do influence the resultant color of the ceramic restorations.

CONCLUSIONS

Within the limitations of this study the following conclusions can be drawn:

1. CRs and masking abilities are affected by the types and thickness of the ceramics.
2. NRPA demonstrated the least masking ability among the three ceramics tested. IPSe and VM7 were similar in their masking abilities, but IPSe exhibited higher CR percentages than did VM7.
3. All the materials tested in this study were not capable of completely masking the underlying black background, although the masking ability improved when the thickness was increased from 1.0 to 1.5 mm.
4. There is a strong linear relationship between masking ability and CR.
5. The clinician needs to understand the limitations of the ceramic veneers in masking severely stained teeth. In addition, each ceramic system has distinctive optical properties that may influence the final appearance of the restoration. Therefore, the type of ceramic should be chosen according to each clinical situation.

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Microleakage of IPS Empress 2 Inlay Restorations Luted With Self-adhesive Resin Cements

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Clinical Relevance

Although self-adhesive resin cements reduce the duration of the luting process, their universal practice requires caution because the microleakage performance of some brands is very poor.

SUMMARY

Objective: To assess the microleakage of three self-adhesive and one etch-and-rinse resin cements when luting IPS Empress 2 (Ivoclar Vivadent, Liechtenstein) all-ceramic inlay restorations to the prepared cavities in extracted human molars.

Methods: The cylindrical Class V cavities were prepared on the buccal surfaces of 40 extracted human third molars using diamond burs. The IPS Empress 2 ceramic inlays were placed with Multilink Sprint (Ivoclar Vivadent), RelyX Unicem (3M ESPE, USA), G-Cem (GC, Japan), or Variolink II (Ivoclar Vivadent) as the control group. After storage in distilled water at 37°C for 24 hours, samples were subjected to 1000 thermal cycles between baths of 5°C and 55°C, with a dwell time of 30 seconds. The microleakage scores were examined on the occlusal and gingival margins at 30× magnification after each sample was stained with 0.5% basic fuchsin and sectioned into three parts using a thin diamond blade (Isomet, Buehler, USA) (n=40). The extent of microleakage on both occlusal and gingival margins of the restorations was scored and recorded. The microleakage data were analyzed using Kruskall-Wallis and Mann-Whitney U-tests.

Results: Statistically significant differences were observed between the groups in both

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margins according to the Kruskall-Wallis and Mann-Whitney U-tests ($p<0.05$). Microleakage scores on the occlusal margins were Variolink II < RelyX Unicem < G-Cem = Multilink Sprint. Microleakage scores on the gingival margins are Variolink II = RelyX Unicem < G-Cem < Multilink Sprint.

Conclusion: Self-adhesive resin cements displayed higher microleakage scores on the occlusal margins, whereas on the gingival margins RelyX Unicem showed comparable microleakage results with the control samples.

INTRODUCTION

Increasing esthetic demands have resulted in the development of tooth-colored restorative materials. Among these materials, glass ceramic inlay techniques have some advantages such as satisfactory physico-chemical properties, abrasion resistance, and color retention compared with the composite resins that have led to some problems when used in stress-bearing areas of the mouth.[1] IPS Empress 2 (Ivoclar Vivadent, Schaan, Liechtenstein) glass ceramic is a heat-pressed, lithium disilicate–reinforced material using the "lost-wax" technique. This all-ceramic material has been introduced for single-unit restorations as well as for three-unit fixed partial dentures in the anterior region extending to the second premolar. The final restoration, made of a lithium disilicate–framework ceramic, offers clinical benefits in terms of machinability, polishability, and reduced wear of opposing tooth structure, with the advantages of increased biocompatibility, natural appearance, and superior esthetics.[2-4]

Marginal adaptation of ceramic inlays is one of the important features that could influence the durability of these restorations.[5,6] Microleakage at the interface between the teeth and the restorative material still remains a problem, and unfortunately, no technique is available that completely eliminates this leakage.

Ceramic inlays are generally cemented using resin-based cements because these cements generally display superior mechanical properties[7,8] and can increase the retention[9] and fracture resistance of overlying ceramic materials.[10] In addition, the flexural strengths of resin cements are higher than glass ionomer and resin-modified glass ionomer cements; thus, they are especially recommended for high-strength clinical situations.[8,11] To date, a number of resin-based luting cements have been introduced by different manufacturers. Various pretreatment steps to prepare the tooth's surface are necessary prior to the use of the resin cements to achieve adhesion to dentin. Depending on the type of luting cement, these steps include etching with phosphoric acid, priming, and bonding. In order to obtain optimal adhesive cementation, use of dentin bonding agents prior to the cementation of inlays is gaining wider acceptance. In previous studies, acceptable results were obtained with the bonding strategies described above.[12,13] However, the clinical application of these steps is time consuming and technically sensitive. Thus, cements that form a comparable adaptation but avoid the complex bonding procedures are desirable.

Recently, new resin-based systems, named *self-adhesive* resin cements, have been put on the market. These cements were developed to meet the demand among dentists for luting agents that offer easy, quick, and universal application. During the application of these materials, no conditioners or bonding agents need to be applied to the dental hard tissues. Bonding values and mechanical properties of these products are claimed to be comparable to those of similar products currently available by the manufacturer.[14,15]

The purpose of this *in vitro* study was to compare the adaptation of three self-adhesive and one etch-and-rinse resin cements on the microleakage of IPS Empress 2 all-ceramic inlay restorations by a dye penetration method.

METHODS AND MATERIALS

A total of 40 extracted, caries-free human third molars were used in this study. Immediately after extraction, the teeth were scraped of any residual tissue tags, pumiced, and washed under running tap water. The teeth were stored in distilled water at +4°C until required, a period not exceeding one week. Standardized, nonbeveled cylindrical Class V cavities were prepared on the buccal aspects of each tooth with round internal angles, 1 mm below the cementoenamel junction, using cylindrical (3.8 mm in diameter and 1.8 mm in length) (041-038C, MDT Micro Diamond Technologies Ltd, Afula, Israel) and 6° conical diamond burs (702.8KR, Abrasive Technology, London, UK). Standardization of the cavity size was accomplished by using the cylindrical diamond burs in similar dimensions with the prepared cavities and using the hand piece in a parallelometer during preparation. After preparation, the teeth were randomly divided into four groups. The bonding agents and resin cements used in this study are described in Table 1.

Table 1: *The Bonding Agent and Resin Cements Used in This Study*

Cement	Variolink II	Multilink Sprint	RelyX Unicem	G-Cem
Type	Etch-and-rinse resin cement	Self-adhesive resin cement	Self-adhesive resin cement	Self-adhesive resin cement
Etching	Scotchbond Etchant	—	—	—
Priming	Syntac primer	—	—	—
Bonding	Syntac adhesive	—	—	—
Manufacturer	Ivoclar Vivadent, Liechtenstein	Ivoclar Vivadent, Liechtenstein	3M ESPE, USA	GC, Japan
Mixing	Hand	Automix syringe	Hand	Capsule
Active components	Base paste: Bis-GMA, UDMA, TEGDMA, filler	Base paste: Dimethacrylates, ytterbium trifluoride, glass filler, silicon dioxide, initiators, stabilizers and pigments	Base paste: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers	Powder: Fluoro-alumino-silicate glass, initiator, pigment
	Catalyst paste: Bis-GMA, UDMA, TEGDMA, filler	Catalyst paste: Dimethacrylates, ytterbium trifluoride, glass filler, silicon dioxide, adhesive monomer, initiators, stabilizers and pigments	Catalyst paste: Methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments	Liquid: Urethane dimethacrylate, dimethacrylate, 4-Methacryloyloxyethyl trimellitate, distilled water, phosphoric acid ester monomer, silicon dioxide, initiator, inhibitor

Definitions: Bis-GMA, bisphenol-glycidyl methacrylate; TEGDMA, triethylene-glycoldimethacrylate; UDMA, urethane dimethacrylates.

Impressions were made with polyvinyl siloxane material (Imprint II VPS, 3M ESPE AG, Seefeld, Germany) and poured into a vacuum-mixed polyurethane die material (Alpha Die MF, Schültz-Dental GmbH, Rosbach, Germany) according to the manufacturer's instructions. IPS Empress II ceramic inlays were fabricated according to the manufacturer's instructions and then glazed.

Ceramic inlays were etched with the hydrofluoric acid for 20 seconds, and then a layer of silane coupling was applied to the ceramic bonding surface for 60 seconds and air-dried.

Products used for cementation were as follows: in group 1, Multilink Sprint (Ivoclar Vivadent, Schaan, Liechtenstein); in group 2, G-Cem (GC, Tokyo, Japan); in group 3, RelyX Unicem (3M ESPE AG) self-adhesive resin cements; and in group 4, Variolink II (Ivoclar Vivadent) etch-and-rinse resin cement. All resin cements were applied to the diamond bur–prepared dentin surface with smear layer; however, self-adhesive luting agents were applied without separate dentin conditioning.

The cavities of self-adhesive resin cement groups were cleaned with fluoride-free pumice and water after cavity preparation. In groups 1 and 2, the cavities were rinsed with water and air-dried for two to four seconds to remove excess moisture, leaving the dentin surface with a slightly glossy appearance before cementation with Multilink Sprint and RelyX Unicem.[16,17] In group 3, the cavity surfaces were rinsed with water and gently dried by blowing with oil-free air until the prepared surfaces appeared dry before cementation with G-Cem.[18] After tooth preparation, the self-adhesive resin cements were applied to the cavities. Inlay restorations were seated into the cavities using light pressure and cured briefly (one to two seconds) with light. Then the excess material was removed using a scaler. The

restoration margins were covered with glycerin gel. The luting agents were light-cured for 20 seconds. The light activating unit was Optilux 501 (Kerr, Orange, CA, USA), which was tested prior to each sample. The output of this unit did not drop below 500 mW/cm^2. After the final polymerization the glycerin gel was rinsed off.

In group 4, the enamel and dentin margins of inlay cavities were first cleaned with fluoride-free pumice and water and then etched with a phosphoric acid gel (Uni-etch, Bisco, IL, USA) for 15 seconds. They were rinsed thoroughly with water for approximately five seconds and air-dried for two to four seconds to remove excess moisture, leaving the dentin surface with a slightly glossy appearance.[19] One drop of the primer (Syntac, Ivoclar Vivadent) was applied to the cavity surfaces for 15 seconds and gently air-dried. A layer of bonding resin (Syntac, Ivoclar Vivadent) was applied with a brush for 10 seconds, spread gently with air. After application of enamel bonding agent (Heliobond, Ivoclar Vivadent) to the cavity and the bonding surface of the ceramic inlay restorations, the cavities were filled with Variolink II (Ivoclar Vivadent) and inlays were placed into the cavities using light pressure and cured briefly (one to two seconds) with light. Then the excess material was removed using a scaler. The restoration margins were covered with glycerin gel. The resin cement was light-cured for 40 seconds. After final polymerization the glycerin gel was rinsed off.

Excess material was removed with finishing diamond burs and flexible discs. The restoration margins were finished with silicone polishers (Astropol F and Astropol P, Ivoclar Vivadent).

After cementation, specimens were stored in distilled water at 37°C for 24 hours and then subjected to 1000 thermal cycles between baths of 5°C and 55°C, with a dwell time of 30 seconds. The teeth were subsequently coated with nail varnish 1 mm short of the restoration margins to seal open dentin tubules. The dye penetration test was conducted in a 0.5% basic fuchsin dye solution for 24 hours. The teeth were then rinsed, and Class V restorations were sectioned into three parts longitudinally in a bucco-lingual plane with a slow-speed diamond blade (Struers, Ballerup, Denmark). The sections from the centers of the restorations were 2-mm thick, whereas the other sections were 1-mm thick. In this way, four surfaces (either mesial or distal surfaces of 1-mm-thick samples and both mesial and distal surfaces of 2-mm-thick samples) were obtained from one restoration for microleakage

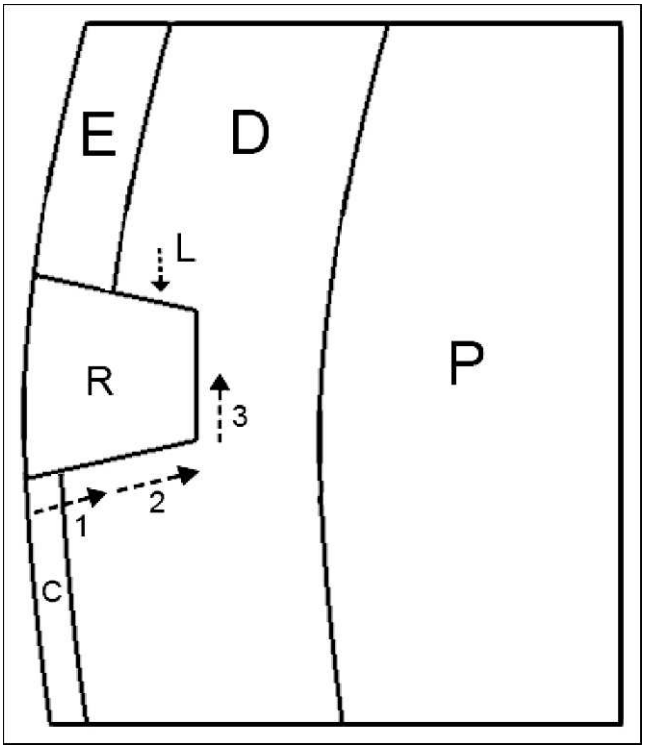

Figure 1. *Dye penetration between restoration and tooth. E, enamel; D, dentin; C, cementum; R, inlay restoration; L, luting cement; P, pulp.*

evaluation. A total of 160 surfaces (n=40) were evaluated at 30× magnification under a stereomicroscope (Olympus Co, Tokyo, Japan) by two examiners who were calibrated prior to the study.

The extent of the microleakage was scored according to the following criteria (Figure 1):

0 = no leakage visible;
1 = penetration of dye along the cavity wall, but less than half the length;
2 = penetration of dye along the wall, but short of the axial wall;
3 = penetration of dye to and along the axial wall.

Leakage scores at occlusal and gingival margins for each group were compared with the nonparametric Kruskall-Wallis test. Multiple comparisons were performed as pairwise comparisons using the Mann-Whitney U-test. Significance was considered at the 0.05 level.

RESULTS

The microleakage scores are shown in Table 2. The mean and median dye penetration scores, standard deviation, minimum, maximum, and interquartile range values are shown in Table 3. According to the

Table 2: *Frequency of Microleakage Scores in Study Groups, n (%)*

	Occlusal Margin				Gingival Margin			
	0	1	2	3	0	1	2	3
Variolink II	18 (45)	13 (32.5)	7 (17.5)	2 (5)	24 (60)	8 (20)	6 (15)	2 (5)
Multilink Sprint	4 (10)	18 (45)	12 (30)	6 (15)	1 (2.5)	6 (15)	13 (32.5)	20 (50)
RelyX Unicem	12 (30)	13 (32.5)	11 (27.5)	4 (10)	22 (55)	10 (25)	5 (12.5)	3 (7.5)
G-Cem	6 (15)	10 (2.5)	16 (40)	8 (20)	15 (37.5)	9 (22.5)	11 (27.5)	5 (12.5)

Kruskall-Wallis and Mann-Whitney U-tests, there were significant differences between the groups in both margins ($p<0.05$).

Nearly half of the Variolink II samples (45%) revealed no visible leakage at the occlusal margins, followed by 30% of RelyX Unicem, 15% of G-Cem, and 10% of Multilink Sprint samples. Only 20% or less of all groups showed dye penetration along the axial wall at the occlusal margin. Variolink II samples showed the lowest leakage scores at the occlusal margins ($p<0.05$). RelyX Unicem's leakage score was higher than that of Variolink II ($p<0.05$), whereas it was lower than those of the Multilink Sprint and G-Cem samples ($p<0.05$). There were no statistically significant differences between the occlusal margin leakage scores of G-Cem and Multilink Sprint samples.

More than half of the Variolink II (60%) and RelyX Unicem (55%) samples revealed no visible leakage at the gingival margins, followed by 37.5% of G-Cem and 2.5% of Multilink Sprint samples. Half of the Multilink Sprint samples (50%) showed dye penetration along the axial wall at the gingival margin, whereas the ratio of score 3 was under 13% for the other tested agents. Multilink Sprint samples showed the highest leakage scores at the gingival margins ($p<0.05$). G-Cem's leakage score was lower than that of the Multilink Sprint samples ($p<0.05$), whereas it was higher than that of the RelyX Unicem and Variolink II samples ($p<0.05$). There were no statistically significant differences between the gingival margin leakage scores of the RelyX Unicem and Variolink II samples.

There were no significant differences between the microleakage at the enamel and dentin margins in Variolink II samples. The microleakage at the enamel margins was greater than that at the dentin margins in the RelyX Unicem and G-Cem samples ($p<0.05$), whereas the microleakage at the dentin margins was greater than that at the enamel margins in the Multilink Sprint samples ($p<0.05$).

Table 3: *Descriptive Analysis of Study Groups*

	Occlusal Margin						Gingival Margin					
	Mean	SD	Median	Min	Max	Interquartile Range	Mean	SD	Median	Min	Max	Interquartile Range
Variolink II	0.83	0.903	1	0	3	1	0.65	0.921	0	0	3	1
Multilink Sprint	1.5	0.877	1	0	3	1	2.30	0.823	2.5	0	3	1
RelyX Unicem	1.18	0.984	1	0	3	2	0.73	0.96	0	0	3	1
G-Cem	1.65	0.975	2	0	3	1	1.15	1.075	1	0	3	2

Abbreviations: Max, maximum; Min, minimum; SD, standard deviation.

DISCUSSION

Different techniques have been described for studies of margin quality. The most widely accepted method is the dye penetration test.[20] In our study, 0.5% basic fuchsin solution was used for the dye penetration test. All restorations had previously undergone thermal cycling in order to subject the restorations to thermal expansion and contraction challenges. The different thermal expansion coefficients of tooth tissue from the restorative materials may lead to gap formation.[21] As such, to assess the *in vitro* performance of resin materials, thermal cycling is the common method used to simulate the long-term stresses to which the resin restorations are exposed.[22]

In the present study, the performance of self-adhesive resin cements on the microleakage of IPS Empress II inlays was evaluated in Class V cavities. The reason for studying Class V cavities was that 1) Class V cavities have unfavorable configuration factor resulting in high contraction scores within an adhesively fixed resin material, 2) Class V restoration margins are located in enamel as well as in dentin, 3) preparation and restoration of Class V lesions are minimal and relatively easy, thereby somewhat reducing practitioner variability, and 4) it is easier to standardize the preparation of Class V cavities than Class II cavities.[23,24]

An essential requirement for a perfect restoration is the perfect adhesion to the tooth surface.[20] The adhesion of ceramic restorations to tooth surface depends on the properties of the dental cements and their bonding procedures. The tooth pretreatment with acid, primer, and bonding is very important for resin cement because these procedures affect the condition of the dentin and the smear layer.

Self-adhesive resin cements without pretreatment with acid, primer, and bond demonstrated higher microleakage scores than etch-and-rinse resin cement at the occlusal margins. In addition, the microleakage at the enamel margins was greater than that at the dentin margins in the RelyX Unicem and G-Cem samples. The greater leakage at the occlusal margins with self-adhesive resin cements may be attributed to their insufficient ability to etch the smear layer–covered enamel surface and lack of development of adequate micromechanical retention. High viscosity that the cements have after mixing and the short interaction time that they have with the tooth surface before light-curing may be the reasons for the inadequate micromechanical retention on enamel. The initial low pH may not be sufficient to etch the enamel when the etching time is not adequate or when the neutralization reactions take place rapidly. The use of resin cement including a separate acid application step prior to the adhesive application results in good micromechanical retention because it adequately etches enamel.[25] Previous *in vitro* studies also reported that the etching potential of the self-adhesive cements was lower than that of resin cements applied by a phosphoric acid.[26,27] Moreover, significantly better marginal adaptation in enamel margins was indicated with resin cements when compared with self-adhesive ones.[28,29]

Similar to enamel margins, Variolink II showed good performance at dentin margins. However, varying results were obtained at dentin margins with self-adhesive cements, similar to the previous findings.[29,30] RelyX Unicem's leakage score was similar to that of Variolink II, while it was lower than those of the Multilink Sprint and G-Cem samples. The results of a majority of previous studies were consistent and demonstrated that RelyX Unicem performed comparably to other multistep resin cements on dentin.[31,32] The authors reported that the specific multifunctional phosphoric-acid methacrylates of this cement react with the tooth surface in multiple ways, resulting in an effective seal. Besides the formation of complex compounds with calcium ions, different kinds of physical interactions such as hydrogen bonding or dipole-to-dipole interactions were supposed to favorably affect the adhesion of this self-adhesive cement.[33]

The different microleakage results of the self-adhesive cements may be explained by their different functional monomers and different chemical compositions. Differences in pH values may affect the etching ability of these cements to enamel and dentin, resulting in less than ideal adhesion with subsequent microleakage. G-Cem includes 4-methacryloxy-ethyl trimellitate anhydride, which bonds by a chelating reaction to calcium ions in apatite.[34] In addition, this cement was applied to the dry dentin surfaces, contrary to the other cements tested, because it is a water-based system and these systems require drier dentin surfaces for improved adhesion.[35] However, its leakage scores were higher than those of RelyX Unicem. The relatively weak bonding potential and the high molecular weight of the functional monomer may be the reason for the failure of the supposed chemical reaction within a clinically reasonable time.[36]

Multilink Sprint showed the worst performance at the dentin margins. In addition, the microleakage at

the dentin margins was greater than that at the enamel margins in Multilink Sprint samples. The reason for this result may be the mild discrepancy between demineralization and the infiltration depths recorded for Multilink Sprint by Monticelli and others,[30] resulting in a deeper diffusion of noncured, nonneutralized acidic monomers. These residual monomers may retain their etching potential, forming an unprotected dentin zone and jeopardizing adhesion.[37]

CONCLUSION

Within the limitations of this *in vitro* study, self-adhesive resin cements displayed higher microleakage scores on the occlusal margins than the control (Variolink II) group. On the gingival margins, RelyX Unicem showed comparable microleakage results with the control group.

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Clinical Techniques/Case Report

Functional and Aesthetic Guidelines for Stress-Reduced Direct Posterior Composite Restorations

S Deliperi

Clinical Relevance

A step-by-step "stress-reducing direct composite" technique is recommended to reduce postoperative sensitivity and improve the clinical performance of composite restorations.

SUMMARY

Amalgam has been used in the restoration of structurally compromised posterior teeth for many years. When placing large amalgam restorations, replacement of weak cusps with restorative material is recommended to prevent tooth fracture. This recommendation can be modified with new guidelines using modern adhesive techniques. Semidirect and indirect inlay/onlay composite restorations have progressively replaced amalgam restorations over the past 20 years. Lately, single visit direct resin-bonded composite (RBC) restorations have also been used as a viable alternative to conventional indirect restorations. This paper is intended to introduce a step-by-step protocol for the direct restoration of structurally compromised posterior teeth using RBCs with stress-reducing protocols.

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INTRODUCTION

The most recent American Dental Association (ADA) statement on resin-bonded composites (RBCs) endorses the use of posterior composites in 1) small and moderately sized restorations, 2) conservative tooth preparations, and 3) areas where esthetics are important.[1] These include Classes I and II, replacement of failed restorations, and primary caries. Teeth needing either larger or cusp replacement restorations are usually treatment planned for both indirect laboratory-fabricated composite resin and ceramic inlay/onlay restorations. A meta-analysis of studies conducted in the 1990s reported an annual failure rate of 2.2% for direct posterior composite restorations, 2.9% for resin composite inlays, and 1.9% for ceramic restorations.[2] As the basic chemistry of indirect RBCs remains similar to that of the direct materials, differences in mechanical proper-

ties are minimal and not expected to be clinically significant.[3] Despite the ADA recommendation, dentists are stretching the clinical indications for direct RBC restorations.[4,5] Brunthaler and others[6] found a linear correlation between the size of the restoration and the observation period and the failure rate; conversely, Brackett and others[7] reported no difference in the clinical performance of medium-size vs large direct RBCs.

RBCs have been used to restore posterior teeth since the 1970s[8] and have been researched extensively in the last 40 years; the drawbacks of composite resin are well known. Concerns still exist with regard to composite wear,[9] less-than-ideal bonding to dentin,[10] stress from polymerization shrinkage,[11] and technique sensitivity.[12]

In fairness, one must realize that the evolution of both composite materials, adhesive, and light-curing systems has advanced rapidly over the last two decades. However, both researchers and clinicians have been waiting for further material technology improvement from the manufacturer side; conversely, they have not focused enough on the development of a step-by-step protocol to improve the clinical performance of RBC restorations.

As matter of fact, many factors contribute to the achievement of clinical success with direct posterior RBC restorations: 1) analysis of the occlusion, 2) complete excavation of dental caries and proper cavity preparation, 3) analysis of residual tooth structure, 4) proper selection and application of the dentin bonding System, 5) control of polymerization stresses by using appropriate layering and curing techniques, and 6) occlusal force equilibration. The goal of this paper is to provide esthetic and functional guidelines for the restoration of structurally compromised posterior teeth using a "stress-reducing direct composite" (SRDC) technique.

MATERIALS AND METHODS

Step-by-Step Protocol Through Case Presentation

A 27-year-old female patient presented with a failing restoration in a lower molar tooth replacing both the distal marginal ridge and a facial cusp (#36). The patient's tooth was restored with a direct composite resin 1 year earlier (Figure 1).

Step 1: Analysis of the Occlusion—Preoperative occlusal analysis showed concentration of the occlusal load on the residual facial wall of tooth #36 and an absence of an upper molar palatal centric stop (Figure 2). Wear facets are notable on the remaining facial cusps in the area of the remaining occlusal contacts (Figure 1), upper left molar, and premolar teeth did not present any restoration. Because of the unbalanced occlusion, a fracture of the remaining wall can occur under mastication. After completing the analysis of occlusion and presenting a treatment plan to the patient for both a direct and indirect restoration, an SRDC restoration was planned on tooth #36.

Step 2: Complete Excavation of Dental Caries and Proper Cavity Preparation—A rubber dam was placed, and the existing restoration was removed using # 2 and #4 round burs (Brasseler, Savannah, GA, USA). The cavity was prepared in a very conservative manner, just removing the decayed dental tissue and trying to preserve the remaining sound tooth structure according to the basic guidelines for direct adhesive preparations. A caries indicator (Sable Seek, Ultradent Products, South Jordan, UT, USA) was applied to the dentin; stained nonmineralized and denatured dental tissues were removed with a spoon excavator. Residual enamel sharp angles and unsupported prisms were smoothed using the SD and SB partially diamond-tipped ultrasonic tips (EMS, Nyon, Switzerland); the SB instrument was also used to smooth sharp angles located within the dentin (Figure 3). No bevels were placed on either the occlusal or the gingival margins.

Step 3: Analysis of Residual Tooth Structure—Once the preparation was completed, it was determined that the facial-lingual occlusal extension (isthmus) was greater than two-thirds the intercuspal distance, the proximal extension was greater than half the distance to the line angle, and the facial-distal cusp was missing. However, the thickness of the residual walls greater than 3 mm and the mesiodistal extension almost half the distance to the marginal ridges were considered sufficient to give enough support to an SRDC restoration.

Step 4: Proper Selection and Application of the Dentin Bonding System—A circular matrix (Omni-Matrix, Ultradent Products) was placed around tooth #36 and interproximal matrix adaptation secured by only tightening it; perfect adaptation to the gingival margin was achieved without using any dental wedge. The use of dental wedges in teeth having cervical margins below the gingival level may create a step on the restoration because the matrix is pushed onto the cavity in an attempt to achieve its adaptation to the cervical area. Both the bonding and the marginal adaptation to the cervical area may be compromised. The tooth was etched for 15 seconds using a 35% phosphoric acid (UltraEtch,

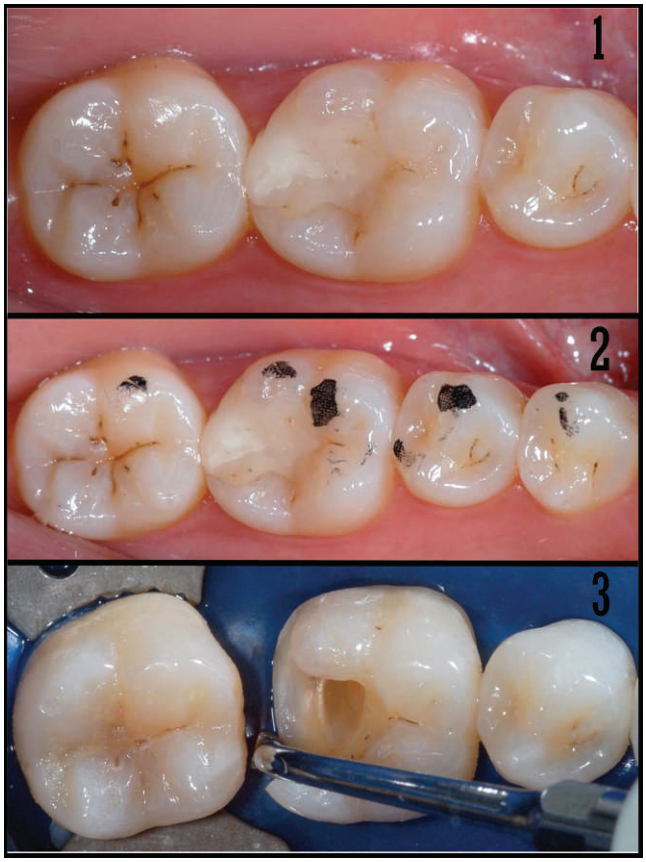

Figure 1. *Preoperative view of tooth #36 showing an incongruous tooth-colored restoration*
Figure 2. *Before starting anesthesia, occlusion was checked, and centric stops were recorded*
Figure 3. *Cavity preparation was completed using partially diamond tipped ultrasonic tips*

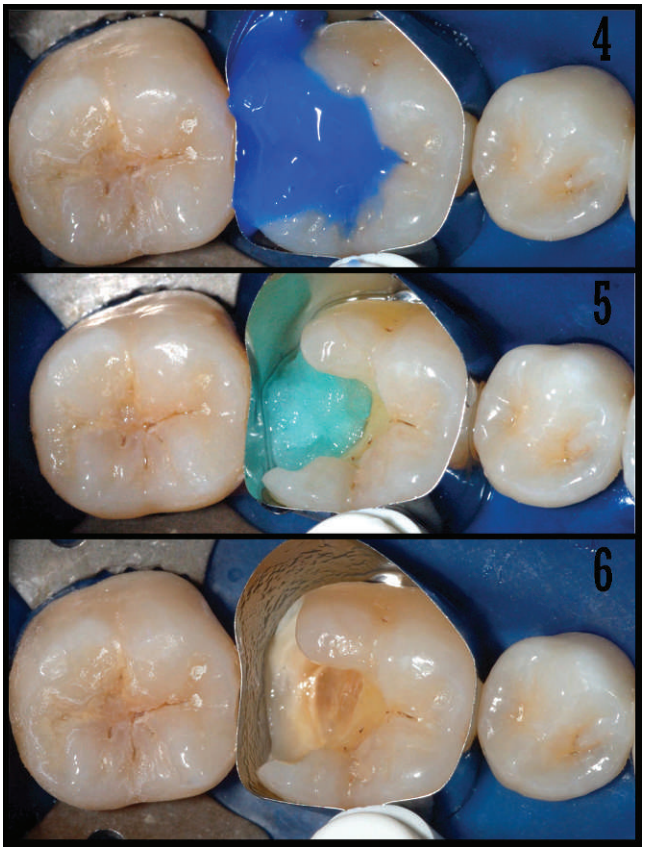

Figure 4. *A circular matrix was placed and etching performed using 35% phosphoric acid*
Figures 5 and 6. *A 2% digluconate chlorexidine solution was applied on dentin, followed by an ethanol-based adhesive system application on both enamel and dentin*

Ultradent Products; Figure 4). The etchant was removed and the cavity rinsed with water spray for 30 seconds, being careful to maintain a moist surface. The cavity was disinfected with a 2% chlorexidine antibacterial solution (Consepsis, Ultradent Products; Figure 5). A fifth-generation 40% filled ethanol-based adhesive system (PQ1, Ultradent Products) was placed in the preparation, gently air thinned, and light cured for 20 seconds using an LED curing light (UltraLume V, Ultradent Products; Figure 6).

Step 5: Control of Polymerization Stresses by Using Appropriate Layering and Curing Techniques—Vit-l-escence microhybrid composite resin (Ultradent Products) was used to restore the teeth. Stratification was initiated using multiple 1- to 1.5-mm triangular-shaped (wedge-shaped) increments; apico-occlusal placed layers of A4 shade were used to reconstruct the cervical third of the proximal surface. At this point, the circular matrix was replaced with a sectional matrix to achieve a more predictable contact point with the second molar tooth. Both the proximal surface and the external shell of the disto-lingual cusp buildups were completed using the Pearl Smoke (PS) enamel shade (Figure 7). Stratification of dentin was started by placing a 1- to 1.5-mm even layer of A3.5 flowable composite (PermaFlo, Ultradent Products) on deeper dentin (Figure 8), which was followed by the application of dentin wedge-shaped increments strategically placed to only two bonded surfaces, decreasing the cavity configuration or C-factor ratio (Figure 9). The C-factor is defined as the ratio between bonded and unbonded cavity surfaces; increasing this ratio also increases the stress from polymerization shrinkage.[13] For the same reason, single increments of PS enamel shade were applied to one cusp at a time (Figure 10); each cusp was cured separately, achieving the final primary and secondary occlusal morphology (Figure 11). In order to reduce stress from polymerization shrinkage, the

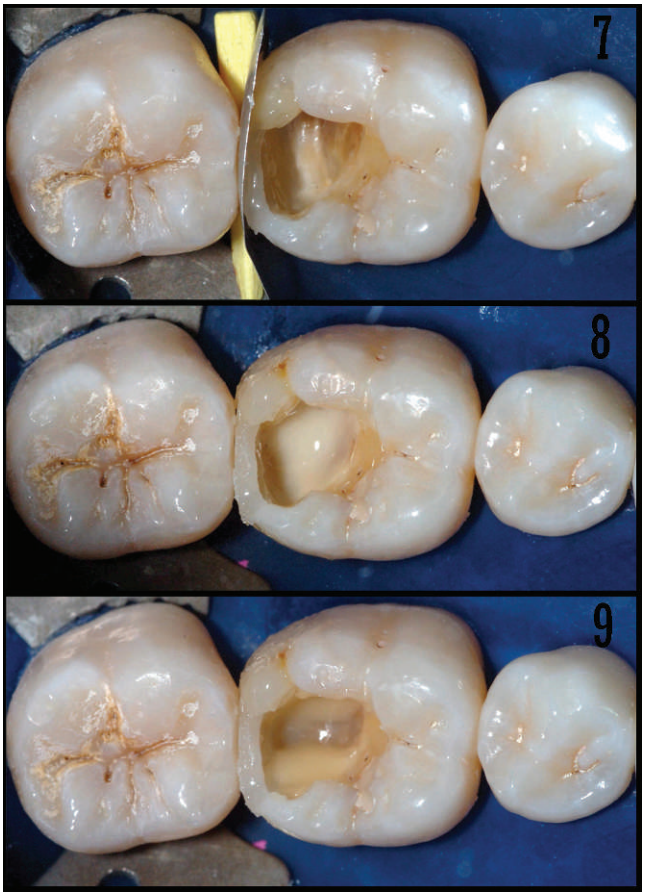

Figure 7. *The circular matrix was replaced with a sectional matrix, and the peripherical enamel skeleton was built up first using a combination of dentin and enamel wedge-shaped increments*
Figure 8. *Dentin stratification was started placing a 1-mm layer of A3.5 flowable composite resin on deep dentin*
Figure 9. *Dentin stratification was performed by using wedge-shaped increments of composite dentin shades*

authors utilized a previously described polymerization technique, based on a combination of pulse (enamel) and progressive (dentin) curing technique through the tooth. The pulse curing protocol is adopted for the proximal and occlusal enamel buildup polymerization; it is accomplished by using a very short curing time (1 or 2 seconds) per each increment. The progressive curing technique is used for the polymerization of the dentin increments; it is performed by placing the light tip in contact with the external cavity walls to start the polymerization through the wall (indirect polymerization) at a lower intensity. Final polymerization is then provided at a higher intensity and extended curing time. Initial occlusal and proximal adjustment of the restoration was performed using #7404 and #7902 carbide burs (Brasseler). The patient was recalled after 48 hours to complete the occlusal adjustment and perform the final polishing. Figure 12 shows the restoration at the six-month recall.

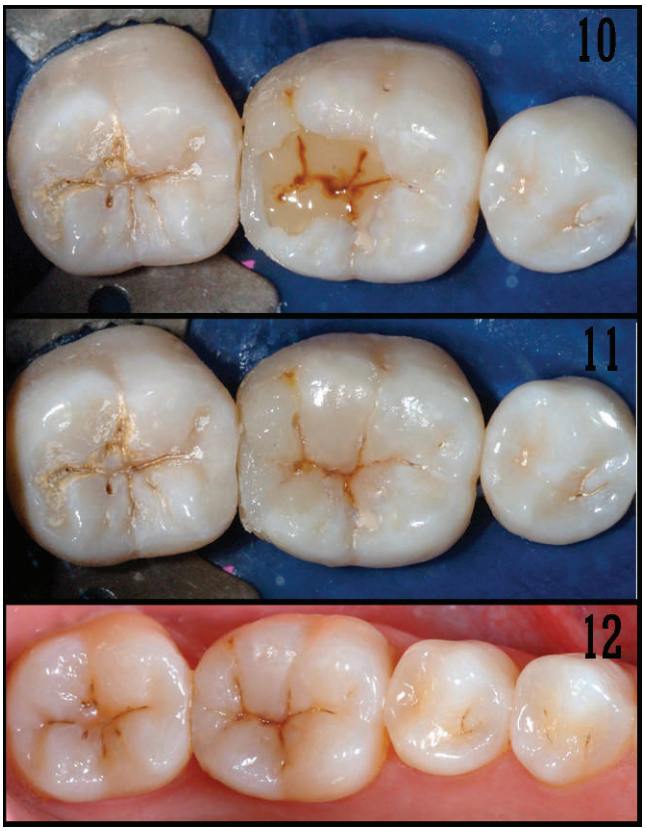

Figure 10. *A brown composite tint was placed at the end of dentin stratification*
Figure 11. *Restoration was completed with the application of PS shade to each cusp in order to develop cusp ridges and supplemental morphology*
Figure 12. *Result at the six-month recall*

Step 6: Occlusal Force Equilibration—Occlusion was verified, avoiding excessive load on the residual facial cusp and creating a centric stop in the composite restoration at the center of the tooth-restoration complex. The centric stops located on the tooth structure and composite resin are of the same intensity; they do not differ from the ones on the adjacent premolar teeth (Figure 13). Figures 14 and 15 show the pre- and postoperative X-rays.

DISCUSSION

When restoring a significant amount of occlusal anatomy, the patient's occlusion is a major determining factor in the success of large RBC restorations. The preoperative analysis of occlusion and the equal distribution of the load on the residual tooth structure and the restorative material, once the restoration is completed, are important to maintain the tooth-RBC complex over time.

The analysis of occlusion is performed at two different levels: the tooth to be restored and the opposing dentition. The tooth/teeth to be restored should be analyzed with the goal of detecting an uneven distribution of the occlusal contacts and assessing the presence of any wear facets related to both malocclusion and parafunction habits. Overload on either the restoration or the remaining tooth structure may lead to premature failure. The antagonist teeth need to be checked to assess the presence of any anatomically incongruous restoration potentially responsible for the incorrect occlusion detected in the opposing dentition; if this is the case, replacement of the restoration in the lower and upper teeth should be considered. Conversely, having a functional restoration in the antagonist dentiton will not require a replacement; the material (ceramic vs composite resin) used to restore the antagonist teeth may influence the choice of the restorative material for the restoration of the opposing tooth.[14] Given that the wear of posterior RBCs is similar to the reported enamel wear,[15] the selection of RBCs is the ideal choice having either virgin teeth or teeth restored with composite resin in the opposing dentition. Conversely, the partial or complete coverage of the opposing teeth with ceramic restorations may guide the clinician to select an indirect ceramic restoration to better match the wear rate. Although ceramic is considered the most "enamel-like" material, increased wear of either opposing natural teeth[16] or composite resin[17] remains a primary concern.

The thickness of the residual cusp wall both at the base and the cusp tip is a key element in the decision to preserve or eliminate cusps. Cusp coverage with a 2-mm overlap of restorative material is recommended when cusp base thickness is less than 2 mm and occlusal margins located at the cusp tip.[18] This decision needs to be supported by the analysis of the remaining tooth structure, including the connection of the cusp with a marginal ridge and its thickness, and the occlusal load distribution. Removing the unsupported enamel prisms and smoothing the sharp angles on both enamel and dentin are just the first steps to achieve a reliable bond to the dental substrate. These need to be coupled with both prevention of hybrid layer degradation and final occlusal equilibration. RBC restorations rely on both macromechanical and micromechanical retention; increasing cavity size results in restorations depending more on micromechanical retention provided by a specific adhesive technique.[19] Adhesive systems produce bond strengths that allow clinicians to bond

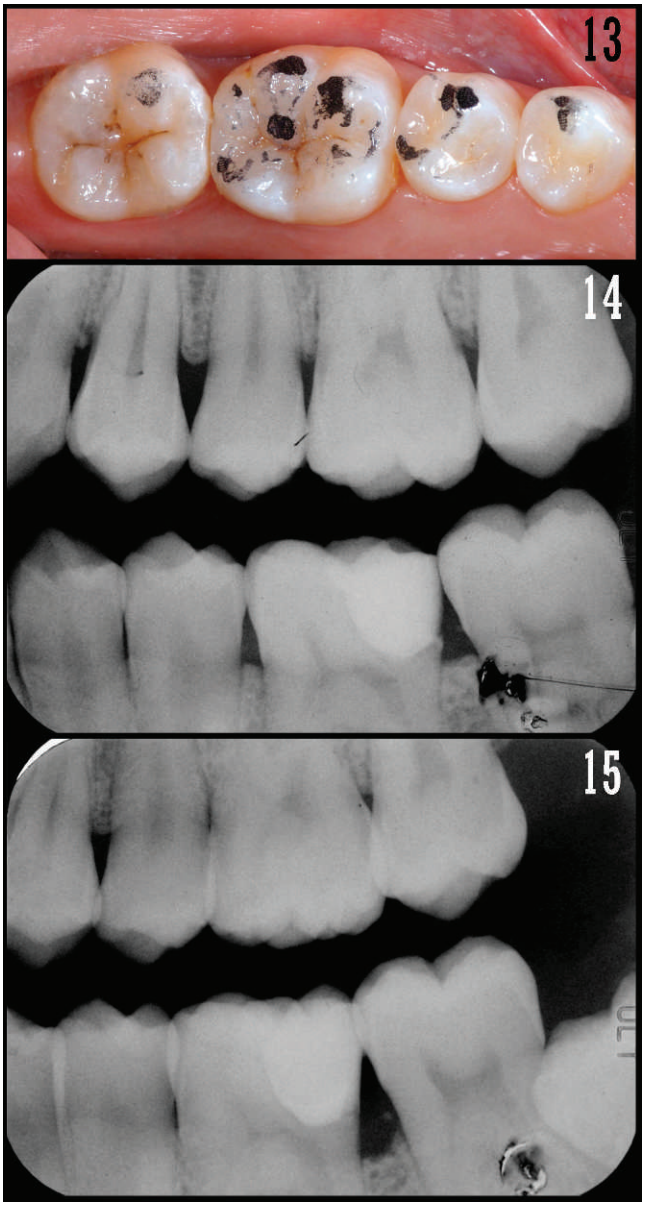

Figure 13. *Occlusal view of the final restorations after occlusion checking*
Figures 14 and 15. *Pre- and postoperative X-rays*

to tooth structure without the use of aggressive retentive cavity preparations. However, immediate dentin bonding may be challenged by the overlaying composite shrinkage stress; Magne and others[20] reported increased bond strength following immediate dentin sealing after the completion of tooth preparation for semidirect and indirect restorations. The protocol for SRDC restorations adopts a layering technique based on an enamel wall buildup first, followed by dentin stratification; this first step requires selective curing to be accomplished, allowing for initial dentin bonding maturation. Neverthe-

less, major concerns have been recently expressed regarding interfacial aging due to degradation of the hybrid layer related to water sorption, hydrolysis of the resin, and disruption of the collagen network.[10] Matrix metalloproteinases-2 (MMP-2) are endopeptidases present in large amounts in human dentin; MMP-2 may be involved in the degradation of the polymer matrix of the hybrid layer as well as the collagen fibrils. As a result, deterioration of the dentin-composite bond may compromise the longevity of both direct and indirect RBC restorations. Occlusal loading may contribute to this process because of the development of fatigue. Chlorexidine was demonstrated to be effective in the inhibition of MMPs. The application of a similar inhibitory agent in the clinical bonding procedure may result in a more satisfactory performance of bonding interfaces over time.[21]

Stress from polymerization shrinkage is one of clinician's main concerns when placing direct RBC restorations. Postoperative sensitivity, marginal enamel fractures, premature marginal breakdown, and staining may result from the stress developed at the tooth restoration interface. Three different strategies to reduce polymerization stress have been identified[9,22]: 1)modification of the placement technique, 2) altered curing schemes, and 3) use of a resilient liner on dentin. Combining composite stratification with wedge-shaped increments and polymerization with a low-intensity approach is mandatory to reduce stress in the restoration. Multiple wedge-shaped increments are placed, trying to contact no more than two bonded cavity walls; the technique allows a decrease in stress from polymerization shrinkage by reducing the composite mass (per increment) and transforming the high-C-factor configuration into multiple low-C-factor configurations (maximizing the unbonded free surface to enhance stress relief). In addition to this sophisticated stratification technique, a combination of progressive and pulse curing polymerization is used on dentin and enamel, respectively, to further decrease the stress from polymerization shrinkage.[22] By adopting a similar soft-start curing protocol, physical and mechanical properties of composite resin may also be improved; more time is available for composite flow in the direction of the cavity walls, resulting in stress release during polymerization shrinkage and increased cross-linking. The quality of the polymer network, which is not equivalent to the degree of conversion, is influenced by the modified curing scheme. A recent research study[23] corroborated previous findings[24,25] supporting the fact that polymerization protocols based on low intensity and increased curing time result in longer polymer chain formation; conversely, frequency of cross-linking increases using higher-intensity and short curing times, leading to multiple short polymer chains formation and reduced degree of cure.

The application of a thin layer of flowable composite (.5-1 mm) limited to the dentin floor has been suggested as an adjunctive strategy to counteract stress from polymerization shrinkage.[26,27] According to Hooke's law, stress depends on both shrinkage and elastic modulus; because of their low stiffness, flowable composite may deform to absorb some of the overlaying composite shrinkage strain.

CONCLUSION

An SRDC technique is based on a detailed pre- and postoperative analysis of occlusion. A well-equilibrated occlusion may contribute to the prevention of either changes in the occlusal morphology or tooth-RBC complex failure; accurately preserving and preparing the remaining sound tooth structure, selecting strategies to prevent the degradation of the hybrid layer, and adopting specific layering and curing schemes may protect the RBC restoration from both polymerization shrinkage and occlusal loading stresses.

Conflict of Interest Declaration

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

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A Clinical Study of Direct Composite Full-Coverage Crowns: Long-Term Results

V Alonso • M Caserio

Clinical Relevance

Composite full-coverage crowns are a viable option for teeth with amelogenesis or microdonts and are especially suitable for patients still undergoing growth.

SUMMARY

Objective: Long-term assessment of the clinical behavior of direct composite full-coverage crowns using transparent strip crowns as a matrix.

Method: A retrospective observational study without controls of 21 restorations was performed: nine teeth with hypoplasia, six conoid teeth, and six with microdontia. The mean patient age was 22.5 ± 8.2 years. The clinical procedure consisted of cleaning the tooth, acid etching and application of adhesive, after which a transparent strip crown was filled with composite and placed on the tooth. The gingival contour was polished using multifluted burs and interproximal spaces polished with polishing strips. Patients were examined after a period of 12.5 (±4.6) years by two observers who recorded the plaque index and evaluated the restorations in accordance with the modified U. S. Public Health Service (USPHS) criteria.

Results: Except for one case, all the scores obtained on the basis of the USPHS criteria were within the acceptable range. There were no cases of secondary caries. The statistically significant variations were anatomical form, marginal adaptation, marginal discolouration, and surface roughness.

Discussion: This technique is simple and non-invasive. It is a viable long-term treatment option for teeth with amelogenesis or microdonts and is especially suitable for patients still undergoing growth.

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INTRODUCTION

The dental anomalies that most commonly affect the upper lateral incisors are microdontia, conoidism, or a combination of both.[1] Microdontia affects 1.5% to 2% of the population.[2]

The term "amelogenesis imperfecta" covers a clinically and genetically heterogeneous group of hereditary disorders. Epidemiological studies of

differing populations have shown prevalence to range widely, from one in 700 to one in 14,000. It is classified into three groups—hypoplasia, hypomaturation, and hypocalcification[3]—and affects the enamel of both deciduous and permanent dentitions. More rarely, amelogenesis imperfecta is associated with other dental and oral disorders, such as taurodontism, and predominantly extraoral systemic syndromes, such as cone-rod dystrophy, oculodento-digital syndrome, tricho-dento-osseous syndrome, nephrocalcinosis, or Usher's syndrome.[4] Hypoplasic teeth exhibit a varying decrease in the enamel thickness, along with pitting and other irregularities. However, their hardness and transparency are preserved.

Histological alterations are seen in the enamel of teeth affected by amelogenesis imperfecta. Such alterations may affect the quality of the adhesive bond that may be achieved. In particular, hypocalcification reduces the quality of the bond because of the lack of mineralized structure. Some authors consider that deproteinization, through application of sodium hypochlorite a minute after acid etching, can improve enamel bonding in primary teeth,[5,6] while others prefer to limit the application of orthophosphoric acid to a maximum of 30 seconds in order to prevent further demineralization.[7]

These histological alterations affect the esthetics of the anterior sector. Such teeth are typically restored by indirect methods, using veneers,[8-10] porcelain,[11-13] or metal-ceramic[14] crowns. Another option is composite reconstruction,[15-20] indicated in growing patients and as a transitional restoration in readiness for future prosthetic rehabilitation.

Strip crowns have been used successfully for many years for restoring carious deciduous anterior teeth,[21-28] serving in the anterior sector as a matrix for a composite reconstruction. Little has been published on their use in permanent teeth.[29,30] There are very few long-term studies on direct composite restorations that modified the size and form of affected teeth in adult patients. In fact, strip crowns[31,32] were not used in any study.

The aim of this work was to study the long-term clinical outcome of direct composite complete crowns fabricated using transparent strip crown matrices on caries-free permanent teeth with microdontia, conoidism, or hypoplasia.

MATERIALS AND METHODS

This observational retrospective study without controls assessed a total of 21 restorations provided between 1992 and 2006: 14 in men and seven in women with a mean age of 22.5 ± 8.2 years. All restorations were in the anterior sector, 16 upper and five lower, and without endodontic treatment. There were six conoid teeth, six with microdontia (all upper lateral incisors), and nine with hypoplasia. At the time of the restoration, five patients were receiving orthodontic treatment, none had bruxism, and only one was a smoker (Table 1).

All participating patients signed an informed consent form. The study was approved by the Ethics Committee of the Faculty of Medicine and Dentistry of the University of Santiago de Compostela.

The restorations in this study were performed at a private clinic by the same operator. The clinical procedure used was as follows. Infiltration anesthesia was administered. A mouth opener was applied and gauze placed on the tongue. Nonimpregnated retraction cord was introduced in the gingival sulcus to improve access to this area. Wedges were placed in cases where interproximal contacts could interfere with the gingival adjustment of the crown. Additionally, it was necessary to separate adjacent teeth with wedges to compensate for the thickness of the matrix and avoid diastemas. The choice of crown was made according to form and size, aiming for a mesiodistal diameter that matched as closely as possible the tooth's gingival contour. The crown should normally be of a slightly larger size to compensate for the thickness of the matrix and the removal of material during polishing and esthetic recontouring. The crown was then trimmed back gingivally to obtain the correct height, and a hole was made in the palatal area of the matrix to allow any excess resin to escape when placing the crown on the tooth. The tooth was subsequently cleaned with pumice powder, taking care not to cause the gums to bleed. This was followed by etching with 37% orthophosphoric acid for 20 seconds, rinsing, and drying. Before curing the adhesive, it was verified that there was no contact with adjacent teeth. The adhesives used were Scotchbond 2 in eight cases, Prime & Bond 2.0 in four cases, Prime & Bond 2.1 in two cases, and Prime & Bond NT in seven cases.

The composites used were TPH Spectrum (Dentsply-Detrey, Konstanz, Germany) in 12 cases, Herculite XRV (Kerr, Orange, CA, United States) in seven cases, and Filtek A110 (3M ESPE, Seefeld Germany) in two cases. Medium or "body" opacity and a single color were used for all teeth. Frasaco® strip crowns (Franz Sachs & Co, Tettnang, Germany) were used in all cases. They have a thickness of 0.20 to 0.30 mm. Six different sizes are available

Table 1: *Teeth Included in the Study*

Age	Sex	Tooth	Anomaly	Year of Treatment	Adhesive	Composite	Plaque Index	Restorations Years
47	F	22	Conoid	1992	Scotchbond 2	XRV	0	18
31	M	12	Microdontia	1992	Scotchbond 2	XRV	2	18
32	M	22	Microdontia	1993	Scotchbond 2	XRV	2	17
16	M	21	Hypoplasia	1994	Scotchbond 2	TPH	0	16
16	M	11	Hypoplasia	1994	Scotchbond 2	TPH	0	16
16	M	31	Hypoplasia	1994	Scotchbond 2	TPH	1	16
16	M	41	Hypoplasia	1994	Scotchbond 2	TPH	1	16
22	F	22	Conoid	1994	Scotchbond 2	TPH	1	16
17	M	22	Hypoplasia	1995	Prime&Bond 2.0	TPH	0	15
17	M	12	Hypoplasia	1995	Prime&Bond 2.0	TPH	0	15
17	M	32	Hypoplasia	1995	Prime&Bond 2.0	TPH	1	15
17	M	42	Hypoplasia	1995	Prime&Bond 2.0	TPH	1	15
23	F	12	Microdontia	1997	Prime&Bond 2.1	XRV	0	11
23	F	22	Microdontia	1997	Prime&Bond 2.1	XRV	0	11
32	F	12	Microdontia	2000	Prime&Bond NT	TPH	0	10
33	F	22	Microdontia	2001	Prime&Bond NT	TPH	0	9
26	F	12	Conoid	2001	Prime&Bond NT	A110	0	9
19	M	43	Hypoplasia	2002	Prime&Bond NT	TPH	1	8
13	M	22	Conoid	2001	Prime&Bond NT	A110	2	4
16	M	22	Conoid	2006	Prime&Bond NT	XRV	2	4
16	M	12	Conoid	2006	Prime&Bond NT	XRV	2	4

for the upper incisors and three for the lower incisors. When filling the strip crown forms, it was important to avoid the formation of pores, especially in the corners of the incisal edge. Deformation of the crown by pressing too hard with the fingers was avoided. Any excess material escaping from the gingival area and palatal opening was removed with an explorer. The vestibular and palatal surfaces were light cured, and the crown was finally removed by breaking it with an explorer.

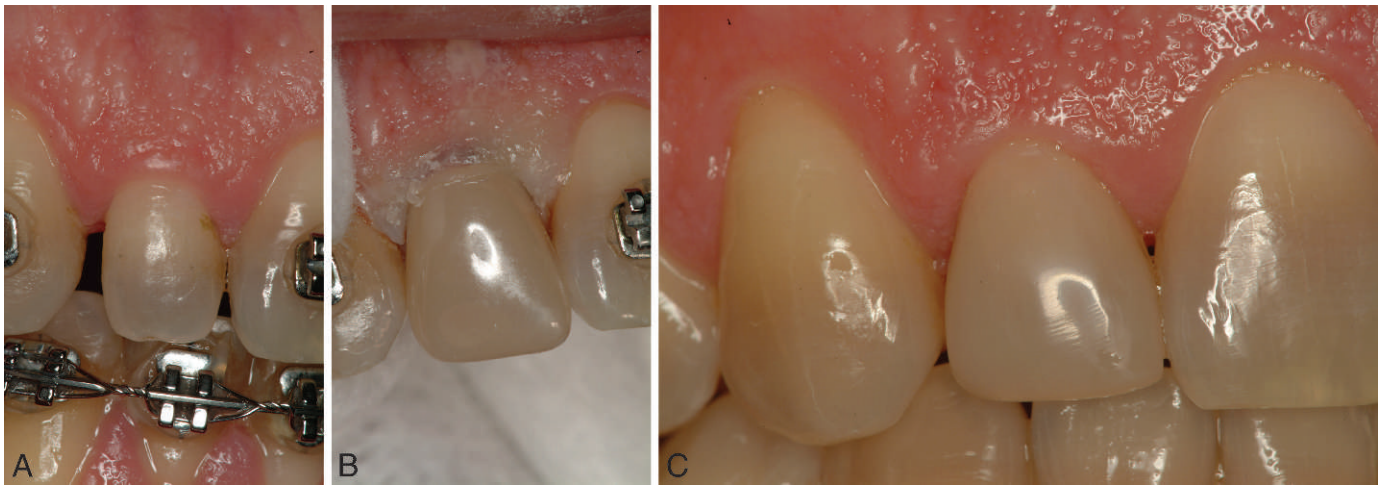

Figure 1. *(A) Tooth 12 with microdontia; the only procedure required is cleaning of the tooth with pumice powder. (B) The strip crown with composite filling is placed on the tooth; excess material is removed prior to polymerization. (C) Restoration is complete after one week.*

The gingival area was polished with multifluted tungsten burs, and an explorer was subsequently used to verify the uniformity of the surface and the absence of ridges. Strips were used in the interproximal area, and vestibular and incisal areas were finished and recontoured with discs. Occasionally, occlusal adjustment required removal of composite resin from the palatal area, possibly exposing the tooth surface.

Photographs were taken pre-, intra-, and postoperatively and during the examinations using a Medical Nikkor 120-mm lens (Fig. 1). The examinations were conducted by two external evaluators, postgraduate dental students specifically trained for this study. The postoperative evaluation was based on photographs taken seven days after completion of the restoration.

Restorations were assessed clinically in 2008 and 2010 (after 12.5 ± 4.6 years). The mean patient age was 35.0 ± 10.2 years. Dental health was evaluated by means of the Silness and Löe[33] plaque index in accordance with U.S. Public Health Service (USPHS) criteria modified by van Dijken[34,35] (Table 2). The SPSS (version 17, IBM, New York, USA) software was used for the statistical analysis and the Kaplan-Meier analysis for estimating survival curves.

RESULTS

Based on the analysis of the immediate postoperative photographs, the examiners gave a score of 0 in all categories of the USPHS criteria. In the examinations, the criteria obtaining statistically significant ($p<0.05$) values were anatomical form, surface roughness, marginal discoloration, and marginal adaptation (Table 3), with the greatest changes being found in the last three (43%, 62%, and 43%, respectively). However, all scores were in the "acceptable" range except for one case.

According to the USPHS criteria, a color change was observed in only one restoration, but there were no cases of secondary caries. The anatomical form of the crowns varied in six cases. Of these, four were due to locally reduced occlusal surface, one was due to slight undercontouring, and the last, which obtained a score of 2 (not acceptable), was due to fracturing where the dentin was exposed (Fig. 2).

Regarding marginal adaptation, in nine cases the margin was detectable with the explorer, showing an invisible gap (score of 1). As for marginal discoloration, in 11 cases there was slight staining that was removable by polishing, obtaining a score of 1, and two cases showed a stain that could not be polished out (score of 2). Nine restorations presented small pores and obtained a score of 1 for surface roughness (Figure 3 and 4).

Of the 21 composite crowns, only one had to be repaired after 10 years because of partial fracture. In three cases, the patients decided to change them for ceramic crowns at the end of the orthodontic treatment after four and 11 years (Figure 5).

Survival analysis after two years of follow-up was 95.2%, 88.9% after 10 years, and 75.2% after 11 years. From 11 years on, the survival rate remained constant.

Table 2: *Modified USPHS Criteria for Direct Clinical Evaluation of the Restorations*

Category	Score		Criteria
	Acceptable	Unacceptable	
Anatomical form	0		The restoration is continuous with tooth anatomy
	1		Slightly under- or overcontoured restoration; marginal ridges slightly undercontoured; contact slightly open (may be self-correcting); occlusal height reduced locally
		2	Restoration is undercontoured, dentin or base exposed; contact is faulty, not self-correcting; occlusal height reduced, occlusion affected
		3	Restoration is missing or traumatic occlusion; restoration causes pain in tooth or adjacent tissue
Marginal adaptation	0		Restoration is continuous with existing anatomic form; explorer does not catch
	1		Explorer catches, no crevice is visible into which explorer will penetrate
	2		Crevice at margin, enamel exposed
		3	Obvious crevice at margin, dentin or base exposed
		4	Restoration mobile, fractured, or missing
Color match	0		Very good color match
	1		Good color match
	2		Slight mismatch in color, shade, or translucency
		3	Obvious mismatch, outside the normal range
		4	Gross mismatch
Marginal discoloration	0		No discoloration evident
	1		Slight staining, can be polished away
	2		Obvious staining can not be polished away
		3	Gross staining
Surface roughness	0		Smooth surface
	1		Slightly rough or pitted
	2		Rough, cannot be refinished

Table 2: Continued.

Category	Score		Criteria
	Acceptable	Unacceptable	
	3	Surface deeply pitted, irregular grooves	
Secondary caries	0		No evidence of caries contiguous with the margin of the restoration
		1	Caries is evident contiguous with the margin of the restoration

DISCUSSION

The crowns were chosen according to size and shape, with a mesiodistal diameter that fitted as well as possible to the gingival contour of the tooth. Crowns should be slightly longer than the tooth to compensate for the thickness of the matrix and material removal during polishing and esthetic recontouring. Pore formation should be avoided, especially in the incisal angles, when filling the transparent crown. During insertion onto the tooth, distortion of the crown due to excessive finger pressure should also be avoided.

This procedure simplifies the fabrication of the restoration. No composite modeling or layering is required, just recontouring and polishing. A full coverage crown is completed in a single step. In orthodontic treatment where microdontia is very marked or associated with conoidism, applying this clinical procedure provides a greater facial surface for bracket adhesion[36] and simultaneously improves tooth esthetics. Therefore, in such cases a tooth cannot be treated when a bracket has already been attached.

In this work, all the restorations were full coverage composite crowns with no restoration-tooth interface on the visible surfaces, a fact that possibly enhanced the long-term esthetic results. Of the 21 composite crowns, 20 showed a good color match, and 15 preserved well their anatomical shape after 12.5 $\pm$ 4.6 years. In this study, 52% of the restorations were carried out in patients younger than 19 years of age. In the view of the authors, this technique could be the treatment of choice in patients still undergoing growth.

Peumans and others[31,32] studied direct composite restorations correcting form and position in the anterior sector. They reported that after five years, these restorations maintained a perfect color in 56% of cases, but only 20% retained their anatomical

Table 3: *USPHS Criteria Values at Follow-Up*[a]

USPHS Criteria Value	Anatomical Form	Marginal Adaptation	Color Match	Marginal Discoloration	Surface Roughness	Secondary Caries
0	15	12	20	8	12	21
1	5	9	1	11	9	0
2	1	0	0	2	0	—
3	0	0	0	0	0	—
4	—	0	0	—	—	—
	p=0.031*	p=0.004*	p=1.000	p=0.000*	p=0.004*	p=1.000*

[a] *At baseline, a score of 0 was given in all USPHS criteria. n=21.*
** Statistically significant* $p<0.05$.

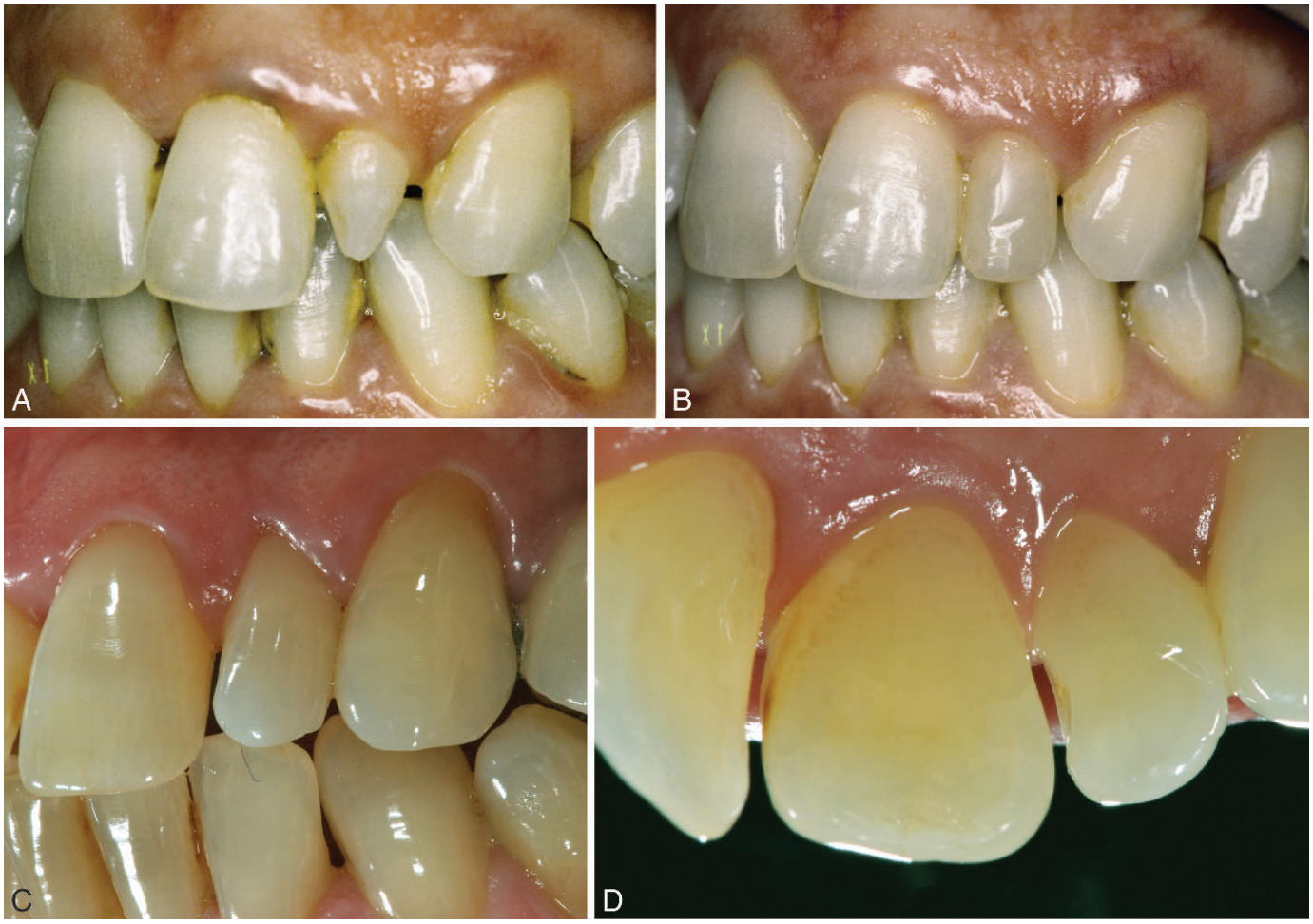

Figure 2. *(A) 47-year-old woman with conoid tooth 22. (B) Completed restoration; an indentation on the buccal surface can be observed, caused by excessive pressure exerted with the fingernail when placing the transparent strip crown. Composite used: Herculite XRV (Kerr). (C and D) State of the restoration after 18 years; this was the only case included in the study where an unacceptable score was obtained (anatomical form) due to fracturing of the restoration.*

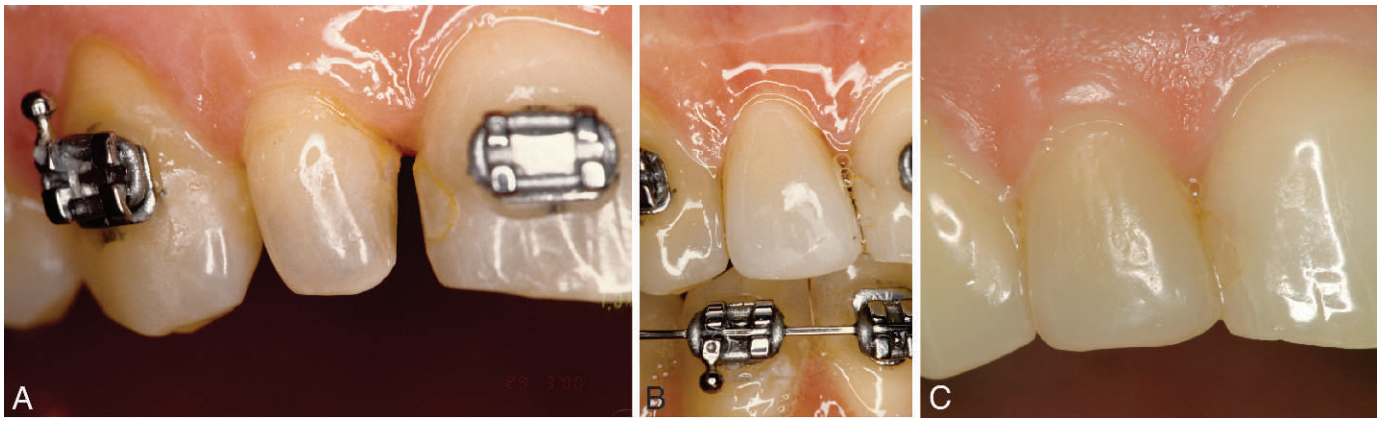

Figure 3. *(A) 23-year-old woman with microdontia in both upper lateral incisors. At the time of the restoration, the subject was undergoing orthodontic treatment. (B) Restoration is complete after one week. The composite used was Prodigy (Kerr). (C) Appearance of the restoration after 11 years.*

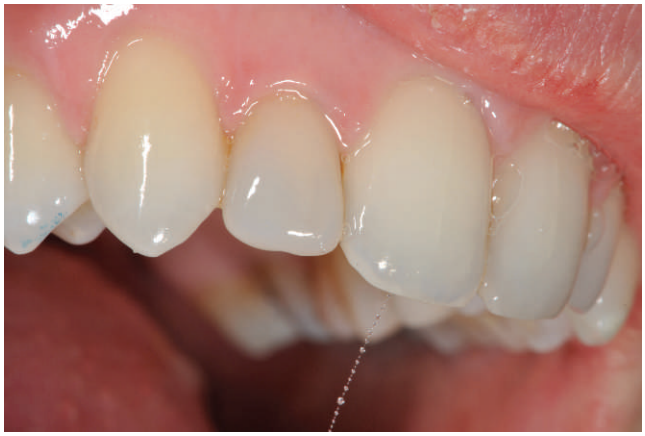

Figure 4. *Conoid tooth 12 restored with composite A110 (3M ESPE); appearance after 9 years. Observe the healthy gingival margin and the long-term esthetic behavior of the composite.*

form as a result of restoration material loss. They considered that the size of the restoration was a determining factor for esthetics. Moreover, they found that central incisors performed best, followed by canines and then lateral incisors. In 89% of their cases, they reported cervical region discoloration due to chip fractures, leading to loss of adaptation and consequent microfiltration. Additionally, they found no recurrence of caries, and only 5% of their restorations presented perfect margins.

No cases of secondary caries were observed in this study. An influencing factor could be that decayed teeth were not restored without prior cavity preparation. Marginal adaptation obtained scores of zero in 12 cases (57%) and one in the remainder. Marginal discoloration scored zero in eight cases (38%) and one in 11 cases (52%).

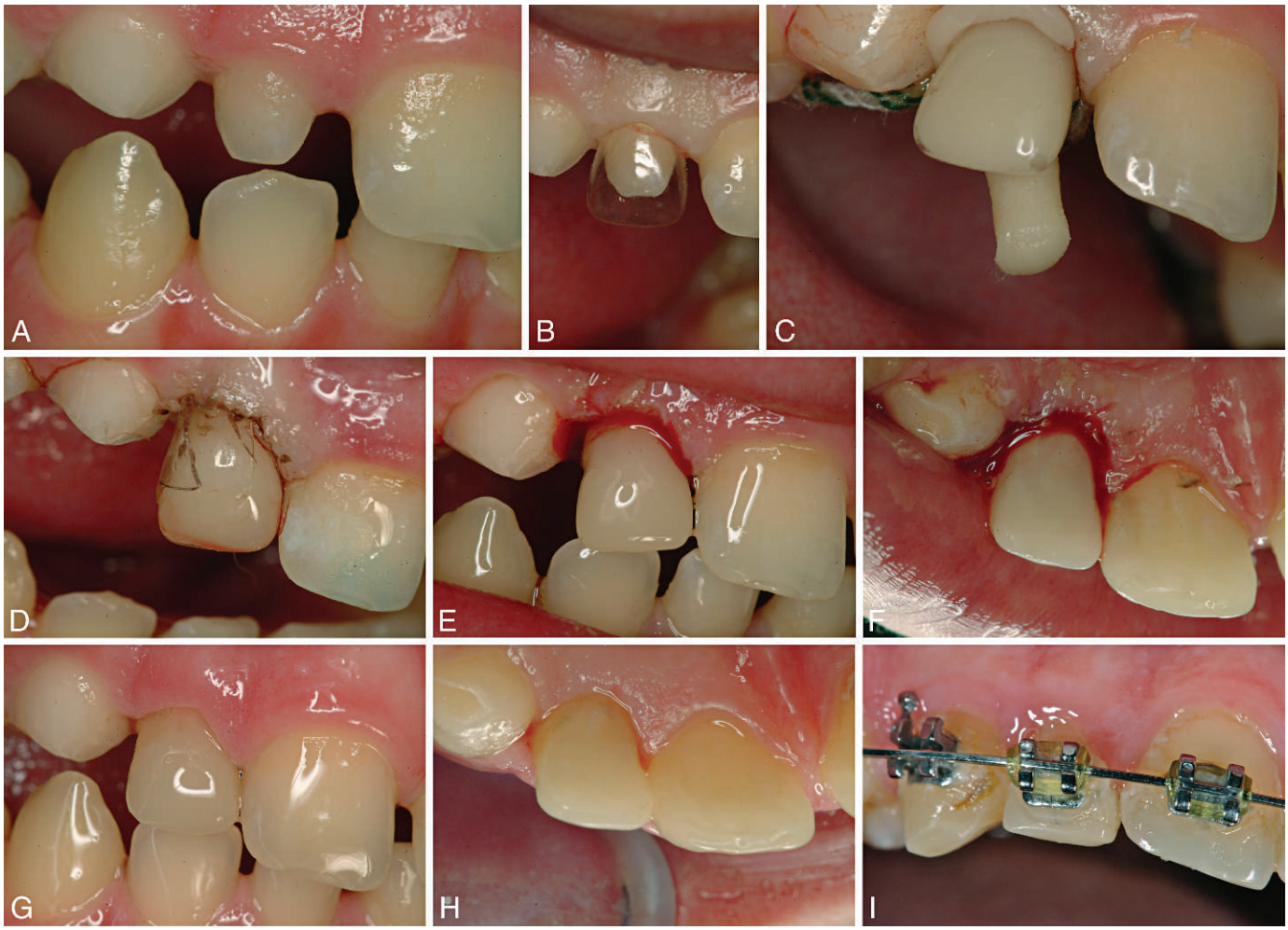

Figure 5. *(A) A 16-year-old male with microdontia of the upper right lateral incisor that will be orthodontically treated. (B) Try-in of the strip crown to evaluate the gingival fit, the mesiodistal diameter, and its size. (C) Insertion of the strip crown with composite, excess composite exits through the hole previously made in the palatal region of the matrix. (D) The strip crown is removed by breaking it through forcing a probe up from the gingival margin. (E and F) Gingival polishing with multifluted tungsten burs with non cutting tips. (G and H) Revision after seven days. The bracket is later placed. (I) Evaluation after four years.*

In a study comparing metal-ceramic crowns with composite reconstructions, it was concluded that while composites suffered more fractures, they were at least reparable, especially in the anterior sector. However, failures in metal-ceramic crowns tended to involve root-canal treatments and extractions. They also reported that there were no statistically significant differences in durability between the two types of restorations over a 10-year period.[37] In this study, only one fracture occurred and was easily reparable, requiring only composite to be added to the existing restoration. Endodontic therapy was not required later in any case.

Little exists in the literature regarding composite reconstruction of permanent teeth using a strip crown as a matrix.[29,30] However, there are several studies analyzing their performance in carious deciduous teeth[21-28] and teeth with amelogenesis imperfecta.[38] A review of the literature[24] concluded that while esthetic results are satisfactory, more prospective studies are needed to validate the technique. Kupietzky and others[22] used this technique on 112 carious deciduous incisors in 40 children and found none had lost the complete restoration after assessment at 18 months, the retention rate being 88%. Another study by the same authors[25] examined 145 restorations of deciduous upper incisors with caries. After three years, not a single restoration had been lost, and only two of them showed radiographic evidence of pulpal pathology. In another study, Ram and others[23] concluded that more than 80% survived successfully for at least two years and reported that the retention rate was lower in teeth with caries affecting three or more surfaces.

In some studies, hypoplasia in the anterior sector was treated using porcelain veneers,[10] porcelain crowns,[12] or metal-ceramic crowns.[14] Others, on the other hand, opted for composite restorations[15-18] but without the use of strip crowns.

This clinical procedure does not require any preparatory tooth drilling, and there is therefore no biological cost attached to it, and the adhesion is entirely on the enamel. It is a reversible, reparable, and modifiable treatment and moreover does not preclude the use of a different technique in the future. Because it is performed in a single clinical session, it could be considered a technique with hardly any contraindications.[30]

The authors believe that the long-term outcomes in the cases performed using this clinical technique are satisfactory. However, further studies with a larger sample size are needed in order to assess the longevity of these restorations for the indications described.

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Laboratory Research

Factors Affecting Dental Air-Turbine Handpiece Bearing Failure

M Wei • JE Dyson • BW Darvell

Objectives: **To investigate the influence of various factors on air-turbine handpiece bearing failure through developing standard protocols for testing the bearing longevity.**

Methods: **Groups of four air-turbine assemblies (Synea TA-98, W&H, Dentalwerk, Bürmoos, Austria) were subjected repeatedly to a full binary combinatorial set of operating conditions: with and without lubrication, simulated clinical loading, and corrosion protection, all with autoclaving, to the point of failure. A control set was lubricated only. Lubrication (Assistina, W&H), autoclaving (ST-Im30b, Eschmann Bros & Walsh, West Sussex, England), simulated clinical loading (0.56 N at 45° to the turbine axis, after autoclaving), and corrosion protection during autoclaving (magnesium sacrificial anode) were used as required. Free-running speed (Hz) and bearing resistance (μNm) were determined (Darvell-Dyson testing machine) at baseline and after every 10 cycles until turbine failure. Three-way analysis of variance (lubrication × loading × corrosion protection) of log(cycles to failure), with $\alpha = 0.05$, was used.**

Results: **All autoclaved turbines had failed by 560 cycles, while the controls failed at 960-1000 cycles. All three main effects were significant: load ($p<10^{-6}$), lubrication ($p<0.0002$), and corrosion protection ($p<0.02$), as was the interaction lubrication × loading ($p<10^{-6}$). No other interaction attained significance.**

Conclusions: **Running under load was the most important factor affecting bearing longevity. While autoclaving clearly has a detrimental effect, lubrication effectively increases longevity. A sacrificial anode may be economically worthwhile to extend life further, but low-load usage patterns, as generally instructed, are confirmed as beneficial.**

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