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Academy of Operative Dentistry • American Academy of Gold Foil Operators • Academy of R.V. Tucker Study Clubs

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Americn Academy of Gold Foil Operators

Distinguished Member Award

Tim Carlson

The American Academy of Gold Foil Operators has chosen Tim Carlson as its 2016 Distinguished Member. The selection committee has made an outstanding decision to a well deserving recipient.

Tim Carlson

Tim was born in Staten Island, but moved to the Midwest when he was 4. He grew up mostly in small towns, Stromsburg, Nebraska pop 1200 through elementary, and Essex Iowa, pop 850 through Junior in HS. Tim's dad was a pastor, so they moved every 5-8 years. He went to his senior year of HS in South Bend, Indiana, pop 100,000, so that was a big jump and quite exciting for a teenager. From there he attended North Park University in Chicago where he met a cute nursing student and they were married after her graduation. Tim attended Indiana University School of Dentistry for both his DDS and MSD in Operative Dentistry degrees. The family lived very frugally in a mobile home during dental school, so Ann was able to support both of them and pay for Tim's education while working as a nurse.

After graduating in 1978, with High Distinction from the Indiana University School of Dentistry, including nearly a clean sweep of the student awards, Tim and Ann left for a two year mission to Haiti.

Upon their return, Mel Lund and Mike Cochran offered him a teaching position in 1980. During the subsequent years, Mike and Tim would share an office together. As typical of dental students, they would frequent their offices with questions and concerns. As a team, they developed the habit of completing each other's sentences. Many times the students didn't know who they were talking to, but Mike says he was always the better looking responder.

Tim is now a full tenured professor at IUSD with joint appointments in the Department of Comprehensive Care and General Dentistry and the Department of Cariology. In his 36 years of teaching, he has had significant influence on over 4000 dental students. He is an active clinician and has practiced one day per week for 33 years.

The missions to Haiti continue and Tim serves as the director of the IUSD International Dental Service Learning Program. Under his supervision the dental students also provide care in Ecuador, Mexico, Guatemala, Kenya and the Lakota Indian Tribe in South Dakota.

He also serves as the managing editor of the journal, Operative Dentistry. He has held this position for 17 years.

Tim was inducted into our academy in 1983. He was the second to receive the Outstanding Clinician Award in 1990. He served as our president in 2000. Of all the academies that he is involved in, the one he greatly enjoys is our own AAGFO. He states, "we share great friends and work alongside the best technical clinicians in the world".

He is a member of the ADA, Indiana Dental Association and the Indianapolis District Dental Society. In addition, according to Mel Lund, Tim holds membership in a number of the other usual and unusual dental organizations both nationally and internationally. He is fluent in Creole and enjoys being a "funster and punster", handyman and computer "guru".

The AAGFO DM Award will be added to the many others that Tim has received, including the Indiana Dental Association Outstanding Faculty Member Award, the IUSD Trustees Teaching Award and the ADA Certificate of Recognition for Meritorious Service for Volunteer International Service.

Of all the decisions he's made including following Christ, he said his best one was asking Ann to marry him. She has been his lifetime partner for 41 years. Being a nurse, I'm sure, has been quite helpful on all those mission trips.

I know that you're all thinking that Tim goes quietly through life serving Christ, dentistry and others in his soft spoken, mild mannered way. But I must tell you that Tim has another (darker) side. All you have to do is put him behind the wheel of a go kart or the stick of a jet fighter simulator and the "animal" comes out. But to no surprise, he excels and distinguishes himself in these areas, too.

On weekends, Tim enjoys riding motorcycles with friends and driving with Ann in their Mazda Miata convertible. They put the top down, turn up the heat or AC and disregard the weather. They even drive in the rain staying above 30 mph and avoiding stop signs. They love to travel both in and out of the US. But lately their travels take them to Minneapolis to see the grandkids.

I would be remiss without mentioning one other mentor and dear friend, Ron Harris. Mike, Mel, Ron and Tim have had many adventures in teaching and continue to enjoy a long lasting friendship. He now joins them as our 2016 American Academy of Gold Foil Operators Distinguished Member.

Congratulations Tim or as they say in Creole, Konpliman and byen fe (well done).

Rick Nash

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Clinical Research

A Single-Blind Randomized Trial About the Effect of Hydrogen Peroxide Concentration on Light-Activated Bleaching

AP Mena-Serrano • E Garcia • I Luque-Martinez • RHM Grande • AD Loguercio • A Reis

Clinical Relevance

The use of light-emitting diode/laser light activation could be considered for in-office dental bleaching when low concentrations of hydrogen peroxide are used. However, the conflicting results between the two instruments used to evaluate color changes deserve further study.

SUMMARY

Objective: To compare the bleaching efficacy and tooth sensitivity (TS) of two hydrogen peroxide (HP) concentrations (20% and 35%) used for in-office bleaching associated or not with a light-emitting diode (LED)/laser light activation.

Method: Seventy-seven patients with a right maxillary canine darker than A3 were selected for this single-blind randomized trial. The participants were distributed in four groups: bleaching with 35% HP, 35% HP + LED/laser, 20% HP, and 20% HP + LED/laser. The anterior teeth were bleached in two sessions, using a 35% or 20% HP gel with a one-week interval. Each session had three applications of 15 minutes. For the light-activated groups, the LED/laser energy (Whitening Laser Light Plus, DMC) was employed according to the manufacturer's instructions. The color change was evaluated by subjective and objective methods. Participants recorded TS with five-point verbal and visual analog scales. Color change in ΔE was evaluated by analysis of variance and Tukey tests (α=0.05) and in ΔSGU with Kruskall-Wallis and Dunn test. The absolute risk of TS and TS intensity were evaluated by Fisher exact test and Kruskall-Wallis test, respectively (α=0.05).

Results: All groups achieved the same level of whitening, except for the 20% HP group, which

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showed the lowest degree of whitening in the subjective analysis. The use of light did not increase the absolute risk or intensity of TS. No significant difference among groups was observed when color changes were assessed with the spectrophotometer.

Conclusion: According to the value-oriented shade guide, the use of LED/laser light activation was able to increase the degree of whitening of the 20% HP group, but this association was not useful for the 35% HP gel. The spectrophotometer, however, did not detect significant differences among groups.

INTRODUCTION

In-office tooth bleaching is an effective technique that is commonly used in dental practice to improve the esthetics of discolored teeth.[1] It offers quicker whitening results with reduced applications than at-home bleaching techniques.[1,2] It also avoids the ingestion of the whitening product, the use of bleaching trays, and the gingival irritation that frequently occurs when such a procedure is undertaken.[3]

Within the in-office bleaching approach, hydrogen peroxide (HP) is the active molecule that acts as a strong oxidizing agent through the formation of free radicals, reactive oxygen molecules, and HP anions.[4] Some studies suggest that teeth are whitened by the oxidizing action of these radicals on the organic dentin matrix,[5,6] which results in constituents that reflect less light and thus create a whitening effect.[4,7-10]

However, tooth sensitivity (TS) is a remarkably common side effect that patients often report, mainly with in-office bleaching.[11-13] The mechanism that causes this painful outcome is still not fully understood,[14] but it seems to be associated with the ability of HP to penetrate the dental structure and reach the pulp chamber.[15-18] At a high concentration, HP and its related by-products can exceed the antioxidant capacity of the pulp cells and cause oxidative stress, leading to cell damage.[19-22]

In an effort to reduce this side effect, some manufacturers have released in-office bleaching gels with lower HP concentrations. Based on the assumption that the HP diffusion through dentin is proportional to the original concentration of the bleaching agent,[17,23] low-HP products would be less harmful to the living pulp cells. However, the whitening effect that is produced by the low-HP gels is inferior to the traditional 35% HP concentration.[2,24]

The association of light sources with low-HP gels may improve the bleaching outcome because light sources increase the oxygen dissociation rate and may reduce the time that is required for the bleaching protocol to occur.[25,26] Although the benefits of this association are still controversial,[25-31] a recent systematic review and meta-analysis of the literature[32] concluded that the advantages of light-activated bleaching with low-HP concentrations (i.e., 15%-20%) must still be investigated. Therefore, the aim of this study was to evaluate the impact of HP concentration at levels of 20% and 35% and light activation on color change and TS for in-office bleaching procedures. The null hypotheses that were tested postulated that 1) the different HP concentrations or light activation would not result in different degrees of color change and 2) the different HP concentrations or light activation would not result in various levels of the absolute risk of TS.

METHODS AND MATERIALS

This clinical study was approved (protocol 07943/10) by the Ethics Committee of the State University of Ponta Grossa. The protocol of this study was registered at clinicaltrials.gov under registration number NCT01231243. The experimental design followed the CONSORT statement.[33] Based on pre-established criteria, 76 volunteers from the cities of Ponta Grossa (Paraná, Brazil) and São Paulo (São Paulo, Brazil), were selected for this study. Two weeks before the bleaching procedures, all of the volunteers received a dental screening and a dental prophylaxis with pumice and water in a rubber cup, and they signed an informed consent form.

Study Design

This was a single-blind randomized clinical trial with an equal allocation rate. The study took place in the clinics of the schools at the State University of Ponta Grossa, Paraná, and the University of São Paulo, São Paulo, from June 2010 to June 2012.

Inclusion and Exclusion Criteria

The patients who were included in this clinical trial were men and women of any age who were in good general and oral health. These participants were recruited by wall announcements at both universities. The participants were required to have six maxillary and mandibular anterior teeth without caries lesions or restorations. The right maxillary

canine was shade A3 or darker, as judged by comparison with a value-oriented shade guide (VITA Classical Shade Guide, Vita Zahnfabrik, Bad Säckingen, Germany). Pregnant or lactating women and smokers were not included in this trial. Participants with anterior restorations, bruxism habits, severe internal tooth discoloration (tetracycline stains, fluorosis, pulpless teeth), and recessed or exposed dentin were also excluded. In addition, participants who took anti-inflammatories, analgesics, or antioxidants were not included in the study.

Sample Size Calculation

The primary outcome of this study was color change of the participants' teeth. A previous study[34] reported that two bleaching sessions with the product Whiteness HP Maxx 35% (FGM Dental Products, Joinville, SC, Brazil) without light activation produced a whitening effect of about 7 ± 2 SGUs. To detect a difference of 2 SGUs between the means of any pair of the study groups, with a power of 80% and an alpha of 5%, a minimum sample size of 17 patients per group was required. This threshold of perceptibility was based on the fact that "untrained" people, such as the patients, do not detect easily changes of one shade guide unit at the lighter end of the classical guide.

Random Sequence Generation and Allocation Concealment

Participants were randomly divided into four groups according to the combination of the main factors: HP (20% or 35%) and light activation (with or without). A third person who was not involved in the research protocol performed the randomization procedure by using computer-generated tables. We used blocked randomization (block sizes of 2 and 4) with an equal allocation ratio (www.sealedenvelope.com). Opaque and sealed envelopes containing the identification of the groups were prepared by a third person not involved in the study intervention.

Study Intervention

The participants and the operator were not blinded to the procedure, as the use of light could not be masked. However, the examiners who evaluated the color changes with the value-oriented shade guide (VITA Classical Shade Guide, Vita Zahnfabrik) were not aware of the allocation of the participants within the study groups.

This study employed the 35% HP Whiteness HP Maxx (FGM Dental Products). Its manufacturer also produced specifically for this study a 20% HP bleaching product that shares the same features as the 35% HP Whiteness HP Maxx. The light activation source used was light-emitting diode (LED)/laser equipment (Whitening Lase Light Plus, DMC Odontologica, São Carlos SP, Brazil). This light source is composed of a matrix of LEDs with a wavelength of 470 nm, three infrared laser diodes with a wavelength 830 nm, and a light intensity of 200 mW/cm^2.

Bleaching Procedure

We isolated the gingival tissue of the teeth to be bleached by using a light-cured resin dam (Top Dam, FGM Dental Products). In compliance with the manufacturer's directions, we applied the HP gels (20% and 35%) during three 15-minute applications for both groups. The products were refreshed every 15 minutes during the 45-minute application period. We performed two bleaching sessions with a one-week interval. The light-activated groups received an LED/laser energy (Whitening Lase Light Plus) according to the manufacturer's directions. The buccal surfaces were activated for 1 minute, and then the device was turned off for 2 minutes. This procedure was repeated three times for each 15-minute gel application. All of the participants were instructed to brush their teeth regularly (i.e., four times a day) with a fluoridated toothpaste (Sorriso Fresh, Colgate-Palmolive, São Paulo, SP, Brazil) that was provided by the study investigators.

Color Evaluation

The examiners recorded the color prior to the commencement of the study and at periods of one week and 30 days after the bleaching treatment by using subjective (value-oriented shade guide VITA Classical Shade Guide, Vita Zahnfabrik) and objective evaluation tools (Easyshade spectrophotometer, Vident, Brea, CA, USA).

For the subjective examination, the shade guide's 16 tabs were arranged from highest (B1) to lowest (C4) value, thus denoting the color A3 as number 9. The measurement area of interest for shade matching was the middle one-third of the buccal surface of the right maxillary canine. For calibration purposes, five participants whom we did not include in the study sample participated in the training phase. The two examiners, who were blinded to the allocation assignment, scheduled these patients for bleaching and evaluated their teeth against the shade guide at the baseline at one week and again 30 days after the procedure. The two evaluators presented superior

color-matching competency according to the ISO/TR 28642.[35] This means that they have an agreement of at least 85% (Kappa statistic) before beginning the study evaluation (85% of correctly matched pairs of tabs in shade guides). If disagreements occurred during the evaluation, they needed to reach a consensus before the participant was dismissed.

For the objective evaluation, a dense silicone Speedex (Coltène Whaledent AG, Altstaetten, Switzerland) was used to make a preliminary impression of the maxillary arch of the patients. The impression, which was extended to the maxillary canine, served as a standard color measurement guide for the spectrophotometer. A window was created on the labial surface of the silicone guide so that the right maxillary canine could be evaluated. The window was made by using a metallic device with well-formed borders as a radius of 3 mm.[28] Only one of the operators conducted the assessment on all of the participants by using Vita Easyshade before the procedure and one week and 30 days after the bleaching process.

The shade was determined by using the following parameters that were detected by the Easyshade device: L*, a*, and b*, in which L* represents the value from 0 (black) to 100 (white) and a* and b* represent the shade, where a* is the dimension along the red-green axis and b* is the dimension along the yellow-blue axis. The color comparison before and after the treatment was assessed through the differences (ΔE) that were observed between the two colors. Such differences were calculated with the formula $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.[2,28]

Tooth Sensitivity Assessment

The patients recorded their perception of TS during the first and second bleaching sessions according to two pain scales. A five-point rating scale (0 = *none*, 1 = *mild*, 2 = *moderate*, 3 = *considerable*, and 4 = *severe*)[28,34,36] and a visual analogue scale (VAS)[37-39] were employed in this study. The VAS scale is a 10-cm horizontal line that denotes the words *no pain* at one end and *worst pain* at the opposite end. We asked the subjects to indicate whether they experienced TS in the intervals: during the treatment up to 30 minutes and from 30 minutes up to 48 hours after the bleaching process. The worst score/numerical value that was obtained in both bleaching sessions was considered for statistical purposes.

If the patient scored zero (no sensitivity) in all time assessments from both bleaching sessions, this patient was considered to be insensitive to the bleaching protocol. In all other circumstances, the patients were considered to have sensitivity to the bleaching procedure. This dichotomization allowed us to calculate the absolute risk of TS, which represented the percentage of patients who reported TS at least once during treatment. We also calculated the overall TS intensity. In addition, the participants were instructed to record the painful tooth on an appropriate form.

Statistical Analysis

The analysis followed the intention-to-treat protocol and involved all of the participants who were randomly assigned. The statistician was blinded to study groups. The color change (primary outcome) was used to determine the efficacy of the bleaching treatment. The color change (ΔSGU and ΔE) between the baseline vs one week and baseline vs 30 days was calculated for each group. The ΔE were subjected to two-way repeated-measures analysis of variance (groups vs time as the main factors) and Tukey test. The ΔSGU data were subjected to Kruskall-Wallis and Dunn test.

We compared the study group's absolute risk of TS by using the Fisher exact test. The confidence interval for the effect size was calculated. The study groups' TS intensity at each assessment period (for both scales) was statistically analyzed with the Kruskal-Wallis test. Comparisons between assessment points (during and following the bleaching process), within each group, were performed by applying the Wilcoxon signed-rank test. The type of tooth that was reported to be the most painful was analyzed by Fisher exact test or the chi-square test. In all of the statistical tests, the alpha was preset at 0.05.

RESULTS

A total of 263 participants were examined; 77 participants were selected (Figure 1). The mean age (years) of the participants, the percentage of women vs men, and the baseline SGU are described in Table 1. One can observe comparable data among treatment groups by ensuring the comparability of baseline features. None of the patients discontinued the intervention or presented adverse effects during the intervention. No medication and/or desensitizer were necessary to be prescribed/applied in the participants from this study for the relief of bleaching-induced TS.

Color Change

Significant whitening was observed in the study groups under the subjective and objective evaluation

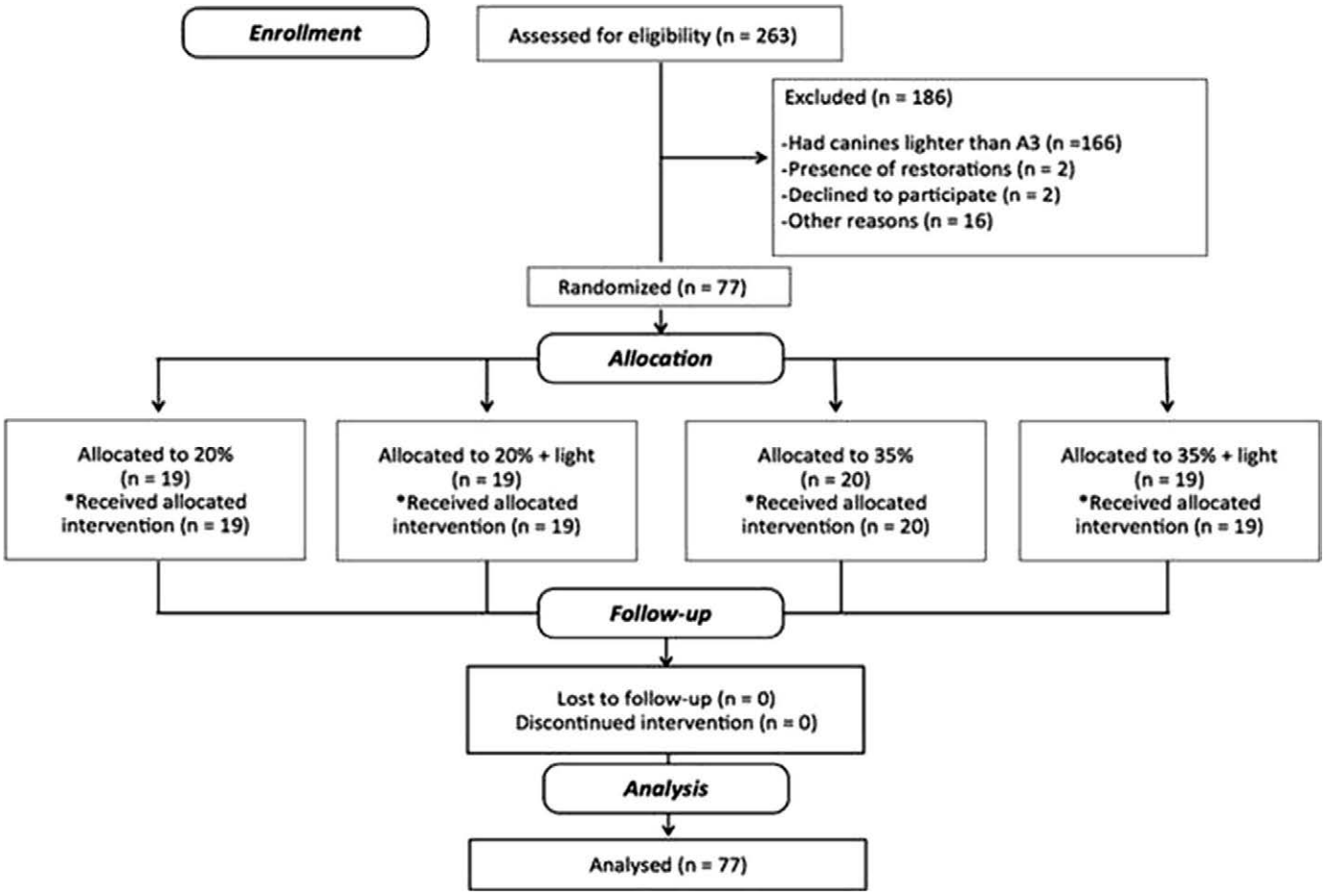

Figure 1. *Flow diagram of the trial including detailed information on the excluded participants.*

methods. A whitening of approximately 6 to 8 SGU and a ΔE of 12 to 14.5 was detected for the groups (Table 2). A lower amount of whitening was observed for the group 20% under subjective evaluation (Table 2; $p=0.006$). No significant difference among groups was detected under the objective evaluation (Table 2; $p>0.05$).

Tooth Sensitivity

With regard to the absolute risk of TS, no significant difference was observed between groups (Table 3; $p=0.4229$). With regard to the TS intensity, the statistical analysis of both pain scales detected no significant difference among the groups for the two assessment points (Table 4; $p>0.05$). Regarding the TS intensity of each group in the two assessment points, the pain during bleaching was statistically lower than that observed in the post-bleaching period for the 35% + light group in the five-point verbal scale and for the 35% group in the VAS (Table 4; $p<0.05$). Table 5 demonstrates that pain was rarely experienced in the premolars, while the anterior teeth experienced that symptom most often in the 35% groups ($p<0.03$).

DISCUSSION

Bleaching procedures have become the most conservative and popular techniques that are used to solve

Table 1: *Demographic Features of the Participants in Each Study Group*

Feature	20%	20% + Light	35%	35% + Light
Age, mean ± SD	22.9 ± 4.0	22.0 ± 4.4	23.0 ± 3.4	22.0 ± 3.6
Female, n (%)	13 (68)	12 (63)	13 (65)	12 (63)
Baseline SGU, median (25-75 percentile)	12 (11-14)	12 (11-12)	12 (10.5-15)	11 (9-12)

Table 2: *Means and Standard Deviations of ΔSGU and ΔE at the Different Assessment Points for the Treatment Groups*[a]

Color Evaluation	Assessment Time	Group			
		20%	20% + Light	35%	35% + Light
ΔSGU[b]	Baseline vs one week	6.7 ± 2.6 B	7.9 ± 1.8 A	8.0 ± 2.2 A	8.2 ± 1.2 A
	Baseline vs 30 days	6.1 ± 2.6 B	8.2 ± 1.3 A	8.2 ± 2.5 A	8.4 ± 1.4 A
ΔE[c]	Baseline vs one week	12.0 ± 4.9 a	11.8 ± 4.0 a	13.5 ± 2.3 a	14.5 ± 3.5 a
	Baseline vs 30 days	13.2 ± 4.1 a	11.8 ± 4.0 a	12.4 ± 3.7 a	14.1 ± 2.9 a

[a] *Comparisons are valid only within each color evaluation scale.*
[b] *Identical uppercase letters indicate statistically similar means (Kruskall-Wallis and Mann-Whitney test, α=0.05).*
[c] *Identical lowercase letters indicate statistically similar means (two-way repeated-measures analysis of variance and Tukey test, α=0.05).*

tooth discoloration. Consequently, many authors have focused their studies on determining the best clinical approach that produces the fewest side effects.[1,11,13,24,31,39,40] Although only a 10% carbamide peroxide product has the American Dental Association's seal of acceptance,[41] there are some other commercially available bleaching products (i.e., over-the-counter, at-home, and in-office bleaching) that have yielded successful outcomes.[1,12,31,42-44]

In the present investigation, color changes were evaluated in the canines instead of the incisors, as commonly done in bleaching studies. This procedure was already done in other studies in the literature.[45,46] A significantly stronger overall increase in whitening was observed in canines than in incisors,[45,46] which was 1.4 to 1.6 times more pronounced than incisors, probably because of their darker baseline color.[47-49] By measuring the color in canines, the recruitment of patients became easier as patients with baseline incisors A3 or darker is not common, while this is more frequent for canines.

All of the in-office bleaching techniques that were investigated in this clinical trial showed significant whitening after two bleaching sessions. Both the 35% and 35%+ light groups showed a color change of approximately eight SGU, which supports the outcome of previous studies that evaluated two bleaching sessions that each consisted of three 15-minute applications.[11,12,30]

Color matching is a complex issue because of the color discrimination ability that differs from individual to individual. The visual color selection depends on several factors, such as the shape, size, position, surrounding illumination, and background color. A variation in any factor may result in an altered perception of color.[50-53] To eliminate the subjective variables for shade analysis, improve the communication and reproduction of color, and increase the efficiency of esthetic restorative works, an instrumental color assessment has been developed. Some studies demonstrated that this equipment can be more accurate than human shade assessment.[53-55] Other studies explain that previous training in shade matching and clinical experience in dentistry play a more significant role in demonstrating shade-matching accuracy[56] and that clinical education and professional experience has a positive impact on the participants' ability to match correctly tooth shades.[50,52,57]

In the present study, a lack of agreement between the color evaluation tools was observed. Significant differences were observed with the subjective tool, while the objective color evaluation was not capable to detect such differences. Usually, the opposite is more common (i.e., differences in color change are not detected with the subjective tool), but it is when an instrumental method is employed.[26,58] When this occurs in a clinical study, authors are put in a dilemma about which data to discuss. Although the spectrophotometer gives accurate results, this instrument is yet not currently used in clinical practice. On the other hand, shade guide units are the most used tools for color evaluation in the clinicians' armamentarium. This controversy between these two instruments, however, should not be interpreted as a flaw of the present study. One should look at this as a need for future randomized clinical trials on this topic, and researchers should be encouraged to run further studies with this aim.

Irrespective of the instrument for color change, the results of this study are consistent with previous

Table 3: *Absolute Risk of Tooth Sensitivity (%) for the Treatment Groups Along With the 95% Confidence Interval (CI) for the Arch*[a]

Group	Tooth Sensitivity, %	95% Confidence Interval
20%	63 A	41-80
20% + light	73 A	51-88
35%	80 A	58-92
35% + light	85 A	64-95

[a] *Fisher exact test,* p=0.4229.

Table 4: *Medians and Interquartile Ranges of Tooth Sensitivity Intensity Reported by Patients at Different Assessment Times for the Treatment Groups in the Upper Arch Using the Five-Point Verbal Scale and the Visual Analog Scale*[a]

Assessment Time	Five-Point Verbal Scale				Visual Analog Scale			
	20%	20% + Light	35%	35% + Light	20%	20% + Light	35%	35% + Light
During bleaching up to 30 minutes	0 (0-1) aA	0 (0-1) aA	1 (0-2) aA	0 (0-1) aA	7 (0-22) aA	16 (0-56) aA	0 (0-27.5) aA	20 (0-52) aA
30 minutes up to 48 hours	0 (0-1.75) aA	0 (0-1.75) aA	1 (0-2) aA	2 (0-3) bA	0 (0-30) aA	17 (0-52) aA	36.5 (0-71) bA	45 (0-57) aA

[a] *Each pain scale was individually analyzed. At each treatment, the two periods were compared with Wilcoxon signed rank (α=0.05), and differences are represented by different lowercase letters. For each assessment time, the treatments were compared with Kruskall-Wallis and Mann-Whitney U test, and the differences are represented by different uppercase letters.*

studies that revealed that the use of light irradiation did not improve the bleaching efficacy of 35% HP.[27-32,59] At first glance, this result seems to contradict the well-known finding that light can heat and photo-activate the HP, thereby increasing the rate of the oxygen decomposition to produce oxygen-free radicals.[60] In fact, from chemical theories, one knows that, in simplest chemical reactions, the highest concentration of reactants raises collisions per unit time. Hence, the reaction rate increases. However, if the reaction is complex and involves a series of consecutive steps, there might be a limit to which the increased concentration leads to faster reaction rates. We hypothesize that 35% HP alone already produces enough free radicals to oxidize the organic component of dentin, and thus, the increase in free radicals that are produced by the light activation might be useless. Consequently, the further increases in HP radicals that are produced by light activation do not lead to faster bleaching because of the presence of unknown rate-determining steps in the oxidizing mechanism of tooth bleaching.

This concept is strengthened by the findings of the 20% group observed in the subjective evaluation. On average, the use of 20% HP yielded a whitening of six SGUs, which was statistically lower than the mean eight SGUs detected in the other groups that led us to partially reject the first null hypothesis. In this case (20% group), it seems that the limiting factor of the oxidizing reaction rate was the amount of free radicals; thus, the association with light, which likely increases the amount of free radicals, produced a faster reaction rate and a whitening degree that was similar to that of the 35% HP gel associated or not with light.

These results corroborate previous clinical studies.[25,26] A detailed analysis of the literature reveals that when the association of light effectively increased the bleaching rate, low-HP concentrations were employed. For instance, Tavares and others[25] showed favorable results for the use of a light source associated with a 15% HP gel applied in a single one-hour session. Similarly, Ontiveros and Paravina[26] observed improved whitening when a 25% HP was irradiated with a light source during two 45-minute in-office bleaching sessions.

One should not interpret, however, that the use of low-HP concentrations could not achieve the same level of whitening that is produced by the other techniques investigated in this study. In an *in vitro* study, Sulieman and others[2] established a direct correlation between the concentration of the HP gel and the number of applications needed to achieve a satisfactory whitening effect. Thus, another clinical session of 20% HP alone would probably produce a similar outcome to the other in-office bleaching techniques, but requires further study.

Bleaching-induced TS is a common side effect that occurs during bleaching treatments,[11-13] and the present study is in agreement with such observations from the previous literature. The risk of TS in this study varied from 63% to 85%, and it is within the range reported in the literature. Although the reported risk of TS is variable in clinical trials, it very often exceeds 50%. A recent study that

Table 5: *Number of Patients (%) Who Reported Tooth Sensitivity at Least Once in the Different Tooth Types, in the Maxillary Arch*[a]

Tooth Type	Group			
	20%	20% + Light	35%	35% + Light
Central incisors	7 (37) a	7 (37) a	6 (32) a	9 (45) a
Lateral incisors	6 (32) a	11 (56) a	11 (56) a	7 (35) a
Canines	6 (32) a	8 (42) a	8 (42) a	10 (50) a
Premolars	2 (11) a	4 (21) a	2 (11) b	2 (10) b
p-value[a]	0.274	0.1382	0.002	0.03

[a] *Fisher exact or chi-square tests (α=0.05). Comparisons are valid only within columns. The same lowercase letters indicate statistically similar groups.*

evaluated the individual patient data of 11 clinical trials on bleaching produced a more accurate estimate of these risks. For in-office bleaching, the risk of TS was reported to be 62.9% (95% confidence interval [CI] 56.9-67.3), which was not very different from that reported for at-home bleaching (51% [95% CI = 41.4-60.6]).[49] Although the risk of TS was reported to be similar between in-office and at-home bleaching, the intensity of TS was very different between these bleaching protocols. On a 0 to 4 pain scale, the overall mean intensity of bleaching-induced TS for in-office bleaching was 2.8 ± 2.9, while that for at-home bleaching was 0.5 ± 0.9.[49]

In the present study, we could not detect, however, a difference between bleaching protocols. As a result, we did not reject the second null hypothesis. Although some studies reveal that light activation produces more persistent bleaching-induced TS,[11,61,62] this study failed to show such a trend. However, this finding should be interpreted with caution, as we have not calculated the sample size of the present study to detect clinical and relevant changes in the bleaching-induced TS but rather on color change. Thus, we cannot rule out the fact that a true difference between the study groups may exist.

A few studies in the literature have attempted to investigate what tooth type is the most sensitive to the bleaching protocol.[31,63] In this study, anterior teeth (incisors and canines) were reported to be more painful than premolars, which is in agreement with previous studies.[31,63] In a review of the literature, Haywood[64] reported that bleaching-induced TS usually affects the smaller teeth, such as the maxillary laterals and the mandibular incisors. These reports are in agreement with a recent histological study of pulp tissue after in-office bleaching.[22] Notable damage of the pulp tissue was observed in the incisors but not in premolars.[22] The thinner enamel and dentin layers of the incisors, in comparison with premolars, may allow the easy passage of HP to the pulp; thus, there is less time for the production and release of protective enzymes against damage by HP.[22]

Lastly, we should mention the limitations of the present study. Most of the participants who participated in this study were young, which prevents us from generalizing the results of this study to older patients. The conflicting results between the two instruments used for color change evaluation highlight the need for further research on this topic.

CONCLUSION

Within the limitations of the present study, the treatment with supplementary light only showed significantly higher degree of whitening when used with 20% HP gel when evaluated with the value-oriented shade guide unit. No significant difference in color change was reported when this outcome was evaluated with the spectrophotometer.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects' oversight committee guidelines and policies of State University of Ponta Grossa. The approval code for this study is: 07943/10.

Conflict of Interest

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

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Six-month Follow-up of Cervical Composite Restorations Placed With a New Universal Adhesive System: A Randomized Clinical Trial

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Clinical Relevance

At six months the universal adhesive Xeno Select didn't fulfill the American Dental Association criteria for full approval when using all of the bonding strategies suggested by the manufacturer. Its behavior depends on the bonding strategy used.

SUMMARY

Purpose: The objective of this double-blind, randomized clinical trial was to evaluate the six-month clinical performance of a new universal adhesive (Xeno Select, Dentsply) in non-carious cervical lesions (NCCLs) using two evaluation criteria: World Dental Federation (FDI) and the US Public Health Service (USPHS).

Methods and Materials: A total of 124 restorations were randomly placed in 31 patients according to the following groups: ER-D = etch-and-rinse/dry dentin; ER-M = etch-and-rinse/moist dentin; SE-et = selective enamel etching; and SET = self-etch. The composite resin EVOLUX (Dentsply) was placed incrementally. The restorations were evaluated after one week (baseline) and at six months using the FDI and USPHS criteria. Statistical analyses were performed using appropriate tests (α=0.05).

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Results: Fifteen restorations were lost or fractured at six months (one for ER-D, three for ER-M, five for SE-et, and six for SET) ($p>0.05$ at six-month recall). When ER (ER-D and ER-M) was compared with SE (SE-et and SET) there was a significant difference in the retention rate after six months ($p=0.001$). Marginal staining and postoperative sensitivity to air were only observed in three (one for ER-M and two for SET) and two restorations (two for ER-D) in both evaluation criteria ($p>0.05$), respectively. Forty-seven restorations were considered to have minor discrepancies in marginal adaptation at the six-month recall using the FDI criteria (13 for ER-D, 10 for ER-M, 11 for SE-et, and 13 for SET; $p>0.05$ between groups). However, for all groups, a significant difference was detected when baseline and six-month data were compared ($p<0.05$).

Conclusions: The six-month clinical behavior of Xeno Select Universal Adhesive depends on the bonding strategy used. The universal adhesive did not fulfill the American Dental Association criteria for full approval when used in the self-etch mode.

INTRODUCTION

Adhesive systems led to a significant revolution in restorative dentistry, which has provided dentists with the opportunity to offer pleasing, direct esthetic restorations, which are typically able to be completed during a single clinical appointment and with satisfactory mechanical properties.[1]

Etch-and-rinse (ER) adhesive systems require previous dissolution of the mineral components in enamel and dentin with phosphoric acid before monomer infiltration into the dental substrates. For most dental adhesives, the depth and pattern of the etching play a significant role in the enamel bond strength values,[2,3] and in dentin, prior dentin demineralization with phosphoric acid is essential to expose collagen fibrils for resin infiltration.[4,5] Three-step and two-step ER adhesive systems have been extensively evaluated *in vitro* and *in vivo*, and, in general, the results have been quite satisfactory.[6-8] Regardless of the number of steps, the main disadvantage of the ER system is that there is a risk of collagen fibril collapse during drying of the demineralized dentin, which leads to a consequent decrease in bond strength.[1] To avoid this, demineralized dentin should be kept moist, which is a difficult task clinically since adequate moisture depends on the solvent employed in the material[9] and on the individual's interpretation of the manufacturer's directions.[10] This complex bonding protocol of ER adhesives may lead to clinical mistakes,[1,9,10] which may be clinically translated as postoperative sensitivity or adhesive failure after some years of function.

On the other hand, self-etch (SE) adhesive systems have a simpler bonding protocol.[11] The demineralization of the dental substrates is produced by a nonrinsing acidic primer, and except in the case of some SE systems,[12] the whole extension of the demineralized dentin depth is impregnated by resin monomers. Unfortunately, SE adhesives produce relatively low bond strength values and inferior marginal adaptation to enamel when compared to ER systems.[13,14] SE adhesives do not produce a retentive etching pattern on enamel, such as that produced by phosphoric acid, which may produce restorations with higher rates of marginal discoloration—a clinical problem in anterior restorations. Although selective etching of enamel margins prior to the application of SE adhesives[15,16] can minimize this limitation, an accidental dentin etching may occur and jeopardize bonding efficacy to dentin.[17,18]

Considering that both adhesive strategies have advantages and limitations, their selection may vary depending on the clinical scenario. In light of this fact, more versatile adhesives, called multimode or universal adhesives, were developed recently. Theoretically, these universal adhesives are indeed SE adhesives that can be used with or without acid etching in both enamel and dentin.[19-23] Different research centers have shown that universal adhesives applied on dentin in both ER or SE strategies have high bond strength values,[20,21] and this can be attributed to the acidic monomers that promote chemical bonding to a tooth.[21,22]

As these materials are relatively new in the literature, few clinical trials have been conducted to evaluate their clinical performance.[19,24,25] Most of the clinical data available are related to 10–methacryloyloxydecyl dihydrogen phosphate10-MDP–containing adhesives. According to an *in vitro* study,[23] only universal adhesives that contain 10-MDP showed high and stable dentin bonding after six months of water storage, but such a finding warrants clinical validation.

To the authors' knowledge, no clinical study has so far evaluated the clinical performance of a universal adhesive that contains functional monomers other than 10-MDP. Therefore, the objective of this double-blind, randomized clinical trial was to evaluate the

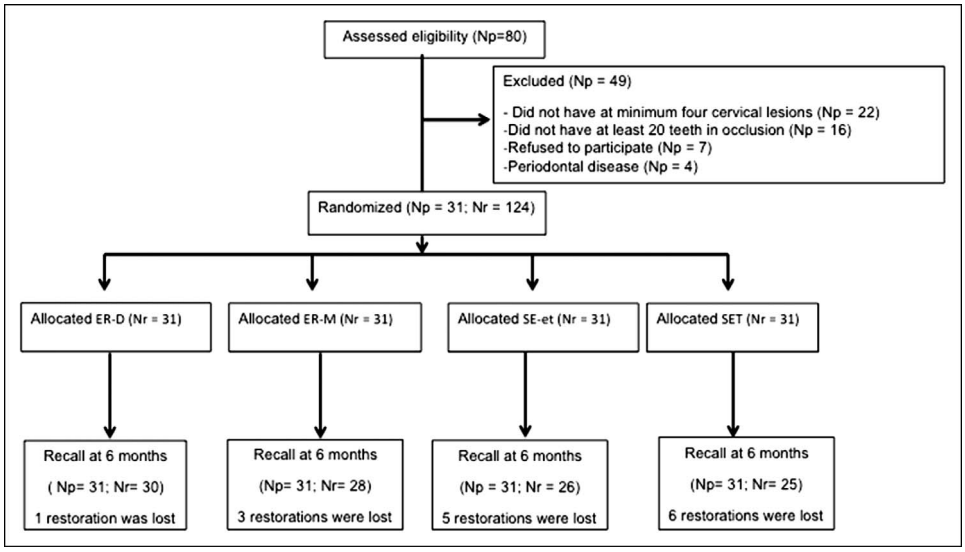

Figure 1. *Flow chart of the study design.*

influence of different application strategies on the clinical behavior of a new universal multimode adhesive (Xeno Select adhesive system) placed in noncarious cervical lesions (NCCLs) over the course of six months using the evaluation criteria of the World Dental Federation (FDI) and the US Public Health Service (USPHS). The null hypothesis tested was that bonding to NCCLs using the SE strategy—regardless of whether or not it is associated with selective enamel etching or using the ER strategy—applied on dry or moist dentin would result in similar retention levels over six months of clinical service.

METHODS AND MATERIALS

Study Design

The experimental design followed the Consolidated Standards of Reporting Trials (CONSORT) statement.[26] This was a randomized, double-blind clinical trial, registered in the REBEC clinical registry under protocol RBR-4wh4sh. The study was carried out in the clinic of the School of Dentistry at the local university from June 2014 to November 2014. All participants were informed about the nature and objectives of the study, but they were not aware of which tooth received the specific treatments under evaluation.

Participant Recruitment

The local Ethics Committee on Investigations Involving Human Subjects reviewed and approved the protocol and issued a consent form for this study (protocol 800.273/14). Written informed consent was obtained from all participants prior to starting the treatment.

Sample Size Calculation

The sample size calculation was based on the retention rate of Xeno III, IV, and V, and predecessors of the Xeno Select adhesive system from the same manufacturer (Dentsply, DeTrey, Konstanz, Germany). The retention rate was reported to be 96% at 18- to 24-month follow-up.[27-30] Using an α of 0.05, a power of 80%, and a two-sided test, the minimal sample size was 31 restorations in each group in order to detect a difference of 25% among the tested groups.[31]

Eligibility Criteria

A total of 80 participants were examined by two calibrated dental students to determine if they met the inclusion and exclusion criteria (Figure 1). Those who qualified for the study were recruited in the order in which they reported for the screening session, thus forming a convenience sample.

The evaluations were performed using a mouth mirror, an explorer, and a periodontal probe. Participants had to be in good general health, at least 18 years of age, have an acceptable oral hygiene level, and present with at least 20 teeth under occlusion. Participants were required to have at least four NCCLs to be restored in four different teeth. These lesions had to be noncarious, nonretentive, and deeper than 1 mm and had to involve both the enamel and dentin of vital teeth without mobility. The cavo-surface margin could not involve more that 50% of enamel.[32]

Table 1: *Dentin Sclerosis Scale*[33]

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

All patients were given oral hygiene instructions before the operative treatment was performed. Patients with extremely poor oral hygiene, severe or chronic periodontitis, or heavy bruxism habits were excluded from the study.

Randomization and Allocation Concealment

A staff member not involved in the research protocol performed the randomization process with computer-generated tables. Details of the allocated groups were recorded on cards contained in sequentially numbered, opaque, sealed envelopes. Opening the envelope on the day of the restorative procedure revealed the allocation assignment. The operator was not blinded to group assignment when administering interventions; however, participants and evaluators were blinded to the group assignment.

Interventions: Restorative Procedure

All of the patients selected for this study received dental prophylaxis with a suspension of pumice and water in a rubber cup and signed an informed consent form two weeks before the restorative procedures were initiated.

The degree of sclerotic dentin from the NCCLs was measured according to the criteria described by Swift and others[33] (Table 1). The cavity dimensions in millimeters (height, width, and depth), the geometry of the cavity (evaluated by profile photograph and labeled at $<45°$, 45°-90°, 90°-135°, and $>135°$), the presence of an antagonist, and the presence of attrition facets were observed and recorded. Preoperative sensitivity was also evaluated by applying air for 10 seconds from a dental syringe placed 2 cm from the tooth surface and with an explorer. These features were recorded to allow comparison of the baseline features of the dentin cavities among experimental groups.

In order to calibrate the restoration procedure, the study director placed one restoration of each group in order to identify all steps involved in the application technique. Then the two operators, who were resident dentists with more than five years of clinical experience in operative dentistry, placed four restorations, one in each group, under the supervision of the study director in a clinical setting. The restoration failures were shown to the operators prior to starting the study. At this point, the operators were considered calibrated to perform the restorative procedures.

The calibrated operators restored all teeth under the supervision of the study director. All participants received four restorations, one of each experimental group, in different lesions previously selected according to the inclusion criteria.

Before restorative procedures, the operators anesthetized the teeth with a 3% mepivacaine solution (Mepisv, Nova DFL, Rio de Janeiro, RJ, Brazil) and cleaned all lesions with pumice and water in a rubber cup, followed by rinsing and drying. Then shade selection was made using a shade guide. Following the guidelines of the American Dental Association (ADA),[34] no additional retention or bevel was prepared.

A rubber dam was placed, and then the NCCLs received the Xeno Select adhesive system (also known in some countries as Prime & Bond One Select, Dentsply) applied in different modes: an ER approach, keeping the dentin dry (ER-D) or moist (ER-M), and a SE approach with (SE-et) or without (SET) selective enamel etching, which defined the four different groups. The compositions, application modes, and batch numbers are described in Table 2. Some details of the restorative procedures include the following:

- Etch-and-rinse/dentin dry group (ER-D)—The 37% phosphoric acid (Dentsply, Rio de Janeiro, Brazil) was applied on enamel (30 seconds) and dentin (15 seconds). Then cavities were rinsed thoroughly for 30 seconds and slightly air-dried for five seconds to dry dentin without causing dentin dehydration. The adhesive system was applied sufficiently, wetting all cavity surfaces uniformly, and was gently agitated on the entire enamel and dentin

Table 2: *Materials (Batch No.), Compositions, and Application Mode*

Materials (Batch No.)	Composition	Application Mode[a]
Xeno Select; Dentsply, DeTrey, Konstanz, Germany (1401001212)	Bifunctional acrylate, acidic acrylate, functionalized phosphoric acid ester, water, tertiary butanol, initiator, stabilizer	Etch & Rinse (ER-D and ER-M) 1. Apply etchant in enamel (30 s) and dentin (15 s), rinse for 30 s, air-dry to remove excess water; 2. Keep dentin dry (ER-D)[b] or moist (ER-M); 3. Apply the adhesive for 20 s with vigorous agitation, gently air thin for 5 s. Light-cure for 10 s.
		Selective enamel etching (SE-et) 1. Apply etchant for 15 s in enamel, rinse for 15 s, air-dry to remove excess water; 2. Keep dentin dry (do not overdry); 3. Apply the adhesive for 20 s with vigorous agitation, gently air thin for 5 s. Light-cure for 10 s.
		Self-etching (SET) 1. Do not use etchant; 2. Keep dentin dry (do not overdry); 3. Apply the adhesive for 20 s with vigorous agitation, gently air thin for 5 s. Light-cure for 10 s.
37% Tooth Conditioner Gel; Dentsply, Rio de Janeiro, Brazil (941685F)	Phosphoric acid, surfactant, Aerosil 200, deionized water, and pigment	With the aid of the applicator tip, apply the acid conditioner to the dental structures in question, keeping it in contact with enamel for at least 15 s and with dentin for no more than 15 s. After conditioning, wash the surfaces with plenty of water for at least the same amount of time as used for the conditioning, vacuuming with a saliva sucker.
EvoluX; Dentsply, Rio de Janeiro, Brazil (Shade A3–681887E; Shade A3.5–697997E; Shade A1–673729E)	Silanized barium aluminum borosilicate glass, silanized barium fluoro aluminum boro silicate glass, dimethacrylate Bis-GMA and Bis-EMA, nanoparticulated silica, dyes, photoinitiators, inhibitors	Insert in the cavity increases of up to 2 mm and light-cure each area of the surface of the restoration with a dental curing light appliance light power of 1200 mW/cm^2) for 30 s.

Abbreviations: Bis-EMA, bisphenol A dimethacrylate; Bis-GMA, bisphenol A glycidyl dimethacrylate.
[a] *According to the manufacturer's instructions.*
[b] *Manufacturer does not indicate application in dry dentin.*

surface for approximately 20 seconds, according to the manufacturer's recommendations (Table 2). Then the adhesive was evaporated by gentle air thinning for five seconds and light-cured (Radii Cal, SDI, Victoria, Australia) for 10 seconds (1200 mW/cm^2).

- Etch-and-rinse/dentin moist group (ER-M)—All of the restorative procedures were similar to those described for the ER-D group. The only difference was that after acid rinsing, dentin was kept visibly moist.
- Selective enamel etch group (SE-et)—The 37% phosphoric acid (Dentsply) was applied for 15 seconds only on enamel. Then it was rinsed thoroughly for 15 seconds and air-dried for five seconds, until the dentin was dried but not over dried. Following this, the adhesive was applied similarly to the way it was applied to the ER-D group.
- Self-etch group (SET)—The adhesive system was applied as described in the ER-D group, without any previous acid etching. Then the adhesive was evaporated by gentle air thinning for five seconds and light-cured (Radii Cal, SDI) for 10 seconds (1200 mW/cm^2).

After adhesive application, EvoluX (Dentsply) resin composite was used in up to three increments, each one being light cured (Radii Cal, SDI) for 30 seconds. The restorations were finished immediately with fine and extra-fine #2200 diamond burs (KG Sorensen, Barueri, SP, Brazil). Polishing was performed with rubber points (Enhance, Dentsply) one week after placement of the restorations.

Clinical Evaluation

Two experienced and calibrated dentists, not involved with the restoration procedures and therefore blinded to the group assignment, performed the evaluation. For training purposes, the examiners observed 10 photographs that were representative of each score for each criterion. They evaluated 10 to 15 patients each on two consecutive days. These subjects had cervical restorations but were not part of this project. An intraexaminer and interexaminer agreement of at least 85% was necessary before beginning the

Table 3: *World Dental Federation (FDI) Criteria Used for Clinical Evaluation*[36]

	Esthetic Property	Functional Properties		Biological Properties	
	1. Staining Margin	2. Fractures and Retention	3. Marginal Adaptation	4. Postoperative Hyper-Sensitivity	5. Recurrence of Caries
1. Clinically very good (A)	1.1 No marginal staining	2.1 Restoration retained, no fractures/cracks	3.1 Harmonious outline, no gaps, no discoloration	4.1 No hypersensitivity	5.1 No secondary or primary caries
2. Clinically good (B) (after correction very good)	1.2 Minor marginal staining, easily removable by polishing	2.2 Small hairline crack	3.2.1 Marginal gap (50 μm) 3.2.2 Small marginal fracture removable by polishing	4.2 Low hypersensitivity for a limited period of time	5.2 Very small and localized demineralization; No operative treatment required
3.Clinically sufficient/ satisfactory (C) (minor shortcomings with no adverse effects but not adjustable without damage to the tooth)	1.3 Moderate marginal staining, not esthetically unacceptable	2.3 Two or more or larger hairline cracks and/or chipping (not affecting the marginal integrity)	3.3.1 Gap $<$ 150 μm not removable 3.3.2. Several small enamel or dentin fractures	4.3.1 Premature/ slightly more intense 4.3.2 Delayed/weak sensitivity; no subjective complaints, no treatment needed	5.3 Larger areas of demineralization, but only preventive measures necessary (dentin not exposed)
4. Clinically unsatisfactory (D) (repair for prophylactic reasons)	1.4 Pronounced marginal staining; major intervention necessary for improvement	2.4 Chipping fractures that damage marginal quality; bulk fractures with or without partial loss (less than half of the restoration)	3.4.1 Gap $>$ 250 μm or dentin/base exposed 3.4.2. Chip fracture damaging margins 3.4.3 Notable enamel or dentin wall fracture	4.4.1 Premature/ very intense 4.4.2 Extremely delayed/weak with subjective complaints 4.4.3 Negative sensitivity intervention necessary but not replacement	5.4 Caries with cavitation (localized and accessible and can be repaired)
5. Clinically poor (E) (replacement necessary)	1.5 Deep marginal staining not accessible for intervention	2.5 (Partial or complete) loss of restoration	3.5 Filling is loose but *in situ*	4.5 Very intense, acute pulpitis or nonvital; Endodontic treatment is necessary and restoration has to be replaced	5.5 Deep secondary caries or exposed dentine that is not accessible for repair of restoration
Acceptable or not acceptable (n, %, and reasons	Esthetic criteria	Functional criteria		Biological criteria	

evaluation.[35] After recording the parameters during evaluation using a standardized paper case report form, the evaluation paper had to be sent back to the research staff, so that evaluators were blinded to group assignment during follow-up recalls.

The restorations were evaluated by FDI[36] and the classical USPHS criteria (adapted by Bittencourt and others[37] and Perdigão and others[7]) at baseline and after six months of clinical service. Only the clinically relevant measures for evaluation of the performance of adhesives were used and scored (Tables 3 and 4). The primary clinical endpoint was restoration retention/fractures, but the following secondary endpoints were also evaluated: marginal staining, marginal adaptation, postoperative sensitivity, and recurrence of caries. The evaluation of the spontaneous postoperative sensitivity was performed one week after the restorative procedure.

These variables were ranked according to the criteria in the following scores: 1) FDI criteria (clinically very good, clinically good, clinically sufficient/satisfactory, clinically unsatisfactory, and clinically poor) and USPHS criteria (alfa, bravo, and charlie). In the case of marginal staining and marginal adaptation, the semiquantitative criteria (SQUACE) proposed by Hickel and others[36] was used. Each evaluator outlined the extent of the observed event on the sketch of each restoration using a pen according to defined criteria (marginal staining and marginal adaptation); after that, each

Table 4: *Modified US Public Health Service (USPHS) Criteria According to Perdigão and Others[7] and Bittencourt and Others[37]*

	Marginal Staining	Retention	Fracture	Marginal Adaptation	Postoperative Sensitivity	Recurrence of Caries
Alfa	No discoloration along the margin	Retained	None	Restoration is continuous with existing anatomic form	No postoperative sensitivity directly after the restorative process and during the study period	No evidence of caries contiguous with the margin
Bravo	Slight and superficial staining (removable, usually localized)	Partially retained	Small chip, but clinically acceptable	Detectable V-shaped defect in enamel only Catches explorer going both ways	—	—
Charlie	Deep staining cannot be polished away	Missing	Failure due to bulk restorative fracture	Detectable V-shaped defect to dentin-enamel junction	Sensitivity present at any time during the study period	Evidence of presence of caries

margin was assessed quantitatively as a proportion of the total length of the margin.

Both examiners evaluated all of the restorations once and independently. When disagreements occurred during the evaluations, they had to reach a consensus before the participant was dismissed.

The restoration retention rates were calculated according to the ADA guidelines[34] Cumulative failure percentage = $[(PF + NF)/(PF + RR)] \times 100\%$, where *PF* is the number of previous failures before the current recall, *NF* is the number of new failures during the current recall, and *RR* is the number of currently recalled restorations.

Statistical Analysis

The statistical analyses followed the intention-to-treat protocol according to the CONSORT (Consolidated Standards of Reporting Trials) suggestion.[26] Descriptive statistics were used to describe the distributions of the evaluated criteria. Statistical analysis for each individual item was performed for each evaluation criteria (FDI and USPHS criteria).

Two different statistical analyses were performed after we had collected data for six months. We used the Friedman repeated-measures analysis of variance by rank to compare the differences in the ratings of the four groups at six months, and we used the Fisher exact test to compare the differences in the ratings of ER vs SE groups. In this case, the data of the ER-D and ER-M were merged, as were those from the SE-et and SET groups.

We compared the baseline vs the six-month finding within each group using the McNemar test. The Cohen kappa statistic was used to test for interexaminer agreement. Data from SQUACE were categorized into three scores, as follows: 1) marginal discrepancies involving less than 10% of the total length of the restoration; 2) those involving between 10% and 30%; and 3) those involving more than 30%,[19,24] and the groups were compared with Kruskal-Wallis and Mann-Whitney tests. In all statistical tests, we pre-set the level of significance to 5%.

RESULTS

The restorative procedures were implemented exactly as planned, and no modification was performed. Forty-nine out of 80 patients examined for eligibility were not enrolled in the study because they did not fulfill the inclusion criteria. Thus, a total of 31 subjects (15 men and 16 women) were selected. One hundred and twenty-four restorations were placed, 31 for each group (Figure 1).

All baseline details relative to the research subjects and characteristics of the restored lesions are displayed in Table 5. The overall Cohen kappa statistics showed excellent agreement between the examiners in the six-month (0.94) follow-up. All research subjects were evaluated at baseline and at the six-month recall.

Retention/Fracture

Fifteen restorations were lost or fractured after six months (one for ER-D, three for ER-M, five for SE-et, and six for SET). According to both evaluation criteria, the six-month retention rates (95% confidence interval [CI]) were 96.8% (83.8%-99.4%) for ER-M, 90.3% (75.1%-96.7%) for ER-D, 83.9% (67.4%-92.9%) for SE-et, and 80.7% (63.7%- 90.8%) for SET,

Table 5: *Characteristics of the Research Subjects and the Noncarious Cervical Lesions (NCCLs) per Group*

Characteristics of Research Subjects	**No. of Lesions**			
Gender distribution				
Male	15			
Female	16			
Age distribution, y				
20-29	0			
30-39	1			
39-49	6			
>49	24			
Characteristics of NCCL Lesions	**No. of Lesions**			
	ER-D	**ER-M**	**SE-et**	**SET**
Shape, degree of angle				
<45	1	1	1	0
45-90	6	6	10	7
90-135	16	18	11	17
>135	8	6	9	7
Cervico-incisal height, mm				
<1.5	4	5	4	3
1.5-2.5	12	10	12	12
2.5-4.0	11	13	10	12
>4.0	4	3	5	4
Degree of sclerotic dentin				
1	15	16	15	15
2	7	8	5	10
3	6	6	8	4
4	3	1	3	2
Presence of antagonist				
Yes	29	28	30	29
No	2	3	1	2
Attrition facet				
Yes	16	15	20	19
No	15	16	11	12
Preoperative sensitivity (spontaneous)				
Yes	0	1	0	1
No	31	30	31	30
Preoperative sensitivity (air dry)				
Yes	7	9	12	9
No	24	22	19	22
Preoperative sensitivity (touch)				
Yes	7	11	14	9
No	24	20	17	22
Tooth distribution				
Anterior				
Incisor	7	5	3	4
Canines	4	4	4	3
Posterior				
Premolar	19	22	21	19
Molar	1	0	3	5

Table 5: Continued.

Characteristics of Research Subjects	No. of Lesions			
Arc distribution				
Maxillary	17	15	15	10
Mandibular	14	16	16	21

Abbreviations: ER-D, etch-and-rinse, dry dentin; ER-M, etch-and-rinse, moist dentin; SE-et, self-etch with selective enamel etching; SET, self-etch without selective enamel etching.

with no statistical difference identified between any pair of groups ($p>0.05$; Tables 6 and 7).

When the data from the six-month results from each group were compared with their baseline findings, a significant difference was found only for SET ($p=0.03$; Tables 6 and 7). Also, when the overall retention rate of the ER approach (93.6%, 95% CI 84.5%-97.5%) was compared with the overall retention rate of the SE approach (82.2%, 95% CI 70.9%-89.8%), significant differences in the retention rates were detected after six months ($p=0.001$; Tables 6 and 7).

Postoperative Sensitivity

Only two restorations had postoperative sensitivity to air at the six-month recall using both criteria (two for ER-D). No significant difference was detected between any pair of groups at the six-month mark or when the six-month findings of each group were compared to the baseline data. The

Table 6: *Number of Evaluated Restorations for Each Experimental Group Classified According to the World Dental Federation (FDI) Criteria*[36]

FDI Criteria	(*)	Baseline				6 mo			
		ER-D	ER-M	SE-et	SET	ER-D	ER-M	SE-et	SET
Marginal staining	A	31	31	31	31	31	27	26	23
	B	——	——	——	——	——	1	——	2
	C	——	——	——	——	——	——	——	——
	D	——	——	——	——	——	——	——	——
	E	——	——	——	——	——	——	——	——
Fractures and retention	A	31	31	31	31	30	28	25	25
	B	——	——	——	——	——	——	——	——
	C	——	——	——	——	——	——	1	——
	D	——	——	——	——	1	——	——	——
	E	——	——	——	——	——	3	5	6
Marginal adaptation	A	31	31	31	31	17	18	15	12
	B	——	——	——	——	13	10	11	13
	C	——	——	——	——	——	——	——	——
	D	——	——	——	——	1	——	——	——
	E	——	——	——	——	——	——	——	——
Postoperative (hyper-) sensitivity	A	31	31	31	31	29	28	26	25
	B	——	——	——	——	2	——	——	——
	C	——	——	——	——	——	——	——	——
	D	——	——	——	——	——	——	——	——
	E	——	——	——	——	——	——	——	——
Recurrence of caries	A	31	31	31	31	31	28	26	25
	B	——	——	——	——	——	——	——	——
	C	——	——	——	——	——	——	——	——
	D	——	——	——	——	——	——	——	——
	E	——	——	——	——	——	——	——	——

Abbreviations: ER-D, etch-and-rinse, dry dentin; ER-M, etch-and-rinse, moist dentin; SE-et, self-etch with selective enamel etching; SET, self-etch without selective enamel etching.

Table 7: *Number of Evaluated Restorations for Each Experimental Group According to the Modified US Public Health Service (USPHS) Criteria*[7,37]

USPHS Criteria	Baseline				6 mo			
	ER-D	ER-M	SE-et	SET	ER-D	ER-M	SE-et	SET
Marginal staining								
Alfa	31	31	31	31	31	27	26	23
Bravo	—	—	—	—	—	1	—	2
Charlie	—	—	—	—	—	—	—	—
Retention								
Alfa	31	31	31	31	31	28	26	25
Bravo	—	—	—	—	—	—	—	—
Charlie	—	—	—	—	—	3	5	6
Fracture								
Alfa	31	31	31	31	30	28	25	25
Bravo	—	—	—	—	—	—	1	—
Charlie	—	—	—	—	1	—	—	—
Marginal adaptation								
Alfa	31	31	31	31	30	28	26	25
Bravo	—	—	—	—	—	—	—	—
Charlie	—	—	—	—	1	—	—	—
Postoperative sensitivity								
Alfa	31	31	31	31	29	28	26	25
Bravo	—	—	—	—	2	—	—	—
Charlie	—	—	—	—	—	—	—	—
Recurrence of caries								
Alfa	31	31	31	31	31	31	31	31
Bravo	—	—	—	—	—	—	—	—
Charlie	—	—	—	—	—	—	—	—

Abbreviations: ER-D, etch-and-rinse, dry dentin; ER-M, etch-and-rinse, moist dentin; SE-et, self-etch with selective enamel etching; SET, self-etch without selective enamel etching.

overall postoperative sensitivity of ER vs SE was also not statistically significant ($p>0.05$; Tables 6 and 7).

Marginal Adaptation

Forty-seven restorations were considered to have minor discrepancies in marginal adaptation at the six-month recall using the FDI criteria (13 for ER-D, 10 for ER-M, 11 for SE-et, and 13 for SET). No significant difference was detected between any pair of groups at the six-month recall for either evaluation criteria ($p>0.05$). In addition, the overall ER data were not statistically different from the overall SE at the six-month evaluation for marginal adaptation ($p>0.05$).

Despite these minor discrepancies, only one restoration was considered to have clinically relevant discrepancies in marginal adaptation (one for ER-D; Table 6). When the USPHS criteria were used, one restoration was scored as "charlie" for marginal adaptation (one for ER-D; Table 7). However, for all groups, a significant difference was detected when baseline and six-month data were compared ($p=0.0001$), as well as when the overall ER data and overall SE data were compared with their baseline data ($p=0.0001$).

No significant difference was detected between any pair of groups (ER-D, ER-M, SE-et, and SET) or ER (ErD and ErM) vs SE (SE-et and SET) at the six-month recall for either criteria ($p>0.05$).

Marginal Staining

Marginal staining was observed in three restorations (one for ER-M and two for SET) in both evaluation criteria. No significant difference was found between groups at six months or when the overall ER data were compared to the SE data. It was not detect any significant differences within each group when baseline and six-month data were compared for both criteria ($p>0.05$; Tables 6 and 7). When SQUACE[36]

Table 8: *Number of Evaluated Restorations for Each Experimental Group According to the Semiquantitative Score (SQUACE)*[36]

Classification	ER-D	ER-M	SE-et	SET
Less than 10%	17	18	15	12
Between 10% and 30%	13	9	9	11
Between 31% and 50%	1	1	2	2
Statistical analysis[a]	A	A	A	A

Abbreviations: ER-D, etch-and-rinse, dry dentin; ER-M, etch-and-rinse, moist dentin; SE-et, self-etch with selective enamel etching; SET, self-etch without selective enamel etching.
[a] *Different letters indicate significant differences between groups (Kruskal-Wallis,* $p>0.05$*).*

was used, we did not detect a statistical difference among groups at the six-month evaluation ($p>0.05$; Table 8).

Other Parameters

No restoration showed recurrence of caries after six months for either criteria. One restoration had clinical problems related to fracture at six months for both criteria. When the FDI criteria for "acceptable" vs "not acceptable" restorations were applied, 16 restorations were ranked as "not acceptable" (14 restorations were lost, one restoration fractured, and one showed severe lack of marginal adaptation). Among these 16 restorations, only five were placed with the ER approach, while the remaining 11 restorations were placed with the SE approach (Table 9).

DISCUSSION

Most of the studies using universal adhesives found in the literature were laboratory tests, although it is known that the validity of bond strength testing is questionable.[38,39] Clinical studies in NCCLs provide the ultimate proof for the evaluation of the performance of adhesive systems, mainly because the most important parameter for evaluation of restorations in NCCLs is retention.[6] This is considered a true endpoint, because if restorations are lost, none of the other parameters can be evaluated.

The results of the present study showed that after six months of clinical service a total of 15 restorations failed as a result of debonding—11 bonded with the SE approach and four bonded with the ER approach (ER-M and ER-D)—which highlighted a poor bonding efficacy of the Xeno Select when used in the SE strategy. This poor bonding ability may be related to the kind of chemical bond produced by this adhesive with the dental substrates.

In general, SE adhesives differ from one another in many aspects, especially in their resin monomer composition, water content, and acidity.[40] They usually dissolve the smear layer and do not remove the dissolved calcium (Ca) phosphates.[41] According to the manufacturer of the Xeno Select, two acidic monomers are responsible for the interaction of this adhesive with the dentin surface: an "inverse" functionalized phosphoric acid ester and an acryloylamino alkylsulfonic acid, which was also present in the predecessor Xeno V.[42]

The manufacturer claims that the presence of a phosphoric acid ester group in the functionalized monomer makes it very similar to dipentaerytritol-pentacrylate–phosphoric acid–monomer (PENTA). There is evidence (by Raman spectroscopy findings) that the ester group of PENTA can establish a covalent bond with the Ca from dentin and enamel.[43] However, not only chemical bonding is essential to provide a strong bonded interface; the Ca salt produced by this chemical interaction should also be stable in an aqueous environment,[44,45] which ultimately depends on the stability of the formed

Table 9: *Restorations Acceptable or Not Acceptable According to the World Dental International (FDI) Criteria After Six Months*[36]

	Esthetic				Functional								Biological							
	Staining Margin				Fractures and Retention				Marginal Adaptation				Postoperative (Hyper-) Sensitivity				Recurrence of Caries			
	ER-D	ER-M	SE-et	SET	ER-D	ER-M	SE-et	SET	ER-D	ER-M	SE-et	SET	ER-D	ER-M	SE-et	SET	ER-D	ER-M	SE-et	SET
Acceptable	31	28	26	25	30	28	26	25	30	28	26	25	31	28	26	25	31	28	26	25
Not acceptable	0	0	0	0	1	3	5	6	1	0	0	0	0	0	0	0	0	0	0	0
Reasons	14 restorations were lost, one restoration fractured, and one restoration showed severe lack of marginal adaptation																			

bond to Ca, according to the "adhesion-decalcification" concept.[41,46-48]

Different functional monomers, such as "inverse" functionalized phosphoric acid ester and acryloylamino alkylsulfonic acid from Xeno Select, initially bond to Ca of hydroxyapatite, but they readily debond.[41,49] This debonding was recently confirmed by Zhou and others[50] Through the use of attenuated total reflection (ATR) spectroscopy, the authors evaluated the chemical interaction of functional monomers with dentin. The authors did not observe any signal of chemical bonding of the Xeno V monomers to dentin surfaces, indicating low affinity of Xeno V (the predecessor of Xeno Select) to this substrate.[50]

It is also known that during interaction with the hydroxyapatite surface mild and ultramild SE adhesives leach little Ca, as they bind strongly enough to mineral content. On the other hand, intermediary strong or strong SE adhesives also bind to Ca ions from the hydroxyapatite, but they also demineralize the substrate more and tend to produce more soluble Ca salts.[46,47] According to the Xeno Select data, this adhesive system can be considered to be of intermediate strength (pH<2.0, manufacturer instructions), along with the predecessor Xeno V, which was classified as a strong SE adhesive system (pH=1.38 for Xeno V).[50,51]

The PENTA ester group is considered a weak acid, but this low pH is likely obtained after addition of the acryloylamino alkylsulfonic acid to the adhesive formulation. According to the manufacturer's instruction, this second acidic monomer was added to the formulation to adjust the acidity of Xeno Select.[42] Compared to other mild SE adhesives, the Xeno V produced cleaner dentin surfaces with more exposition of dentin tubules, likely due to the lower pH of Xeno V in comparison to that of the other mild SE adhesives tested.[50] Using field-emission scanning electron microscopy and ATR findings, these authors[50] also observed that the mild SE adhesives contained functional monomers with a higher affinity to dentin, likely due to the type of functional monomers presented in their composition.

This was also confirmed by a systematic review[51] of clinical studies in NCCLs. The authors showed that the mild and ultramild SE systems contained functional monomers with the chemical potential to produce restorations with higher quality and durability in cervical lesions.[51] This was also confirmed by the few clinical studies[19,24,25,52] that evaluated ultramild universal adhesives in NCCLs. Mena-Serrano and others[24] showed that after six months of clinical service, only three restorations were lost (94% retention rate), and this feature was very similar to the more recent publication of Lawson and others[52] (one restoration lost; 97% retention rate). Even after two and three years of clinical service, retention rates as high as 89% to 93% were observed.[25,52]

The good performance of composite restoration in cervical lesions from these studies[19,24,25,52] is likely due to the presence of the acidic functional monomer 10-MDP in the SE adhesives.[41,46-48] This functional monomer is responsible for the formation of a stable salt with the Ca in hydroxyapatite. The stability of this Ca salt has been correlated[40] with the high bond strength of MDP to enamel and dentin immediately and after long-term water storage.

When the six-month data of this material are compared with that of other 10-MDP–containing universal adhesives in the SE strategy, we clearly show that 10-MDP adhesives show higher retention rates (94%-97%)[19,24,25,52] than are reported in this clinical trial (80.7%), which reinforces that MDP might have an important role on the bonding performance of universal adhesives when used in the SE mode. Future studies comparing universal adhesives with and without MDP should be conducted.

It is also interesting to observe that even when the SE was applied after selective enamel etching (SE-et), the retention pattern did not improve significantly.[53,54] The clinical trials[53,54] that compare the benefits of selective enamel etching before application of SE adhesives do not report improved retention rates of composite resin restorations in NCCLs, and this finding has also been observed for one universal adhesive.[19,24,25] On the other hand, selective enamel etching with SE adhesives (SE-et) can reduce marginal discoloration at the restoration interface after medium/long-term clinical service.[53,54]

The better performance of Xeno Select with the ER approach may be related to the fact that after phosphoric acid etching, the adhesive is no longer dependent on the chemical bonding produced by the acidic monomers with the dental substrates.[55] In this case, micromechanical bond is responsible for the good retention of the adhesive so long as the material produces a well-impregnated hybrid layer[55] and a strong polymer inside the hybrid layer.[1]

In the present investigation, the results of Xeno Select when applied in wet (ER-M) and dry (ER-D)

conditions showed similar results in terms of retention rates, as well as all other parameters evaluated. The similar results of the ER-M and ER-D groups were not surprising, as other clinical trials that compared wet and dry bonding with ER systems did not report higher retention rates for the wet bonding protocol,[56,57] mainly if the adhesive was applied actively,[57,58] as was done for Xeno Select (manufacturer's recommendation).

In the case of Xeno Select, this new adhesive system contains a special solvent named tert-butanol (2-methyl-2-propanol), which is able to re-expand collapsed collagen fibrils produced by air-drying. Indeed, during microscopic analysis of resin-dentin interfaces produced by an adhesive system (XP-Bond) containing tert-butanol, the formation of very similar hybrid layers and resin tags in dentin under dry and moist conditions was observed.[59] Although tert-butanol has a higher molecular weight than ethanol, the evaporation rate is almost the same, with a latent heat of vaporization of 41 kJ/mol for tert-butanol and of 42 kJ/mol for ethanol.[60] The vapor pressure of the different kinds of solvents at 20°C is given as 2330 Pa for water, 4133 Pa for tert-butanol, 5900 Pa for ethanol, and 23,300 Pa for acetone.[61]

Regardless of the bonding strategy used, the present study observed a significant deterioration of the marginal adaptation even at the short-term evaluation. These results are similar to those of earlier published articles[19,24,25] that compared universal adhesives using ER and SE approaches using a more sensitive criteria for evaluation (SQUACE). Although different clinical trials have shown that marginal discrepancies of a composite restoration usually develop rather rapidly,[19,24,25,52] most of the marginal defects were small and clinically acceptable.[51]

While there is a general consensus that marginal defects can affect the final performance of resin composite restorations, to the extent of our knowledge no study has so far observed an association between marginal defects and loss of retention. These defects can cause early marginal discoloration, which may jeopardize the restoration esthetics. Fortunately, restoration re-polishing can amend these discrepancies without causing any damage to the integrity of the restoration.[62]

As a result of the efforts of the FDI, new criteria for evaluating dental restorations were published in 2007 and named the "FDI criteria."[36] Since then, only a few publications have used the FDI criteria,[19,24,25,63,64] as most clinical studies on NCCL restorations continue using the USPHS criteria.[6-8,52] Recent publications[19,24,25,63] that compared the six-month clinical behavior of several adhesion strategies using both FDI and USPHS-modified criteria concluded that the FDI criteria are more sensitive than the USPHS-modified criteria to small variations in the clinical outcomes of NCCLs. This finding was corroborated in the present study, as the marginal discrepancies were more frequently measured in the FDI criteria in relation to USPHS criteria. We opted to keep both criteria in the present study to allow comparison of the present results with those of studies that used both criteria.

Clinical trials have greater value when published after long-term follow-ups. However, the large number of failures in such a short-term follow-up justifies the publication of these data, as this can provide clinicians with further evidence before selecting a universal adhesive for purchase and use in their clinical offices. Additionally, these findings may also stimulate further studies based on this material, improving when they use the SE approach in their formulation, as Xeno Select did not fulfill the criteria for provisional acceptance according to the ADA guideline of having a failure rate of less than 5% after six months of clinical performance.[34] Future clinical recalls are already scheduled in order to assess the long-term performance of Xeno Select in NCCL restorations.

CONCLUSIONS

Within the limitations of this study, the six-month clinical behavior of Xeno Select Universal Adhesive (Dentsply) depends on the bonding strategy used. The universal adhesive did not fulfil the ADA criteria for full approval when used in the SE mode.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subject's oversight committee guidelines and policies of: Fluminense Federal University. The approval code for this study is: 800.273/14.

Conflict of Interest

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

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Laboratory Research

Influence of Conditioning Time of Universal Adhesives on Adhesive Properties and Enamel-Etching Pattern

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Clinical Relevance

The active and prolonged application of universal adhesives in the self-etch mode may be a viable alternative comparable to enamel etching with phosphoric acid.

SUMMARY

Objectives: To evaluate the effect of application protocol in resin–enamel microshear bond strength (μSBS), *in situ* degree of conversion, and etching pattern of three universal adhesive systems.

Methods and Materials: Sixty-three extracted third molars were sectioned in four parts (buccal, lingual, and proximals) and divided into nine groups, according to the combination of the main factors—Adhesive (Clearfil Universal, Kuraray Noritake Dental Inc, Tokyo, Japan; Futurabond U, VOCO, Cuxhaven, Germany; and Scotchbond Universal Adhesive, 3M ESPE, St Paul, MN, USA)—and enamel treatment/application time (etch-and-rinse mode [ER], self-etch [SE] application for 20 seconds [SE20], and SE application for 40 seconds [SE40]). Specimens were stored in water (37°C/24 h) and tested at 1.0 mm/min

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(μSBS). The degree of conversion of the adhesives at the resin–enamel interfaces was evaluated using micro-Raman spectroscopy. The enamel-etching pattern was evaluated under a scanning electron microscope. Data were analyzed with two-way analysis of variance and Tukey test (α=0.05).

Results: In general, the application of the universal adhesives in the SE40 produced μSBS and degree of conversion that were higher than in the SE20 ($p<0.01$) and similar to the ER mode. The deepest enamel-etching pattern was obtained in the ER mode, followed by the SE40.

Conclusions: The active and prolonged application of universal adhesives in the SE mode may be a viable alternative to increase the degree of conversion, etching pattern, and resin–enamel bond strength.

INTRODUCTION

The current adhesive systems can be classified based on the number of application steps or according to the adhesion strategies in etch-and-rinse (ER) and self-etch (SE).[1] Recently, a new group of dental adhesives has been introduced to the market as "universal" or "multimode" adhesives. They are essentially one-step SE adhesives that may be associated with phosphoric acid etching.[2,3] This versatile new adhesion philosophy advocates the use of the simplest option of each strategy, that is, one-step SE or two-step ER,[2-4] with the advantage that the dentist can decide which adhesive strategy to use for each specific clinical situation.

In spite of this, bonding to enamel is still a concern. Similar to what has been reported for one-step SE adhesive applied on enamel,[5,6] reduced bonding effectiveness is observed in enamel when universal adhesives are used in the SE mode.[7-9]

The most recognized technique for improving the bonding of one-step SE to enamel is to use selective enamel etching,[10] which led dental manufacturers and researchers to advocate selective enamel etching for one-step SE and for the majority of universal adhesives. A shortcoming of this technique is that accidental dentin etching may occur, particularly in small cavity preparations or when a low-viscosity etchant is used.[4,11]

Although prior phosphoric acid etching increases the immediate bond strength of universal adhesives to dentin when applied as ER for the majority of universal adhesives,[2-4,12,13] earlier signs of degradation are observed in the ER mode compared to the SE approach.[14,15] Additionally, some universal adhesives applied as SE showed stable bond strength and reduced the nanoleakage at the interface after six months of aging.[15]

To improve adhesion of one-step SE adhesives applied to enamel[16] without the use of phosphoric acid etching, some authors suggested increased application time or multiple adhesive layers. This approach increases the contact of acidic resin monomers with the enamel surface, creating a more retentive pattern. While some authors reported improved enamel bond strength using these aforementioned techniques, this is not consensual, probably because it seemed to be dependent on others factors, such as the pH of the adhesive tested as well as the application technique for each adhesive.[17-22]

Therefore, taking into consideration the recent launch of universal adhesives on the market, the aim of the present study was to compare the effect of different active application times on resin–enamel microshear bond strength (μSBS), *in situ* degree of conversion, and etching pattern of three universal adhesive systems. The following null hypotheses were tested: 1) the different active application times will not influence the μSBS of the different adhesives, 2) the different active application times will not influence the degree of conversion of the adhesives at the resin–enamel interfaces, and 3) the different active application times will not influence the enamel-etching pattern of the universal adhesives.

METHODS AND MATERIALS

Sixty-three extracted, caries-free human molars were used. The teeth were collected after obtaining the patients' informed consent under a protocol approved by the Ethics Committee Review Board of the local university. The teeth were disinfected in 0.5% chloramine, stored in distilled water, and used within six months of extraction.

The roots of all teeth were removed by sectioning at the enamel-cementum junction. Each dental crown was then sectioned in the diagonals across the long axis of the tooth to produce four enamel specimens (buccal, lingual, and proximals).[9] Two hundred and sixteen enamel specimens, which originated from 54 teeth, were ground wet with 180- and 600-grit SiC paper for 60 seconds each and used for evaluation of μSBS and *in situ* degree of conversion at the resin–enamel interfaces. The other 36 enamel specimens, which originated from

Table 1: *Adhesive System (Batch Number), Composition, and Application Mode of the Adhesive Systems According to the Manufacturers' Instructions*

Adhesive (Batch Number)	Composition[a]	Application Mode	
		Self-Etch[b]	Etch-and-Rinse
Clearfil Universal (01416)	1. Etchant: 35% phosphoric acid, colloidal silica, polyethyleneglycol, pigment, and water (K-ETCHANT) 2. Adhesive: HEMA, MDP, Bis-GMA, ethanol, camphorquinone, hydrophilic aliphatic dimethacrylate, silane coupling agent, colloidal silica, water, and accelerators	1. Apply bond and rub for 20 s[a] or 40 s 2. Dry by blowing mild air for 5 s 3. Light cure for 10 s at 1200 mW/cm^2	1. Apply etchant for 10 s 2. Rinse thoroughly 3. Dry 4. Apply adhesive as for the self-etch mode
Futurabond U (1346518)	1. Etchant: 34% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminum oxide (Scotchbond Universal Etchant) 2. Adhesive: Liquid 1: Acidic adhesive monomer HEMA BISGMA, HEDMA, UDMA Catalyst Liquid 2: Ethanol initiator, catalyst	1. Apply the adhesive to the entire preparation with a microbrush and rub it for 20 s[a] or 40 s. If necessary, rewet the disposable applicator during treatment 2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent is evaporated completely 3. Light cure for 10 s at 1200 mW/cm^2	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Air dry 2 s 4. Apply adhesive as for the self-etch mode
Scotchbond Universal Adhesive (D-82229)	1. Etchant: 34% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminum oxide (Scotchbond Universal Etchant) 2. Adhesive: MDP phosphate monomer, dimethacrylate resins, Bis-GMA, HEMA, methacrylate-modified polyalkenoic acid copolymer, camphorquinone, filler, ethanol, water, initiators, silane	1. Apply the adhesive to the entire preparation with a microbrush and rub it in for 20 s[a] or 40 s. If necessary, rewet the disposable applicator during treatment 2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent is evaporated completely 3. Light cure for 10 s at 1200 mW/cm^2	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Air dry 2 s 4. Apply adhesive as for the self-etch mode

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; MDP, methacryloyloxydecyldihydrogen phosphate; Bis-GMA, bisphenolglycidyl methacrylate; UDMA, urethanedimethacrylate; HEDMA, hexamethylenedimethacrylate.
[a] *As per manufacturer's instructions.*
[b] *The intensity of the light-curing procedure was standardized for all materials.*

nine teeth, were not ground and used for the evaluation of the etching pattern produced on the enamel surface.

Experimental Design

The enamel specimens were randomly assigned into nine experimental conditions according to the combination of the independent-variable adhesive system—Clearfil Universal (Kuraray Noritake Dental Inc, Tokyo, Japan), Futurabond U (VOCO, Cuxhaven, Germany), and Scotchbond Universal Adhesive (3M ESPE, St Paul, MN, USA; also known as Single Bond Universal in some countries) (Table 1)—and enamel treatment/application time: ER mode, SE mode with application time of 20 seconds, and SE mode with application time of 40 seconds.

μSBS

Each enamel specimen was embedded in a polyvinyl chloride tube (10 mm high × 13 mm diameter) using a chemically cured acrylic resin (Jet Clássico, São Paulo, Brazil) in a way that the enamel surface was left exposed on the top of the cylinder. The delimitation of the bonding area was performed according to Shimaoka and others.[23] Six to eight perforations, with an internal diameter of 0.8 mm were made in an acid-resistant, double-faced adhesive tape (AdelbrasInd e Com Adesivos Ltda, São Paulo, Brazil) that was adapted to the enamel surface. This procedure was performed with the Hygienic Ainsworth–style rubber-dam punch (Coltene, Alstätten, Switzerland) and adapted to the enamel surface. The variation in

the number of perforations for each enamel surface was dependent on the dimensions of the enamel specimens.

The randomization of the specimens for the μSBS was done using block randomization. A person not involved in the research protocol performed this procedure using computer-generated tables. The universal adhesives were applied to the enamel surface as described in Table 1. A single operator performed all bonding procedures according to the description below:

1) ER mode: The phosphoric acid gel of each adhesive system was applied and left undisturbed for the time recommended for each manufacturer. Then the surfaces were water rinsed with an air–water syringe for 10 seconds.
2) SE mode, 20-second application: Each adhesive was applied actively on the enamel for 20 seconds without phosphoric acid etching. The manual pressure exerted on the microbrush (Microbrush International, Grafton, WI, USA) during application was equivalent to 35 g.[24,25]
3) SE mode, 40-second application time: Each adhesive was applied actively on the enamel for 40 seconds without phosphoric acid etching. As reported earlier, the manual pressure exerted on the microbrush during application was equivalent to 35 g.

After the application of the adhesive system, polyethylene transparent Tygon tubes (Tygon Medical Tubing Formulations 54-HL, Saint Gobain Performance Plastics, Akron, OH, USA) with the same internal diameter of the perforations and a height of 0.5 mm were positioned on the perforations over the double-faced tape, ensuring that their lumen coincided with the circular areas exposed by the perforations. Resin composite (Filtek Z350, 3M ESPE) was carefully packed inside each tube, and a clear Mylar matrix strip was placed over the filled Tygon tube and pressed gently into place. The resin composite was light cured for 20 seconds using an LED light-curing unit set at 1200 mW/cm^2 (Radii-cal, SDI Limited, Bayswater, Victoria, Australia). A radiometer (Demetron L.E.D. Radiometer, Kerr Sybron Dental Specialties, Middleton, WI, USA) was used to check the light intensity every five specimens. These procedures were carried out under 10x magnifying loupes.

After storage of the specimens in distilled water for 24 hours at 37°C, the Tygon tubes and the double-faced adhesive tape were carefully removed with a blade, exposing the composite cylinders. Each specimen was examined under a stereomicroscope at 10× magnification. The bonded cylinder was discarded if there was evidence of porosities or gaps at the interface.[26]

The specimens were attached to a shear-testing fixture (Odeme Biotechnology, Joaçaba, SC, Brazil) and tested in a universal testing machine (Kratos IKCL 3-USB, Kratos Equipamentos Industriais Ltda, Cotia, São Paulo, Brazil). Each specimen was positioned onto the universal testing machine, and a thin wire (0.2-mm diameter) was looped around the base of each composite cylinder. The orthodontic wire contacted the composite resin cylinder in half of its circumference. The setup was kept aligned (resin–enamel interface, the wire loop, and the center of the load cell) to ensure the correct orientation of the shear forces.[27] The crosshead speed was set at 1 mm/min until failure.

The μSBS values (MPa) were calculated by dividing the load at failure by the surface area (mm^2). After testing, the specimens were examined in an optical microscope (SZH-131, Olympus Ltd, Tokyo, Japan) at 100× magnification to define the location of the bond failure. The type of failure was determined based on the percentage of substrate-free material as adhesive (A; failure at the resin–enamel interface), cohesive (C; failure exclusively within enamel or resin composite), and/or mixed (M; failure at the resin–enamel interface that included cohesive failure of the neighboring substrates).

In Situ Degree of Conversion

Four enamel specimens were randomly assigned for each group as described earlier for the μSBS. The adhesives were applied, and composite resin build-ups were constructed on the bonded enamel using the same materials and protocols described for the μSBS test. After storage of the restored teeth in distilled water at 37°C for 24 hours, the resin–enamel specimens were longitudinally sectioned across the bonded interface with a low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to obtain two resin–enamel slices.

The resin–enamel slices were wet polished with 1500-, 2000- and 2500-grit SiC paper for 15 seconds each. Then they were ultrasonically cleaned for 20 minutes in distilled water and stored in water for 24 hours at 37°C. The micro-Raman spectrometer (Bruker Optik GmbH, Ettlingen, Baden-Württemberg, Germany) was first calibrated for zero and then for coefficient values using the following micro-Raman parameters: 20-mW neon laser with 532-

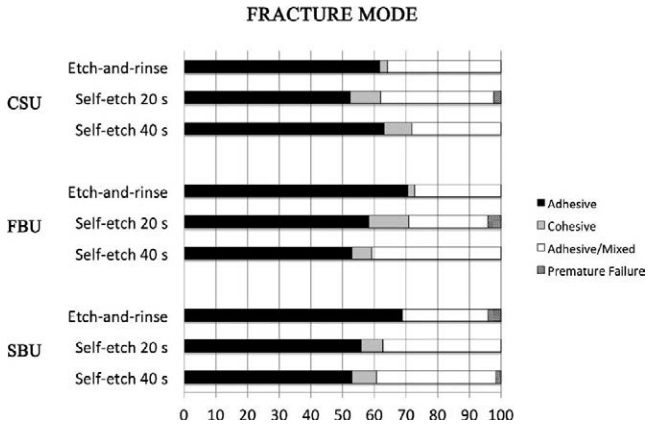

Figure 1. *Number of specimens (%) according to fracture mode for all experimental groups.*

nm wavelength, spatial resolution ≈3 μm, spectral resolution ≈5 cm^{-1}, accumulation time of 30 seconds with five coadditions, and magnification of 100× (Olympus UK, London, UK) using a ≈1-μm beam diameter.[28]

Spectra were taken at the resin–enamel adhesive interface at three different sites for each specimen. Spectra of uncured adhesives were taken as references. Post-processing of spectra was performed using the Opus Spectroscopy Software, version 6.5 (Bruker Optik). The ratio of double-bond content of monomer to polymer in the adhesive was calculated according to the following formula: Degree of conversion (%) = $(1 - R_{cured}/R_{uncured}) \times 100\%$, where R is the ratio of aliphatic and aromatic peak areas at 1639 cm^{-1} and 1609 cm^{-1} in cured and uncured adhesives, respectively.

Enamel-Etching Pattern

The enamel-etching pattern was evaluated on the enamel surface under a scanning electron microscope (JSM 5600LV, JEOL, Tokyo, Japan). For this purpose, the adhesives were applied in the SE strategy (20 and 40 seconds) according to the experimental conditions (Table 1), but the adhesives were not light cured. For the ER mode, the phosphoric acid gel was applied on enamel for 15 seconds, rinsed for 10 seconds, and air-dried according to each manufacturer's instructions (Table 1). Then the adhesives were applied without the light-curing step.

The enamel surfaces were then immediately stored in acetone for 24 hours to dissolve the resinous material from the enamel surface. Then the specimens were rinsed off in deionized water (5 minutes), 96% alcohol bath (5 minutes), and deionized water (5 minutes) in order to dissolve and remove the SE primer and the adhesive resins.[29]

All specimens were dried and dehydrated in a desiccator for 12 hours, and the conditioned enamel surfaces were sputter coated with gold/palladium in a vacuum evaporator (SCD 050, Balzers, Schaan, Liechtenstein). The entire surface of treated enamel was examined under a scanning electron microscope (JSM 5600LV, JEOL). Photomicrographs of representative surface areas were taken at 1000×, 1200×, and 5000× magnification.

Statistical Analysis

The resin–enamel μSBS values with adhesive/mixed failure mode and obtained from the same enamel specimen were averaged for statistical purposes. Similarly, the values of the degree of conversion from the same resin–enamel specimen were averaged so that the experimental unit in this experiment was the enamel specimen.

Specimens with cohesive and premature failures were not included in data analysis of the μSBS due to their low frequency in the experiment. Data from μSBS and degree of conversion were analyzed separately using two-way analysis of variance (adhesive vs. enamel treatment/application time) and Tukey *post hoc* test at a level of significance of 5%. The enamel-etching pattern was evaluated only qualitatively.

RESULTS

Microshear Bond Strength

The majority of the specimens (92.2% to 100%) showed adhesive/mixed failures (Figure 1). For all adhesives, the SE mode with an application time of 40 seconds yielded bond strength values that were statistically superior to that obtained in the SE mode for 20 seconds ($p<0.005$; Table 2). For Futurabond U and Scotchbond Universal, the SE modes for 40 seconds were statistically similar to the respective adhesives in the ER mode ($p>0.62$; Table 2). For Clearfil Universal, the application in the SE mode for 40 seconds produced adhesive interfaces with higher bond strength values than that obtained in the ER mode ($p<0.0002$; Table 2).

In Situ Degree of Conversion

For all adhesives, their application in the SE mode for 40 seconds produced a statistically higher degree of conversion than that obtained in the SE mode for 20 seconds ($p<0.003$; Table 3) and ER strategies ($p<0.00002$; Table 3). The lowest degree of conver-

Table 2: *Means and Standard Deviations of the Microshear Bond Strength (MPa) Values of the Different Experimental Groups*

Adhesives	Application Mode		
	Etch-and-Rinse	Self-Etch (20 s)	Self-Etch (40 s)
Clearfil Universal	19.1 ± 1.8 BC[a]	20.1 ± 1.2 B	23.1 ± 2.0 A
Futurabond Universal	17.1 ± 2.0 C	15.9 ± 1.3 D	18.2 ± 1.1 C
Scotchbond Universal	19.3 ± 1.7 BC	16.9 ± 1.1 D	20.4 ± 1.5 B

[a] *Similar letters indicate groups that are statistically similar (analysis of variance, Tukey test;* p>*0.05).*

Table 3: *Means and Standard Deviations of the Degree of Conversion (%) Values for Each Experimental Condition*

Adhesives	Application Mode		
	Etch-and-Rinse	Self-Etch (20 s)	Self-Etch (40 s)
Clearfil Universal	67.3 ± 2.6 D[a]	83.2 ± 3.8 B	92.3 ± 3.5 A
Futurabond Universal	60.2 ± 3.3 E	76.2 ± 2.7 C	93.5 ± 4.2 A
Scotchbond Universal	73.1 ± 4.3 C	76.0 ± 2.7 C	89.9 ± 4.6 A

[a] *Similar letters indicate groups that are statistically similar (analysis of variance, Tukey test;* p>*0.05).*

sion was obtained in the ER protocol, except for Scotchbond Universal, in which the degree of conversion in the SE mode for 20 seconds was statistically similar to the ER condition (p=0.45; Table 3). For the other two adhesives (Clearfil Universal and Futurabond U), the degree of conversion in the SE mode for 20 seconds was superior to the degree of conversion measured in the ER protocol (p<0.002; Table 3).

Enamel-Etching Pattern

The ER strategy resulted in the deepest and most pronounced etching pattern, mainly when compared with the SE mode applied for 20 seconds (Figure 2). In the SE mode applied for 20 seconds, all universal adhesives resulted in featureless morphology in which the superficial enamel layer was removed without exposure of the subsurface enamel. The application for 40 seconds in the SE mode improved the etching pattern, resulting in exposure of the periphery of the prisms, with signs of hydroxyapatite dissolution (Figure 2).

DISCUSSION

The results of this study confirmed previous findings[5,6,30,31] that adhesives applied in the SE mode for shorter application times resulted in reduced resin–enamel bond strength values compared to the ER protocol, which led us to reject the first null hypothesis. This means that even for universal adhesives, bonding to enamel is still a concern for clinicians when they are used in the SE mode.

Acidic monomers presented in the SE composition partially dissolve the smear layer and etch the enamel, and their effect is more pronounced when applied on ground enamel. But their acidity is not sufficient to produce retentive etching patterns equivalent to those produced by 35% phosphoric acid.[30-33] Several studies have shown a shallow intercrystallite infiltration of the monomers and a slight formation of interprismatic resin tags,[30,31] indicating a very superficial interaction of the SE adhesives, applied for shorter periods, with the enamel surface. This may be the reason why reduced resin–enamel μSBS was usually reported for SE adhesives.[32,34]

The universal adhesive systems evaluated in the present study have variable pH values around 2.5. The adhesives Clearfil Universal and Futurabond Universal have pH values of approximately 2.3, while Scotchbond Universal has a pH of 2.7.[4,9,35] Within this pH range, SE adhesives applied for only 20 seconds do not etch enamel as effectively as phosphoric acid.[6,9,30,31,36,37]

On the other side, Clearfil Universal and Futurabond U adhesives showed a higher degree of conversion when applied as SE for 20 seconds in comparison with ER, as was previously shown by Loguercio and others.[9] This indicates the rejection of the second null hypothesis.

Taking into consideration that, in the ER strategy, a double conditioning (phosphoric acid + acidic monomers from SE adhesives) occurred, these authors speculated that as Clearfil Universal and Futurabond U are more acidic when compared to Scotchbond Universal, the lower pH potentiates the demineralization mechanism and improves the superficial interaction.[6,32,36,38,39] Additionally, the uneven topography of etched enamel surfaces may increase the entrapment of solvents and air into the deeper prismatic and interprismatic portions of the etched enamel, leading to a lower *in situ* degree of conversion.[40]

The present study showed that the prolonged application of the adhesives in the SE mode might compensate for the higher pH of the universal adhesives. Improved enamel-etching pattern and

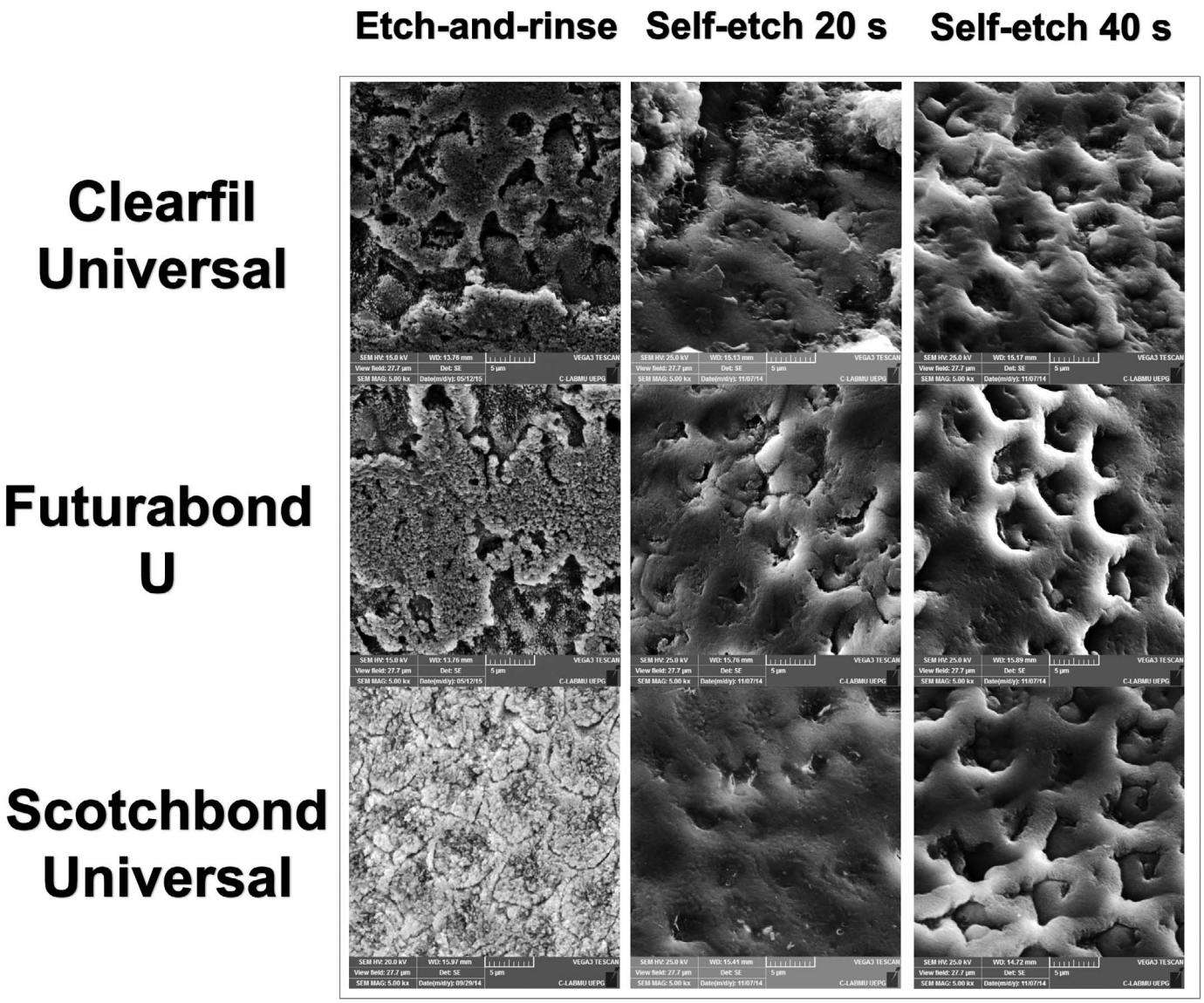

Figure 2. *Representative morphology of enamel treated with different applications protocol with the three universal adhesives used in this study. The etch-and-rinse group resulted in the best-defined etching pattern for all adhesives. Observe that all adhesive showed a more eroded pattern in comparison with Scotchbond Universal. A 20-second application showed signs of mild etching effect, with few islands without evidence of acid conditioning. A 40-second application of adhesives showed a better etching pattern with signs of interprismatic conditioning.*

increased resin–enamel bond strength values were observed when the universal adhesives were applied for 40 seconds instead of the conventional 20 seconds. This may be the result of improved diffusion and interaction of acidic resinous monomers with the underlying enamel, increasing not only the potential of etching but also resin impregnation into the underlying enamel.[17,22]

Although the etching pattern achieved with the SE mode for 40 seconds was still not as retentive as that produced in the ER mode, the ultramorphological images of the present investigation support the findings that a deeper demineralization occurred with the universal adhesives when they were used in the SE mode for 40 seconds, leading us to reject the third null hypothesis. It should be mentioned that a more retentive etching pattern could have been achieved had we removed the outer enamel layer before adhesive application.[30,31]

Apart from that, the literature findings are not consensual on whether increased application times produce higher resin–enamel bond strength values.[17-22] Several methodological differences could explain these differences. For example, in two studies where similar results were obtained, it is

not clear whether the adhesives were applied passively or actively.[19,21]

In the present experimental study, the good results associated with the increased application time in the SE mode cannot be dissociated from the benefits of the active application method. In all groups, the adhesives were applied actively, as this method was shown to improve enamel demineralization and interaction of resin monomers with prismatic and interprismatic areas by carrying fresh resin monomers to the deeper enamel layers during active application.[9,18,37,41-43]

Additionally, active and prolonged application of universal adhesives in the SE mode may increase solvent diffusion outward, mainly for adhesives composed of solvents with low vapor pressure, such as water.[33] This solvent evaporation may allow room for changes in polymer topology by reducing in the intrinsic fraction of nanopores, allowing increased cross-linking and improved mechanical properties of the polymer inside the enamel hybrid layer.[44,45] This may be an explanation of why the highest degree of conversion was obtained when adhesives were applied in the SE mode for 40 seconds.

In short, the prolonged and active application of the universal adhesives in the SE mode improved the etching pattern of the enamel surface and produced a higher degree of conversion and higher resin–enamel bond strengths. Future clinical studies should be conducted to evaluate if this protocol may increase the retention rates and the quality of the margins of composite resin restorations placed in cervical lesions without the need of selective phosphoric acid etching of the enamel margins.

Due to the difficulty of SE adhesives bonding to enamel, several manufacturers have been recommending selective enamel etching, although no significant difference exists in the retention rate of composite resin restorations in non-carious cervical lesions when selective enamel etching is compared with SE adhesives without selective enamel etching.[46-50] The only benefit of selective enamel etching with SE adhesives is minor reduction of marginal discoloration at the restoration interface.[46,47]

On the other hand, at least one clinical study showed that higher retention rates after 18 months were associated with the prolonged time of active SE application in noncarious cervical lesions, but, in this specific study, the increase of application time was associated with more coats of the SE adhesive. In addition, the mentioned adhesive is no longer available for use.[51] Further clinical trials should be conducted to validate the results obtained with the prolonged SE application of universal adhesives in this laboratory study, mainly because the prolonged and active application of SE adhesives makes the application procedure easier when compared to selective enamel etching, mainly because the former omitted the use of phosphoric acid.[52]

CONCLUSIONS

In summary, the active and prolonged application of universal adhesives in the SE mode may be a viable alternative to increase the degree of conversion, etching pattern, and resin–enamel bond strength.

Acknowledgments

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of: COEP/UEPG. The approval code for this study is: 0123/2009.

Conflict of Interest

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

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Delayed Photo-activation Effects on Mechanical Properties of Dual Cured Resin Cements and Finite Element Analysis of Shrinkage Stresses in Teeth Restored With Ceramic Inlays

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Clinical Relevance

Clinicians should take into account the mechanical properties when selecting resin cements. Slowing down the polymerization reaction allows more viscous flow with no effect on mechanical properties. Delayed photo-activation may be beneficial for cementation of indirect restorations in vital teeth.

SUMMARY

Objective: The aim of this study was to investigate the effect of delayed photo-activation on elastic modulus, Knoop hardness, and post-gel shrinkage of dual cure resin cements and how this affects residual shrinkage stresses in posterior teeth restored with ceramic inlays.

Methods and Materials: Four self-adhesive (RelyX Unicem, 3M ESPE; GCem, GC; Mono-Cem, Shofu; and seT, SDI) and two conventional (RelyX ARC, 3M ESPE; and AllCem, FGM) dual cure resin cements for cementing posterior ceramic inlays were tested. Strain gauge and indentation tests were used to measure the post-gel shrinkage (Shr), elastic modulus (E), and Knoop hardness (KHN) when photo-activated immediately and 3 and 5 minutes after placement (n=10). Shr, E, and KHN results were analyzed using two-way analysis of variance followed by Tukey honestly significant difference post hoc tests (α=0.05). The

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experimentally determined properties were applied in a finite element analysis of a leucite ceramic inlay (Empress CAD, Ivoclar Vivadent) cemented in a premolar. Modified von Mises stresses were evaluated at the occlusal margins and cavity floor.

Results: Shr, E, and KHN varied significantly among the resin cements ($p<0.001$). Highest overall Shr values were found for RelyX Unicem; GCem had the lowest. Increasing the photo-activation delay decreased Shr significantly. Delayed photo-activation had no effect on E ($p=0.556$) or KHN ($p=0.927$). RelyX Unicem had the highest E values; seT and MonoCem had the lowest E values. AllCem and RelyX Unicem had the highest KHN and MonoCem had the lowest KHN. Cements with high Shr and E values caused higher shrinkage stresses. Stresses decreased with delayed photo-activation for all cements.

Conclusions: KHN and E values varied among the different resin cements. Residual shrinkage stress levels decreased with increasing photo-activation delay with all resin cements.

INTRODUCTION

In recent years, esthetic restorations became more popular as an alternative to amalgam restorations with good clinical longevity.[1] In teeth with large cavities, where direct composite restorations can be compromised, indirect techniques such as adhesive ceramic inlays or onlays have been indicated to improve marginal adaptation and proximal contact.[2,3] Close to 90% of ceramic inlays and onlays survive after 12 years.[1] Despite their good longevity, postoperative sensitivity, marginal fracture, secondary caries, marginal deterioration, and discoloration are frequently identified as the causes for failure of this restorative procedure.[1,3-5] These failure mechanisms have been associated with polymerization shrinkage of resin cements used for luting ceramic inlays and onlays.[5-7]

Dual cure resin cements are widely used for luting ceramic restorations because they combine desirable features of photo- and chemical-cured systems and have shown good clinical performance.[1,8,9] Manufacturers recommend clinicians to wait before light curing the cements, because a time delay between the cement mixing step and photo-activation may favor the ability of dual-curing resin cement to bond to dentin.[10-12] Additionally, a delay in photo-activation of dual-cured resin cements may reduce polymerization shrinkage stress.[13] No studies have assessed how time lapse between manipulation of resin cements and photo-activation affects mechanical properties and residual shrinkage stresses in teeth restored with ceramic inlays.

The purpose of this study was to test the effect of delayed photo-activation of resin cements on 1) their mechanical properties (elastic modulus and Knoop hardness) and post-gel shrinkage and 2) residual shrinkage stresses in a premolar restored with a ceramic inlay restoration. The null hypotheses were that there would be no difference in mechanical properties and residual shrinkage stress among the resin cements due to photo-activation timing.

METHODS AND MATERIALS

Post-gel Shrinkage Measurements

Linear post-gel shrinkage of resin cement was determined using the strain gauge method.[14] The materials used were six dual cure resin cements: four were self-adhesive: RelyX Unicem (3M ESPE, St Louis, MO, USA), GCem (GC, Tokyo, Japan), MonoCem (Shofu, Tokyo, Japan), ad seT (SDI, Melbourne, Australia), and two are conventional resin cements: AllCem (FGM, Joinville, SC, Brazil) and RelyX ARC (3M ESPE). Composition and manufacturer information are listed in Table 1. Resin cement samples were shaped into hemispheres and placed on top of a biaxial strain gauge (CEA-06-032WT-120, Measurements Group, Raleigh, NC, USA) that measured shrinkage strains in two perpendicular directions. A strain conditioner (2101A Series, Micro Measurements Group) converted electrical resistance changes in the strain gauge to voltage changes through a quarter-bridge circuit with an internal reference resistance. Resin cement was photo-activated for 40 seconds with the light tip of the light-curing unit (XL 3000, 3M ESPE) placed at 1-mm distant from the surface of the resin cement. The radiant exposure was 32 J/cm^2 (800 mW/cm^2 $\times$ 40 seconds) measured by halogen light radiometer (Demetron, Danbury, CT, USA). Three photo-activation timings were tested: 1) light curing immediately, 2) after 3 minutes, or 3) after 5 minutes. Strain development (post-gel shrinkage) during polymerization shrinkage was monitored for 10 minutes, starting from the beginning of photo-activation. Ten specimens were tested for each resin cement at each time of photo-activation. The shrinkage strain was determined as the average of the strains in both perpendicular directions. The shrinkage strain values at 10 minutes were converted to volume percent

Table 1: *Resin Cement Compositions (Manufacturer Information)*

Resin cement	Wt%	Vol%	Composition	Manufacturer
RelyX Unicem	70	50	Base paste: fiberglass, phosphoric acid esters methacrylate, triethylene glycol dimethacrylate, silica treated silane, sodium persulfate Catalyst paste: fiberglass, substitute dimethacrylate, silane treated silica, p-toluenesulfonate sodium, and calcium hydroxide	3M ESPE
MonoCem	60	46	Mono-, di- and multifunctional acrylate resins, dual-initiators, fillers	Shofu
AllCem	68	57	Bis-GMA, Bis-EMA, TEGDMA, camphorquinone, initiators, stabilizers, microfillers of Ba-Al-silicate, silica nanoparticles microfiller and nanofillers	FGM Dental Products
GCem	71	57	4-MET, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer, fluoro-alumino-silicate glass, initiator, pigment	GC
seT	67	45	Methacrylate ester phosphoric acids, UDMA, photoinitiator, fluoride aluminum silicate glass and pyrogenic silica	SDI
RelyX ARC	67	45	Paste A: silane treated ceramic, TEGDMA, Bis-GMA, silane-treated silica, functionalized dimethacrylate polymer; 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-benzeneethanol. Paste B: silane-treated ceramic, TEGDMA, Bis-GMA, silane-treated silica, functionalized dimethacrylate polymer	3M ESPE

Abbreviations: BisEMA, bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; DMA, dimethacrylates; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MPS, 3-methacryloyloxypropyl trimethoxysilane; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

shrinkage by multiplying the strain values by 3% and 100%. Two-way analysis of variance (ANOVA) followed by Tukey honestly significant difference (HSD) post hoc tests (α=0.05) was used for the statistical analysis.

Elastic Modulus and Knoop Hardness Measurements

Five test specimens per resin cement were fabricated using a 2-mm-thick stone mold with a 5-mm-diameter circular opening. The stone mold was placed on a glass slide, filled with resin cement, and covered with another glass slide. The resin cement was light cured for 40 seconds (XL 2500, 3M ESPE) through the top glass slide at the three photo-activation times (immediately and after 3 or 5 minutes). Five Knoop indentations (MicroMet 5104, Buehler, Lake Bluff, IL, USA) were made to obtain an average Knoop hardness value for each specimen. The Knoop indentations were also used to determine the elastic modulus.[15-17] The decrease in the length of the indentation diagonals caused by elastic recovery of a material is related to the hardness/elastic modulus ratio (H/E) according to the following empirical relationship: $b'/a' = b/a - A\,(H/E)$, where b/a is the ratio of the diagonal dimensions a and b in the fully loaded state, given by a constant 0.140647. b'/a' is the ratio of the altered dimensions when fully recovered, and A = 0.45 is a proportionality constant. Two-way ANOVA followed by Tukey HSD post hoc tests (α=0.05) were used for the statistical analysis of the parameters elastic modulus and Knoop hardness.

Residual Stress Calculation: Finite Element Analysis

To calculate residual stresses in a restored tooth, a two-dimensional (2D) finite element simulation was carried out for a leucite ceramic (Empress CAD, Ivoclar Vivadent, Schaan, Liechtenstein) inlay restoration with the cavity floor in dentin. The geometric model was based on a digitized buccolingual cross section of a premolar. Coordinates were obtained using ImageJ software (public domain, Java-based image processing and analysis software developed at The National Institutes of Health, Bethesda, MD, USA). Only the cervical portion of the root was simulated because the rest of the root did not affect the coronal stress distribution.[16] A simplified boundary condition was assumed at the cut-plane of the root (zero displacements in horizontal and vertical directions). The elastic modulus of enamel was 84 GPa and Poisson's ratio 0.30; the dentin elastic modulus was 18 GPa and the Poisson's ratio was 0.23.[18] The elastic modulus of leucite ceramic was 65 GPa and Poisson's ratio was 0.19.[19] The shrinkage and elastic modulus values of the six resin cements were obtained from the experimental data. The Poisson's ratio was chosen to be the same for all resin cements at 0.35.[19]

The finite element analysis (FEA) was performed using MSC.Mentat (preprocessor and postprocessor)

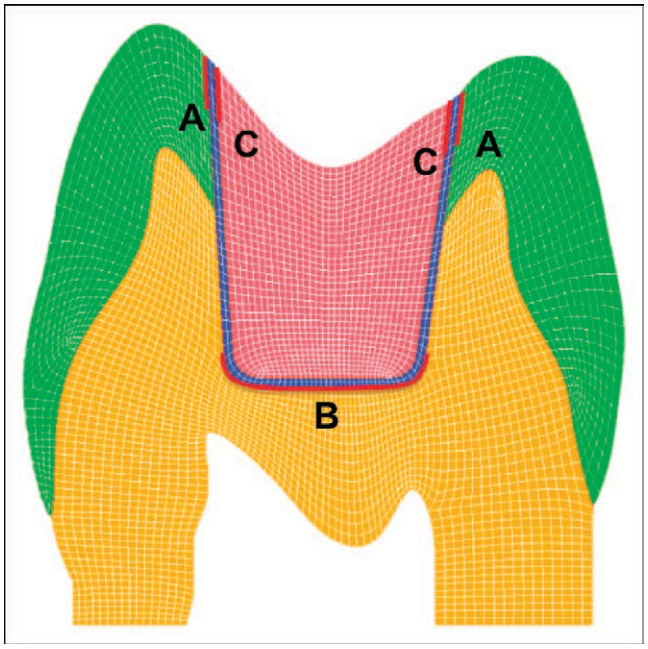

Figure 1. *Mesh of digitized ceramic inlay restored premolar showing the locations where the stresses were recorded: (A) Enamel aspect of the restoration margin corresponding to with marginal gap formation. (B) Dentin aspect of pulpal floor corresponding to the potential area of post-operative sensitivity. (C) Ceramic aspect of the inlay margin corresponding to with ceramic marginal fracture.*

and MSC.Marc (solver) software (MSC Software Corporation, Santa Ana, CA, USA). A plane strain condition was assumed for the tooth cross sections. Polymerization shrinkage was simulated by thermal analogy. Temperature was reduced by 1°C, while the linear post-gel shrinkage value was entered as the coefficient of thermal expansion. Modified von Mises equivalent stress was used to express the stress conditions, using compressive-tensile strength ratios of 3.3, 37.3, 3.0, and 3.9 for the ceramic, enamel, dentin, and resin cement, respectively.[16,19] Enamel stress values were recorded in nodes along the interface at the occlusal margin (Figure 1A). Dentin stress values were recorded in nodes along the interface at the pulpal floor (Figure 1B). Stress values in the ceramic aspect were recorded in nodes along the interface at the occlusal margin (Figure 1C). The mean values of the 5% highest stresses were determined.

RESULTS

Post-gel Shrinkage

The mean values and standard deviations for the volumetric post-gel shrinkage of six resin cements are presented in Table 2. Two-way ANOVA revealed statistical differences among the resin cements ($p<0.001$) and timing of photo-activation ($p<0.001$); however, no difference was found for interaction between resin cement and timing ($p=0.319$). The Tukey HSD test showed that the post-gel shrinkage values were progressively lower with delayed photo-activation, irrespective of resin cements. GCem had the lowest post-gel shrinkage values; seT had intermediate post-gel shrinkage, whereas RelyX Unicem, RelyX ARC, Monocem, and AllCem had high post-gel shrinkage values irrespective of photo-activation timing.

Elastic Modulus

The mean values and standard deviations for the elastic modulus of six resin cements are presented in Table 3. Two-way ANOVA revealed statistical differences among the resin cements ($p<0.001$); however, no difference was found for photo-activation timing ($p=0.556$) and for the interaction between resin cement and timing ($p=0.061$). RelyX Unicem had the highest elastic modulus values; GCem, RelyX ARC, and AllCem had intermediate values; and seT and MonoCem showed the lowest values, irrespective of photo-activation timing.

Knoop Hardness

The mean values and standard deviations for Knoop hardness of six resin cements are presented in Table

Table 2: *Mean and Standard Deviation of Post-Gel Shrinkage (Volume %)*

Resin cements	Post-gel shrinkage			
	Immediate	3 minutes	5 minutes	Pooled average
RelyX ARC	0.97 (0.05)	0.73 (0.11)	0.64 (0.09)	0.78 (0.16)^{C}
AllCem	0.94 (0.06)	0.72 (0.08)	0.65 (0.06)	0.77 (0.14)^{C}
RelyX Unicem	0.93 (0.05)	0.82 (0.09)	0.77 (0.03)	0.84 (0.10)^{C}
MonoCem	0.91 (0.10)	0.83 (0.07)	0.71 (0.09)	0.82 (0.12)^{C}
seT	0.78 (0.13)	0.66 (0.08)	0.58 (0.13)	0.67 (0.16)^{B}
GCem	0.61 (0.03)	0.52 (0.06)	0.33 (0.02)	0.49 (0.17)^{A}
Pooled average	0.86 (0.15)^{c}	0.71 (0.13)^{b}	0.61 (0.17)^{a}	

Mean values with same letters are not significantly different (p>0.05). Uppercase letters compare among resin cements and lowercase letters compare among time of photo-activation.

Table 3: *Mean and Standard Deviation of Elastic Modulus (GPa)*

Resin cements	Elastic modulus			
	Immediate	3 minutes	5 minutes	Pooled average
RelyX Unicem	18.6 (1.9)	18.4 (1.9)	18.1 (2.0)	18.4 (2.0)[A]
GCem	14.0 (1.7)	13.7 (1.5)	13.4 (1.8)	13.7 (1.6)[B]
RelyX ARC	12.3 (1.1)	12.7 (1.2)	12.8 (1.7)	12.6 (1.2)[B]
AllCem	11.7 (1.3)	11.5 (0.9)	12.0 (1.1)	11.7 (1.1)[B]
seT	9.5 (1.0)	9.6 (0.9)	9.7 (1.0)	9.6 (1.0)[C]
MonoCem	9.0 (0.8)	9.1 (0.9)	9.7 (0.7)	9.2 (0.8)[C]
Pooled average	12.5 (3.4)[a]	12.6 (3.2)[a]	12.5 (3.5)[a]	

Mean values with same letters are not significantly different ($p>0.05$). Uppercase letters compare between resin cements and lowercase letters compare among photo-activation timings.

4. Two-way ANOVA revealed statistical differences among the resin cements ($p<0.001$); however, no difference was found for time of photo-activation ($p=0.927$) or for the interaction between resin cement and timing ($p=0.306$). AllCem and RelyX Unicem had the highest mean Knoop hardness values; RelyX ARC, seT, and GCem had intermediate mean values, and MonoCem showed the lowest values, irrespective of photo-activation timing.

Residual Stress in FEA

Stress distributions are shown in Figures 2 (tooth structure) and 3 (ceramic inlay). They indicate stress concentrations (modified von Mises equivalent stress) in the tooth and inlay at the occlusal margins and in the dentin at the pulpal line angles. The highest levels of residual stresses were identified by calculating the mean values of the 5% highest modified von Mises stresses in the enamel aspect along the enamel/resin cement interfacial margin, the ceramic aspect along the ceramic/resin cement interfacial margin, and the dentin aspect along the pulp floor dentin/resin cement interface (Table 5). The highest maximum stresses were found for the RelyX Unicem and the lowest for the seT. In all cases, the stress level decreased with increasing delays in photo-activation (Table 6).

DISCUSSION

Volumetric shrinkage is a consequence of the polymerization process, whereby the conversion of monomer molecules results in a cross-linked polymer network.[20] During this polymerization reaction, resinous materials like cements change from a viscous to a predominantly solid substance, which can be characterized by the development of the elastic modulus.[21,22] Residual shrinkage stresses are generated when the surrounding tooth structure restricts volumetric changes after the elastic modulus has developed.[23] It can be theorized that this restriction is high for cementation of indirect restorations compared with direct composite resin restorations.

FEA was used to assess the stresses that polymerization shrinkage in the resin cement may pose on the tooth structure and ceramic inlay. This numer-

Table 4: *Mean and Standard Deviation of Knoop Hardness (KHN)*

Resin cements	Knoop hardness			
	Immediate	3 minutes	5 minutes	Pooled average
AllCem	51.0 (2.3)	50.5 (2.0)	50.7 (2.9)	50.7 (2.5)[A]
RelyX Unicem	49.3 (4.3)	49.8 (4.6)	49.8 (3.9)	49.6 (2.5)[A]
RelyX ARC	47.0 (4.0)	46.9 (3.2)	46.4 (3.5)	46.8 (3.5)[B]
seT	45.6 (3.4)	45.8 (3.1)	46.2 (2.7)	45.9 (2.5)[B]
GCem	44.0 (2.2)	44.8 (2.0)	43.6 (2.9)	44.1 (2.5)[B]
MonoCem	31.8 (2.1)	30.6 (3.8)	30.8 (2,2)	31.1 (2.5)[C]
Pooled average	44.8 (6.9)[a]	44.6 (6.8)[a]	44.8 (6.5)[a]	

Mean values with same letters are not significantly different ($p>0.05$). Uppercase letters are used to compare between resin cements and lowercase letters to compare among photo-activation timings.

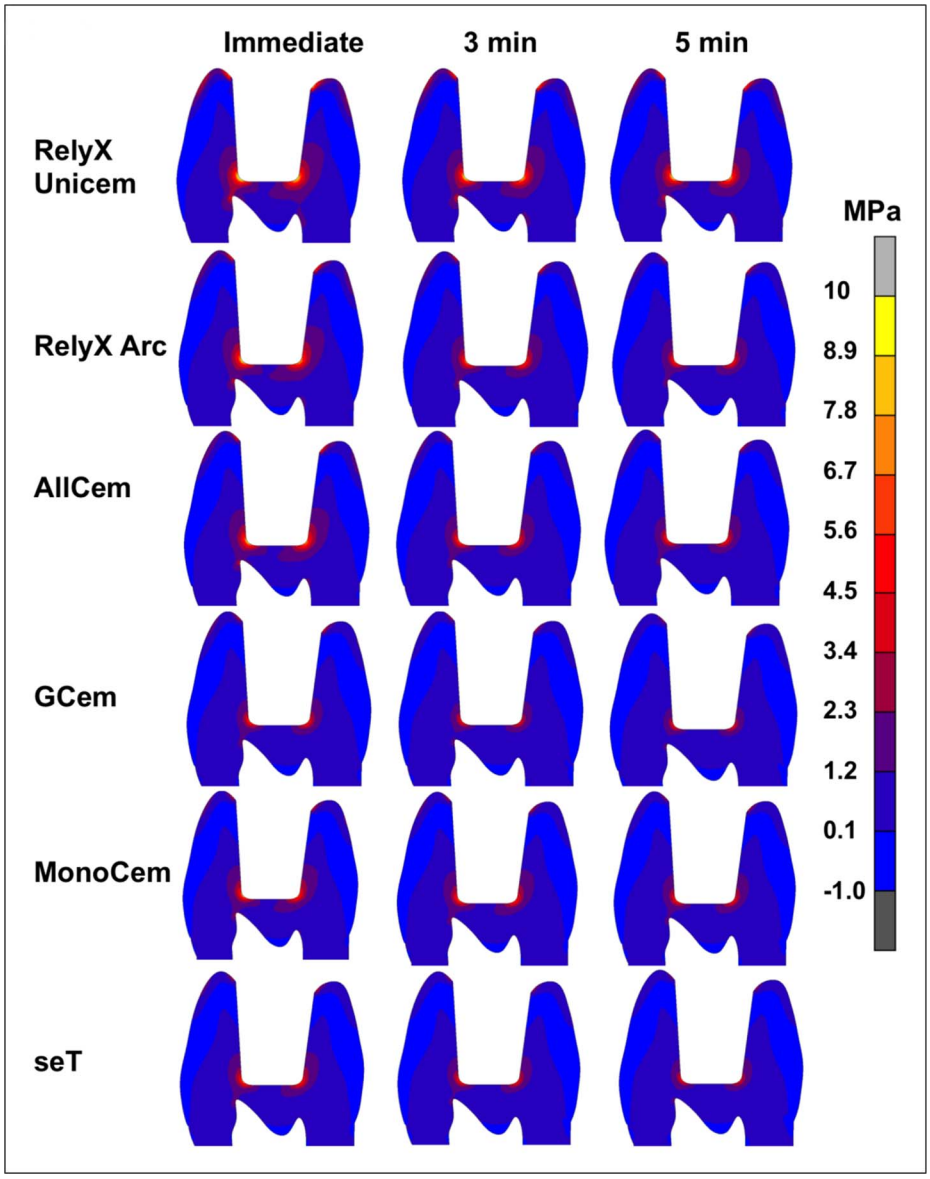

Figure 2. *Residual shrinkage stress distributions (modified von Mises stress) in the tooth structure of a premolar restored with a ceramic inlay using six dual cure resin cements that were cured at three photo-activation timings (immediate and 3 or 5 minutes delayed).*

Table 5: *Mean and Standard Deviation of the Highest 5% Modified von Mises Equivalent Stress Values (MPa) Recorded in the Enamel and Ceramic Along the Occlusal Margins and in the Dentin at the Pulpal Floor*

Resin cements	Enamel margins			Ceramic margins			Dentin, pulpal floor		
	Immediate	3 minutes	5 minutes	Immediate	3 minutes	5 minutes	Immediate	3 minutes	5 minutes
RelyX Unicem	25.4 (2.0)	22.2 (1.8)	20.9 (1.7)	26.1 (2.6)	22.8 (2.2)	21.4 (2.1)	29.1 (1.0)	25.4 (0.9)	23.8 (0.8)
RelyX ARC	17.9 (1.3)	13.9 (1.0)	12.3 (0.9)	18.4 (1.5)	14.3 (1.2)	12.6 (1.1)	21.0 (1.0)	16.3 (0.8)	14.3 (0.7)
AllCem	16.5 (1.2)	12.5 (0.9)	11.7 (0.8)	17.0 (1.4)	12.8 (1.0)	12.1 (1.0)	19.5 (1.0)	14.7 (0.8)	13.8 (0.7)
GCem	12.7 (1.0)	10.6 (0.8)	9.8 (0.7)	13.1 (1.1)	11.0 (0.9)	10.1 (0.9)	14.8 (0.3)	12.4 (0.5)	11.4 (0.5)
MonoCem	12.4 (0.8)	11.4 (0.8)	10.0 (0.7)	12.8 (0.9)	11.8 (0.8)	10.3 (0.8)	15.0 (1.0)	13.9 (0.9)	12.1 (0.8)
seT	11.4 (.08)	9.6 (0.7)	8.5 (0.6)	11.7 (0.9)	9.9 (0.7)	8.8 (0.7)	13.6 (0.8)	11.5 (0.7)	10.2 (0.6)

Table 6: *Percentage Reduction in Stress Levels (Modified von Mises) of All Analyzed Regions When Photo-Activation Was Delayed 3 or 5 Minutes.*

Resin cements	Stress reduction		
	Immediate	3 minutes	5 minutes
AllCem	—	−34%	−42%
RelyX ARC	—	−29%	−46%
seT	—	−19%	−34%
GCem	—	−20%	−30%
MonoCem	—	−18%	−24%
RelyX Unicem	—	−15%	−22%

ical method takes into account the geometrical restrictions experienced by the shrinking cement layer and combines them with the two major material properties involved in stress development: shrinkage and elastic modulus. Post-gel shrinkage was applied for the calculation because not all shrinkage induces stresses—only shrinkage that occurs when the resin cement has developed elastic properties. The elastic properties, which are the properties that prevent relief by viscous flow, were characterized by the elastic modulus. The determined stress distributions were therefore not determined by any individual property (shrinkage or

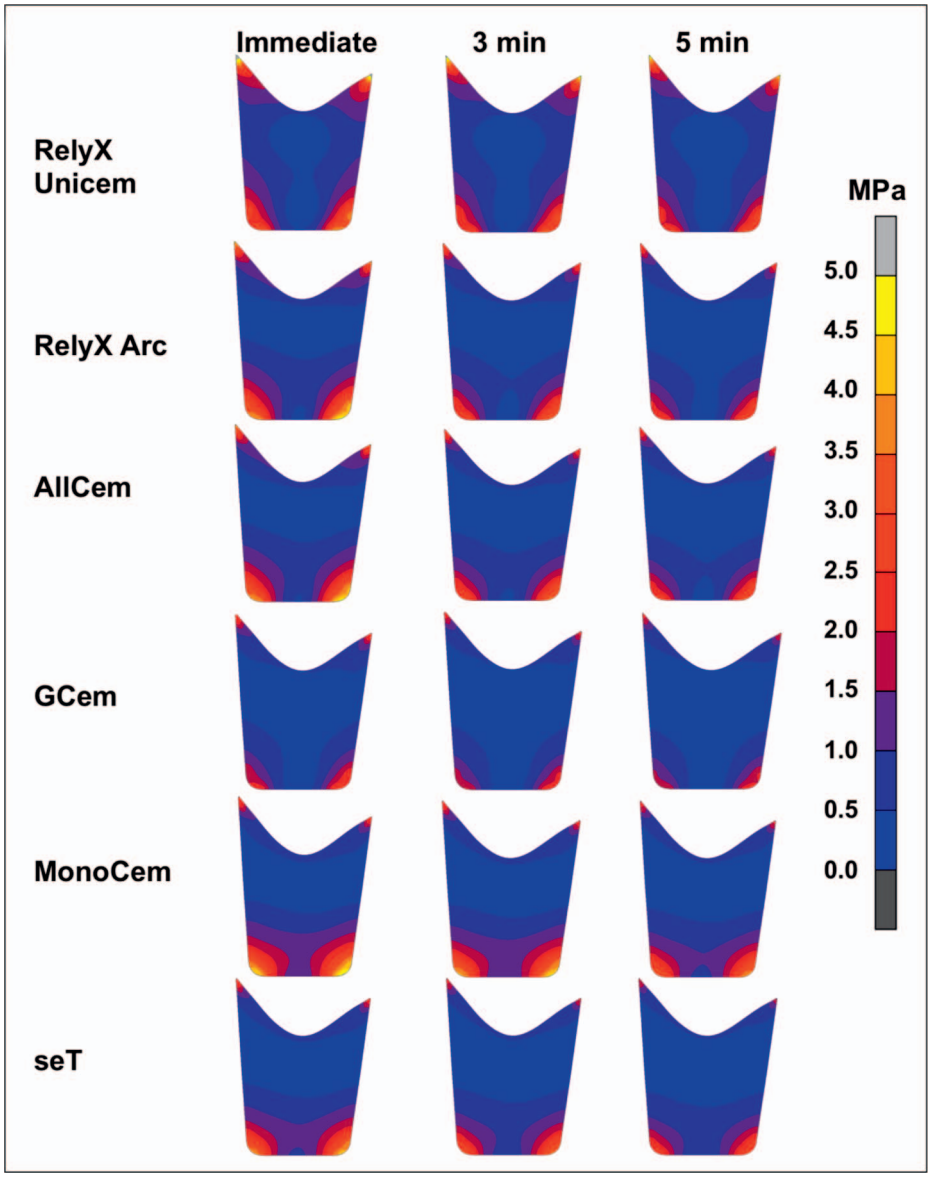

Figure 3. *Residual shrinkage stress distributions (modified von Mises stress) in a ceramic inlay using six dual cure resin cements that were cured at three photo-activation timings (immediate, and 3 or 5 minutes delayed).*

elastic modulus) or cavity configuration (bonding area), but were the result of the combination of all elastic properties and restriction factors (bonding and stiffness of tooth and inlay structures). However, by standardizing the restriction factors in our analysis, the differences between residual shrinkage stresses depended only on the shrinkage and elastic modulus of the resin cements.

The highest post-gel shrinkage values were found for RelyX ARC, AllCem, RelyX Unicem, and MonoCem. RelyX Unicem also had the highest elastic modulus, followed by GCem and RelyX ARC. The highest residual shrinkage stress level was found in RelyX Unicem, followed by RelyX ARC and AllCem. GCem, which had the lowest post-gel shrinkage but a relatively high elastic modulus, and MonoCem, which had the lowest elastic modulus but high post-gel shrinkage, developed intermediate shrinkage stress levels, whereas seT with relative low post-gel shrinkage and elastic modulus values resulted in the lowest shrinkage stress. This confirms that, all other conditions being equal, it is the combination of post-gel shrinkage and elastic modulus that determines the shrinkage stress levels.

Given full polymerization, mechanical properties such as elastic modulus and hardness were unaffected by the delayed photo-activation. However, delayed polymerization is advantageous by reducing the post-gel shrinkage values and thus decreased residual shrinkage stress levels in the restored tooth complex. Similar behavior has been shown previously for restorative composites, where photo-activation at lower light intensities reduced post-gel shrinkage.[24,25] This was explained by the slower reaction that allowed more viscous flow during polymerization and thus reduces the post-gel portion of the total shrinkage.[26] Delaying the photo-activation of dual-cure resin cements used with endodontic posts has shown a similar effect,[13] slowing down the polymerization reaction, allowing more viscous flow and therefore reducing the post-gel shrinkage without jeopardizing other mechanical properties as evidenced by the elastic modulus and Knoop hardness values that were unaffected by the delayed curing.

The decrease in post-gel shrinkage due to delayed photo-activation resulted in shrinkage stress reductions of 15%-34% with the 3-minute delays and 22%-42% with the 5-minute delays, depending on the resin cement type. Conventional resin cements (AllCem and RelyX ARC) showed higher reduction than self-etching resin cements (GCem, MonoCem, seT, and RelyX Unicem), probably because the monomer composition of these cements is more reactive immediately after mixing. It has been suggested that photo-activation immediately after resin cement mixing may negatively affect the self-curing mechanism, because rapid formation of a cross-linked polymer on light exposure leads to entrapment of the activators and initiators needed for the self-cure reaction.[27] In our study, delayed photo-activation did not negatively affect the mechanical properties (Knoop hardness and elastic modulus), which is consistent with previous reports.[28,29]

Failure of indirect restorations is often attributed to marginal fracture, marginal deterioration, discoloration, and secondary caries.[3,4] Cement close to the restoration margin has been shown to have high stress concentrations,[7] and this coincides with the area where bond and ceramic failure initiates. We also found residual shrinkage stress concentrations at the enamel and ceramic/resin cement interface close to the margin. Functional stresses in these areas can be elevated by these residual stresses concentrations, raising the overall stress levels at the occlusal margins and potentially causing bond failure, gap formation, or initiating cracks in the ceramic restoration. Gaps retain debris and pigments, resulting in marginal discoloration. Marginal discoloration is a frequent reason for replacement of indirect ceramic restorations because it can be misinterpreted as secondary caries. The cavosurface angle is also the thinner and thus weaker part of ceramic inlay and onlay restorations. Stress concentrations at this thin ceramic margin have been associated with formation of microcracks in ceramic restorations.[30,31] This study found residual shrinkage stress concentrations in the margins of the ceramic restoration, which may add an additional challenge to this vulnerable area. Avoiding or reducing shrinkage stresses at the margins could therefore increase the longevity of ceramic restorations. This study suggests that if clinicians delay photo-activation for 3-5 minutes after mixing the resin cements, the reduction in post-gel shrinkage will decrease shrinkage stresses and consequently lower the risk of marginal failure of indirect ceramic restorations. The 5-minute delay reduced post-gel shrinkage significantly more than the 3-minute delay and further reduced the residual shrinkage stress. It should also be emphasized that the delayed photo-activation did not decrease the mechanical properties of the resin cement as evidenced by the elastic modulus and hardness values shown in Tables 3 and 4.

The main reason for using dual cure resin cement is that light cannot reach the floor of the cavity; therefore, the cure of the cement at the pulp floor depends on its auto cure component. Any accompanying shrinkage stress may cause postoperative sensitivity. Postoperative sensitivity is a frequent cause of indirect restoration failures.[32] Confined polymerization shrinkage in tight interfacial spaces may cause fluid movement within the dentinal tubules and cause postoperative sensitivity.[33] Dentin deformation caused by shrinkage stress of the resin cements may also stimulate nerves directly or may exert mechanically induced dentinal fluid flow that triggers nerve activity.[34] We found high stress concentrations at the pulpal floor for all resin cements. This may explain the relatively high percentage of postoperative sensitivity (13.3%) observed with dual cure conventional resin cement.[31]Although postoperative sensitivity tends to reduce with time, it is an undesirable incident.[35,36] Our study shows that delaying photo-activation may reduce shrinkage stresses at the pulpal floor and thus can reduce the incidence of postoperative sensitivity.

In this study, the inlay was simulated in a premolar only for simplification of the model because the findings can be extrapolated to clinically more common onlays and crowns. The results of in vitro studies should be carefully interpreted before being extrapolated to a clinical context. However, the general behavior of resin cements that are subjected to delayed photo-activation is consistent with previously reported observations and should also happen under clinical conditions. Considering no negative effect on the mechanical properties was found, this study suggests a potential strategy for clinicians to cement indirect ceramic restorations with delayed photo-activation. The current analysis indicates that the best results were found when photo-activation was delayed for 5 minutes after mixing the resin cements, which is clinically feasible considering the time used for seating the indirect restoration.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn: 1) Knoop hardness and elastic modulus values varied among the different resin cements; 2) post-gel shrinkage decreased significantly with increasing the photo-activation delay; 3) delayed photo-activation had no effect on elastic modulus and Knoop hardness of all resin cements; 4) resin cements with high post-gel shrinkage and elastic modulus values caused higher shrinkage stresses; 5) the residual stress is higher on the pulp floor than on margins of the ceramic inlay; and 6) residual stresses on all regions analyzed decreased with 3 and 5 minutes of delayed photo-activation for all resin cements compared with immediate activation.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Federal University of Uberlândia in Brazil.

Conflict of Interest

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

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Degree of Conversion of Self-etch Adhesives: *In Situ* Micro-Raman Analysis

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Clinical Relevance

The degree of conversion is a fundamental parameter with which to evaluate adhesives' clinical performance and stability in the oral environment; based on the results of this study, all of the tested self-etch adhesives showed a clinically acceptable degree of conversion.

SUMMARY

Purpose: Degree of conversion (DC) affects the physicochemical properties of dental adhesives. The aim of this study was to measure the DC within the hybrid layer of four one-step self-etch adhesives using Raman microspectroscopy. The hypothesis tested was that there was no difference among the tested adhesives.

Methods and Materials: The selected one-step self-etch adhesives (Clearfil S^3 Bond Plus, I-BOND, G-BOND, and Adper Easy Bond) were applied on human dentin disks and polymerized in accordance with the manufacturers' instructions. Specimens were transversally cut to expose the bonded interfaces to the micro-Raman beam, and Raman spectra were collected along the dentin/adhesive interface. Measurements were performed at 1-μm intervals. The relative intensities of bands associated with the C=C bond (at 1640 cm^{-1}) and an internal stable peak (1610 cm^{-1}) were determined to calculate the degree of conversion within the hybrid layer. Data were statistically analyzed with Kolmogorov-Smirnov and Bartlett tests and Kruskal-Wallis and Mann-Whitney *U*-tests.

Results: The DC ranked as follows: G-BOND (93%±6%) ≥ Adper Easy Bond (92%±6%) ≥ I-BOND (89%±7%) > Clearfil S3 Bond Plus (80%±14%) ($p<0.05$).

Conclusions: Based on the results of this study, all of the tested self-etch adhesives showed a

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clinically acceptable DC that was material dependent.

INTRODUCTION

Dentin bonding systems can be divided into etch-and-rinse or self-etch materials depending on their bonding application procedures. The current self-etch adhesives are characterized by the application of an acidic primer/adhesive mixture that can be simultaneously applied on enamel and smear layer–covered dentin: this procedure avoids the discrepancy between etched collagen network and resin infiltration and reduces the technique sensitivity that characterizes the etch-and-rinse approach.[1]

One-step self-etch adhesives combine etching, priming, and bonding into a single product. They are easy and fast to use; however, previous studies[2-5] reported that immediate bond strength to dentin of one-step self-etch adhesive systems is generally lower in comparison to that associated with multi-step systems. Additionally, significant bond strength reduction and increased interfacial nanoleakage expression were reported[6,7] for one-step self-etch adhesives in long-term studies.

A good collagen impregnation and a high degree of conversion (DC) of the adhesive monomers are fundamental for the longevity of composite restorations. It was hypothesized that the poor performance of self-etch adhesives could depend upon shallow resin tag penetration produced by the self-etching process, an inefficient curing caused by their acidic nature,[8] or solvent retention and phase separation phenomena due to the coexistence of both hydrophilic and hydrophobic moieties in the same product.[9,10] A low rate of monomer-to-polymer conversion in polymers leads to weaker polymer networks, low physicochemical properties, and, therefore, higher susceptibility to degradation processes.[11-13] Moreover, self-etch adhesives behave as permeable membranes,[13,14] presenting unreacted monomers in their hybrid layer.[15] The hydrophilicity of one-step self-etch adhesives into the hybrid layer can lead to increased water uptake, plasticization of the polymer network, and elution of unreacted monomers.[13,16,17]

Micro-Raman spectroscopy is a helpful tool for the *in situ* evaluation of DC of adhesive resins because it can directly measure the percentage of converted double-bonds within the adhesive layer in a nondestructive manner. Indeed, a spatially resolved chemical analysis of the hybrid layer can be performed through micro-Raman spectroscopy, detecting chemical changes in bands of interest, such as vinyl and phenyl C=C bonds, after minimal specimen preparation steps. Although other spectroscopy techniques, such as micro–Fourier transform infrared spectroscopy (FTIR), can also provide a high–spatial resolution map of molecular vibrational changes, micro-Raman spectroscopy is less sensitive to water and requires minimal specimen preparation.[15,18,19] Although the DC of dental adhesives was previously investigated *in vitro,* there is still a lack of knowledge of the DC *in situ* into the hybrid layer of commercial adhesives.

The aim of this study was the evaluation of the DC within the hybrid layer of four commercial one-step self-etch adhesives. The tested hypothesis was that no differences in terms of DC were present among the adhesives systems investigated in this study.

METHODS AND MATERIALS

Tooth Preparation and Bonding Procedures

Noncarious human third molars (n=12) stored in 0.5% chloramine in water at 4°C were collected after informed consent was obtained from patients under a protocol approved by the University of Trieste, Italy. To ensure that the sample size was adequate, the power of the statistical test had been previously verified (Software G*Power 3, Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany). A low-speed diamond saw with water cooling (Isomet 5000 Buehler Ltd, Lake Bluff, IL, USA) was used to expose middle/deep dentin, and a uniform smear layer was obtained with 180-grit silicon carbide (SiC) paper to simulate the bur's action. To ensure that any pulp was not exposed, an accurate surface inspection under a 50× light microscope (Leica, Wetzlar, Germany) was performed.

The dentin disks were then randomly and equally assigned to the tested one-step self-etch adhesives (n=3), as follows: group 1: Clearfil S^3 Bond Plus (Kuraray Medical Inc, Tokyo, Japan); group 2: I-BOND (Heraeus Kulzer, Hanau, Germany); group 3: G-BOND (GC America Inc, Alsip, IL, USA); group 4: Adper Easy Bond (3M ESPE, St Paul, MN, USA). Adhesive compositions and application modes are listed in Figures 1 through 4.

Dentin bonding systems were applied in accordance with manufacturers' instructions (Figures 1 through 4) and polymerized using a quartz-halogen lamp (Elipar 2500; 3M ESPE) with an irradiance of 600 mW mm^{-2}, as previously assayed using a 400-500-nm radiometer (100 Optilux radiometer; SDS Kerr, Danbury, CT, USA), maintaining the light tip at a distance of 1 mm from the adhesive surface. A 2-

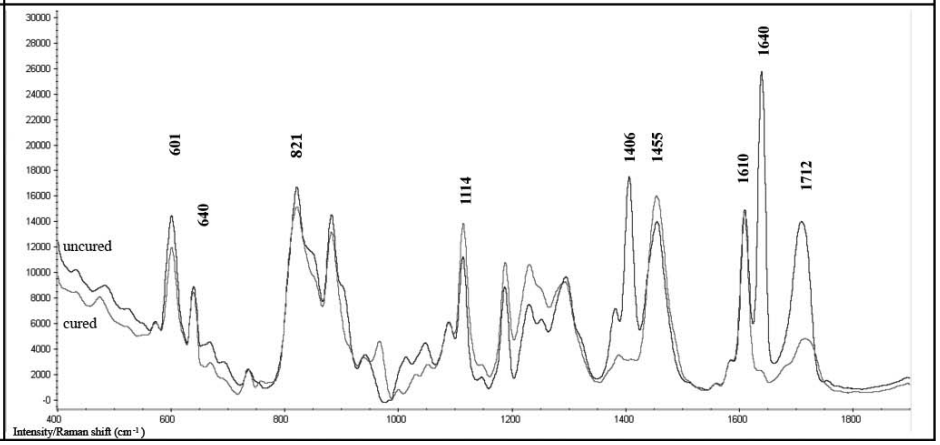
Adhesive system	Composition	Application mode	Raman spectrum
Group 1: CLEARFIL S^3 BOND PLUS	MDP, Bis-GMA, HEMA, Colloidal silica, Ethanol, Water, dl-Camphorquinone, Photo initiator, Hydrophilic aliphatic dimethacrylate, Hydrophobic aliphatic methacrylate, Photo polymerization accelerator, Chemical polymerization accelerator, Sodium fluoride, Others	Apply to tooth surface for 10 s. Dry with mild air for more than 5 s until the bond does not move. Light cure for 10 s.	

Figure 1. *Composition, application mode (as recommended by the respective manufacturer), and Raman spectra of cured and uncured Clearfil S^3 Bond Plus. The decrease in the C=C peak after polymerization is visible when comparing the spectra of the uncured adhesive. Refer to Table 1 for other peaks. Abbreviations for all the figures: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloxydecyl dihydrogen phosphate; 4-META, 4-methacryloxyethyltrimellitic acid anhydride; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.*

mm layer of resin composite (Filtek Z250; 3M ESPE) was applied on the adhesive surface and light-cured for 20 seconds. Each specimen was then cut transversally to obtain two 2-mm slabs. The two middle slabs were selected and then polished to expose the adhesive interface for micro-Raman analysis.[20] Polishing was performed for 30 seconds with wet SiC papers (up to 800 grit), followed by 6-, 1-, and 0.05-μm diamond pastes, with distilled water rinsing between each step. Slabs were then cleaned with soap, sonicated for one minute, and rubbed with a cotton swab saturated with 5% sodium hypochlorite to remove smeared proteins produced by polishing from the surface. The specimens were dry-stored in a dedicated cupboard at controlled room temperature (22°C) with silica gel to obtain a dry environment.

Micro-Raman Spectroscopy

The equipment used consisted of a computer-controlled laser Raman microprobe connected to a DM/LM optical microscope with a 100× objective (NA 0.9; N Plan EPI objective; Leica, Wetzlar, Germany) and

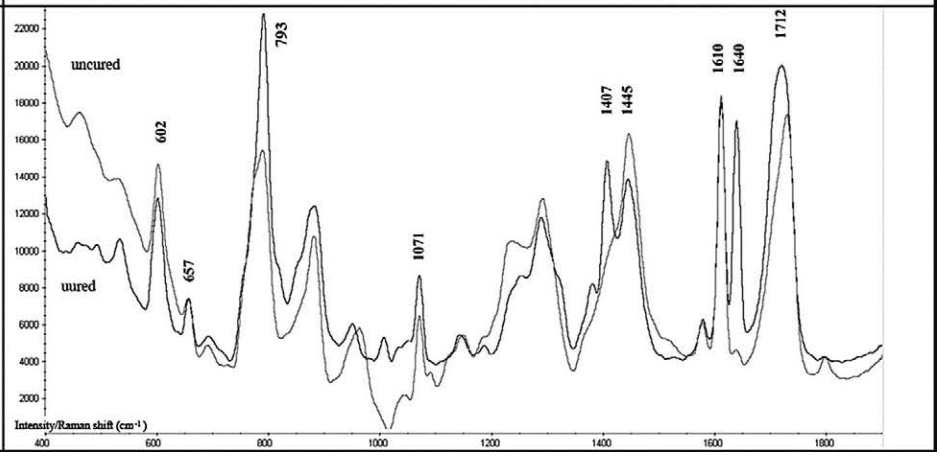
Adhesive system	Composition	Application mode	Raman spectrum
Group 2: I-BOND	Acetone, Water, Methacrylates, 4-META, UDMA, Photo initiator	Apply to dentin and agitate slightly for 20 s. Air-dry at least for 5 s. Light cure for 20 s.	

Figure 2. *Composition, application mode (as recommended by the respective manufacturer), and Raman spectra of cured and uncured I-BOND. The C=C peak decreased after polymerization. Refer to Table 1 for other peaks.*

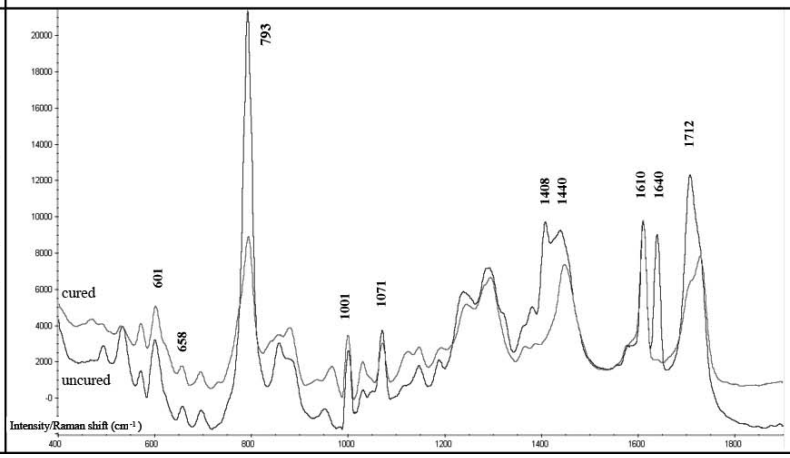
Adhesive system	Composition	Application mode	Raman spectrum
Group 3: G-BOND	Acetone, Distilled water, 4-META, UDMA, TEGDMA, Dimethacrylate component, Phosphoric ester monomer	Brush, apply one coat and wait 5 s Thin with high-pressure syringe air. Light cure for 10 s.	

Figure 3. *Composition, application mode (as recommended by the respective manufacturer), and Raman spectra of cured and uncured G-BOND. The decrease in the reaction peak is clearly visible in the spectrum of the cured adhesive. Refer to Table 1 for other peaks.*

a CCD detector attached to a modular research spectrograph (Renishaw InVia; Renishaw PLC, New Mills, Wotton-under-Edge, Gloucestershire, UK). The 100× objective increased the precision of the beam, resulting in a laser spot size of ≤ 1 μm. A monochromatic, near-infrared diode 785-nm laser was used to induce the Raman scattering effect. The spectral range of this model was from 100 to 3450 cm^{-1}, with an average spectral resolution of 5 cm^{-1}. A wavelength and intensity calibration was performed with a silicon standard using the calibration system integrated with the software (WiRE 2.0; Renishaw PLC) before each experiment following the manufacturer's specifications.

The dentin slabs were analyzed by acquiring spectra in three line-scans for each slab, starting in the sound mineralized dentin and ending in the adhesive resin layer. One Raman spectrum was collected every 1 μm along the dentin-adhesive interface in the intertubular region clearly detected by the microscope camera (laser wavelength" 785 nm; Renishaw InVia; Renishaw PLC) using a computer-controlled motorized *x-y-z* stage, with an exposure time of 40 seconds and a laser power on the

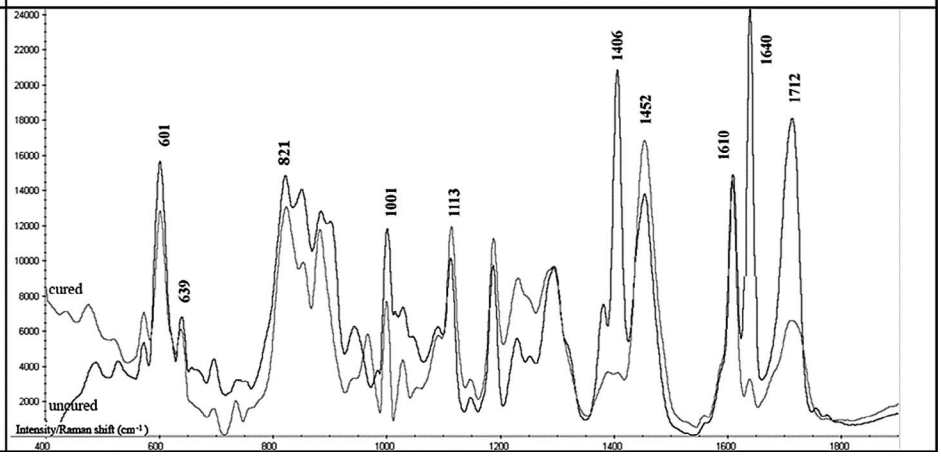
Adhesive system	Composition	Application mode	Raman spectrum
Group 4: ADPER EASY BOND	HEMA, Bis-GMA, Methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, Methacrylate functionalized, Polyalkenoic acid (VitrebondTM Copolymer), 7 nm silica filler, Ethanol, Water, Camphorquinone based initiators, Stabilizers	Apply to dentin and rub for 20 s. Gentle air-dry for 5 s. Light cure for 10 s.	

Figure 4. *Composition, application mode (as recommended by the respective manufacturer), and Raman spectra of cured and uncured Adper Easy Bond. The decrease in the C=C peak at 1640 cm^{-1} is visible in the cured adhesive. Refer to Table 1 for other peaks.*

Table 1: *Attribution of the Peaks of Interest to the Corresponding Functional Group*[21]

Functional Group	Range Signal, cm^{-1}
O-C=O bending	590-700
C-O-H bending	620-680
Mono-substituted benzene	690-800
Para-substituted benzene	800-850
P-O-C antisymmetrical stretch	915-1055
C-O-C antisymmetrical stretch	1070-1280
OH in-plane bending (acid)	1400-1440
CH_2 scissors vibration CH_3 antisymmetrical deformation	1440-1475
C=C aromatic ring stretch	1590-1615
C=C aliphatic stretch	1630-1680
C=O carbonyl ester stretch	1650-1870

specimen of ~8 mW. Data were acquired over the spectral region from 400 to 1900 cm^{-1}. They were analyzed with software developed for spectrographic analysis (Grams/AI 7.02; Thermo Galactic Industries, Salem, NH, USA).

Hybrid Layer Detection and Conversion Calculation

The spectra were acquired, starting from the dentin, and the appearance of peaks associated with the adhesive and dentin components indicated the beginning of the hybrid layer. This allowed investigation of the impregnation pattern of the adhesives into the dentin structure. To detect the hybrid layer within the adhesive interface the relative intensities of bands associated with mineral dentin components (the PO_4^{3-} functional group at 960 cm^{-1}) and adhesive (vinyl C=C at 1640 cm^{-1} and phenyl C=C at 1610 cm^{-1}) were identified (Table 1).[21] The major peak at 960 cm^{-1} is associated with the mineral PO_4^{3-} group, while the bands at 1245 (C–N), 1450 (CH_2), and 1667 cm^{-1} (C=O) are associated with the dentin organic components[19] (Table 1).[21] Because mild and ultramild self-etch adhesives demineralize dentin only superficially and create a very thin hybrid layer,[22,23] only the first two spectra (2 μm) in which the mineral and the adhesive coexisted were considered as the hybrid layer (ie Figure 5a, spectra at micron 6). Even when both of these spectra were

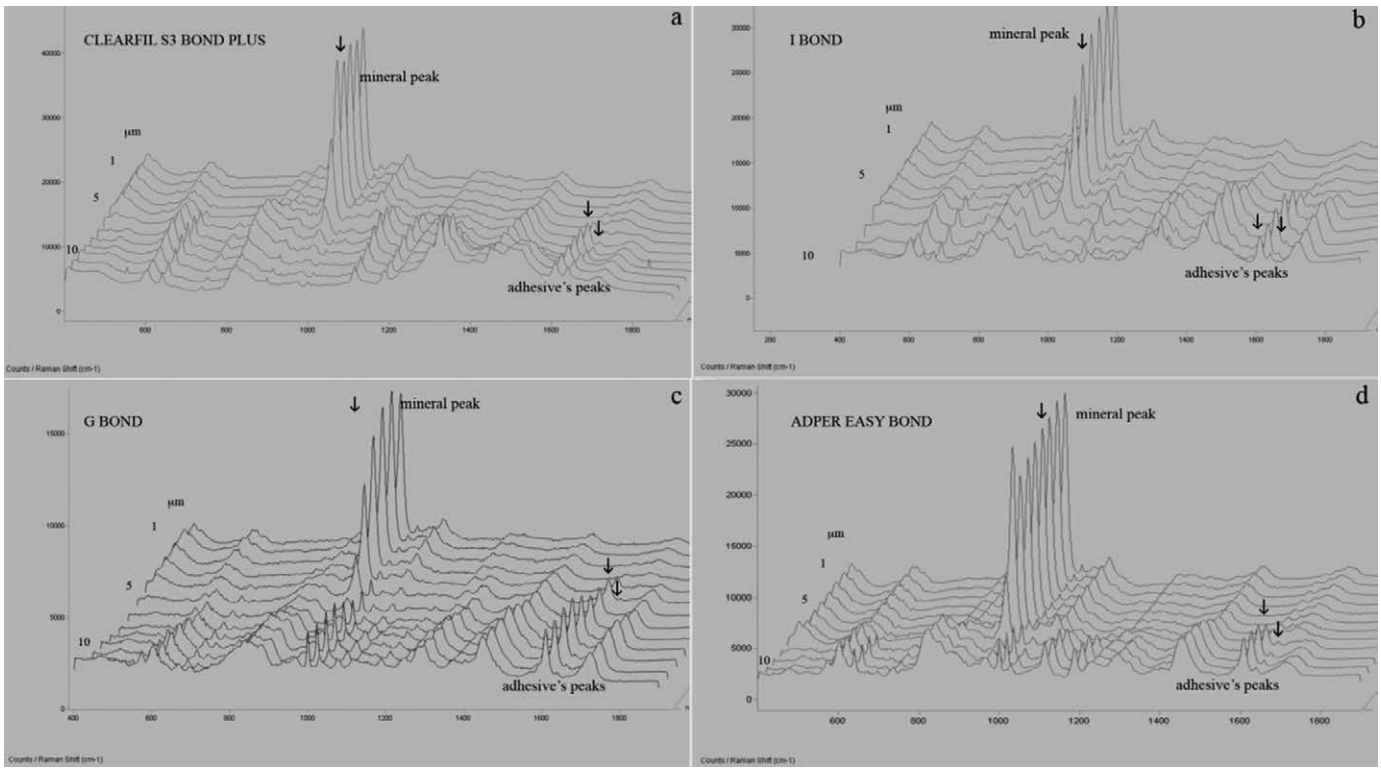


Figure 5. *(a and c) Spectra 1-5 were in mineralized dentin, spectrum 6 was taken along the hybrid layer, and spectra 7-12 were representative of the adhesive layer. (b) Spectra 1-6 were in mineralized dentin, spectrum 7 was taken along the hybrid layer, and from spectra 8 they were in the adhesive layer. (d) Spectra 1-8 were in mineralized dentin, spectrum 9 was taken along the hybrid layer, and the last two were representative of the adhesive layer.*

Table 2: *Degree of Conversion (DC) of the Adhesives Tested**

Adhesive System	DC, %
Cleafil S^3 Bond Plus	80 ± 14^{a}
I-BOND	89 ± 7^{b}
G-BOND	93 ± 6^{c}
Adper Easy Bond	92 ± 6^{bc}

*Means of DC followed by the same superscript letter indicate no statistical difference at the 95% confidence level ($p<0.05$).

still present, more superficial spectra were considered to represent the adhesive layer only, in which the demineralized buffered mineral might be dispersed. Raman spectra of each uncured adhesive were also collected to identify the reference and reaction peaks needed for conversion calculations, and these spectra were compared with the spectra of cured adhesive on dentin (Figures 1 through 4). The phenyl C=C peak at 1610 cm^{-1}, which was stable and unmodified during polymerization, was selected as a reference peak, while the vinyl C=C 1640 cm^{-1} peak was selected as a reaction peak. The DC of the adhesive within the hybrid layer was calculated using the ratio between the reaction (A_{rxn}) and the internal reference (A_{ref}) peak areas, as follows:

$$\%conversion = \left\{1 - \frac{[A_{rnx}(p)/A_{ref}(p)]}{[A_{rnx}(u)/A_{ref}(u)]}\right\} \times 100,$$

where u refers to unpolymerized and p to the polymerized adhesive system, respectively.

Statistical Analysis

The data were checked for normality and equal variances using Kolmogorov-Smirnov and Bartlett tests. As data were not normally distributed, they were analyzed using the nonparametric Kruskal-Wallis test for multiple comparisons. The Mann-Whitney U-test was used for pairwise comparison between the groups. Statistical significance was set at $p < 0.05$.

RESULTS

When comparing the uncured vs cured Raman spectra of the tested adhesives, the C=C peak clearly decreased after polymerization in each adhesive system (Figures 1 through 4). The vinyl signal, set as reference peak, was originated from the bisphenol A diglycidyl ether dimethacryalate (Bis-GMA) resin component for Clearfil S^3 Bond Plus and Adper Easy Bond, and the 4-methacryloxyethyltrimellitic acid anhydride (4-META) in G-BOND and I-BOND (Figures 1 through 4). As expected, the OH acidic bending group peak of the acidic monomers also disappeared in the polymerized specimens for all of the adhesives because it was buffered by the mineral components of dentin. The OH acidic bending group was clearly visible in the unpolymerized G-BOND and I-BOND spectra, where it was originated with the opening of the anhydride ring of the 4-META monomer because of the instability of 4-META in a wet environment. Generally, all specimens showed that the hydroxyapatite peak at 960 cm^{-1} decreased in intensity from dentin to adhesive. Representative Raman spectra acquired in line-scans across the adhesive interface are shown in Figure 5. Overlapping spectra acquired in the same region demonstrated the technique's reproducibility.

Means and standard deviations (SDs) of DC percentages and statistical differences ($p<0.05$) among adhesives are reported in Table 2.

The DC ranked as follows: G-BOND (93% ±6%) ≥ Adper Easy Bond (92%±6%) ≥ I-BOND (89%±7%) > Clearfil S^3 Bond Plus (80%±14%).

DISCUSSION

The present study based on micro-Raman spectroscopic data presented detailed information on the monomer DC at the adhesive/dentin interface of four one-step self-etch adhesives when applied on human dentin, even though a limiting factor to a complete analysis of the interaction between the chemical components of these materials and dentin is that the real composition of commercial materials is not disclosed by manufacturers.

Among different spectroscopic techniques, micro-Raman spectroscopy is the most appropriate to use to study the adhesive/dentin interface from the chemical point of view because it is not negatively influenced by the presence of water, does not require complicated specimen preparation (which does not affect the tested specimen), and offers detailed chemical information (ie, the double-bond converted percentage *in situ* within the hybrid layer).[10,20,24]

The hypothesis tested—that no differences in terms of DC would be found among the adhesives systems—was rejected because statistically significant differences were found between the DCs of the adhesives tested. Self-etching adhesives have been classified according to their pH as strong (pH≤1), intermediate (pH~1.5), and mild (pH~2).[25] I-BOND and G-BOND can be defined as mild self-etch adhesives because their reported pH is ~2,[26] as was the case with Clearfil S^3 Bond Plus, the pH of

which was measured in our lab, while Adper Easy Bond can be described as ultramild because its stated pH is slightly higher (~2.7).[27] Mild self-etch adhesives demineralize dentin only superficially and create a submicron hybrid layer,[22] as do the the ultramild self-etch adhesives[23]; on the contrary, strong self-etch adhesives demineralize dentin comparably to etch-and-rinse adhesives.[2,28] The different composition of the tested materials, as shown in Figures 1 through 4, likely influenced the results. Clearfil S^3 Bond Plus showed the lowest DC values and the highest standard deviation. It was the only adhesive tested that contains methacryloyloxydecyl dihydrogen phosphate (MDP), a monomer capable of forming strong ionic bonds with the hydroxyapatite's calcium.[29] MDP is a quite hydrophobic etching monomer, with a long carbonyl chain.[30] The steric hindrance of the carbonyl chain could hamper the conversion of the vinyl groups in the aliphatic bond. Another hypothesis is that the monomer mixture of Clearfil S^3 Bond Plus promotes phase separation between the domains rich in hydrophilic monomers (2-hydroxyethyl methacrylate [HEMA]) and those rich in hydrophobic monomers (Bis-GMA) that do not polymerize properly with camphorquinone.[31,32] Adper Easy Bond also contains Bis-GMA and HEMA and showed a higher DC. Recent studies[33] demonstrated that Adper Easy Bond showed good microtensile bond strength values when compared with other self-etch adhesives. DC is not the only factor contributing to the longevity of dental restorations: despite its inferior polymerization results (as recorded in this study), in previous investigations[34] Clearfil S^3 Bond Plus showed an improved bond strength when compared with two contemporary one-step self-etch adhesives. Other factors, such as the nature of the chemical interaction between the MDP contained in Clearfil S^3 Bond Plus and the dentin substrate, could have positively influenced its bond strength.

El-Damanhoury and Gaintantzopoulou[35] recently reported lower DC values for both I-BOND and Adper Easy Bond) than were reported for this study, while they confirmed the results contained herein for Clearfil S^3 Bond Plus. These controversial results can be attributed to the different specimen treatments and study setup. In that study, the DC of adhesives applied on dentin disks was calculated by attenuated total reflectance FTIR after removing the oxygen-inhibited layer with acetone and focusing on the adhesive surface (ie, the DC was assayed in the adhesive layer). Conversely, the micro-Raman technique used in the present investigation allowed assessment not only of the DC on the specimen surface but also the assessment of DC micron by micron across the hybrid layer, in which different measurements were obtained since the acidic monomers contained in the self-etch adhesives are neutralized by the dentin calcium ions. Indeed, it is well known that the chemical interaction between acidic monomers and dentin mineral in self-etch adhesives is crucial for an adequate polymerization, as residual acidic monomers might compromise the polymerization, and thus the DC, of the adhesive.

Both I-BOND and G-BOND contain 4-META, which is an acidic monomer with an aromatic group that is soluble in acetone.[26] 4-META is used both as an adhesion-promoting monomer,[36] because it is able to establish an ionic bond with calcium in hydroxyapatite, and as a demineralizing monomer. As the composition of these two adhesives is only partially disclosed by their manufacturers, we referred to the study of Van Landuyt and others,[26] who reported that both I-BOND and G-BOND contain urethane dimethacrylate (UDMA), while G-BOND also includes triethylene glycol dimethacrylate (TEGDMA) in its composition. It could be speculated that G-BOND contain less UDMA than I-BOND because of the presence of TEGDMA and that perhaps the long C-C chain of UDMA (PM: 470.56) interfered with the polymerization of the vinyl groups more than did TEGDMA (PM: 286.32).

A low percentage of monomer-to-polymer conversion is related to low physicochemical properties of adhesives[11,12] in resins with lower degrees of conversion, higher elution of monomers occurs,[11,37] negatively affecting the longevity of the bond.[11,12] Thus, *in situ* analysis of the DC is an important factor in predicting adhesive performance *in vivo*.

Previous studies[18,38] demonstrated that simplified adhesives such as one-step self-etch adhesives exhibit a lower DC and higher adhesive permeability than do the two-step self-etch as a result of the higher presence of hydrophilic monomers and solvents. Solvents are included to remove intrinsic water from the wet dentin matrix, promoting adhesive impregnation of the demineralized dentin.[39] If residual water and solvents are trapped within the adhesive layer, this can result in low polymerization,[40] rendering residual unreacted monomers more prone to leaching.[41,42] A previous study[43] demonstrated that high residual ethanol percentages may compromise the polymerization of dental adhesives as a result of residual solvent entrapped within the polymer network.

Clearfil S^3 Bond Plus and Adper Easy Bond contain ethanol and water as solvents, while I-BOND and G-BOND are acetone-based adhesives (Figures 1 through 4). Thus, the type of organic solvent used may also have influenced the differences in DC of the tested adhesives. Nevertheless, no correlation between solvent type and DC was found in this study, as both the adhesive with the lowest DC (Clearfil S^3 Bond Plus) and that with the highest DC (Adper Easy Bond) contained ethanol and water. The ability of Adper Easy Bond to efficiently cure confirm the previously published reports.[44,45] This might be explained by the different type of photoinitiators blended within the adhesive system, which is undisclosed by manufacturers. It has been shown that the photoinitiating system should be selected in relation to adhesive system composition, especially the presence of hydrophilic elements. Water-compatible photoinitiators are recommended in highly hydrophilic adhesive formulations to guarantee an appropriate DC of the adhesive layer and to diminish aging phenomena.[31] Recently, photoinitiators such as TPO [ethyl 4-dimethylaminobenzoate and diphenyl(2,4,6-trimethylbenzoyl)-phosphine oxide] have become popular, and their inclusion in new adhesive formulations (even if not disclosed by manufacturers) might also explain the higher DC of the adhesives tested in this study compared to previously marketed one-step self-etch adhesives.

CONCLUSIONS

Within the limitations of this study, the results showed that the DC, which is one of the factors contributing to the longevity of an adhesive, varied among the tested self-etch adhesives and was material dependent. Further studies are necessary to evaluate whether the DC of the tested materials can be correlated to their stability over time and to their bond strength performance.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Department of Medical Sciences, the University of Trieste, Italy.

Conflict of Interest

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

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Effect of Minocycline on the Durability of Dentin Bonding Produced with Etch-and-Rinse Adhesives

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Clinical Relevance

Chemical-modified tetracyclines can be considered an alternative to retard the degradation of dentin-bonding interfaces.

SUMMARY

Objectives: To evaluate the effect of minocycline and chlorhexidine pretreatment of acid-etched dentin on the longevity of resin-dentin bond strength (μTBS) and nanoleakage of two-step etch-and-rinse adhesives.

Methods: Before application of Prime & Bond NT and Adper Single Bond 2 in occlusal dentin, the dentin surfaces were treated with 37% phosphoric acid, rinsed, air-dried, and rewetted with water (control group), 2% minocycline, or 2% chlorexidine digluconate. Composite buildups were constructed incrementally, and specimens were longitudinally sectioned to obtain bonded sticks (0.8 mm^2) to be tested in tension (0.5 mm/min) immediately or after 24 months of water storage. For nanoleakage, two specimens of each tooth/period were immersed in the silver nitrate solution, photo-developed, and polished with SiC paper for analysis under energy-dispersive X-ray spectroscopy/scanning electron microscopy.

Results: Reductions of the μTBS and increases in the nanoleakage were observed for both

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adhesives when the rewetting procedure was performed with water. Stable bonds were observed for the 2% minocycline and 2% chlorexidine digluconate groups after 24 months.

Conclusions: The use of 2% minocycline as pretreatment of acid-etched dentin is one alternative to retard the degradation of resin-dentin interfaces over a 24-month period as well as 2% chlorexidine digluconate.

INTRODUCTION

In spite of the high immediate resin-dentin bonds created by simplified etch-and-rinse adhesives, dentin adhesive interfaces suffer substantial deterioration after water aging.[1,2] It is hypothesized that this biodegradation involves a cascade of events,[3] starting with the incomplete infiltration of the exposed collagen fibril matrix after acid etching[1,2] and followed by extraction of the resins[4] via water-filled nanometer-sized voids within the hybrid layer. The third stage involves the enzymatic attack of the exposed collagen fibrils by endogenous matrix metalloproteinases (MMPs) and cysteine cathepsins.[5,6]

The MMPs are a large family of zinc- and calcium-dependent endopeptidases presented in the human dentin[6-8] that are capable of degrading all extracellular matrix components.[9] By suppressing the collagenolytic and gelatinolytic activities of dentin, one can reduce the degradation of the collagen fibrils,[10] producing bonded interfaces less prone to degradation over time. In fact, previous studies demonstrated that the pretreatment of acid-etched dentin with chlorhexidine digluconate,[11-14] the most tested MMP inhibitor, yielded stable bond strength values after aging.

Several other MMP inhibitors are described in the literature,[15,16] such as ethylene diamine tetra-acetic acid[17,18], tetracycline and chemical-modified tetracyclines (CMTs),[18-21] galardin,[22] polyvinilphosphonic acid,[23] quaternary ammonium compounds,[24,25] green tea polyphenols,[26] and carbodiimides,[27] among others. However, few attempts were made to investigate their effect on prolonging the lifetime of dentin-bonded interfaces.

Recently, Osorio and others[21] and Toledano and others[18] reported that doxycycline, a CMT, was capable of inhibiting collagen degradation in demineralized dentin in periods ranging from 24 hours up to 3 weeks. In another laboratory investigation, the authors reported that the application of 2% minocycline, which is also a CMT, as dentin pretreatment did not affect the immediate performance of etch-and-rinse adhesives,[28] appearing to be a good priming agent to prevent the degradation of the resin-dentin interfaces. More recently, Feitosa and others[29] showed that the inclusion of doxycycline in nanotube-modified adhesive can inhibit MMP activity without jeopardizing the degree of conversion or bond strength to dentin. However, as pointed out in a recent review published by Perdigão and others,[16] the use of CMTs and their analogues in dentin bonding as a way to prolong resin-dentin bonds is still scarce in the literature and should be the focus of further investigation because of the potential role of CMTs on MMP inhibition. Therefore, the aim of this study was to evaluate the effect of a 2% aqueous solution of minocycline when compared to a 2% aqueous solution of chlorexidine digluconate as pretreatment of acid-etched dentin on the longevity of resin-dentin bond strength and nanoleakage (NL) of two-step etch-and-rinse adhesives.

METHODS AND MATERIALS

Tooth Preparation and Experimental Design

Thirty extracted, caries-free human third molars were used. The teeth were collected after the patient's informed consent and the local university review board approved this study (protocol #6280/2009). Teeth were disinfected in 0.5% chloramine, stored in distilled water in the refrigerator (4°C), and used on average 2-3 months after extraction.

A flat and superficial dentin surface was exposed on each tooth after wet grinding the occlusal enamel on 180-grit SiC paper. The enamel-free, exposed dentin surfaces were further polished on wet 600-grit silicon-carbide paper for 60 seconds to standardize the smear layer.

After preparation, teeth were divided into six groups (n=5), according to the combination of the main factors adhesive (Adper Single Bond 2 [3M ESPE, St Paul, MN, USA] and Prime & Bond NT [Dentsply De Trey, Konstanz, Germany]) and rewetting solution (2% aqueous solution of minocycline [MO], 2% aqueous solution of chlorhexidine digluconate [CHX], and water). The composition and batch number of the adhesives are described in Table 1.

Restorative Procedures and Specimen Preparation

The dentin surfaces were conditioned with 37% phosphoric acid gel (Condac 37, FGM, Joinville, Brazil) for 15 seconds, water rinsed for 15 seconds, and air-dried for 30 seconds. In the control group,

TABLE 1: *Adhesive Systems: Composition, Groups and Application Mode*

Adhesive Systems	Composition	Rewetting Solution	Application Mode
Prime & Bond NT	Caulk Tooth Conditioner Gel 34% phosphoric acid Adhesive: UDMA, PENTA, R 5-62-1 resin, T resin, D resin, silanated colloidal silica, cetylamine hydroxyfluoride, initiator, stabilizer, and acetone	Water 2% minocycline 2% chlorhexidine	a: acid etch (15 s); b: rinse (15 s); c: air-dry (30 s); d: rewetting (15 s); e: one coat of adhesive; f: air-dry for 10 s at 20 cm; g: repeat e and f; h: light cure (10 s, 600 mW/cm^2).
Adper Single Bond 2	Scotchbond Etchant 35% phosphoric acid Adhesive: Bis-GMA, HEMA, dimethacrylates, nanofilled colloidal silica (5 nm) polyalkenoic acid copolymer, initiators, water, and ethanol	Water 2% minocycline 2% chlorhexidine	a: acid etch (15 s); b: rinse (15 s); c: air-dry (30 s); d: rewetting (15 s); e: one coat of adhesive; f: air-dry for 10 s at 20 cm; g: repeat e and f; h: light cure (10 s, 600 mW/cm^2).

Abbreviations: UDMA – urethane dimethacrylate; PENTA – dipentaerythritol pentacrylate monophosphate; Bis-GMA: bisphenol A diglycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate

the dentin surfaces were rewetted with water for 15 seconds, while in the experimental groups, dentin surfaces were rewetted with 2 wt% aqueous solution of MO (Fleming Drugstore, Ponta Grossa, Brazil) or 2 wt% aqueous solution of CHX (FGM) for 15 seconds. All substances were used within 1 week after preparation.

The excess solution was removed with a blotting paper. The adhesive systems were applied according to the manufacturer's instructions (Table 1) and light cured with a quartz-tungsten-halogen light (VIP, Bisco, Schaumburg, IL, USA; 600 mW/cm^2) for 10 seconds. Resin composite buildups (Opallis, FGM) were constructed in three increments of approximately 1.5 mm and individually light activated for 40 seconds. All bonding procedures were carried out by a single operator at 24°C and 50% relative humidity.

The bonded teeth were stored in distilled water at 37°C for 24 hours. After storage, specimens were longitudinally sectioned in both "x" and "y" directions across the bonded interface with a diamond saw (Extec Corp., Enfield, CT, USA) at 300 rpm to obtain bonded sticks with a cross-sectional area of approximately 0.8 mm^2. The number of premature failures during specimen preparation was recorded.

The cross-sectional area of each stick was measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) to the nearest 0.01 mm and recorded for subsequent calculation of the microtensile bond strength (μTBS). The bonded sticks that originated from the same tooth were randomly divided for immediate or 24-month testing. The specimens for the 24-month group were stored at 37°C in hermetically sealed vials containing distilled water.

Two resin-bonded sticks, from each tooth and at each storage period, were used for NL evaluation for each experimental condition, and the remaining bonded sticks were tested for microtensile bond strength.

(μTBS) Microtensile Bond Strength Test

Resin-dentin bonded sticks were attached to a μTBS jig (Odeme Prod. Odont. Ltda, Joaçaba, Brazil) with cyanoacrylate adhesive and tested under tension (Emic, São José dos Pinhais, Brazil) at 0.5 mm/min until failure. The μTBS values were calculated by dividing the load at failure by the cross-sectional bonding area.

The failure modes were evaluated at 400× (HMV-2, Shimadzu, Tokyo, Japan) and classified as cohesive (failure exclusive within dentin or composite [C]), adhesive (failure at resin-dentin interface [A]), or adhesive/mixed (failure at resin-dentin interface that included cohesive failure of the neighboring substrates [A/M]).

Nanoleakage (NL)

Two resin-bonded sticks, from each tooth and at each storage period, were used for NL evaluation for each experimental condition (n=10). Ammoniacal silver nitrate was prepared according to the protocol previously described by Tay and others.[30] The sticks were placed in the ammoniacal silver nitrate in darkness for 24 hours, rinsed thoroughly in distilled water, and immersed in photo-developing solution for 8 hours under a fluorescent light to reduce silver ions into metallic silver grains within voids along the bonded interface. Specimens were polished with a wet 600-, 1000-, 1200-, 1500-, 2000-, and 2500-grit SiC paper and 0.25 μm diamond paste (Buehler Ltd, Lake Bluff, IL, USA) using a polishing cloth.

They were ultrasonically cleaned, air-dried, and acid etched for 3 seconds in a 50% phosphoric acid

TABLE 2: *Number and Percentage of Specimens (%) According to Fracture Pattern in Each Experimental Condition as Well as Total Number of Specimens Tested (TN)*

Rewetting Solution	Prime & Bond NT								Adper Single Bond 2							
	Immediate				24-Mo				Immediate				24-Mo			
	A/M	C	PF	TN	A/M	C	PF	TN	A/M	C	PF	TN	A/M	C	PF	TN
Control	36 (80)	0 (0)	9 (20)	47	45 (78)	1 (2)	12 (20)	58	39 (93)	0 (0)	3 (7)	41	41 (95)	0 (0)	2 (5)	43
2% minocycline	34 (97)	0 (0)	1 (3)	35	33 (94)	2 (6)	0 (0)	35	45 (98)	0 (0)	1 (2)	45	43 (96)	2 (4)	0 (0)	45
2% chlorhexidine	50 (85)	2 (3)	7 (12)	59	43 (94)	1 (2)	2 (4)	46	41 (93)	2 (5)	1 (2)	44	38 (90)	2 (5)	2 (5)	42

Abbreviations: A/M, adhesive/mixed fracture mode; C, cohesive fracture mode; PF, premature failure.

solution followed by immersion in 2% NaOCl for 10 minutes. Specimens were mounted on aluminum stubs and sputter coated with gold (Sputter Coater IC 50, Shimadzu, Tokyo, Japan). Resin-dentin interfaces were analyzed in a scanning electron microscope operated in secondary electron mode (SSX-550, Shimadzu, Tokyo, Japan) at a working distance of 20 mm and 15 kV.

The percentage of NL in the adhesive and hybrid layers of each bonded stick was measured using energy-dispersive X-ray spectrometry (SSX-550, Shimadzu). This measurement was performed in three random regions (5×5 μm) of the bonded stick (left, center, and right). The total area of the hybrid layer–scanned NL measurement was approximately 75 μm^2. The NL was expressed as a percentage of the total area evaluated.[31]

Statistical Analysis

The mean μTBS and the percentage of NL of all bonded sticks from the same tooth were averaged for statistical purposes. Premature failures were included in the tooth mean with an attributed value of 3.4 MPa. This value corresponds to approximately half of the minimum μTBS value that could be measured in this study, which was 6.9 MPa. This approach avoids overestimation of the bond strength means, and it was previously performed in other studies.[32-34]

The μTBS (MPa) and NL (%) data of each adhesive system were subjected to two-way repeated measures analysis of variance (rewetting solution vs storage time) and Tukey test with a level of significance of 5%.

RESULTS

Approximately 18-25 sticks were obtained per tooth, including those with premature failure. The mean cross-sectional area ranged from 0.81 to 0.96 mm^2, and no difference among groups was detected ($p>0.05$).

The percentage of specimens that suffered premature failure and the frequency of each fracture mode are shown in Table 2. Most of the specimens showed adhesive/mixed fractures in all groups.

μTBS Test

The total number of resin-bonded sticks evaluated in the μTBS test is summarized in the Table 2. For both adhesives tested, the cross-product interaction of rewetting solution vs storage time was statistically significant (Table 3; $p=0.001$). Significant reductions of μTBS values after 24 months were observed only for the control groups ($p<0.05$). The dentin pretreatment with MO and CHX produced stable bond strengths after 24 months of water storage (Table 3).

NL

The cross-product interaction of rewetting solution vs storage time was significant (Table 4; $p>0.001$) for both adhesives. For Prime & Bond NT, lower NL was observed in the immediate period for all rewetting substances. After 24 months, the control group

TABLE 3: *Means and Standard Deviations of the Microtensile Bond Strength Values (MPa) for Each Experimental Condition*[a]

Rewetting Solutions	Prime & Bond NT		Adper Single Bond 2	
	Immediate	24-Mo	Immediate	24-Mo
Control	42.3 ± 3.4 A	23.6 ± 5.3 B	46.2 ± 4.7 a	32.3 ± 4.5 b
2% minocycline	46.3 ± 5.4 A	41.4 ± 3.6 A	49.6 ± 3.6 a	44.2 ± 5.1 a
2% chlorhexidine	44.2 ± 4.3 A	36.3 ± 5.1 A	50.3 ± 5.6 a	43.3 ± 3.5 a

[a] *Groups identified with the same upper- or lowercase letters are statistically similar (Tukey test, p>0.05).*

TABLE 4: *Means and Standard Deviations (%) of Nanoleakage at the Adhesive Interfaces for Each Experimental Condition*[a]

Rewetting Solutions	Prime & Bond NT		Adper Single Bond 2	
	Immediate	24-Mo	Immediate	24-Mo
Control	26.2 ± 3.6 BC	51.3 ± 4.1 D	27.9 ± 5.8 b	39.3 ± 4.2 c
2% minocycline	21.2 ± 3.2 AB	28.1 ± 4.5 C	17.2 ± 5.1 a	24.3 ± 3.9 ab
2% chlorhexidine	17.5 ± 4.3 A	32.3 ± 3.9 C	16.2 ± 4.1 a	26.5 ± 3.4 b

[a] *Groups with the same upper- or lowercase letters are not significantly different (Tukey test, p>0.05).*

showed the highest NL compared with MO and CHX groups (Table 4; $p>0.001$). For Adper Single Bond 2, lower NL was observed in the MO and CHX groups at the immediate period. After 24 months of storage time, the control group showed the highest NL compared with the MO and CHX groups (Table 4; $p>0.001$).

Representative scanning electron microscopic images at the resin-dentin interfaces are shown in Figures 1 and 2. Nanoleakage occurred in all experimental conditions, but the highest NL values were observed in the control group after 24 months of storage (Figures 1D and 2D).

DISCUSSION

As already reported in the literature,[13,14] resin-dentin interfaces from the control groups suffered degradation after long-term water storage. Compared with the immediate bond strength values, the present study detected reductions in the range of 39%-50% for both etch-and-rinse adhesives. On the other hand, the addition of a preliminary treatment of the demineralized dentin with MO or CHX produced stable μTBS and reduced NL after 24 months of water storage in comparison with the control group.

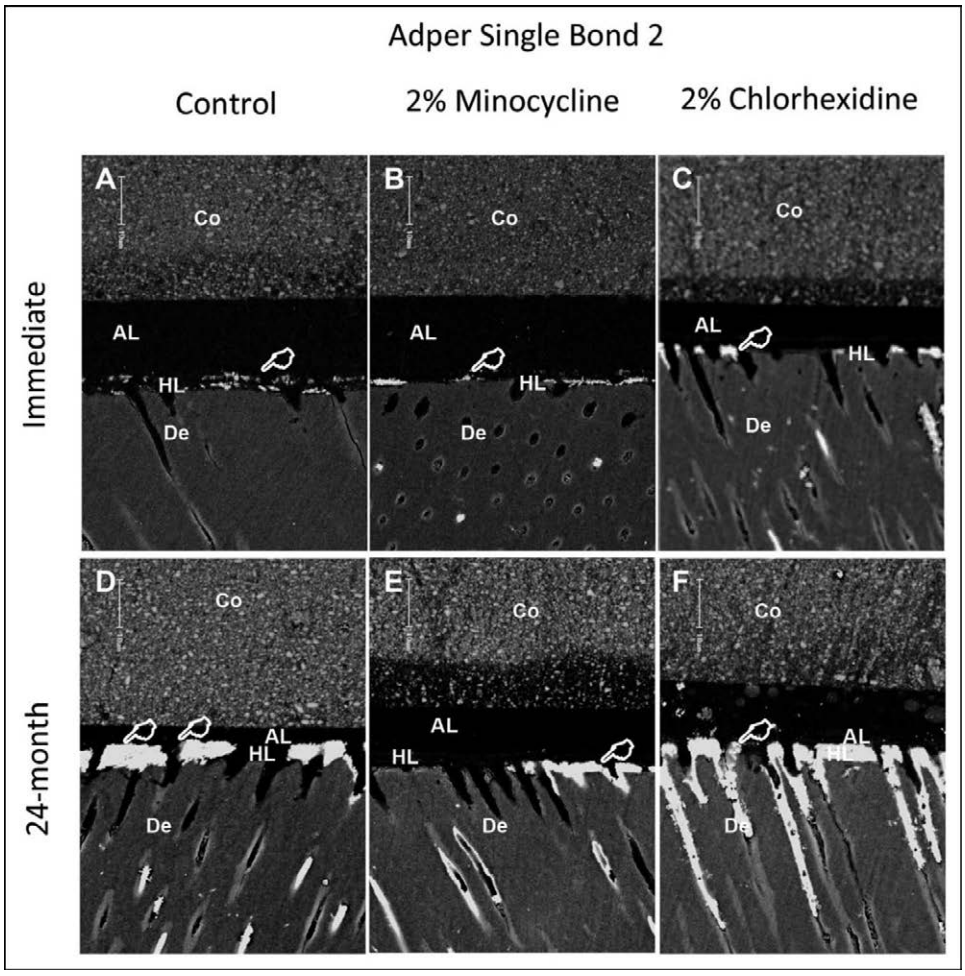

Figure 1. *Representative backscatter SEM images of the resin-dentin interfaces bonded with Adper Single Bond 2. In the immediate time, the amount of silver penetration was lower and practically occurred within the HL (A-C). Few dentin tubules were infiltrated by silver nitrate for all groups (arrows in A-C). After 24 months of water storage, the amount of silver nitrate increased, with deposition of silver deposits occurring throughout the entire thickness of the HL and part of the AL, mainly in the control group (arrows in D) (SEM, scanning electron microscopy; Co, composite; De, dentin; HL, hybrid layer; AL, adhesive layer).*

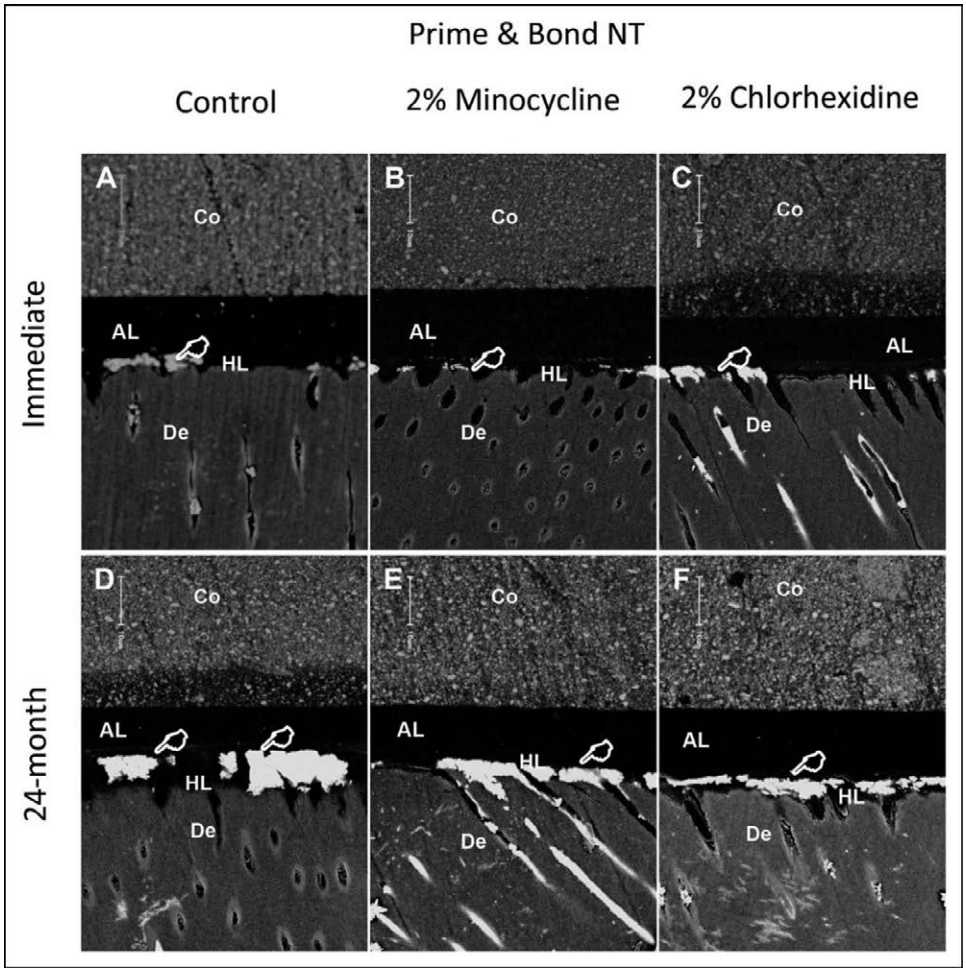

Figure 2. *Representative backscatter SEM images of the resin-dentin interfaces bonded with Prime & Bond NT. Lower percentage of silver penetration was shown in the immediate groups (A-C), mainly in the HL (arrows). In all groups, an increased amount of silver nitrate after 24 months of water storage occurred (D-E). This increase was significantly higher in the control group, with most silver uptake deposits occurring throughout the entire thickness of the HL and part of the AL (arrows in D) (SEM, scanning electron microscopy; Co, composite; De, dentin; HL, hybrid layer; AL, adhesive layer).*

The protective effect of 2% CHX was not surprising. The CHX molecule is characterized by being a strong base with cationic properties when ionized in a solvent such as water, and this cationic part of the molecule connects to the negatively charged part of the collagen or MMPs. It is possible that CHX binds to phosphate groups or to negative carboxyl groups in mineralized dentin crystallites or collagen matrix, respectively,[35] and due to its substantivity can remain bonded in demineralized and mineralized dentin substrates.[36]

CHX was extensively evaluated as dentin pretreatment to prolong the lifetime of bonded interfaces.[37,38] More recently, its incorporation into acidic conditioners and some adhesive systems also produced stable bond strengths after water storage.[14,39-42] The inhibition of MMP-2 and MMP-9 has been attributed to the chelating ability of CHX to zinc.[43] In the case of MMP-8, CHX interacts with the essential sulfhydryl groups and/or cysteine of the active enzyme sites. Additionally, recent investigations reported that CHX could also inhibit the activity of cysteine cathepsins,[5] which constitute another dentin protease likely involved in the degradation of the bonded interfaces.[6]

Similarly to CHX, the chemically modified tetracycline (CMT) tested also produced stable bond strengths after 24 months of water degradation. Apart from the lack of antibiotic activity, CMTs such as minocycline are potent MMP inhibitors.[44] Although the effect of minocycline on dentin MMPs was not investigated, doxycycline, which is another CMT, was shown to be even more effective than CHX in inhibiting MMPs activity. While CHX exerted a partial, time-limited MMP inhibition, minimizing collagen degradation by 57% for only 24 hours, doxycycline fully blocked proteolysis for periods of up to 3-4 weeks.[18,21]

It is fair to hypothesize that both CMTs share similar MMP inhibition potential due to their comparable chemical structure.[19] Doxycycline and minocycline show a basic chemical structure consisting of a tetracyclic naphthacene carboxamide ring

system. These structures differ by doxycycline having one hydroxyl group in carbon 5 of ring B and one methyl group in carbon 6 of ring C. In contrast, minocycline has one amine group on carbon 7 of ring D.[44] Despite these differences, both substances can inhibit collagenases and gelatinases.[45,46] In the present study, we did not investigate the effect of doxycycline on the durability of the resin-dentin bonds since an earlier study reported that doxycycline, when mixed with adhesive solutions, resulted in phase separation and lowered the immediate bond strength of etch-and-rinse adhesives to dentin,[28] and this is probably the reason for recent attempts to encapsulate doxycycline.[29]

CMTs are considered broad-spectrum MMP inhibitors.[19,43] Their inhibitory effects were attributed to their ability to chelate Ca^{+2} and Zn^{+2},[46,47] which are two essential ions for MMPs to maintain their structure and functional active sites.[9] Zinc is bound to the catalytic domain of the enzyme, and calcium is required to produce MMP activation.[18,48] Therefore, by binding to Zn and Ca, CMTs inactivate the endogenous proteases. However, the hypothesis that binding of zinc results in protection of sensitive cleavage sites of metalloproteinases requires further validation since zinc in excess reduces MMP-mediated collagen degradation.[21,49]

Another likely explanation for the MMP inhibition produced by CMT is that it acts by altering MMP conformation rather than by interaction with the catalytic zinc.[18,48] This would explain its long-lasting and more potent effect when compared to CHX.[18,21]

Another important finding of the present investigation was the reduced nanoleakage of the experimental controls after 24 months. The nanoleakage reveals the location of defects at the resin-dentin interface that could work as pathways for degradation of resin-dentin bonds over time. Silver nitrate occupies nanometer-sized spaces around naked collagen fibrils, where resin failed to infiltrate or where residual water was not displaced by the adhesive resin.[50]

This seems to worsen during aging, as naked collagen fibrils are digested by the endogenous proteases, increasing the size and volume of defects in the hybrid layer. In the face of that, we hypothesize that the reduced nanoleakage of the MO and CHX groups after 24 months compared to the control groups shows that less degradation of the collagen fibrils occurred during this water storage period—more indirect evidence of the MMP potential of both MO and CHX.

In spite of the promising results obtained with minocycline in the present study, some authors report that their use in association with dentin bonding systems is limited since they may stain teeth with a purple hue after photo-oxidation of the CMTs.[2,51] A similar concern was already raised for CHX,[51,52] as it can also stain teeth.

Although these concerns are factual, they were never observed under the protocol herein investigated in clinical studies.[53-55] In the present investigation, none of the specimens showed color change over the course of these 24 months, suggesting that this may not be a problem for MO and CHX when used as dentin priming conditioners. Under this protocol, the products were applied only once for a short duration and with a reduced volume of the product. However, further investigations should be conducted to rule out completely this potential side effect of CMTs on dentin bonding.

CONCLUSION

Within the limits of this study, it was found that the use of 2% minocycline as a dentin priming solution after acid etching is an alternative to retarding the degradation of resin-dentin interfaces over a 24-month period.

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Conflict of Interest

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

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Influence of Etching Mode on Enamel Bond Durability of Universal Adhesive Systems

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Clinical Relevance

Total-etch mode has a positive effect on the enamel bond durability of universal adhesives, as was seen with the previous generation of single-step adhesives.

SUMMARY

The purpose of this study was to determine the enamel bond durability of three universal adhesives in different etching modes through fatigue testing. The three universal adhesives used were Scotchbond Universal, Prime&Bond Elect universal dental adhesive, and All-Bond Universal light-cured dental adhesive. A single-step self-etch adhesive, Clearfil S^3 Bond Plus was used as a control. The shear bond strength (SBS) and shear fatigue strength (SFS) to human enamel were evaluated in total-etch mode and self-etch mode. A stainless steel metal ring with an internal diameter of 2.4 mm was used to bond the resin composite to the flat-ground (4000-grit) tooth surfaces for determination of both SBS and SFS. For each enamel surface treatment, 15 specimens were prepared for SBS and 30 specimens for SFS. The staircase method for fatigue testing was then used to determine the SFS of the resin composite bonded to the enamel using 10-Hz frequencies for 50,000 cycles or until failure occurred. Scanning electron microscopy was used to observe representative debonded specimen surfaces and the resin-enamel interfaces. A two-way analysis of variance and the Tukey

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post hoc **test were used for analysis of the SBS data, whereas a modified *t*-test with Bonferroni correction was used for the SFS data. All adhesives in total-etch mode showed significantly higher SBS and SFS values than those in self-etch mode. Although All-Bond Universal in self-etch mode showed a significantly lower SBS value than the other adhesives, there was no significant difference in SFS values among the adhesives in this mode. All adhesives showed higher SFS:SBS ratios in total-etch mode than in self-etch mode. With regard to the adhesive systems used in this study, universal adhesives showed higher enamel bond strengths in total-etch mode. Although the influence of different etching modes on the enamel-bonding performance of universal adhesives was found to be dependent on the adhesive material, total-etch mode effectively increased the enamel bond strength and durability, as measured by fatigue testing.**

INTRODUCTION

In the last decade, self-etch adhesive systems have become prevalent due to the reliability of bonding performance to the tooth structure. In addition, due to chemical bonding to hydroxyapatite and decreased demineralization of dentin, it has been claimed that the incidence of postoperative sensitivity is lower than for etch-and-rinse systems.[1,2] Generally, self-etch adhesive systems can be categorized as either two-step or single-step systems[3]; single-step self-etch adhesives are much easier to use, given that the priming and bonding procedures are combined into one step. This reduction in bonding procedures is beneficial for dental professionals because it allows for reduction in the technique sensitivity in clinical situations.

Recently, a new type of single-step self-etch adhesive categorized as "universal" or "multi-mode" has been introduced.[4-7] Universal adhesives can be used with multiple substrates, such as enamel, dentin, silica-based glass ceramics, zirconia ceramics, and metal alloys, without individual pretreatment.[8,9] This type of multifunctional adhesive is expected to help simplify the bonding procedure when various restorations are bonded to the tooth structure. Furthermore, some universal adhesives use a dual-cure system to allow for better polymerization at the interface and subsequent higher bond strengths when in contact with resin cements. The chemical reaction promoted by the activator may increase the bonding performance when adequate light irradiation is not possible.

For previous generations of single-step self-etch adhesives, some laboratory studies[10,11] have reported lower bonding performance than two-step self-etch systems and etch-and-rinse adhesive systems. In particular, enamel bond durability has been claimed to be a cause for concern due to the poorer mechanical properties of the adhesive layer and lower etching capability.[12] Therefore, to obtain a durable bond to the enamel, selective etching with phosphoric acid prior to application of the single-step self-etch adhesive has been recommended.[13-16] However, in practice, it is extremely difficult to precisely etch only the enamel surface, resulting in the strong possibility of inadvertently overetching exposed dentin. This may lead to decreased dentin bonding quality due to incomplete penetration of the resin monomers into demineralized dentin as well as induced postoperative sensitivity.[1,17] To overcome the weakness of previous generations of single-step self-etch adhesives, universal adhesives have been developed that allow for application of the adhesive with phosphoric acid pre-etching in the total-etch or selective-etch approaches.[4-7]

Although there are several laboratory and clinical studies regarding the bonding performance of universal adhesives,[18,19] only limited information is available on the bond durability of universal adhesives when used in different application modes. To understand the characteristics of universal adhesives, investigation of bonding performance from the perspective of fatigue stress, simulating the oral environment, is important because it clarifies the long-term bond durability. In addition, simulated oral environment testing can provide a rapid and standardized method to examine relative bond durability among materials and help predict expected clinical performance.[15,16]

The purpose of this laboratory investigation was to determine the enamel bond durability of universal adhesives in different application modes through fatigue testing. The null hypotheses to be tested were as follows: 1) universal adhesives would not differ from a single-step self-etch adhesive with respect to bond durability; 2) phosphoric acid pre-etching would not affect the enamel bond durability of universal adhesives.

METHODS AND MATERIALS

Materials

The materials used in this study are shown in Table 1. The following three universal adhesives were

Table 1: *Materials Used in This Study*

Code	Adhesive (Lot No.)	Main Components	Manufacturer
SU	Scotchbond Universal (41256)	MDP, HEMA, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane	3M ESPE, St Paul, MN, USA
PE	Prime & Bond Elect (140710)	Dipentaerythritol pentaacrylate monophosphate, polymerizable dimethacrylate resin, polymerizable trimethacrylate resin, diketone, organic phosphine oxide, stabilizers, cetylamine hydrofluoride, acetone, water	Dentsply Caulk Milford, DE, USA
AU	All-Bond Universal (1300008503)	MDP, bis-GMA, HEMA, ethanol, water, initiators	Bisco Inc, Schaumburg, IL, USA
SP	Clearfil S^3 Bond Plus (4G0011)	MDP, bis-GMA, HEMA, ethanol, water, filler, CQ	Kuraray Noritake Dental Tokyo, Japan
	Pre-etching Agent		
	Ultra-Etch (G017)	35% phosphoric acid	Ultradent Products, Inc, South Jordan, UT, USA
	Resin composite		
	Z100 Restorative (N416713)	Bis-GMA, TEGDMA, silane treated ceramic, 2-benzotriazolyl-4-methyphenol catalysts, accelerators, CQ, pigments, others, zirconia/silica, 0.01-3.5 μm. Filler Load: 84.5% weight, 66% volume	3M ESPE, St Paul, MN, USA

Abbreviations: Bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxy propoxy) phenyl] propane; CQ, dl-camphorquinone; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethyleneglycol dimethacrylate.

used: Scotchbond Universal adhesive (SU; 3M ESPE, St Paul, MN, USA), Prime&Bond Elect universal dental adhesive (PE; Dentsply Caulk, Milford, DE, USA), and All-Bond Universal light-cured dental adhesive (AU; Bisco, Schaumburg, IL, USA). A conventional single-step self-etch adhesive, Clearfil S^3 Bond Plus (SP; Kuraray Noritake Dental, Tokyo, Japan), was used as a control. Ultra-Etch (Ultradent, South Jordan, UT, USA) was used as a 35% phosphoric acid etching agent. A visible light-cured resin composite (Z100 Restorative, 3M ESPE) was used as a restorative material for bonding to enamel.

Specimen Preparation

Deidentified, extracted, caries-free human molars were selected for use in this study under a protocol reviewed and approved by the Ethics Committee for Human Studies of the Nihon University School of Dentistry (No. 2015-06). The enamel bonding sites were prepared by mesiodistally sectioning the teeth and removing approximately two-thirds of the apical root structure. The buccal and lingual tooth sections were mounted with a resin material (Triad DuaLine, Dentsply Trubyte, York, PA, USA) in 25-mm-diameter aluminum rings. The enamel-bonding surfaces were ground flat to 4,000-grit using a water coolant and a series of carbide polishing papers (Struers Inc, Cleveland, OH, USA). Metal rings machined from SAE 304 stainless steel with an internal diameter of 2.4 mm, an external diameter of 4.8 mm, and a length of 2.6 mm were used to condense the resin composite on enamel surfaces for shear bond strength (SBS) and shear fatigue strength (SFS) tests. The bonding procedure resulted in a resin composite cylinder inside the ring that was approximately 2.36 mm in diameter and 2.5 mm in height. The ring was left in place for the tests.

SBS Tests

For each test group, 15 specimens were used to determine the SBS to enamel in total-etch mode (phosphoric acid was applied for 15 seconds prior to application of the adhesive) or self-etch mode (without phosphoric acid etching). The adhesive agents were used in accordance with the manufacturers' instructions, as shown in Table 2. Following treatment of the flat-ground enamel surface with the adhesive agent, the stainless steel metal ring was placed over the enamel surface and secured by clamping with a custom fixture. The light-cured resin composite paste was condensed in the ring and light-irradiated for 40 seconds with a curing unit (Spectrum 800, Dentsply Caulk, Milford, DE, USA) set at a light irradiance average of 600 mW/cm^2. The bonded specimens were stored for 24 hours in 37°C distilled water before testing. The specimens were loaded to failure at a rate of 1.0 mm/min using a universal testing machine (Electron E 1000, Instron, Norwood, MA, USA). A metal rod with a chisel-shaped end was used to apply the load to the metal

Table 2: *Application Protocol for Pre-etching and Self-etching Adhesives*

Method	**Pre-etching Protocol**
Total-etch	Enamel surface was phosphoric acid etched for 15 s, then rinsed with water for 15 s (three-way dental syringe) and air-dried.
Self-etch	Phosphoric acid pre-etching was not performed.
Adhesive	**Adhesive Application Protocol**
SU	Adhesive applied to air-dried enamel surface with rubbing for 20 s. Medium air pressure applied to surface for 5 s. Light-irradiated for 10 s.
PE	Adhesive applied to enamel surface with rubbing for 20 s. Gentle stream of air applied over the liquid for at least 5 s. Light-irradiated for 10 s.
AU	Adhesive applied to enamel surface with rubbing action for 10-15 s/coat. No light cure between coats. Gentle stream of air applied over the liquid for at least 10 s. Light-irradiated for 10 s.
SP	Adhesive applied to air-dried enamel surface for 10 s. Medium air pressure applied to surface for 5 s. Light-irradiated for 10 s.

Abbreviations: AU, All-Bond Universal light-cured dental adhesive; PE, Prime&Bond Elect universal dental adhesive; SP, Clearfil S^3 Bond Plus; SU, Scotchbond Universal.

ring immediately adjacent to the flat-ground enamel surface. The SBS values (MPa) were calculated from the peak load at failure divided by the bonded surface area. After testing, the bonding sites of the enamel surface and resin composite cylinders were observed using an optical microscope (MZ16; Leica Microsystems, Heerbrugg, Switzerland) at 20× magnification to determine the bond failure mode. On the basis of the percentage of substrate area (adhesive, resin composite, and enamel) observed on the debonded cylinders and tooth-bonding sites, the types of bond failure were recorded as 1) adhesive failure, 2) cohesive failure in composite, 3) cohesive failure in enamel, or 4) mixed failure (ie, partially adhesive and partially cohesive).

SFS Tests

The staircase method of fatigue testing reported by Draughn[20] was used for SFS testing, as previously reported.[15,16] Test specimens were prepared as described above for SBS testing. The lower load limit was set near zero (0.4 N), and the initial maximum load applied was 50%-60% of the SBS value determined for each of the adhesive systems tested. The load was applied at a frequency rate of 10 Hz with an ElectroPuls E1000 machine (Instron, Norwood, MA, USA) using a sine wave for 50,000 cycles or until failure occurred. Our preliminary investigations showed that the frequency had no significant effect on the results, and 10 Hz was selected for reasons of practicality.

The load was incrementally increased or decreased (depending on survival or failure) by approximately 10% of the initial load. For each test condition, 30 specimens were used to determine the SFS. Adapting the calculation described by Draughn,[20] the test stress that produces 50% failure is termed the fatigue strength. After testing, the specimens were examined to determine the type of bond failure in the same manner as for SBS, as described in the previous section.

Scanning Electron Microscopy Observations

Restorative-enamel interfaces and representative fracture sites after SFS testing were observed by field-emission scanning electron microscopy (SEM; ERA-8800FE, Elionix, Tokyo, Japan). For ultrastructural observations of the restorative/enamel interface, bonded specimens were embedded in epoxy resin and longitudinally sectioned with a precision low-speed saw (Isomet 111280, Buehler, Lake Bluff, IL, USA). The sectioned surfaces were polished to a high gloss with abrasive discs (Fuji Star Type DDC, Sankyo Rikagaku Co, Saitama, Japan) followed by diamond pastes to a 0.25-μm particle size (DP-Paste, Struers, Ballerup, Denmark). Fracture sites were prepared directly for evaporation coating. SEM specimens of restorative-enamel interfaces were dehydrated in ascending grades of tert-butyl alcohol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for 2 hours) and transferred from the final 100% bath to a critical-point dryer (Model ID-3, Elionix) for 30 minutes. Resin-enamel interface specimens were then subjected to argon-ion beam etching (EIS-200ER, Elionix) for 40 seconds with the ion beam (accelerating voltage 1.0 kV, ion current density 0.4 mA/cm^2) directed perpendicular to the polished surfaces. Finally, all SEM specimens were coated with a thin film of gold in a Quick Coater vacuum evaporator (Type SC-701, Sanyu Denchi Inc, Tokyo, Japan). Observations were carried out using an operating voltage of 10 kV.

Table 3: *Influence of Etching Mode on SBS in MPa (SD)*[a]

Adhesive	Total-etch Mode	Self-etch Mode
SU	46.4 (5.4)^{aA}	27.7 (3.8)^{bB}
PE	42.6 (5.2)^{aA}	28.8 (5.3)^{bB}
AU	42.1 (4.9)^{aA}	24.1 (2.4)^{cB}
SP	43.5 (5.5)^{aA}	27.5 (2.3)^{bB}

Abbreviations: AU, All-Bond Universal light-cured dental adhesive; PE, Prime&Bond Elect universal dental adhesive; SBS, shear bond strength; SFS, shear fatigue strength; SP, Clearfil S^3 Bond Plus; SU, Scotchbond Universal.
[a] Same lowercase letter in vertical columns indicates no difference at 5% significance level. Same capital letter in horizontal rows indicates no difference at 5% significance level.

Statistical Analysis

A two-way analysis of variance (ANOVA) followed by a Tukey honestly significant difference test at an α level of 0.05 were used for analysis of the SBS data. The statistical analysis for SBS was performed using the Sigma Plot software system (version 11.0; SPSS Inc, Chicago, IL, USA). A modified *t*-test with Bonferroni correction was used for the SFS data with a custom program implemented in a spreadsheet (Excel, Microsoft Inc, Redmond, WA, USA).

RESULTS

Shear Bond Strength

The SBS results in different etching modes are shown in Table 3. The two-way ANOVA revealed that the etching mode (total-etch vs self-etch) significantly influenced the SBS values ($p<0.001$), whereas the adhesive system did not ($p=0.390$). However, the interaction between these factors was significant ($p=0.003$).

The mean SBS in total-etch mode ranged from 42.6 ± 5.2 to 46.4 ± 5.4 MPa, whereas the corresponding values for the specimens in self-etch mode ranged from 24.1 ± 2.4 to 28.8 ± 5.3 MPa (Table 3). All adhesives showed significantly higher SBS values in total-etch mode than in self-etch mode. Although there was no significant difference among the tested adhesives in total-etch mode, AU showed a significantly lower SBS value ($p<0.05$) than the other adhesives in self-etch mode.

Shear Fatigue Strength

The SFS results in different etching modes are shown in Table 4. The mean SFS in total-etch mode ranged from 21.0 ± 4.8 to 22.4 ± 4.0 MPa, whereas the corresponding values for the specimens in self-etch mode ranged from 10.0 ± 2.2 to 12.8 ± 1.6 MPa. There was no significant difference among the tested adhesives ($p>0.05$) in both total-etch and self-etch modes. The ratios of SFS:SBS are shown in Table 4. For all adhesives, the total-etch groups demonstrated higher ratios than the self-etch groups.

Failure Mode Analysis of Debonded Specimens

The frequency of different failure modes, comparing the modes seen in SBS testing with those seen in SFS testing, are shown in Table 5. For all adhesives, the predominant mode of failure for specimens in self-etch mode was adhesive failure regardless of the testing methods. However, mixed failure and cohesive failure in enamel increased in total-etch mode for all adhesives in both SBS and SFS.

SEM Observations

Representative SEM images of debonded specimens after SFS testing are shown in Figures 1-4. For the universal adhesives, no clear morphological differences were observed between the self-etch mode and total-etch mode at lower magnifications (Figures 1a,c, 2a,c, and 3a,c). However, at higher magnification, cracks, cleavages, and cohesive failures in enamel could be seen more clearly in the total-etch group (Figures 1d, 2d, and 3d). For SP in self-etch mode, detachment of the boundary at the interface between the adhesive and enamel surface was observed at lower magnification (Figure 4a). Conversely, in total-etch mode, the de-

Table 4: *Influence of Etching Mode on SFS (MPa) and Ratio of SFS/SBS*[a]

Adhesive	Total-etch Mode	Self-etch Mode	Ratio SFS:SBS	
			Total-etch Mode	Self-etch Mode
SU	22.3 (4.6)^{aA}	12.8 (1.6)^{bB}	0.480	0.462
PE	21.0 (4.8)^{aA}	10.7 (1.2)^{bB}	0.493	0.372
AU	21.6 (2.2)^{aA}	10.0 (2.2)^{bB}	0.513	0.415
SP	22.4 (4.0)^{aA}	12.3 (3.5)^{bB}	0.515	0.447

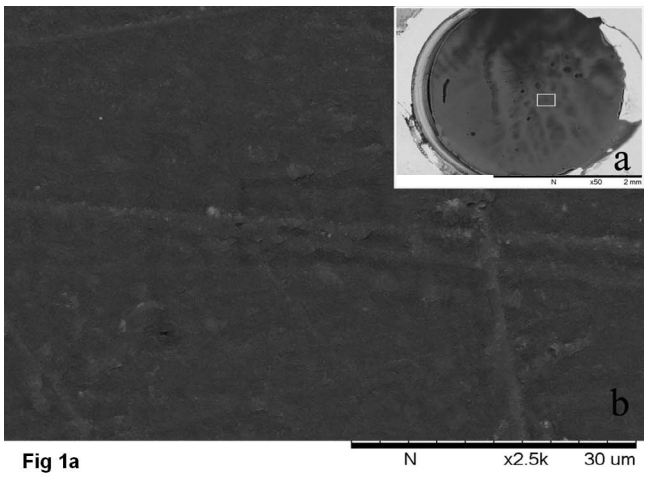
Abbreviations: AU, All-Bond Universal light-cured dental adhesive; PE, Prime&Bond Elect universal dental adhesive; SBS, shear bond strength; SFS, shear fatigue strength; SP, Clearfil S^3 Bond Plus; SU, Scotchbond Universal.
[a] Same lowercase letter in vertical columns indicates no difference at 5% significance level. Same capital letter in horizontal rows indicates no difference at 5% significance level.

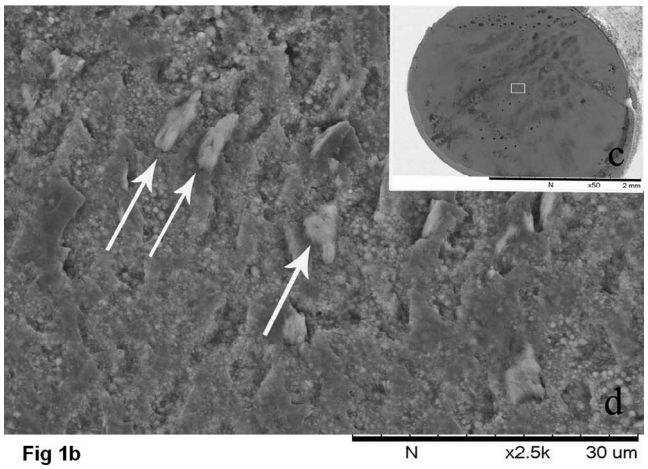
Table 5: *Failure Mode Analysis of Debonded Specimens*[a]

Adhesive	SBS		SFS	
	Total-etch	Self-etch	Total-etch	Self-etch
SU	[60/0/20/20]	[93/0/0/7]	[55/9/27/9]	[100/0/0/0]
PE	[80/0/0/20]	[100/0/0/0]	[77/0/23/0]	[100/0/0/0]
AU	[79/7/7/7]	[100/0/0/0]	[58/0/21/21]	[100/0/0/0]
SP	[67/0/26/7]	[100/0/0/0]	[77/0/23/0]	[100/0/0/0]

Abbreviations: AU, All-Bond Universal light-cured dental adhesive; PE, Prime&Bond Elect universal dental adhesive; SBS, shear bond strength; SFS, shear fatigue strength; SP, Clearfil S[3] *Bond Plus; SU, Scotchbond Universal.*
[a] *Failure mode: [adhesive failure/cohesive failure in resin composite/cohesive failure in enamel/mixed failure] percentage of each failure mode.*

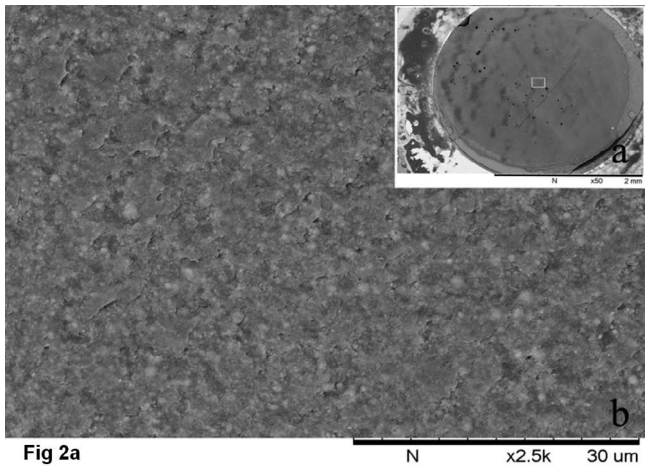
tachment area at the interface between the adhesive and resin composite increased, compared with self-etch mode (Figure 4c). In addition, cohesive failures in enamel and beach marks were more clearly visible (Figure 4d) than in self-etch mode (Figure 4b).





Figures 1-4. *Representative scanning electron microscope images of the fractured resin surface in enamel bonding after SFS.*
Figure 1. *(a): Scotchbond Universal in total-etch mode. (b): Scotchbond Universal in self-etch mode. a,c: 40×; b,d: 1000×.*

Representative SEM images of the resin-enamel interface are shown in Figures 5-8. Although clear differences were observed between samples of the two different treatment modes, the restorative-enamel interface showed excellent adaptation to the enamel surface regardless of the etching mode. However, the resin tags in the enamel surfaces were longer for specimens in total-etch mode than in self-etch mode. The thickness of the adhesive



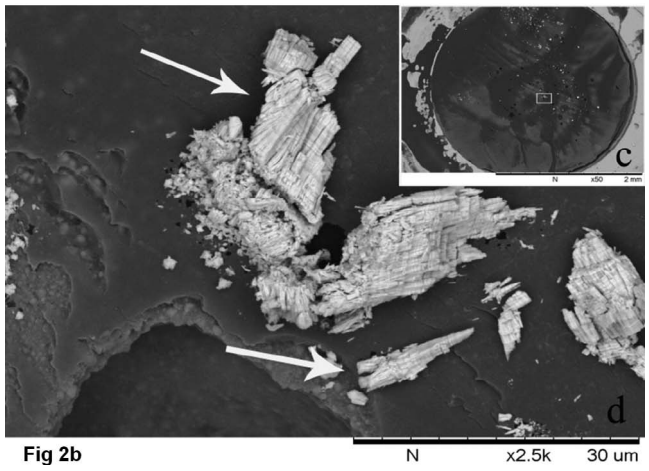


Figure 2. *(a): Prime&Bond elect in total-etch mode. (b): Prime& Bond elect in self-etch mode. a,c: 40×; b,d: 1000×.*

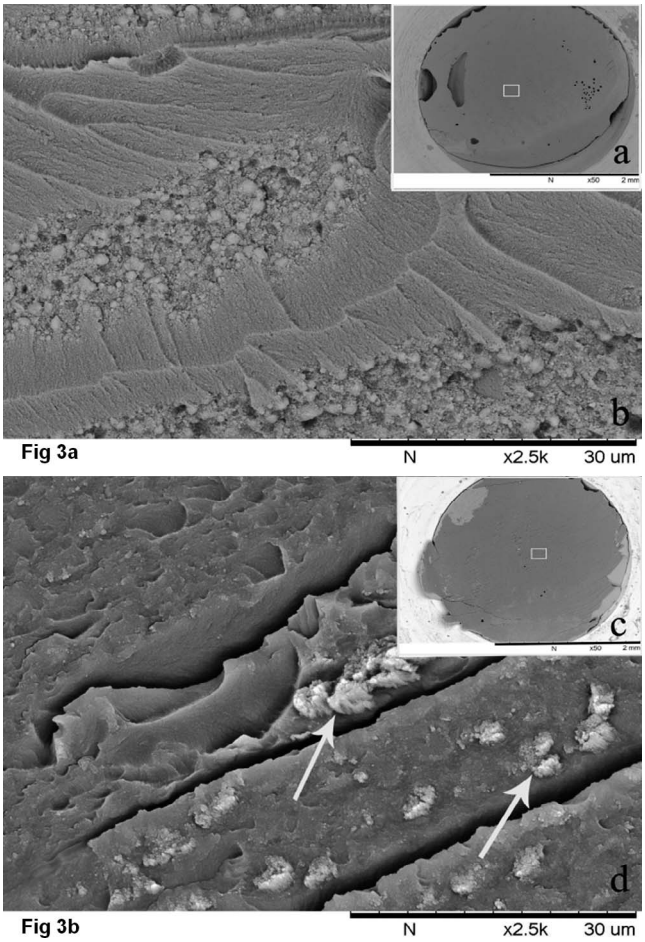


Figure 3. *(a): Clearfil S^3 Bond Plus in total-etch mode. (b): Clearfil S^3 Bond Plus in self-etch mode. a,c: 40×; b,d: 1000×. Arrows indicate cohesive failure in enamel.*

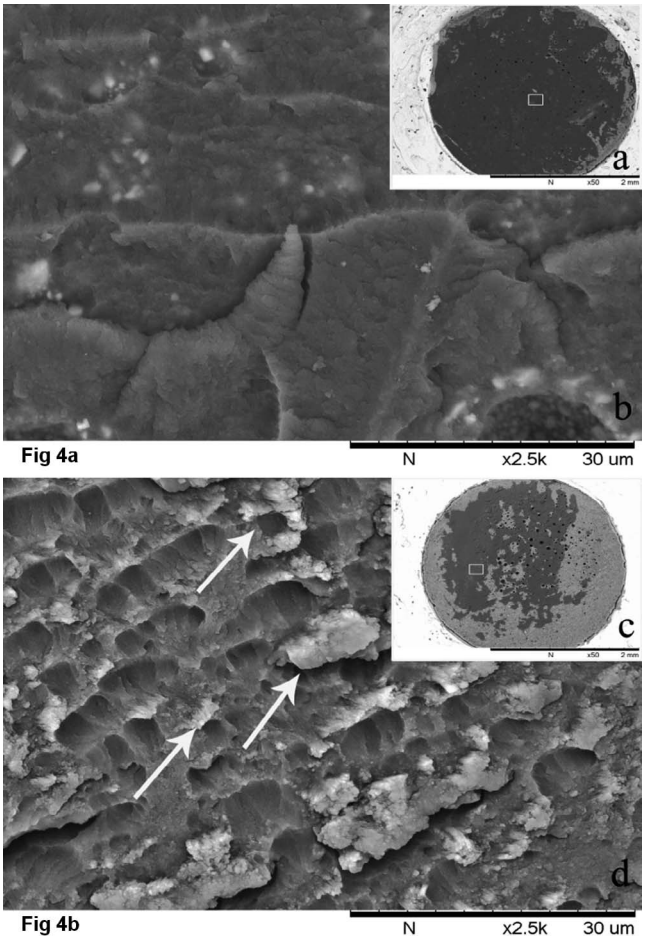


Figure 4. *(a): All-Bond Universal in total-etch mode. (b): All-Bond Universal in self-etch mode. a,c: 40×; b,d: 1000×.*

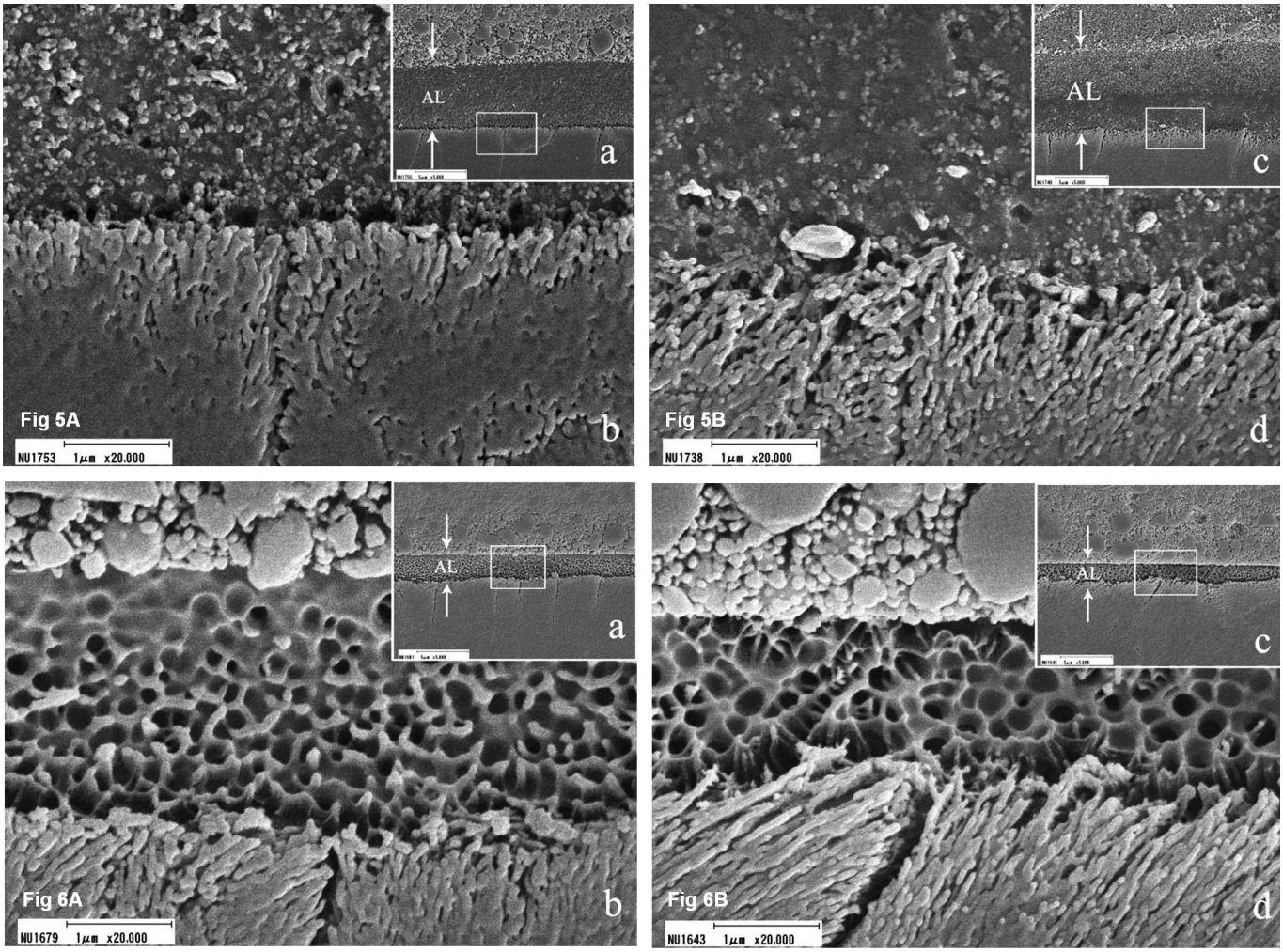
layer was 2-3 μm for PE (Figure 6) and 4-5μm for AU (Figure 7). The adhesive layers of SU (Figure 5) and SP (Figure 8) had similar thicknesses of 7-10 μm.

The nano-fillers in SU and SP (Figures 5 and 8) were clearly observed, although they were more prominent in SP. EL and AU did not contain nano-fillers (Figures 6 and 7) but had distinct adhesive layers. The adhesive layer of AU was largely homogeneous, with obvious inclusion of enamel fragments (arrows in Figure 7). Conversely, PE possessed a clear honeycomb network without enamel fragments (Figure 6).

DISCUSSION

Manufacturers of universal adhesives have claimed that there is no compromise in bonding performance when either total-etch or self-etch modes are used.[21] Multi-mode use of universal adhesives may address the disadvantages of previous generation single-step self-etch adhesives with lower enamel bond durability when used in self-etch mode. If true, this would allow the use of multiple modes in the same restoration. This, in turn, would allow clinicians to adapt appropriately to heterogeneous substrates, which would improve bond durability and reduce postoperative sensitivity. Similarly, when a single mode must be used for the whole of a small restoration, any heterogeneity of the substrate should not have a negative effect. This should have clinical benefits.

However, there is concern that the technology of universal adhesives may not offer a genuine advantage when compared with previous generations of single-step self-etch adhesives.[7,21] In addition, to adapt to multiple substrates without individual pretreatment, the composition of universal adhesives is more complicated than that of the previous generation of self-etch adhesives. However, there is minimal information regarding the influence of



Figures 5-8. *Representative scanning electron microscope images of the resin-enamel interface. The visible material is indicated by the abbreviation AL: adhesive layer.*
Figure 5. *(a): Scotchbond Universal in total-etch mode. (b): Scotchbond Universal in self-etch mode. a,c: 5000×; b,d: 20,000×.*
Figure 6. *(a): Prime&Bond elect in total-etch mode. (b): Prime&Bond elect in self-etch mode. a,c: 5000×; b,d: 20,000×.*

different application modes on the durability of the bond to tooth substrates. Therefore, to clarify the bonding performance of universal adhesives, enamel bond durability was evaluated through fatigue testing.

In self-etch mode, AU showed a significantly lower SBS value than the other adhesives. However, in total-etch mode, there were no significant differences among the adhesives in SBS. The reason that AU demonstrated a lower SBS value in self-etch mode is thought to be related to it having a lower etching capability than other universal adhesives. Chen and others[21] reported that PE and SU are classified as mild self-etch adhesives, whereas AU is classified as an ultramild self-etch adhesive on the basis of pH and transmission electron microscope observations of the adhesives. Therefore, the lower acidity of AU might not allow for strong micromechanical retention, leading to a lower SBS value.

There were no significant differences among all adhesives in SFS, although values in self-etch mode were significantly lower than in total-etch mode. In particular, none of the universal adhesives differed from the conventional single-step self-etch adhesive SP. Therefore, the null hypothesis that universal adhesives would not differ from conventional single-step self-etch adhesives with respect to bond durability was not rejected. However, the other null hypothesis (that phosphoric acid pre-etching would not affect the enamel bond durability of universal adhesives) was rejected.

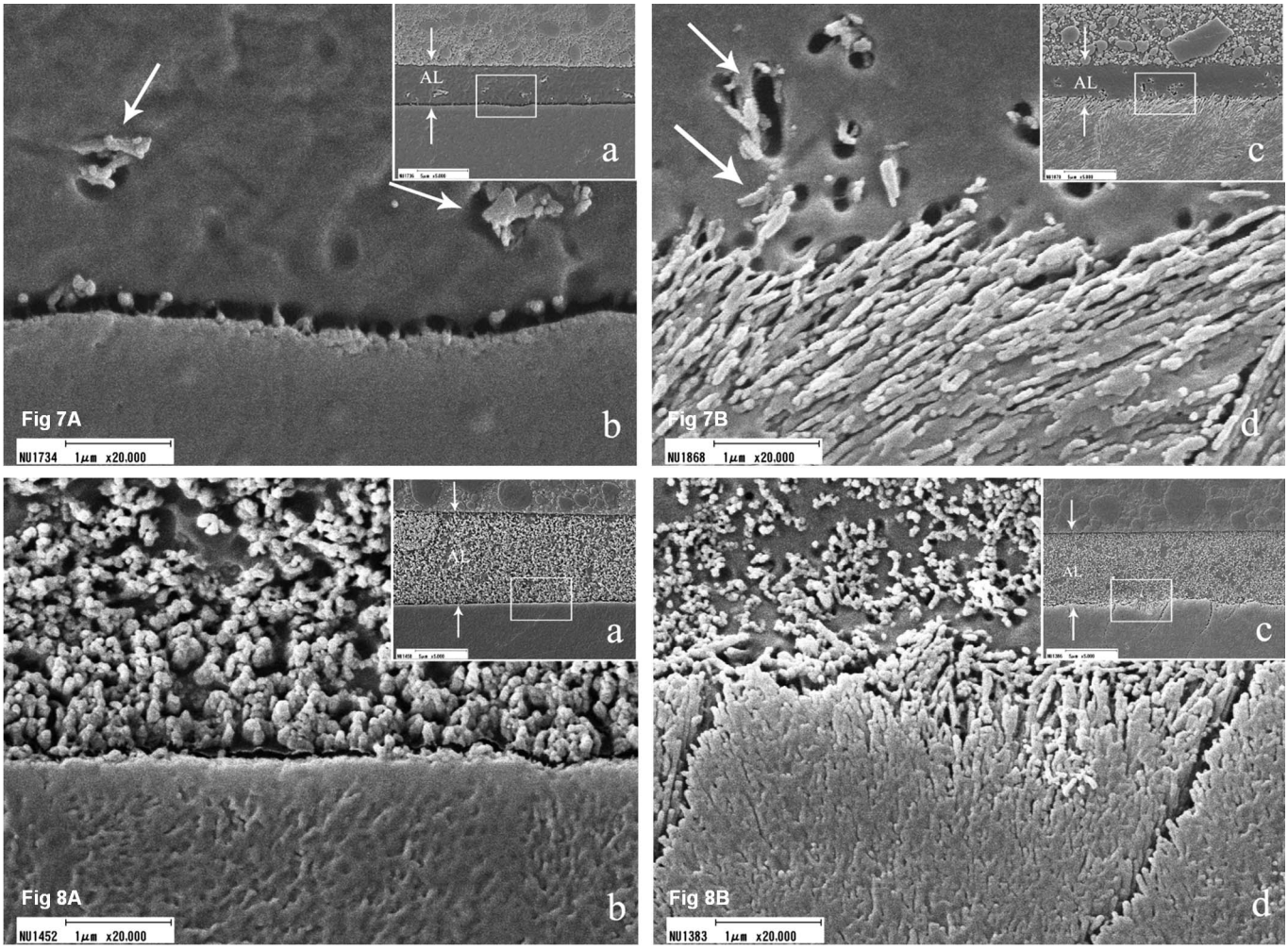


Figure 7. *(a): All-Bond Universal in total-etch mode. (b): All-Bond Universal in self-etch mode. a,c: 5000×; b,d: 20,000×. Arrows indicate enamel fragments in adhesive layers.*
Figure 8. *(a): Clearfil S^3 Bond Plus in total-etch mode. (b): Clearfil S^3 Bond Plus in self-etch mode. a,c: 5000×; b,d: 20,000×.*

The durability of bonded interfaces is threatened by many factors, such as biofilm attack, hydrolytic degradation of the adhesive, and fatigue of the adhesives.[23-25] In addition, dentin bonded interfaces are attacked through enzymatic degradation by matrix metalloproteinases.[26,27] Conversely, unlike dentin, enamel is composed of homogeneous components with minimal organic content. Therefore, the process of enamel bond degradation is simpler than that of dentin bonds due to the absence of collagen fibrils. Hence, deterioration of the adhesive layer in enamel bonds may directly lead to failure of the bond itself, indicating that the composition and properties of the adhesive layer are the primary factors in enamel bond durability.

Fatigue can be defined as the degradation or failure of mechanical properties after repeated subcritical stress at a level below the ultimate fracture strength of the material or interface.[22] Fatigue testing carried out in this study provided not only information related to the endurance characteristics of the bonding systems but also information regarding the ability of the resin-enamel interface zone to resist stress loading. When considering long-term bond durability, the degradation mechanism of the resin-tooth interface is extremely important.

Although there was no statistically significant difference in SFS among the adhesives, AU and PE tended to show lower values than did SU and SP. Although no clinical conclusions can be drawn from this difference because it is not significant, the compositions of the adhesives suggest that these differences may be valid and worthy of further investigation. The adhesive layers of SU and SP contain nano-fillers and are approximately two to three times thicker than those of AU and PE. This

thicker adhesive layer may behave as a shock absorber and the presence of nano-filler may inhibit crack propagation. In addition, the thicker adhesive layer may decrease the relative importance of the oxygen inhibition layer, which is thought to be a vulnerable site.[28] It can thus be proposed that different characteristics of the adhesive layers influenced the enamel bond durability in self-etch mode.

Erickson and others[29] reported that self-etch adhesives produce an etching pattern primarily involving the ends of enamel prisms and fine pitting of the enamel surface, with minimal effect on the interprismatic regions. Bond strength to phosphoric acid–etched enamel is mainly attributable to the penetration of adhesives into the enamel crystals and rods. From the SEM observations of enamel-resin interfaces in self-etch mode, gaps between enamel crystals and rods were not found in any adhesive. However, in total-etch mode, spicular etching patterns and penetration of resin tags were clearly observed for all adhesives. In addition, from the SFS results, total-etch mode significantly increased the enamel bond durability of universal adhesives compared with self-etch mode. Therefore, creating micromechanical retention on the enamel surface through phosphoric acid pre-etching may contribute to better resistance of long-term biomechanical loads when using universal adhesives.

Regarding dentin bonding, we evaluated the influence of different etching modes on the dentin bond durability of three universal adhesives using the same fatigue testing method as the present study.[30] In our study, the SBS and SFS of dentin bonds of a conventional single-step self-etch adhesive showed significantly lower values in total-etch mode when compared with those in self-etch mode. In contrast, the SBS and SFS of the universal adhesives showed equivalent bonding quality to dentin regardless of etching mode, although the dentin bond quality of the universal adhesives did vary among adhesives. We concluded that using universal adhesives in total-etch mode does not have a negative impact on dentin bond quality. However, postoperative sensitivity arises from a different mechanism from bond strength, and increased etching of dentin in the total-etch mode may increase it. This is an important topic for further work.

The clinical implication of this study is that universal adhesives should be used with phosphoric acid pre-etching of enamel, as with the previous generation of single-step self-etch adhesives. In clinical situations, the adherent surface of mineralized tooth tissue is dependent on the cavity configuration. Given that there is no compromise in bonding performance when either total-etch or self-etch mode is used with dentin, total-etch or selective-etch mode should be chosen to achieve a reliable bond to the enamel substrate. In particular, if the cavity is too small to precisely manipulate the phosphoric acid agent for selective etching, the total-etch approach with universal adhesives is most likely appropriate.

CONCLUSIONS

Within the limitations of this *in vitro* study, although AU demonstrated a significantly lower SBS value than the other tested adhesives in self-etch mode, there were no significant differences among the adhesives in both SBS and SFS in total-etch mode and in SFS in self-etch mode. SEM observations of resin-enamel interfaces revealed that the resin tags in enamel surfaces were longer for the samples in total-etch mode than those in self-etch mode. In addition, the thickness and characteristics of the adhesive layers were dependent on the adhesive.

All tested adhesives showed significantly higher values in total-etch mode than in self-etch mode. Therefore, for universal adhesives, total-etch mode has a positive effect on the enamel bond durability, as with the previous generation of single-step adhesives.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Nihon University School of Dentistry. The approval code for the study is 2015-06.

Conflict of Interest

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

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Blue-Light Transmittance of Esthetic Monolithic CAD/CAM Materials With Respect to Their Composition, Thickness, and Curing Conditions

B Stawarczyk • D Awad • N Ilie

Clinical Relevance

The amount of light passing through VITA ENAMIC restorations is reduced and less light-sensitive dual-curing cements should be used for cementation.

SUMMARY

Determining the amount of blue light (360-540nm) passing through nine monolithic computer-aided design/computer-aided manufacturing (CAD/CAM) materials depends on material thickness, initial irradiance, and the distance between the curing unit and the specimen's surface. A total of 180 specimens of two thicknesses (1 mm and 2 mm, n=10/subgroup) were fabricated from TelioCAD, VITA CAD-Temp (VCT), experimental nanocomposite, LAVA Ultimate (LU), VITA ENAMIC (VE), VITA MarkII (VM), IPS EmpressCAD (IEC), IPS e.maxCAD (IEM), and CELTRA DUO (CD). The irradiance passing through the CAD/CAM materials and thicknesses was measured using a light-emitting-diode curing unit with standard-power, high-power, and plasma modes by means of a USB4000 spectrometer. The curing unit was placed directly on the specimen's surface at 2- and 4-mm distances from the specimen's surface. Data were analyzed using a multivariate analysis and one-way analysis of variance with the *post hoc* Scheffé test ($p<0.05$). The highest transmitted irradiance was measured for VM and LU, followed by VCT and IEC, while the lowest values showed VE, followed by IEM and CD. The highest transmitted irradiance was recorded by exposing the material to the plasma mode, followed by the high- and standard-power modes. The measured irradiance was

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decreased by increasing the specimen's thickness from 1 to 2 mm. Fewer differences were measured when the curing unit was placed at 0 or 2 mm from the specimen's surface, and the irradiance passing through the specimens was lower at a distance of 4 mm.

INTRODUCTION

There is a range of polymer-, composite-, or ceramic-based esthetic monolithic computer-aided design/computer-aided manufacturing (CAD/CAM) materials presently available on the market. Dentists can process these materials using CAD/CAM technology in minutes while the patient is seated in the chair. Industrially prefabricated CAD/CAM materials appear to be more structurally reliable for dental applications than materials that are manually processed under dental laboratory conditions. Polymeric-based CAD/CAM materials showed significantly higher mechanical properties compared to conventional temporaries[1-5] and can be used for long-term restorations.[6] Standard monolithic CAD/CAM materials for permanent restorations contain lithium disilicate glass ceramics,[7-9] feldspathic silicate ceramics,[10] and feldspar-based leucite-reinforced glass ceramics[11,12] but also newly developed materials, such as a resin-based block nanocomposite,[13] an experimental isofiller resin-based composite with "nano additives,"[14] and a novel interpenetrating network ceramic (VITA ENAMIC).[15-18] Similarly, a zirconia-reinforced lithium silicate ceramic (CELTRA DUO) offers the opportunity for a permanent restoration. Since the latter materials are quite new, there is little scientific knowledge about them.

In addition to the particular restorative material, the esthetics of a CAD/CAM restoration also depends on the chosen luting cement. Traditional cements, such as glass ionomer and zinc phosphate, are usually very opaque and can therefore distort the color of the esthetic restoration. Esthetic glass-ceramic restorations also demonstrated better long-term clinical stability when cemented with resin composite cements rather than traditional cements.[19,20] An *in vitro* study also reported on the increase of fracture resistance of adhesive-luted crowns compared to traditionally cemented ones.[21] Esthetic restorative materials with lower mechanical properties require reinforcement by adhesive cementation.[21-23] Dual-cure resin adhesive composite cements are often used for these indications. An advantage of these resin composite cements is that they can cure both chemically (autocuring) and via visible-light activation. Such resin composite cements include a catalyst paste with a chemical activator (benzoyl peroxide) and a base paste containing blue-light–cured resin cement as well as an amine responsible for the beginning of the autocuring reaction.[24,25] After mixing both pastes and with a supply of light, the polymerization takes place through physical (photo) and chemical (redox) activation.[24] The working time is controlled by inhibitors of the autocure reaction or by the amount of activators in the polymerization.[26] Nevertheless, when not properly photoactivated, dual-cure resin cements may present reduced degrees of conversion.[27-29] This in turn leads to lower mechanical properties, such as hardness[25] and flexural and compressive strength,[25,30] and higher solubility.[31] Studies have also shown that the absence of light negatively influences the long-term bond strength.[32,33] The impact of light on the polymerization process of dual-cure luting resin composite cements is material dependent.[34] Light-cured resin cements and, in particular, resin composites have thus become an important part of modern, minimally invasive treatment.[35] These resin composites consist of a single paste with a visible-light activation of a photosensitive component (eg, camphorquinone) and an amine. The visible light activates the photosensitive initiator to generate a short-lived excited-state species that complexes with the tertiary amine to promote a sequential electron and proton transfer that creates the active initiating radicalable to start the polymerization.[24]

Direct resin composites are densely filled with inorganic particles and therefore provide high mechanical properties. However, increasing filler parts in resin composites also enhances viscosity, which could reduce the ease of clinical application. Options to improve the rheological behavior by using ultrasonics[36] or preheating[37-39] are of primary interest to the clinician.

Along with improvements of the mechanical properties, cementation using light-luting resin composites has several benefits in clinical applications. Light-cured resin composites have long working times, with the polymerization beginning immediately after the exposure of the material to light.[33]

Many resin composites indicate a high sensitivity to the additional occurrence of blue light, which significantly affects their mechanical properties.[34] The amount of light passing through restoration materials and the translucency of the materials are thus essential elements of cementation with dual-

Table 1: *Product Name, Abbreviation, Material Type, Lot Number, and Color of CAD/CAM Materials Evaluated*

Material (Abbreviation)	Manufacturer	Material Type	Lot Number	Color
Telio CAD (TC)	Ivoclar Vivadent, Schaan, Liechtenstein	PMMA	N73354	LT A2
VITA CAD-Temp (VCT)	VITA Zahnfabrik, Bad Säckingen, Germany	PMMA	CE 0124	2M2
Experimental nanocomposite (TEC)	Ivoclar Vivadent	Resin composite	28923	HT A2
LAVA Ultimate (LU)	3M ESPE, Seefeld, Germany	Resin composite	N372985	HT A2
VITA ENAMIC (VE)	VITA Zahnfabrik	Hybrid ceramic	33000	3M2
VITA Mark II (VM)	VITA Zahnfabrik	Feldspar ceramic	N502353	A2
IPS Empress CAD (IEC)	Ivoclar Vivadent	Leucite glass ceramic.	R39335	LT A2
IPS e.max CAD (IEM)	Ivoclar Vivadent	Lithium disilicate glass ceramic	R37085	LT A2
CELTRA Duo (CD)	DeguDent, Hanau, Germany	Zirconia-reinforced lithium silicate	18015733	LT A2

curing resin composite cements. A previous study investigated the amount of blue light passing through differently colored zirconia ceramics and recommended the use of less light-sensitive dual-cured cements for restorations thicker than 1.5 mm in light-shaded zirconia and 0.5 mm in darker-shaded zirconia.[40]

This study investigated the amount of blue light passing through nine CAD/CAM monolithic materials. Four hypotheses were tested: 1) different CAD/CAM materials, 2) material thickness, 3) curing unit distance to the specimen, and 4) initial irradiance level (curing modes) show no impact on the transmitted irradiance through the CAD/CAM materials.

METHODS AND MATERIALS

Nine different CAD/CAM monolithic materials were selected: TelioCAD (TC; PMMA based; Ivoclar Vivadent, Schaan, Liechtenstein), VITA CAD-Temp (VCT; PMMA based and 10% filled with prepolymers; VITA Zahnfabrik, Bad Säckingen, Germany), experimental nanocomposite (TEC; filled composite; Ivoclar Vivadent) LAVA Ultimate (LU; filled composite; 3M ESPE, Seefeld, Germany), VITA ENAMIC (VE; interpenetrating network ceramic; VITA Zahnfabrik), VITA Mark II (VM; feldspar ceramic; VITA Zahnfabrik), IPS EmpressCAD (IEC; leucite glass ceramic; Ivoclar Vivadent), IPS e.max CAD (IEM; lithium disilicate glass ceramic; Ivoclar Vivadent), and CELTRA DUO (CD; zirconia-reinforced lithium silicate ZLS; DeguDent, Hanau, Germany) (Table 1). CAD/CAM blocks were cut using a low-speed diamond saw in 1- and 2-mm-thick slices (n=10) (Well 3241, Well Diamantdrahtsägen, Mannheim, Germany) under water cooling.

All specimens were polished up to 1 μm with a diamond suspension (Struers, Ballerup, Denmark) and then ultrasonically cleaned for 5 minutes in distilled water. The final dimensions of all specimens were $10 \times 10 \times 1$ mm $\pm$ 0.05 and 10×10 mm $\times 2 \pm$ 0.05 mm.

The analysis of the irradiance passing through the CAD/CAM materials was performed using the blue-violet light-emitting-diode polymerizing unit (VALO, Ultradent, South Jordan, UT, USA) on a laboratory-grade National Institute of Standards and Technology–referenced USB4000 Spectrometer (MARC System, Bluelight Analytics Inc, Halifax, NS, Canada) (n=10). The miniature fiber-optic USB4000 spectrometer uses a 3648-element Toshiba linear charge-coupled-device array detector and high-speed electronics. The spectrometer was spectroradiometrically calibrated using Ocean Optics' NIST-traceable light source (300-1050 nm) (Figure 1). The system uses a CC3-UV Cosine Corrector to collect radiation over a 180-degree field of view, thus mitigating the effects of optical interference associated with light collection sampling geometry.

The irradiance (wavelength ranged from 360 to 540 nm) passing through the nine different CAD/CAM materials and material thicknesses (1 and 2 mm) was measured at the bottom of the specimens at a velocity of 16 records per second. The sensor was triggered at 20 mW. The curing unit was placed directly on the specimen's surface as well as 2 and 4 mm away from the specimen's surface. Three curing modes were examined (standard-power, high-power, and plasma modes), resulting in 180 measurements (2 material thicknesses $\times$ 3 exposure modes $\times$ 3 distances $\times$ 10 specimens) for each ceramic.

Additionally, one randomly selected specimen for each material was analyzed in a scanning electron microscope (SEM; Supra 55 VP, Zeiss, Jena, Germany). For this, the VM and IEC were etched for 60 seconds and IEM and CD for 30 seconds with 9% hydrofluoric acid (Ultradent, lot B6X7B). The specimens were then ultrasonically cleaned and subsequently gold sputtered for 20 seconds. Surface

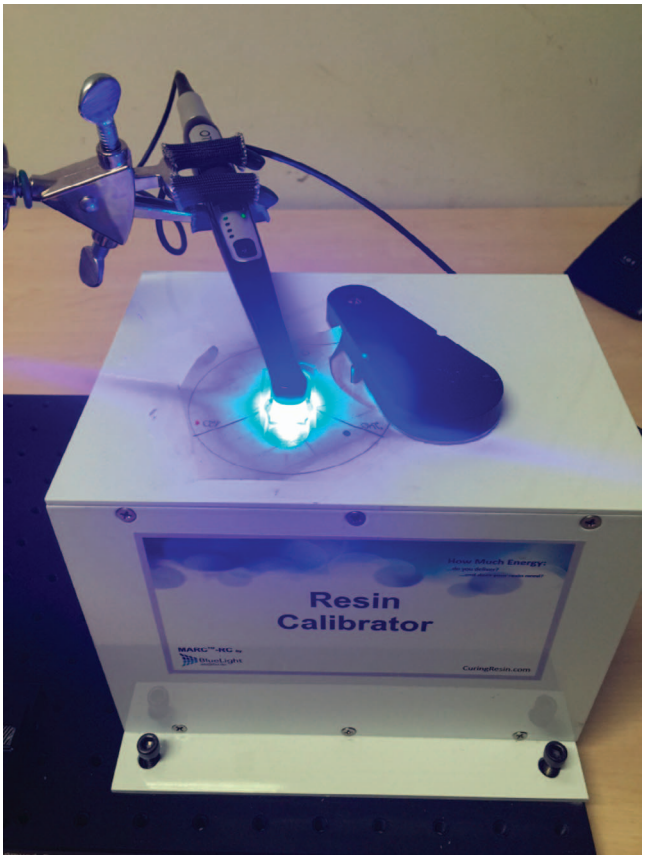

Figure 1. *Testing apparatus.*

topography analyses were performed using an inLens detector at 10 kV with a working distance of 4.5-6.0 mm.

A multivariate analysis (general linear model) assessed the effect of the material, material thickness (1 and 2 mm), distance from the surface (0, 2, and 4 mm), and curing mode (standard-power, high-power, and plasma modes) on the irradiance passing through the CAD/CAM materials. The statistical comparisons between the groups were performed using one-way analysis of variance followed by the *post hoc* Scheffé test; *p*-values smaller than 5% were considered statistically significant (SPSS, version 22.0, SPSS Inc, Chicago, IL, USA).

RESULTS

The greatest influence on the transmitted irradiance was exerted by the curing mode ($\eta_P^2 = 0.991$), closely followed by specimen thickness ($\eta_P^2 = 0.989$), CAD/CAM material ($\eta_P^2 = 0.966$), and distance from the specimen's surface ($\eta_P^2 = 0.904$). All binary combinations of these parameters were also significant ($p<0.05$).

The highest significant values for transmitted irradiance were measured for the materials VM and LU, followed by VCT and IEC, while the lowest significant values were for VE, followed by IEM and CD (Figures 2 through 4). Detailed information about the significant differences between the tested CAD/CAM materials is presented in Table 2.

Among the three tested curing regimens, the highest significant irradiance was recorded by exposing the CAD/CAM material to the plasma mode, followed by the high- and standard-power modes ($p<0.05$). The irradiance measured at 0-mm distance was 3416. ± 8.3 mW/cm^2 for the plasma mode, 1766 ± 0.1 mW/cm^2 for the high-power mode, and 1178 ± 0.5 mW/cm^2 for the standard-power mode. Following the same sequence, an increase in irradiance was identified at 2-mm distance (3797 ± 87.6 mW/cm^2, 1939 ± 3.2 mW/cm^2, and 1272 ± 24.5 mW/cm^2, respectively), and the lowest irradiances were measured at 4-mm distance from the specimen surface (2606 ± 6.6 mW/cm^2, 1346 ± 3.7 mW/cm^2, and 1002 ± 18.0 mW/cm^2).

The transmitted irradiance decreased significantly by increasing the specimen's thickness from 1 to 2 mm ($p<0.05$). Fewer differences were measured when the curing unit was placed at 0 or 2 mm from the specimen's surface, and the irradiance passing through the specimens was lower at a distance of 4 mm ($p<0.05$). SEM pictures of the microstructure of all tested CAD/CAM materials are presented in Figure 5.

DISCUSSION

Tooth-colored monolithic CAD/CAM materials seem to be suitable materials for dental applications; however, a cementation method using resin composite cements remains a key factor in ensuring long-lasting survival and success rates. Previous investigations have shown that the mechanical properties of dual-cure luting cements,[34] as well as the bond strength to dental ceramics, are positively influenced by the amount of light reaching the cements.[41,42]

In general, the highest transmitted irradiance was measured at the bottom of the feldspathic ceramic VM and the resin composite LU in the present study. The lowest values were measured for a hybrid ceramic VE followed by both lithium disilicate ceramics IEM and CD. The first null hypothesis, that the different CAD/CAM materials show no impact on the irradiance through the material, is rejected. It is worth noting that 60.8%-84.0% of the initial irradiance reaching the material surface is

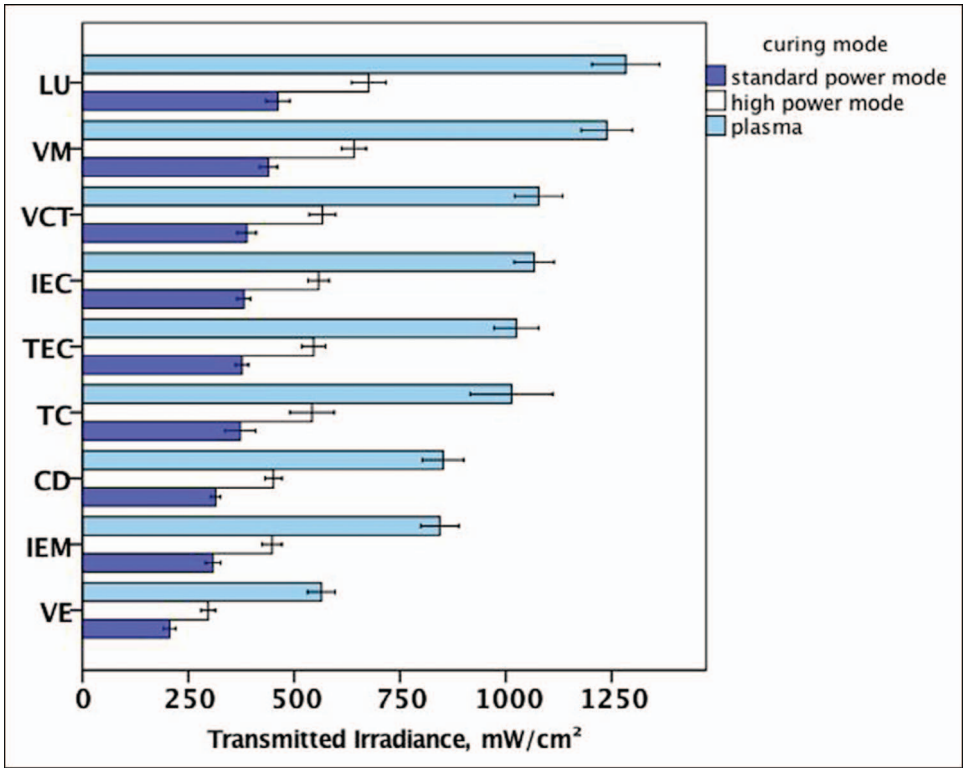

Figure 2. *Transmitted irradiance as a function of material and initial irradiance in 1-mm-thick specimens. The curing unit was positioned directly on the specimen surface.*

lost in passing 1-mm-thick increments of the analyzed materials, and this range changes to 80.6% –95.5% for 2-mm-thick specimens. Within one material, this value is influenced in only a minor way by the level of the initial irradiance. The analyzed materials might be grouped into four categories with respect to this behavior in descending order of their translucency: 1) LU and VM

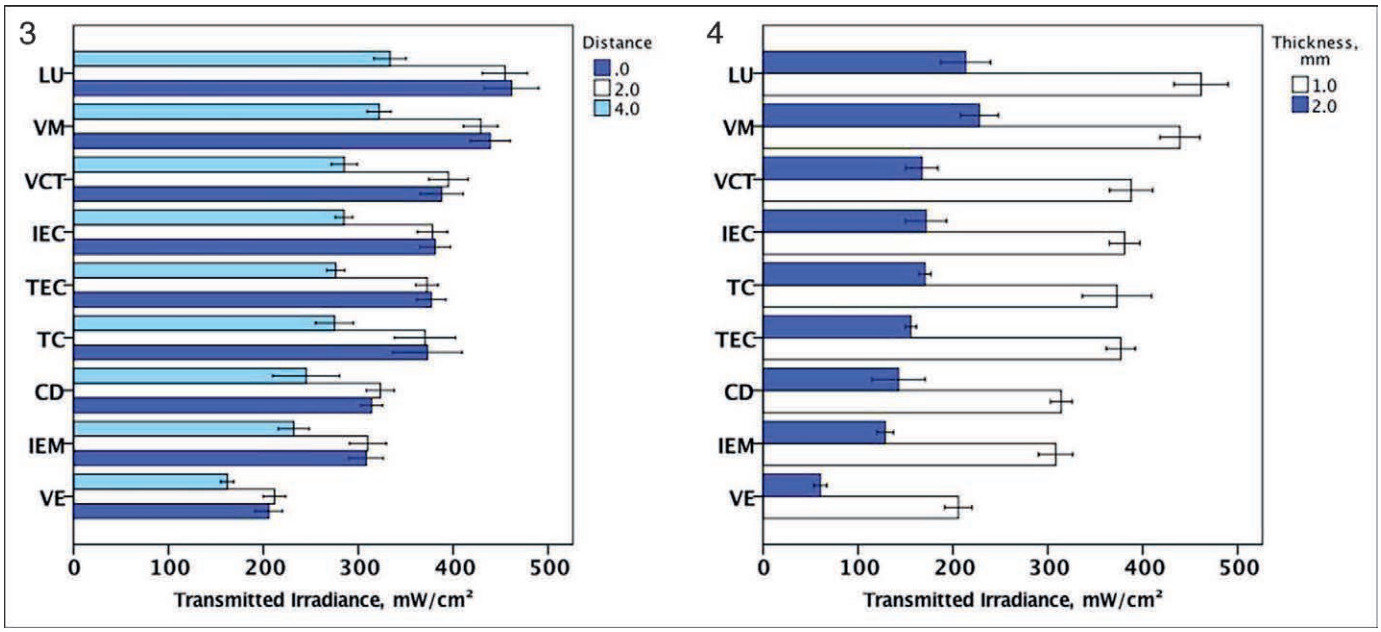

Figure 3. *Effect of distance between light curing and material surface (standard curing mode, 1-mm-thick specimens)*
Figure 4. *Transmitted irradiance as a function of material type and thickness. The curing unit (standard curing mode) was positioned directly on the specimen surface*

Table 2: *Descriptive Statistics for All Tested CAD/CAM Materials With Respect to Material Thickness, Curing Modus, and Distance Between Specimens and Light Unit*

CAD/CAM Material	Distance Between Specimens and Light Unit					
	0 mm		2 mm		4 mm	
	Thickness of CAD/CAM Material		Thickness of CAD/CAM Material		Thickness of CAD/CAM Material	
	1 mm Mean (SD)	2 mm Mean (SD)	1 mm Mean (SD)	2 mm Mean (SD)	1 mm Mean (SD)	2 mm Mean (SD)
Standard-power mode						
TC	373 (18.3) C	171 (3.1) D	371 (16.1) C	168 (3.9) C	275 (9.9) C	129 (2.1) DE
VCT	388 (11.4) C	167 (8.5) D	395 (10.3) D	163 (7.0) C	285 (6.8) C	126 (4.4)* DE
TEC	377 (7.7) C	156 (2.9) CD	372 (5.9) C	155 (4.5)* C	276 (4.8) C	120 (2.6) CD
LU	462 (14.3) E	213 (13.1) E	455 (11.9) F	204 (10.7) D	333 (8.6) D	157 (8.4) F
VE	206 (7.3) A	60 (3.5) A	212 (5.9) A	62 (5.0) A	162 (3.5) A	50 (2.0) A
VM	439 (10.6) D	228 (10.0) E	429 (9.1) E	219 (7.6)* E	322 (6.3) D	178 (4.3) G
IEC	381 (8.1) C	172 (10.9) D	378 (7.9) CD	167 (11.8) C	285 (4.6) C	132 (7.4)* E
IEM	308 (9.1) B	129 (4.4) B	310 (9.7) B	127 (4.9) B	232 (8.3)* B	102 (3.7) B
CD	314 (5.7) B	143 (14.1) BC	323 (7.3) B	139 (10.2) B	245 (17.7) B	111 (7.1) BC
High-power mode						
TC	542 (26.3) C	243 (3.2) DE	537 (23.8) C	236 (3.5) C	397 (13.8) C	183 (3.3) CD
VCT	567 (15.7) C	241 (11.5) DE	575 (15.0) CD	237 (9.4) C	414 (11.0) C	181 (7.4) CD
TEC	546 (13.9)* C	221 (5.8) CD	540 (6.2) C	220 (6.4) C	399 (6.1) C	171 (4.3) C
LU	676 (20.2) E	307 (20.1) F	666 (11.1) E	294 (15.4) D	486 (10.3) D	226 (12.2) E
VE	297 (8.5) A	84 (4.0) A	307 (8.0) A	86 (5.1) A	233 (5.9) A	69 (2.3) A
VM	641 (14.5) D	330 (13.2) F	609 (60.9) D	317 (12.6)* F	473 (9.8) E	257 (6.9) F
IEC	558 (12.5) C	246 (17.7) E	554 (11.7) C	241 (17.7)* C	417 (7.2) C	190 (13.4) D
IEM	448 (11.7) B	183 (5.4) B	450 (14.7) B	181 (5.5) B	338 (12.4) B	140 (5.0) B
CD	451 (10.0) B	200 (18.4) BC	466 (7.7) B	199 (15.0) B	355 (27.9) B	155 (11.1) B
Plasma mode						
TC	1014 (48.7) C	441 (8.3) DE	1007 (44.3) C	431 (8.4) DE	744 (30.2) C	333 (5.9) DE
VCT	1078 (28.3) D	452 (21.4) E	1100 (29.2) E	441 (20.1) DE	793 (20.4) D	340 (13.8) DE
TEC	1025 (26.3) CD	406 (9.9) CD	1017 (13.6) CD	405 (13.3) CD	753 (9.4) CD	315 (7.5) CD
LU	1283 (40.0) E	564 (39.6) F	1261 (38.7) G	543 (33.5) G	922 (19.6) E	418 (22.4) G
VE	546 (16.3) A	155 (7.7) A	582 (14.2) A	158 (10.5) A	443 (9.7) A	125 (5.6) A
VM	1239 (30.2) E	625 (26.5) G	1204 (24.0) F	605 (22.5) G	909 (19.1) E	485 (14.9) G
IEC	1067 (23.6) CD	466 (35.9) E	1064 (22.4) DE	455 (37.2) E	797 (12.7) D	359 (29.7) E
IEM	844 (22.6) B	339 (10.3) B	850 (27.7)* B	336 (10.0) B	636 (21.7)* B	263 (8.5) B
CD	852 (24.4) B	371 (35.3) BC	879 (27.1) B	370 (30.1) BC	672 (59.4)* B	291 (20.7) BC

** Significant differences between the different CAD/CAM materials within one material thickness, cutting modus, and distance between specimens and light unit are marked with different letters.*

(60.8%-62.7% initial irradiance loss when passing 1-mm-thick increments and 80.6%-81.9% when passing 2-mm-thick increments); 2) VCT, IED, TEC, and TC (67.1%-68.3% and 85.8%-86.8 %, respectively); 3) CD and IEM (73.3%-78.8% and 87.9%-89%); and 4) VE (82.5% and 94.9%).

The translucency of ceramic materials and thus the transmitted irradiance are dependent on the crystalline structure, grain size, and pigments as well as the number, size, and distribution of defects and porosity.[43,44] In this study, lithium disilicate glass-ceramic IEM and lithium silicate glass-ceramic CD showed significantly lower transmitted irradiance values than the leucite-reinforced ceramic Empress CAD or feldspathic ceramic VM. A previous study reported higher translucency values for leucite-reinforced IEC than for lithium disilicate glass ceramics and explained this as a result of the different microstructures, with less dense crystals in the leucite-reinforced ceramic than in the lithium disilicate ceramic.[45] These results were confirmed in this study. Lithium disilicate crystals are needle shaped and randomly oriented, representing about two-thirds of the glass-ceramic volume.[46] The

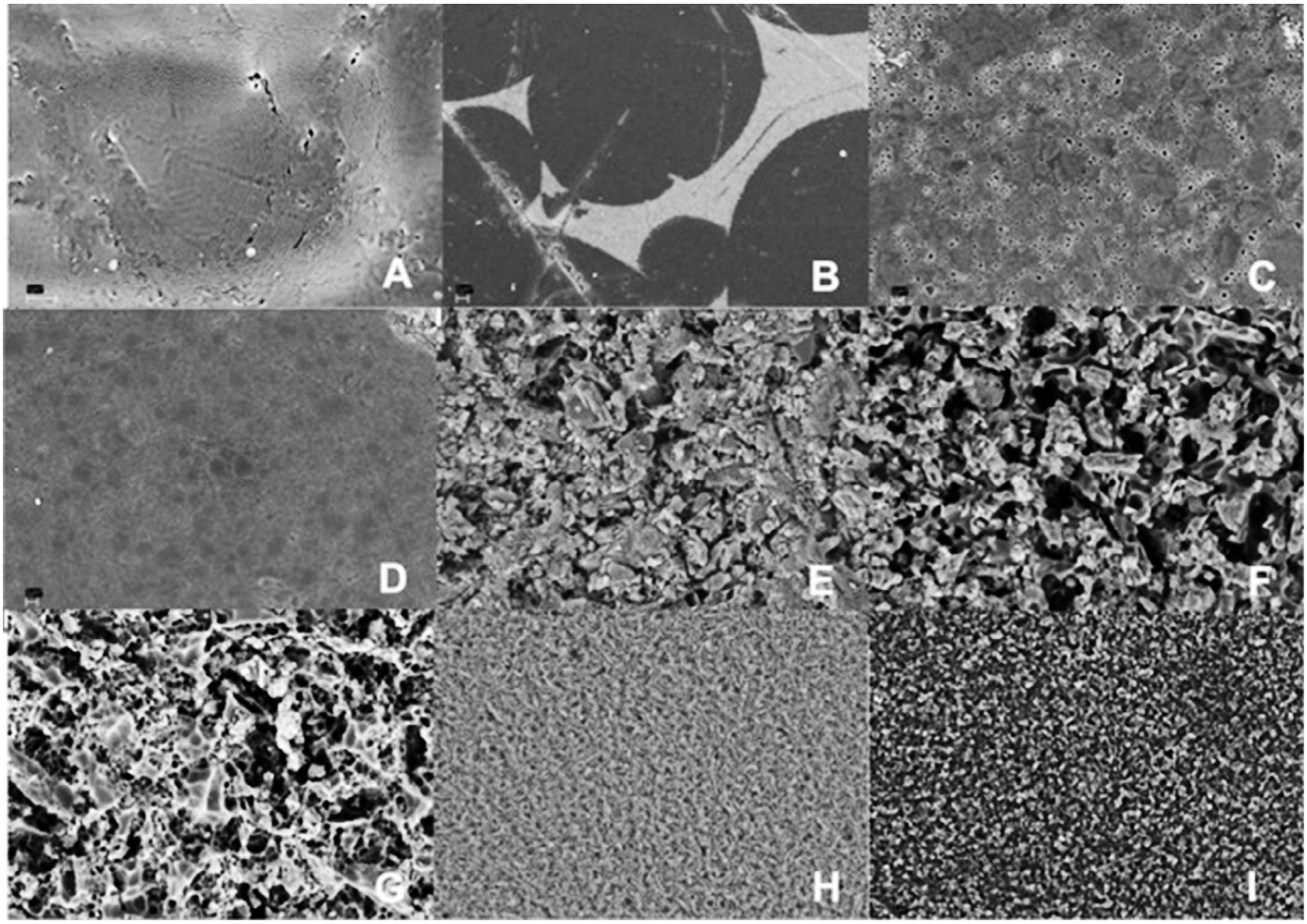

Figure 5. *Scanning electron microscope images of the microstructure of all tested materials: A: TC; B: VCT; C: TEC; D: LU; E: VE; F: VM; G: IEC; H: IEM; I: CD.*

microstructure of the leucite-reinforced ceramic is less dense and characterized by the single crystal formation of leucite ($KAlSi_2O_6$) without interlocking of the crystals.[44,47] Higher-strength ceramics also tend to be less translucent due to the necessary increased crystalline content.[48] Aluminosilicate glass in the lithium disilicate ceramic can result in lower transmitted irradiance values because aluminum compounds cause the ceramic to appear dull and opaque.[49] Feldspathic ceramic (VM) and composites based on tetraethyleneglycol dimethacrylate (TEGDMA) and urethane dimethacrylate (UDMA) (LU) showed the highest transmitted irradiance. In agreement with previous studies,[50,51] VE showed the lowest transmitted irradiance values. VE is a polymer-infiltrated feldspathic ceramic-network material with an 86 wt% ceramic part. The polymer part contains TEGDMA and UDMA monomers. It can thus be assumed that the low transmitted irradiance is related to the density and grain size of the ceramic matrix.

In accordance with previous studies[40,52,53] that investigated the translucency of ceramic materials, this investigation confirmed that material thickness significantly influences the transmitted irradiance. The second null hypothesis was therefore also rejected. In previous studies,[40,52] the glass-ceramic specimens showed a greater decrease in transmitted irradiance compared to zirconia, still in accordance with material thickness, when using standard-power and extra-power curing modes. A lower impact of material thickness on irradiance was observed in exposures to the high-power curing mode. Dental restorations involve various thicknesses, depending on the different conditions of the tooth, and therefore, for use of light-curing cementation, an accurate knowledge of the relationship between irradiance and thickness, depending on the shades, is funda-

mental to improving the long-term stability of ceramic restorations. The present study confirmed this.

Within one type of CAD/CAM material, thickness, or curing mode, no significant difference in transmitted irradiance was recorded until an exposure distance of 2 mm, and this decreased significantly for larger distances (4 mm). This was the result of the particular curing unit used in this study since the variation in irradiance with increasing exposure distance in all three modes showed a slight increase, up to an exposure distance of 2 mm, then decreased exponentially with the distance.[40] For this, the irradiance levels at 0 and 2 mm were comparable. This means that the third null hypothesis is rejected. The special concave glass lens at the tip of the curing unit can explain the impact of the distance between the curing light unit and specimens on the irradiance, where the emitted light is focused to a collimated beam with maximum irradiance at 2 mm. The highest significantly transmitted irradiance was recorded while using the extra-power mode, followed by the high- and standard-power modes. Thus, the fourth hypothesis is also rejected.

In general, it was found that the more translucent a CAD/CAM material, the greater the change in transmitted irradiance as a result of varying thickness. If the microstructure crystals are smaller than the wavelength of visible light (400-700 nm), the glass will look transparent.[49] The material will appear opaque in the case of light scattering and a diffuse reflection.[49] The monolithic CAD/CAM blocks are available in high-translucency (HT) and low-translucency (LT) versions. The LT CAD/CAM materials contain a high number of smaller lithium metasilicate crystals, whereas a small number of crystals are present in the precrystallized state of the HT materials.[46] To the best knowledge of the authors, all materials were ordered in similar A2 colors for the group comparisons; however, materials are offered in different tooth color systems, namely, VITA classic A1-D4 shade guide (classical method) and VITA 3D Master. For this study, the VITA 3D Master colors were translated into the classical colors using VCT and VE. In this study, VE was present in only one tooth color 3M2 (converted A3). Another limitation of this study was that TEC was analyzed in a HT shade, and no information was available for VCT, VE, and VM. It can be assumed that TEC in LT showed comparable transmitted irradiance values to those of the composite LU, but it must be emphasized here that the values obtained provide tendencies for the orientation of the irradiance values.

The transmitted irradiance was evaluated in this study using flat specimens of a standardized thickness. Future investigations should be performed directly on a dental restoration for greater clinical relevance. The influence of the fabrication process of CAD/CAM restorations, such as milling and finishing, could be integrated into these investigations.

Conclusions

Within the limitations of this laboratory investigation, the following conclusions can be drawn:

(1) VITA Mark II and Lava Ultimate showed the highest transmitted irradiance.
(2) The novel interpenetrating network ceramic, followed by the lithium (di)silicate ceramics, showed the lowest transmitted irradiance.
(3) The highest transmitted irradiance was recorded by exposing the material to the plasma mode, followed by the high- and standard-power modes.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Department of Prosthodontics, Dental School, Ludwig-Maximilians University, Munich, Germany.

Conflict of Interest

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

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Effect of Storage Time on Bond Strength Performance of Multimode Adhesives to Indirect Resin Composite and Lithium Disilicate Glass Ceramic

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Clinical Relevance

Multimode adhesives provide reliable bonding to sandblasted indirect resin composites in the long term; however, the use of separate bottles of silane and bonding resin is still recommended for durable bonding to etched glass–based ceramic substrates.

SUMMARY

Purpose: To investigate the bond strength performance of multimode adhesives (MMAs) to indirect resin composite and lithium disilicate glass ceramic after 24 hours or one year of water storage.

Methods and Materials: Thirty flat and polished plates of indirect resin composite (Epricord) and thirty lithium disilicate glass ceramic plates (IPS e.max Press) were prepared. Surfaces were pretreated using sandblasting (indirect resin composite) or hydrofluoric acid (glass-based ceramic). Specimens were bonded with one of two MMAs (Scotchbond Universal [SBU] or All-Bond Universal [ABU]) or ceramic primer and hydrophobic bonding (RelyX Ceramic Primer and

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Adper Scotchbond Multi-Purpose Bond) as a control (n=10). Resin cement cylinders (0.75 mm in diameter × 0.5 mm in height) were bonded to both substrate surfaces using the respective adhesives. After 24 hours or one year of water storage, bonding performance was measured by microshear bond strength (MSBS) testing. Results were analyzed using three-way ANOVA with Bonferroni post hoc tests (α=0.05).

Results: For indirect resin composite, significantly higher MSBS values were found for ABU after 24 hours (ABU > SBU = control); however, no significant difference among the adhesives was observed after one year ($p>0.05$). For glass-based ceramic, significantly different bond strengths were observed among the adhesives after 24 hours (control = ABU > SBU) and one year (control > SBU = ABU; $p<0.05$).

Conclusions: Both MMAs tested can be considered effective alternatives for bonding to sandblasted indirect resin composite after aging, as they showed similar bond performance to that of the control group. However, separate bottles of silane bonding resin showed higher MSBS values and more durable bonding for etched glass–based ceramic.

INTRODUCTION

The demand for esthetic metal-free dental treatments such as inlays, onlays, veneers, and crowns has increased in recent years, which has contributed greatly to the development of adhesive cementing systems for ceramic and indirect resin composite restorations.[1-5] The indirect restorative procedure involves the creation of a machined or laboratory-fabricated restoration, followed by adequate conditioning of the tooth and indirect restoration, and then placement of the restoration with a resin cement.[6] Unlike that with the direct technique, the indirect method can be used to restore the mechanical and biological functions of the tooth with minimal intraoral polymerization shrinkage;[7] this can provide better control of the contact and contour of proximal restorations[8] and therefore enhances the marginal adaptation of the restorative material in the long term.[9]

On the other hand, the bond between indirect restorations and tooth structure can be challenging, since two different interfaces must be considered: that between the dentin–enamel substrate and the resin cement and that between the resin cement and the internal surface of the indirect restoration. For these bonds to form, the tooth substrate and the internal surface of the restoration are conventionally pretreated. During pretreatment of the tooth structure, resin cements can be classified as etch-and-rinse adhesive systems, adhesives containing self-etch primers, or self-adhesive resin cements.[10] Among these categories of resin cements, there is a growing interest in self-adhesive resin cements because of their ease of handling, good esthetics, and suitability for indirect restorations. Such cements combine the properties of adhesiveness and cement into a single step, without the need for additional pretreatment of the tooth. Therefore, compared with conventional resin cements, self-adhesive resin cements are expected to be less technique sensitive during the luting procedure. In addition, studies have shown that self-adhesive resin cements can provide improved sealing ability[11] with durable bonding performance on smear-covered dentin.[12]

Besides significant improvements achieved in the attachment of adhesive cementing systems to tooth structures, bonding between the indirect restoration substrate and the resin cement has also been the focus of several studies.[13,14] Because of the differences in the composition of indirect restorative materials, different internal surface pretreatments seem crucial for their intraoral retention.[15,16] Pressable lithium disilicate–based glass is a ceramic composed of one glassy phase and at least one crystallized phase. Ceramic restorations involve a high degree of crystallization that enhances their mechanical properties.[17] Meanwhile, indirect resin composite restorations are subjected to secondary curing with light and/or heat to increase the resin conversion that enhances their wear resistance; however, this lessens the potential for chemical bonding, as the number of residual-free carbon double bonds is decreased.[16] Conventionally, for reliable adhesion to indirect restorations, the internal surface of the restoration is roughened. As a result, the surface area for bonding and wettability of the adhesive–cement to the restoration is increased; this allows chemical bonds to form between the ceramic or fillers and the cement.[10] Among the surface pretreatments used for indirect restorations, successful bonding to glass-based ceramic surfaces can be achieved by dissolving their glassy phase with hydrofluoric acid (HFA),[17,18] while sandblasting the surface of an indirect resin composite is recommended.[19,20] Both of these methods provide better mechanical and chemical interaction during silane

application prior to the luting agent.[15,21] A silane coupling agent is used conventionally as an adhesion promoter for silica-based materials and forms chemical bonds with the inorganic phase of the indirect restoration and the organic phase of the resin cement.[21,22]

Universal or multimode adhesives (MMAs) are the newest category of simplified one-bottle adhesives and are considered applicable to different substrates such as enamel, dentin, alloys, zirconia, ceramics, and composites. They have been marketed with an indication that they provide a chemical bond between indirect restoration substrates and resin cements without the need for primers or activators. This simplified strategy might be due to the presence of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer in their composition, which can promote adhesion to surfaces based on calcium,[23] metal,[24] or zirconia.[25,26] In addition, MMAs can also contain silane in the same bottle, which is expected to improve their bonding to silica-based ceramic.

The dihydrogen phosphate group from the MDP monomer is responsible for priming and bonding, while its long carboxyl chain provides the hydrophobic properties and hydrolytic stability of acidic monomers. For enamel and dentin substrates, MDP forms a strong ionic bond with calcium from hydroxyapatite that forms calcium salts with low solubility, which may be responsible for the good long-term performance of MDP-containing adhesives.[23,27,28] For non–silica-based substrates, such as metal or zirconia, the hydrophilic phosphate terminal end of MDP interacts chemically with the oxides on the internal surface of restorations, while the hydrophobic methacrylate terminal end copolymerizes the resin monomers of the cement.[29] However, for silica-based indirect restorations such as feldspathic porcelain, leucite-reinforced ceramic, or lithium disilicate glass ceramic, the reaction between silane and MDP promotes the bonding mechanism, improving surface wettability. The free silanol groups form hydrogen bonds with the hydroxyl groups of the indirect restoration. Then, cross-linkages are formed between the methacrylate groups of the cement with organofunctional groups from the silane coupling agent, as well as between the siloxane bonds and the restoration substrate.[22] Because of the versatility of the substrate application, MMAs may also be suitable for intraoral restoration repairs, since they could be a practical alternative to bonding different fractured substrates at the same time.[30] Nevertheless, few studies have investigated the long-term durability of this new category of one-bottle adhesive[31] or their application with different indirect restoration substrates.[32,33]

Therefore, the aim of this study was to determine the bonding effectiveness of two different MMAs with their respective resin cements to indirect resin composite and lithium disilicate glass ceramic substrates after 24 hours or one year of water storage. In addition, scanning electron microscopy (SEM) was used to evaluate fracture mode patterns. The null hypothesis tested was that there would be no significant difference in bond strength among the materials assessed after 24 hours or one year of storage for both substrates.

METHODS AND MATERIALS

A total of 60 standardized rectangular plates (15-mm wide × 6-mm long × 1.5-mm thick) were obtained for this study. Thirty of the plates were fabricated from indirect resin composite (Epricord Dentin A2, Kuraray Noritake Dental, Tokyo, Japan), while the other thirty were fabricated from lithium disilicate–based glass ceramic (IPS e.max Press MO-0 ingots, Ivoclar Vivadent AG, Schaan, Liechtenstein) by using the lost wax and hot press technique, according to the manufacturers' instructions.

For the indirect resin composite, the rectangular plates were prepared by a single increment of Epricord into a silicone mold, which was covered by a thin transparent film (KerrHawe Striproll, KerrHawe, Bioggio, Switzerland), followed by a glass slab, and then light activated using a halogen light-curing unit (intensity = 600 mW/cm^2; Optilux 501, Kerr, Orange, CA, USA) for 40 seconds. The specimens were removed from the mold, and the remaining surfaces were light cured for an additional 40 seconds each.

For the lithium disilicate–based glass ceramic, rectangular wax patterns were fabricated, sprued, and attached to a muffle base with a surrounding paper cylinder. The wax patterns were invested with phosphate-based material (IPS PressVest Speed, Ivoclar Vivadent AG), and the wax was eliminated in an automatic furnace (Vulcan A-550, Degussa-Ney, Yucaipa, CA, USA) at 850°C for one hour. The IPS e.max Press ingots were then pressed into the molds in an automatic press furnace (EP 600, Ivoclar Vivadent AG).

After cooling, both substrate surfaces were wet ground with 400-, 600-, and 800-grit silicon carbide papers (Norton, Vinhedo, SP, Brazil) and ultrasonically cleaned in a water bath for five minutes to remove the remaining debris and air-dried. The

Table 1: *Materials Used in This Study*

Material (Batch Number)	Composition	Application Technique
RelyX Ceramic Primer (N351206); 3M ESPE, St Paul, MN, USA	Ethyl alcohol, water, methacryloxypropyltrimethoxysilane	Apply RelyX Ceramic Primer. Air dry for 5 s.
Adper Scotchbond Multi-Purpose Bond (N205453); 3M ESPE, St Paul, MN, USA	Bond: Bis-GMA, HEMA, triphenylantimony	Apply adhesive for 10 s. Gentle air blow for 5 s. Light cure for 10 s.
RelyX ARC (N339863); 3M ESPE, St Paul, MN, USA	Silane-treated ceramic, TEGDMA, Bis-GMA, silane-treated silica, reacted polycaprolactone polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide	Dispense the cement onto the mixing pad and mix for 10 s. Insert the resin cement into the Tygon tube. Light cure for 40 s.
Scotchbond Universal (472387); 3M ESPE, St. Paul, MN, USA	Adhesive: MDP, Bis-GMA, phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane-treated silica	Apply adhesive for 20 s. Gentle air blow for 5 s.
RelyX Ultimate (467302); 3M ESPE, St. Paul, MN, USA	Silane-treated glass powder, 2-propenoic acid, 2-methyl-1,1′-[1-(hydroxymethyl)-1,2-ethanediyl]ester, reaction products with 2-hydroxy-1,3-propanediyl dimethacrylate and phosphorus oxide, TEGDMA, silane-treated silica, oxide glass chemicals, sodium persulfate, tert-butyl peroxy-3,5,5-trimethylhexanoate, acetate monohydrate	Dispense the cement onto the mixing pad using an intraoral tip. Insert the resin cement into the Tygon tube. Light cure for 40 s.
All-Bond Universal (1200003968); BISCO, Schaumburg, IL, USA	Adhesive: MDP, Bis-GMA, HEMA, ethanol, water, initiators	Apply adhesive. Light cure for 10 s.
Duo-Link (1200006424); BISCO, Schaumburg, IL, USA	Bis-GMA, triethyleneglycol dimethacrylate, urethane dimethacrylate, glass-filler	Dispense the cement onto the mixing pad using an intraoral tip. Insert the resin cement into the Tygon tube. Light cure for 40 s.

Abbreviations: Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; HEMA, hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate.

indirect resin composite specimens were sandblasted with 50-μm aluminum oxide particles (Danville Engineering Inc, San Ramon, CA, USA) for 10 seconds (air pressure, 0.552 MPa; distance from the tip, 1.5 cm). All the resin plates were then subjected to further ultrasonic cleaning in a water bath for another five minutes and were air-dried. On the other hand, the glass-based ceramic specimens were etched with 10% HFA gel (Dentsply Caulk, Milford, DE, USA) for 15 seconds, rinsed with water for 15 seconds, and air-dried.

After surface pretreatment, the plates of each substrate were randomly assigned to three groups according to the bonding materials used (n=10 per adhesive/substrate): one of two one-step MMAs with their respective adhesive resin cements (Scotchbond Universal [SBU] with RelyX Ultimate [3M ESPE, St Paul, MN, USA] or All-Bond Universal [ABU] with Duo-Link [Bisco Inc, Schaumburg, IL, USA]) or a control ceramic primer with hydrophobic bonding resin and an adhesive resin cement (RelyX Ceramic Primer and Adper Scotchbond Multi-Purpose Bond with RelyX ARC [3M ESPE]). The composition of the materials and the application techniques used in this study are included in Table 1.

For microshear bond strength (MSBS) testing, hollow cylinders of 0.5-mm height were cut from micro-bore Tygon tubing (internal diameter, 0.75 mm; Norton Performance Plastics, Akron, OH, USA) and were used as molds for the resin cement luting procedure. The plates were randomly assigned to receive an application of each MMA or control ceramic primer with hydrophobic bonding resin onto the substrate surfaces. Without prior light irradiation, four Tygon tubes were then placed at four locations on the surface of each plate forming a centered straight line, at approximately 3.0 mm apart from the center of each Tygon tube. The resin cements were mixed according to the manufacturers' instructions and carefully inserted into the tubing on their respective adhesive and substrate surfaces. Small resin cement cylinders (approximately 0.75 mm in diameter × 0.5 mm in height) were obtained after polymerization using a halogen light-curing unit (intensity, 600 mW/cm^2; Optilux 501, Kerr) for 40 seconds.

After 24 hours of water storage at 37°C, the Tygon tubing was removed carefully with a thin steel

Table 2: *Mean (SD) Microshear Bond Strength of Adhesive Systems to Indirect Resin Composite and Lithium Disilicate Glass Ceramic (in MPa)*[a]

Material	Indirect Resin Composite Substrate		Lithium Disilicate Glass Ceramic Substrate	
	After 24 h Water Storage	After 1 y Water Storage	After 24 h Water Storage	After 1 y Water Storage
RelyX Ceramic Primer and Adper Scotchbond Multi-Purpose Bond (control)	25.5 (7.9)^{A,a*}	22.7 (2.7)^{A,a**}	35.3 (8.5)^{A,b*}	31.2 (5.9)^{A,b**}
Scotchbond Universal (SBU)	26.6 (5.6)^{A,a}	20.2 (3.1)^{B,a}	23.9 (6.1)^{A,a}	21.3 (5.6)^{A,a}
All-Bond Universal (ABU)	32.7 (3.3)^{A,b}	25.1 (2.0)^{B,a**}	31.5 (7.0)^{A,b}	16.9 (4.4)^{B,a**}

[a] *Data are presented as the mean (standard deviation) in MPa (n=10). Identical capital letters in a row within the same substrate after 24 hours and one year of water storage indicate the absence of any statistically significant difference. Identical lowercase letters in a column indicate the absence of any statistically significant difference. Comparisons within the same material and storage period between different substrates and marked with one asterisk for 24 hours and two asterisks for one year water storage are statistically significant. (Analysis of variance and Bonferroni post hoc test; significance at* $p<0.05$.)

cutting blade to expose the resin cement cylinder. The four resin cement cylinders obtained from each plate were further divided into two subgroups, with two cylinders being tested after a further 24 hours of water storage and the other two being tested after one year of water storage. The average MSBS value obtained from two cylinders on the same plate was considered the mean value of one sample for each storage period (n=10 per adhesive cementing system/storage period). Each plate was fixed with cyanoacrylate glue (Model Repair II Blue; Sankin Industry Co, Tokyo, Japan) to a jig in a universal testing machine (EZ Test, Shimadzu, Kyoto, Japan) in such a way that the straight line formed by the cement cylinders was perpendicular to the force. A thin wire (0.2 mm in diameter) was looped around the cement cylinder, making contact with half of its circumference, and held gently against the cement–indirect restoration substrate interface. A shear force was applied to each specimen at a cross-head speed of 1 mm/min until failure occurred. Tweezers were used to position the wire at the boundary of the cement and the indirect restoration substrates. The resin cement–indirect resin composite interface or the resin cement–lithium disilicate glass ceramic interface, the wire loop, and the center of the load cell were aligned as straight as possible to ensure the desired orientation of the shear test force.

After MSBS testing, the fractured surfaces were mounted onto brass stubs, gold coated, and observed under SEM (JSM5600, JEOL Ltd, Tokyo, Japan). The failure mode pattern of all specimens submitted to MSBS testing was evaluated using SEM micrographs at a magnification of 1000×. For the indirect resin composite and lithium disilicate glass ceramic specimens, the failure mode was determined and classified as follows: prefailure, cohesive failure in resin cement, mixed failure of adhesive and resin cement, failure between adhesive and indirect resin composite or lithium disilicate glass ceramic, or cohesive failure in indirect resin composite or lithium disilicate glass ceramic.

The MSBS data were statistically analyzed using three-way analysis of variance (ANOVA) with the significance level defined as $\alpha=0.05$; bond strengths to indirect resin composite or glass-based ceramic were used as dependent variables, and the adhesive cementing system, storage period, and substrate were used as factors. Bonferroni post hoc tests with UNIANOVA syntax were used for multiple comparisons of significant differences in bond strength means. All statistical analyses were performed using the Statistical Package for Social Sciences (SPSS for Windows, version 16.0, SPSS, Chicago, IL, USA).

RESULTS

The means and standard deviations of the MSBS values obtained in this study are presented in Table 2. Three-way ANOVA showed that bond strength values for indirect resin composite and lithium disilicate glass ceramic substrates were significantly influenced by the adhesive cementing system used ($p<0.001$) and by the storage period ($p<0.001$) but not by the substrate ($p=0.227$). The interactions of these three factors were not significant ($p=0.104$). On the other hand, significant statistical interaction was observed between the adhesive cementing system and the storage period ($p=0.005$) and between the adhesive cementing system and the substrate ($p<0.001$). Significant statistical interaction was not found between the storage period and the substrate ($p=0.458$).

Bonferroni post hoc tests revealed statistically significant differences between the bond strength results after 24 hours or one year of water storage for both substrates. For indirect resin composite, the ABU group displayed higher MSBS values than the

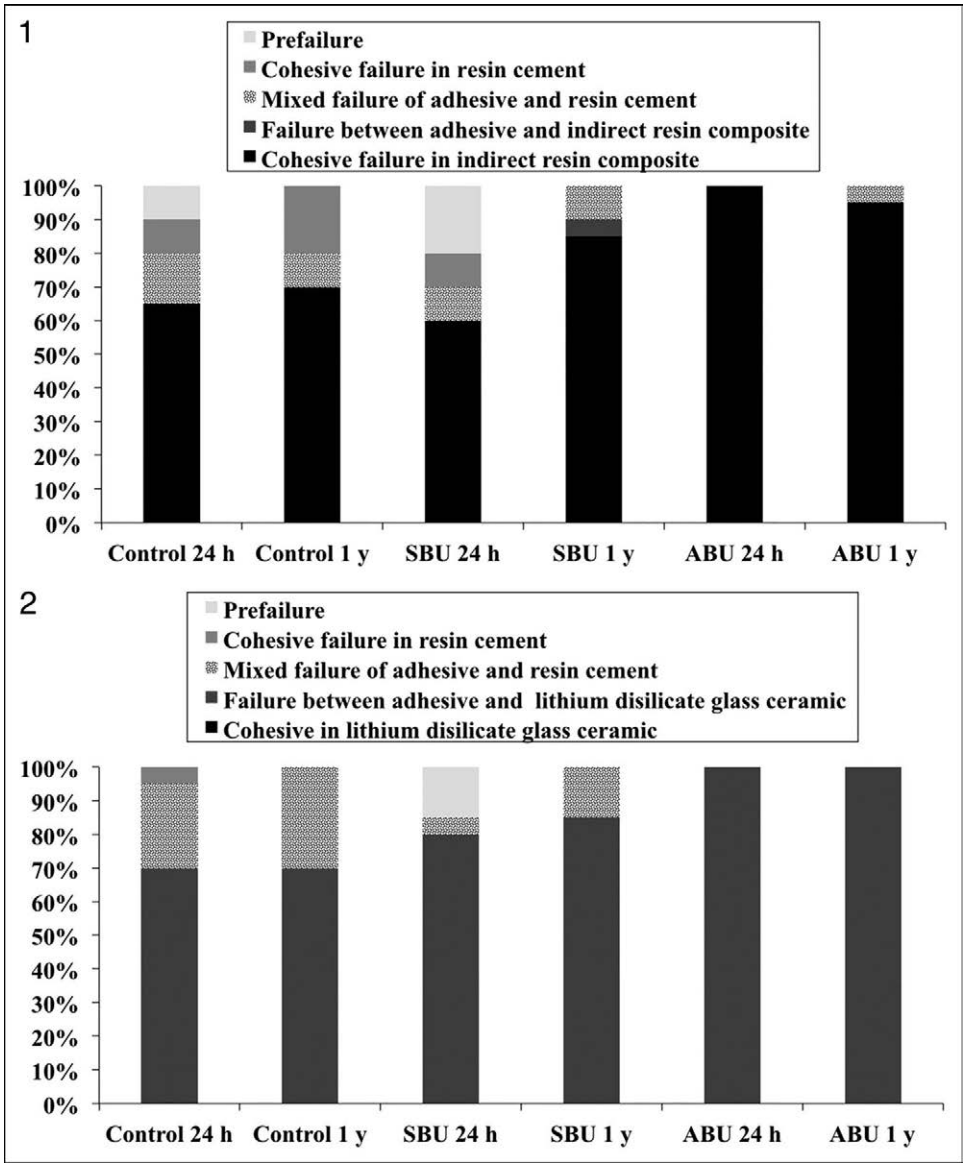

Figure 1. *Distribution (%) of failure modes after 24 hours (24 h) and one year (1 y) of water storage for the indirect resin composite substrate.*

Figure 2. *Distribution (%) of failure modes after 24 hours (24 h) and one year (1 y) of water storage for the lithium disilicate glass ceramic substrate.*

SBU and control groups after 24 hours. After one year of storage, no statistically significant differences were found among all of the materials used for this substrate, while there was a significant decrease in bond strength within the ABU and SBU groups compared with baseline ($p<0.05$; Table 2).

On the other hand, for lithium disilicate glass ceramic after 24 hours, the control group showed significantly higher MSBS values than the SBU group but did not differ from the ABU group. After long-term storage, the control group showed higher MSBS values, but no statistically significant difference was found in MSBS values between the ABU and SBU groups. However, a significant decrease in bond strength was observed within the ABU group compared with the baseline value (Table 2).

Within the control group, there was a significantly higher MSBS to lithium disilicate glass ceramic as compared with indirect resin composite after 24 hours or one year of storage. On the other hand, no statistically significant difference was observed for SBU after the same storage period, regardless of the substrate used. For ABU, bonding to the indirect resin composite substrate resulted in higher bond strength values than bonding to lithium disilicate glass ceramic after one year only.

The distribution of the modes of failure is summarized in Figure 1 for indirect resin composite substrate and Figure 2 for lithium disilicate glass ceramic substrate. Representative high-magnification SEM micrographs of mode patterns are presented in Figure 3. Cohesive failure was mainly

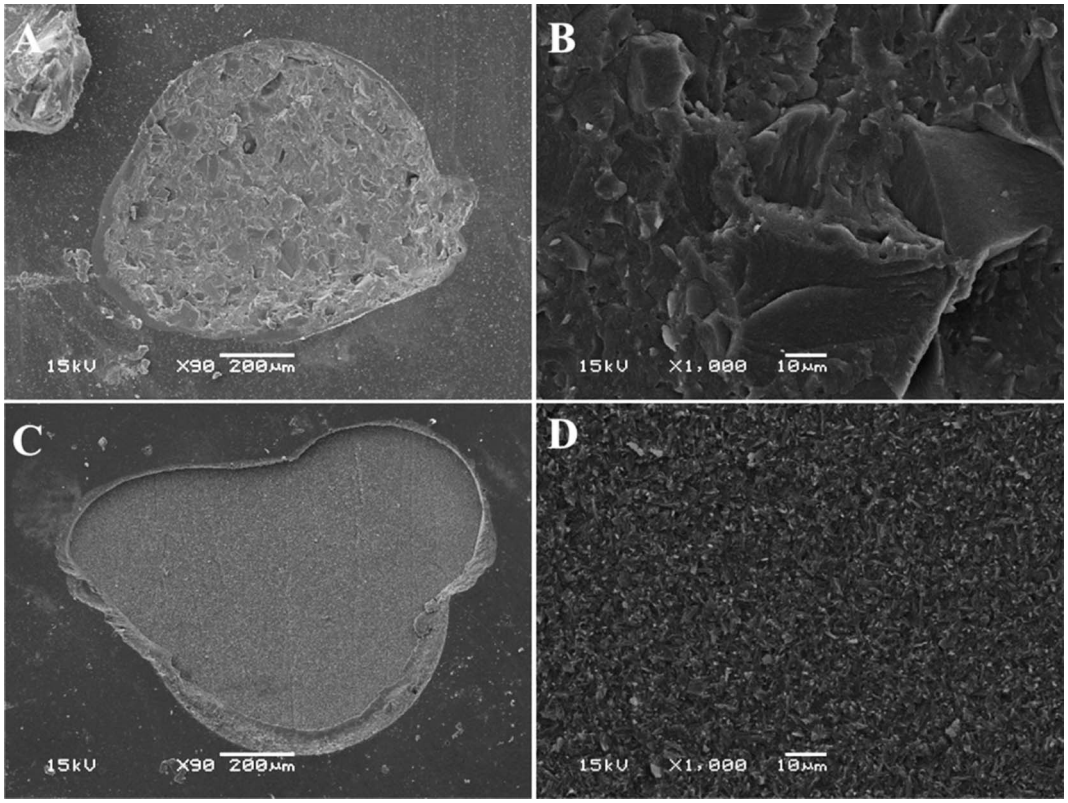

Figure 3. *Scanning electron micrographs of representative main fracture patterns of the indirect resin composite substrate (A and B) and the lithium disilicate glass ceramic substrate (C and D). (A and C): 90× magnification; (B and D): 1000× magnification. (A) and (B) show images of cohesive failure in the indirect resin composite. (C) and (D) show images of failure between the adhesive and lithium disilicate glass ceramic.*

observed in indirect resin composite for all of the materials applied (Figure 3A,B) for both storage times. For lithium disilicate glass ceramic, failure in the adhesive was predominant for both periods of storage (Figure 3C,D).

DISCUSSION

The present study evaluated the effect of storage time on the bonding effectiveness of two MMAs to indirect resin composite and lithium disilicate glass ceramic and compared their bonding performance to a conventional method of two separate bottles of silane agent and hydrophobic bonding resin. The results obtained after 24 hours indicated that, among the materials tested, ABU provided the highest bond strength values to indirect resin composite and showed similar bond performance to the control group for the lithium disilicate glass ceramic. However, the storage time significantly affected the bond performance of both MMAs to indirect resin composite and also significantly decreased the bond strength, particularly for ABU, which was bonded to the lithium disilicate glass ceramic. Since there was a significant difference in bond strength among the materials tested after 24 hours or one year of storage for indirect resin composite and lithium disilicate glass ceramic substrates, the null hypothesis of the current study was rejected.

In this study, the MSBS test was successfully performed for all specimens, and bonding performance was compared among the adhesive systems with two indirect restorative materials. The MSBS test was chosen because of the advantages of bonding tests with small and round bonded areas as well as the ease of sample preparation,[14] since it does not require cutting procedures such as those used for sample preparation in the microtensile bond strength test.[34]

In the current study, sandblasting with aluminum oxide particles was used as a surface pretreatment for indirect resin composite, and HFA etching was used for lithium disilicate glass ceramic. Studies have reported that sandblasting indirect resin composite surfaces can produce higher bond strength compared with acid etching by producing extensive surface roughening.[35,36] On the other hand, conditioning of lithium disilicate glass ceramic substrates

using HFA has been shown to be the most effective pretreatment method to increase bond strength performance, via dissolution of the glassy matrix, roughening of the surface by exposure of crystals, and subsequent enhancement of the potential for micromechanical retention.[14,15,17,18] Thus, the successful creation of micropores for wetting and infiltration of silane monomers, bonding resins, and resin cements was expected because of the application of these pretreatments to different substrates used in the present study.

RelyX Ceramic Primer and Adper Scotchbond Multi-Purpose Bond were used in the control group in the current study. This method uses two separate bottles of silane agent and hydrophobic bonding resin. RelyX Ceramic Primer contains 3-methacryloxypropyltrimethoxysilane diluted in an ethanol-water solution, while Adper Scotchbond Multi-Purpose Bond contains bisphenol-A diglycidyl ether dimethacrylate (Bis-GMA) as a cross-linker and hydroxyethyl methacrylate (HEMA), which has solvent-like properties.[37] The silane molecules from the RelyX Ceramic Primer react with water, forming three silanol groups from the corresponding methoxy groups. These silanol groups form a siloxane network with the silica surface and make covalent bridges with the hydroxyl groups on inorganic substrate surfaces.[20,22] Later, the monomeric ends of the silane molecules on the pretreated surfaces may react with the methacrylate groups of the bonding agent.[38] In this way, a linkage between the substrate, the silane agent, and the hydrophobic bonding resin can be formed.

Silane application is very effective in promoting adhesion and may even be crucial for durable bonding, particularly with silica-based materials.[15,22] Moreover, the subsequent application of a bonding resin as an intermediate agent facilitates the penetration of resin monomers and the resin cement into the irregularities formed by the acid etching/sandblasting pretreatment; this results in micromechanical interlocking[39] and ultimately enhances bond strength.[40] This could explain the optimal results observed for the control group that had similar bond strength values after 24 hours and one year of storage for each substrate. Interestingly, only the control group had significantly higher MSBS values for specimens bonded to glass-based ceramic than those of the indirect resin composite substrate, regardless of the storage period. The use of HFA etching as a surface pretreatment to glass-based ceramic substrate creates a honeycomb-like structure that provides additional micromechanical retention,[15,41] and it could explain the higher MSBS values for specimens bonded to the glass-based ceramic. Apart from that, the control group also demonstrated significantly higher bond strength compared with ABU and SBU for lithium disilicate glass ceramic substrate after one year. The micromechanical retention created by the HFA etching pretreatment combined with the use of a separate bottle of silane agent might have enhanced the bond strength for lithium disilicate glass ceramic substrate in the present study and could explain the good results obtained for the glass-based ceramic in the control group for both storage periods.

SBU contains silane, HEMA, MDP, and Bis-GMA combined into a one-bottle solution. MDP-containing adhesives have been shown to provide a reliable bond to indirect restorative materials[42,43] and tooth substrates.[23] Thus, the presence of silane and MDP monomer was expected to contribute greatly to the bonding mechanism, by improving the wettability and forming cross-linkages with methacrylate groups.[43] However, in the current study, SBU showed similar MSBS values to separate bottles of silane and bonding resin for indirect resin composite substrate for both storage periods. Silane might be unstable when combined with MDP and Bis-GMA resins in a one-bottle solution.[22] Under acidic conditions, such as that in the presence of MDP and water, a self-condensation reaction might occur in the silanol groups of silane.[22] On the other hand, the presence of MDP and the retention created by air abrasion in the indirect resin composite substrate may have promoted chemical and micromechanical attachment, as SBU showed similar bond strength values to the control group for this substrate for both storage periods. Thus, the most common failure mode observed for this bonding material was cohesive within the indirect resin composite. Nevertheless, within the SBU group only, a significant decrease in MSBS was observed after one year of water storage for the indirect resin composite. A recent study reported that air abrasion may influence the durability of the SBU bond, and water could be absorbed into the primed layer as a consequence of aging and could decrease the bonding ability gained by micromechanical retention when air abrasion is performed.[44] Although that study included only the surface pretreatment method and its results were based on SBU bonded to air-abraded zirconia ceramic specimens,[44] we believe that a similar phenomenon may also have occurred in the indirect resin composite specimens in the current study. This could explain the decrease in MSBS

values within the SBU group for this substrate after the long-term storage.

On the other hand, in the present study, for SBU specimens bonded to the lithium disilicate glass ceramic substrate, HFA etching was used prior to SBU application. A recent study showed that HFA etching pretreatment of this substrate is beneficial for SBU bond performance; however, when SBU is used with or without additional silane application on lithium disilicate, a significantly higher bond strength is observed when silane is preapplied.[45] Therefore, this could reinforce the speculation that the silane included in one-bottle universal adhesive might not be fully effective for enhancing the chemical bonding itself. Moreover, incorporating Bis-GMA with the silane may also interfere in the silane-coupling condensation reaction with the hydroxyl groups of silica-based ceramic.[46] This could explain the lower MSBS values observed in the current study for SBU when compared with the control group for lithium disilicate glass ceramic for both storage periods. Nevertheless, the predominant fracture mode observed for this substrate was failure between the adhesive and lithium disilicate glass ceramic substrate, for 24 hours and one year of water storage, which indicates poor adhesion quality. In addition, no statistically significant differences were observed for SBU when both substrates were compared within the same storage period, suggesting that a similar bonding ability can be achieved regardless of the substrate or the surface pretreatment used.

ABU contains HEMA, MDP, and Bis-GMA. While MDP may improve bonding effectiveness to resin-based materials,[47] Bis-GMA has been reported to provide mechanical strength to adhesives by forming densely cross-linked polymers, lower polymerization shrinkage, and rapid hardening.[37] The combination of MDP and Bis-GMA could explain the higher initial MSBS values observed for ABU on the indirect resin composite substrate as compared with the control and SBU groups in the present study. However, water sorption can occur in HEMA-containing adhesives after long-term storage in water, causing hydrolytic degradation of the polymer, followed by elution of degradation products that results in a decrease of bond performance.[48] This is in agreement with the results obtained in the present study, as a statistically significant decrease in MSBS values was observed for ABU after one year of storage when compared with baseline for both substrates. However, these MSBS values did not differ from those of the SBU and control groups on indirect resin composite after the long-term storage. In addition, all the materials used in the current study contained Bis-GMA in their formulation. Because of its high molecular weight, uncured Bis-GMA is highly viscous. This property might also have contributed to producing higher mechanical strength[49] and resulted in similar MSBS values for all of the materials on the indirect resin composite substrate. Moreover, the most common failure mode for both storage periods for ABU on the indirect resin composite substrate was cohesive failure in the indirect resin composite, similarly to the SBU and control groups.

For the lithium disilicate glass ceramic, ABU showed a similarly high level of bond strength to the control group after 24 hours of storage. However, a significant decrease in MSBS bond strength values was observed within the ABU group for glass-based ceramic substrate after long-term storage. Interestingly, for ABU only, the predominant failure mode was between the adhesive and the lithium disilicate glass ceramic for both storage times, suggesting that this interface was the weakest link of the bond. Although a significant decrease in bond strength was observed for ABU for both substrates after one year of storage, MSBS values were significantly lower for the lithium disilicate glass ceramic when compared with the indirect resin composite substrate. This might suggest that the total absence of silane in the ABU group compromised the long-term performance of this material, as compared with its high bond strength at baseline. This is true for both substrates, although it was more pronounced for the lithium disilicate glass ceramic.

Within the limitations of this study, after one year of water storage, MMAs showed comparable bond strength values to the control group for indirect resin composite; however, the use of separate bottles of silane and bonding resin in the control group resulted in durable bonding to the lithium disilicate glass ceramic. The use of a separate silane primer prior to the application of MMAs could be an alternative to improve their chemical bonding to lithium disilicate glass ceramic, as previously reported,[45] as well as to indirect resin composites. Ideally, an intermediate coupling agent should provide enhanced hydrolytic stability[50] and the capability to form strong bonds with different substrates at the same time. Therefore, further investigations are still required for surface pretreatment protocols and long-term interactions of this promising new category of adhesives for bonding to different restorative materials.

CONCLUSIONS

MMAs can provide good bonding performance to sandblasted indirect resin composite after one year of water storage. However, the use of separate bottles of silane and bonding resin resulted in superior bond strength for etched glass–based ceramic substrate after long-term storage.

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Conflict of Interest

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

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Microleakage of Lithium Disilicate Crown Margins Finished on Direct Restorative Materials

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Clinical Relevance

Finishing lithium disilicate all-ceramic crowns on flowable resin composite materials in the esthetic zone should be used with caution. If necessary, finishing lithium disilicate all-ceramic crowns on nanofilled resin composite or resin-modified glass ionomer materials seems to provide the least amount of dye penetration.

SUMMARY

Objective: For some esthetic clinical situations, it is necessary to finish crown margins on direct restorative materials to preserve tissue integrity, bonding integrity, and biological width. The purpose of this research was to investigate microleakage at the interface between bonded lithium disilicate crowns and various direct restorative materials in a class III and class V position.

Methods and Materials: Class III or class V restorations were prepared on one side of extracted incisors with either Tetric EvoCeram, Tetric Evoceram Bulk, Fuji II LC, or Tetric Evoflow. The teeth were prepared for and received a lithium disilicate crown. After load fatiguing, the specimens were thermo-cycled with a fuchsin dye and sectioned. The depth and area of dye penetration were measured with a dimensional grid in micrometers using stereomicroscopy and reported as mean dye depth and area (μm) ± SD. The comparison of multiple categorical independent variables with ratio scale dependent variables was evaluated with an analysis of variance and Tukey's post hoc analysis.

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Results: A statistically significant higher dye penetration was noted for all treatment groups compared with the positive control (side opposite the restoration after sagittal sectioning was used as positive control) regardless of material or placement area ($p<0.05$). In comparing treatment groups, the Tetric EvoFlow experienced a statistically higher dye penetration than did the other treatment groups regardless of material or placement area ($p<0.05$). There was no statistically significant difference between the Tetric EvoCeram, Tetric Evoceram Bulk, and Fuji II LC materials regardless of placement area ($p>0.05$).

Conclusions: Within the limitations of this study, it can be concluded that flowable composite materials as finish lines that interact with resin cements could lead to exacerbated interfacial degradation. Finishing lithium disilicate all-ceramic crowns on flowable resin composite materials in the esthetic zone should be used with caution. If necessary, finishing lithium disilicate all-ceramic crowns on nanofilled resin composite or resin-modified glass ionomer materials seems to provide the least dye penetration depth and area.

INTRODUCTION

In some instances, the clinical situation dictates the necessity to finish crown margins on direct restorative materials in the esthetic zone. Typically class III and V restorations exist that may be above, at, or below the cemento-enamel junction (CEJ). To crown lengthen below existing restorations may compromise tissue integrity, bonding integrity, and biological width on highly esthetic cases. The current published literature does not contain any information for clinicians to provide evidenced-based decisions as to which direct restorative materials would function best in the aforementioned situation, if any. A recent clinical case report in the current literature described the treatment of a maxillary central incisor with class III invasive cervical resorption and a compromised ferrule with resin-modified glass ionomer (RMGI) and a full-coverage zirconia crown.[1] Optimal bond strength is the goal for long-term clinical success of all ceramic restorations. Optimizing bond strength to ceramic, enamel, and direct restorative materials will minimize microleakage associated with water sorption, dentin hypersensitivity, marginal staining, and caries formation.

Resin composite adhesive bonding is well documented within the current literature. Adhesive bonding of resin composite to enamel is very predictable and produces the highest bond strength using the total etch technique.[2] The high mineral content and low water content of enamel allows optimal adhesive bonding while reducing microleakage.[2] Adhesive bonding of a resin composite to dentin and cementum is less predictable due to the higher water content and increased technique sensitivity.[3] The current literature shows that dentin bonding is lower than enamel bonding due to the need for a hybrid layer within the dentinal collagen.[3] Clinical requirements to maintain a moist dentin while preventing water dilution of the adhesive material are difficult at best.[3] The hydrophilic portion of the primer needs to allow the formation of the hybrid layer within the dentinal collagen and tubules to minimize microleakage and sensitivity. The current literature suggests that long-term hydrolysis and proteolysis of the adhesive bond occurs naturally by innate defense mechanisms.[4,5] Additionally, conditioning and bonding to the existing resin composite has been published in the literature with varying results.[6,7] The consensus seems to be a decrease in bond strength across the repaired area in vitro.[6,7] A decrease in bond strength could lead to increased microleakage and a compromised long-term clinical survival of that restoration.[6,7]

RMGI restorative materials are also well documented in the current literature. These materials are advantageous in that they do not require adhesive bonding or mechanical retention within the preparation design.[8] The formulation of a "true" glass ionomer must have water, ion leachable glass, and polyacrylic acid for the acid-base reaction to occur.[8] The additional polyacrylic acid conditioner used as a separate step with this material allows chemical bonding to enamel, dentin, and cementum.[9] The acid-base setting reaction of the RMGI chemically bonds to calcium on the hydroxyapatite crystals and releases fluoride over time.[9] The resin monomer incorporation into the glass ionomer material provides improved esthetics and command light cure.[9] The final set glass can be conditioned with phosphoric acid and adhesively bonded also, as performed in the class II sandwich technique.[10] The current literature suggests that conditioning and adhesively bonding resin composite to a RMGI material yields a clinically acceptable interface.[8-10]

Flowable nano-hybrid resin composite materials have a lower filler content compared with traditional resin composites to improve fluid contact and reduce surface tension on tooth structures, especially at internal line angles.[11] Flowable bulk fill resin

composites have been introduced recently to alleviate the need for small incremental placement. These restorative materials have been shown *in vitro* to experience dye microleakage around class II restorations.[12] There are no studies specifically evaluating the bonding of resin cement to either flowable or bulk fill resin composite materials. Therefore, through association, conditioning and bonding to existing resin composite could lead to a decrease in bond strength across the repaired area *in vitro* as with the nano-filled resin composite.[6,7] A decrease in bond strength could lead to increased microleakage and a compromised long-term clinical survival of that restoration.[6,7]

According to Wolfart,[13] there was no difference in clinical performance of bonded versus cemented lithium disilicate crowns after eight years. There are reported advantages to using a bonded technique versus a cemented technique. Simon and others[14] measured the tensile bond strength of the self-adhesive resin cements and a bonded resin cement for crowns bonded to extracted teeth with preparations having a total taper greater than 30°. It was concluded that some of the new self-etching resin cements can create bonds to nonretentive crown preparations that are stronger than the strength of a ceramic crown. Rojpaibool and Leevailoj[15] investigated the influence of cement film thickness, cement type, and substrate (enamel or dentin) on ceramic compressive fracture resistance. It was concluded that higher fracture loads were related to thinner cement film thickness and RelyX Ultimate resin cement (3M Corporation, St. Paul, MN). Bonding to dentin resulted in lower fracture loads than bonding to enamel.

Dye penetration for marginal sealing has been used for years as an acceptable surrogate to understand fluid flow and marginal integrity of the composites' cohesive and adhesive natures *in vitro*.[16-21] Therefore, the purpose of this research was to investigate the effects of interfacial microleakage when finishing pressed lithium disilicate ceramic crowns on various direct restorative materials in class III and class V positions. Of particular interest was the interface created between the resin cement and various direct restorative materials. The research questions for this evaluation were as follows:

1) Is there a difference in microleakage group means, measured as infiltration depth and area in micrometers, when comparing finish lines placed on class III and class V restorations made with different direct restorative materials (Tetric Evoceram, Ivoclar Vivadent Corp.,Amherst, NY; Tetric Evoceram Bulk, Ivoclar Vivadent Corp., Amherst, NY; Tetric EvoFlow, Ivoclar Vivadent Corp. Amherst, NY; and Fuji II LC, GC America, Alsip, IL) against a positive control for adhesively bonded lithium disilicate crowns in the esthetic zone? The null hypothesis for the first research question was that there will be no difference in microleakage depth or area in comparing treatment groups to the positive control.
2) Is there a difference in microleakage group means, measured as infiltration depth and area in micrometers, when comparing finish lines placed on class III and class V restorations made with different direct restorative materials (Tetric Evoceram, Tetric Evoceram Bulk, Tetric EvoFlow, and Fuji II LC) against treatment groups for adhesively bonded lithium disilicate crowns in the esthetic zone? The null hypothesis for the second research question was that there will be no difference in microleakage depth or area in comparing among treatment groups.

METHODS AND MATERIALS

Eighty newly extracted maxillary incisors were collected, mounted in acrylic, and stored in 1% thymol solution to prevent bacterial growth. Inclusion criteria required that all specimens be free of dental caries and existing direct or indirect restoration. Each specimen was randomly placed in one of four groups that received either a class III (n=10) or class V (n=10) restoration using the following: group 1, a nano-hybrid resin composite (Tetric EvoCeram); group 2, a nano-hybrid bulk fill resin composite (Tetric EvoCeram Bulk Fill); group 3, a nano-hybrid flowable resin composite (Tetric Evoflow); and group 4, an RMGI (Fuji II LC). Ten random specimens from the side opposite the restoration after sagittal sectioning were used as positive controls.

All groups received either a class III preparation or class V preparation as described above. The class III preparations were 8 (Incisal-Gingival) × 8 (Buccal-Lingual) × 2 mm (axially). The preparations were 4.0 mm (50%) above the CEJ and 4.0 mm (50%) below the CEJ (Figure 1). The class V preparations were 8 (Incisal-Gingival) × 8 (Mesial-Distal) × 2 mm (axially). The preparations were 4.0 mm (50%) above the CEJ and 4.0 mm (50%) below the CEJ (Figure 2). All preparations received incisal and gingival retention with a 1/2 round bur (Brasseler Corp., Savannah, GA, USA). All preparations were completed by two calibrated operative dentistry clinicians (one board certified) using the aforementioned prepara-

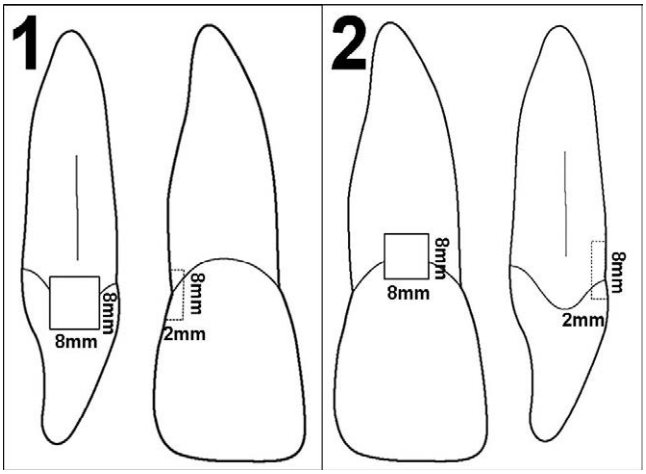

Figure 1. *Dimensional representation of the class III restoration placement.*
Figure 2. *Dimensional representation of the class V restoration placement.*

tion guidelines. Each specimen preparation was completed with a 245 carbide bur (Brasseler Corp.) under copious amounts of water coolant.

Group 1 was restored with a nano-hybrid resin composite restorative material in a crosshatch technique. The preparations were conditioned with 37% phosphoric acid (Total Etch, Ivoclar Vivadent, Amherst, NY) according to the manufacturer's recommendations and bonded with a fifth-generation single bottle bonding agent according to the manufacturer's recommendations (ExciTE F VivaPen, Ivoclar Vivadent). The bonding agent and resin composite restorative material were visibly light cured (VLC) with a calibrated (light intensity of 1200 mW/cm^2) light emitting diode (LED) curing unit (BluePhase and BluePhase Meter, Ivoclar Vivadent Corp) according to the manufacturer's recommendations. Restorations were polished with a serial composite finishing kit until highly polished (Astropol, Ivoclar Vivadent).

Group 2 was restored with a nano-hybrid bulk fill resin composite restorative material in a single application. Group 3 was restored with a nano-hybrid flowable resin composite restorative material in a crosshatch technique. Groups 2 and 3 were restored in the same sequence with the same products as group 1.

Group 4 was restored with an RMGI restorative material in a single application. The preparations were conditioned with 20% polyacrylic acid (GC Cavity Conditioner, GC America Corp., Tokyo, Japan). The RMGI material was placed in bulk according to the manufacturer's recommendations and VLC light cured with a calibrated (light intensity of 1200 mW/cm^2) LED curing unit (BluePhase and BluePhase Meter, Ivoclar Vivadent). Restorations remained hydrated and were polished with a serial composite finishing kit until highly polished (Astropol, Ivoclar Vivadent).

All 80 specimens (groups 1-4) were prepared to receive pressed lithium disilicate full-coverage indirect prostheses with a diamond bur (FG Medium Round End Taper Diamond, Brasseler Corp). All finish margins were a 90° shoulder performed by two calibrated faculty members within the authorship of this publication. The finish lines were placed 2 mm above the CEJ, with an axial reduction of 2 mm (Figure 3). All specimens had 8 mm of finished margin on the direct restorative material at the treatment side. Following preparation of all 80 specimens, a polyvinylsiloxane (PVS) impression (Virtual XD, Ivoclar Vivadent) of each specimen group was taken and labeled according to group and specimen. The PVS impressions were poured with a vacuum-mixed gypsum stone (Jade Stone, Whip Mix Corp., Louisville, KY, USA) to create laboratory analogs and were labeled. Full contour wax crowns were made on the stone analogs and pressed to full contour lithium disilicate ceramic crowns (IPS e.max, Ivoclar Vivadent) at a local dental laboratory. Original specimens were stored in individually labeled containers of 10% thymol at 37°C during the ceramic crown fabrication process.

On return from the local dental laboratory, all 80 ceramic crowns (intaglio pre-etched from the laboratory; 5% hydrofluoric acid) were evaluated for marginal integrity and delivered using a self-etching dual-cured resin adhesive luting system. Monobond Plus primer (Ivoclar Vivadent Corp) was placed on the intaglio of the crowns and let dry for 20 seconds. Multilink Primer A and B were mixed and scrubbed into the preparations for 20 seconds and air dried. A thin layer of the self-etch resin luting agent was placed in the intaglio of the crown and delivered (Multilink Automix, Ivoclar Vivadent). After hand pressure seating and removal of excess marginal material, all crown margins were VLC light cured with a calibrated (light intensity of 1200 mW/cm^2) LED curing unit (BluePhase and BluePhase Meter, Ivoclar Vivadent). All crown margins were polished with a serial composite finishing kit until highly polished (Astropol, Ivoclar Vivadent). Please see Table 1 for experimental design.

All specimens were exposed to cyclic uniaxial loading and thermocycled to measure possible variation in microleakage within experimental

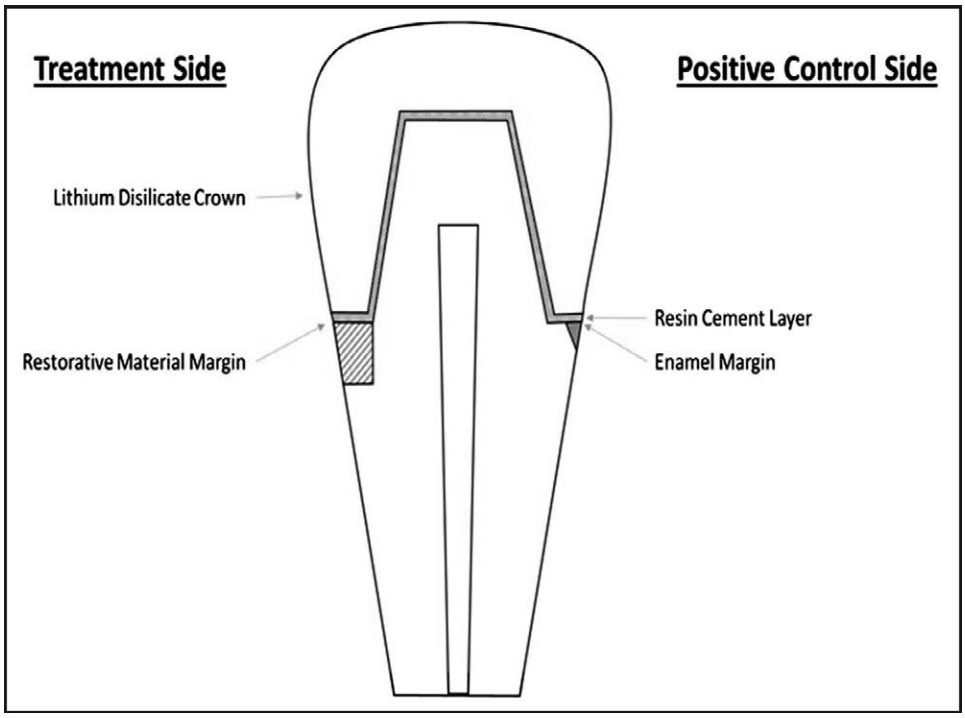

Figure 3. *Pictorial representation of the finish lines for the study of lithium disilicate crowns. Positive control side finished on enamel and treatment side finished on various restorative materials.*

groups and against the controls. All specimens were subjected to dry uniaxial compressive cyclic loading in an Instron (Instron USA, Norwood, MA) testing machine at 12 Hz for 10,000 cycles. A variable compressive load (40-400 N) was applied in the central fossa at an angle of 15° (buccal-lingual) to the long axis of the specimens to mimic shear forces possible experienced clinically. All specimens were then thermocycled with a red 0.5% basic fuchsin dye tracer (0.5 g basic fuchsin dye in 20 mL 95% ethanol diluted to 100 mL with distilled water) from 6°C to 60°C (five-minute dwell time) for 48 hours and immediately sagittal sectioned with a water-cooled diamond saw through the middle of the restorative material in three 2-mm sections. Each section was evaluated immediately for depth and area of dye penetration (micrometers) under stereomicroscopy (50×) on both the treatment side and the control side using a 1-μm boxed dimensional grid. The three sagittal-sectioned specimens were measured and averaged for an overall average dye penetration value.

The depth and area of dye penetration were measured with a dimensional grid in micrometers using stereomicroscopy and reported as mean dye depth and area (micrometers) ± standard deviation. The dimensional grid was boxed with 1-μm squares for easy measurement. The depth of the dye was measured from the cavosurface margin axially to the end of dye penetration in micrometers. The area of dye penetration was measured from the cavosurface margin axially as depth × width in micrometers. Four experimental dental materials were compared with positive control and within-treatment groups regarding mean depth and area of dye penetration in micrometers using analysis of variance (ANOVA) and Tukey post hoc analysis. The significance level was set at $p<0.05$ for these evaluations.

RESULTS

Class III Restoration Dye Penetration

Class III Depth—The descriptive statistics for the class III depth evaluation of all four treatment groups and the positive control are listed in Table 2. Different lowercase letters in Table 2 represent statistically significant differences in group dye depth penetration means. According to the ANOVA, a statistically significant difference exists among the control and four treatment groups (df=4,45; observed $F=92.7$; $p=0.012$, $p<0.05$). A Tukey's post hoc analysis was performed and determined that all four treatment groups were statistically significantly higher in dye penetration depth compared with the control ($p=0.022$, $p<0.05$). The Tetric EvoFlow treatment group was statistically significantly higher in dye penetration depth compared with the Tetric EvoCeram, Tetric EvoCeram Bulk Fill, and Fuji II LC treatment groups ($p=0.024$, $p<0.05$). There was no statistically significant difference in dye depth

Table 1: *Sampling Methodology and Study Design*[a]

Tetric EvoCeram (TEC) (N=20)	Tetric EvoCeram Bulk (TEB) (N=20)	Tetric EvoFlow (TEF) (N=20)	Fuji II LC (FLC) (N=20)	Positive control (opposite side of tooth)
Class III (n=10)	Class III (n=10)	Class III (n=10)	Class III (n=10)	10 random samples from 40 specimens (n=10)
Class V (n=10)	Class V (n=10)	Class V (n=10)	Class V (n=10)	10 random samples from 40 specimens (n=10)
37% phosphoric acid conditioner Adhesive restoration bonding: ExciTE F (N=60)			20% polyacrylic acid conditioner (N=20)	
IPS e.Max All Ceramic Crown Press Luting- Multilink Automix Resin Adhesive Luting System (N=80)				

[a] All materials used according to the manufacturer recommendations.

penetration when comparing the Tetric EvoCeram, Tetric EvoCeram Bulk Fill, and Fuji II LC treatment groups ($p>0.05$).

Class III Area—The descriptive statistics for the class III area evaluation of all four treatment groups and the positive control are listed in Table 3. Different lowercase letters in Table 4 represent statistically significant differences in group dye area penetration means. According to the ANOVA, a statistically significant difference exists among the control and four treatment groups (df=4,45; observed $F=270.6$; $p=0.022$, $p<0.05$). A Tukey's post hoc analysis was performed and determined that all four treatment groups had statistically significantly higher in dye penetration area compared with the control ($p=0.032$, $p<0.05$). The Tetric EvoFlow treatment group was statistically significantly higher compared with the Tetric EvoCeram, Tetric EvoCeram Bulk Fill and Fuji II LC treatment groups ($p=0.012$, $p<0.05$). There was no statistically significant difference in dye area penetration when comparing the Tetric EvoCeram, Tetric EvoCeram Bulk Fill, and Fuji II LC treatment groups ($p\geq0.05$).

Class V Restoration Dye Penetration

Class V Depth—The descriptive statistics for the class V depth evaluation on all four treatment groups and the positive control are listed in Table 4. Different lowercase letters in Table 3 represent statistically significant differences in group dye depth penetration means. According to the ANOVA, a statistically significant difference exists among the control and four treatment groups (df=4,45; observed $F=106.2$; $p=0.011$, $p<0.05$). A Tukey's post hoc analysis was performed and determined that all four treatment groups were statistically significantly higher in dye penetration depth compared with the control ($p=0.023$, $p<0.05$). The Tetric EvoFlow treatment group was statistically significantly higher in dye penetration depth compared with the Tetric EvoCeram, Tetric EvoCeram Bulk Fill, and Fuji II LC treatment groups ($p=0.025$; $p<0.05$). There was no statistically significant difference in dye depth penetration when comparing the Tetric EvoCeram, Tetric EvoCeram Bulk Fill, and Fuji II LC treatment groups ($p>0.05$).

Class V Area—The descriptive statistics for the class V area evaluation on all four treatment groups and the positive control are listed in Table 5. Different lowercase letters in Table 5 represent statistically significant differences in group dye area penetration means. According to the ANOVA, a statistically significant difference exists among the control and four treatment groups (df=4,45; observed $F=271.4$; $p=0.017$, $p<0.05$). A Tukey's post hoc analysis was performed and determined that all four treatment groups were statistically significantly higher in dye penetration area compared with the control ($p=0.019$, $p<0.05$). The Tetric EvoFlow

Table 2: *Class III Depth Descriptive Statistics*[a]

	Class III depth: Descriptive statistics		
	Group mean	Group standard deviations	Sample size
Positive control	190.4[a]	±11.6	N=10 × 3 sections
Tetric EvoCeram (TEC)	249.2[b]	±16.3	N=10 × 3 sections
Tetric EvoCeram Bulk (TEB)	270.0[b]	±17.4	N=10 × 3 sections
Tetric EvoFlow (TEF)	358.6[c]	±28.0	N=10 × 3 sections
Fuji II LC (FLC)	245.2[b]	±23.0	N=10 × 3 sections

[a] Different lowercase letters represent a statistical significant difference in group means.

Table 3: *Class III Area Descriptive Statistics*[a]

	Class III area: Descriptive statistics		
	Group mean	**Group standard deviations**	**Sample size**
Positive control	10,702.1[a]	±1018.7	N=10 × 3 sections
Tetric EvoCeram (TEC)	22,701.7[b]	±1446.7	N=10 × 3 sections
Tetric EvoCeram Bulk (TEB)	24,742.9[b]	±2512.4	N=10 × 3 sections
Tetric EvoFlow (TEF)	36,884.2[c]	±2272.5	N=10 × 3 sections
Fuji II LC (FLC)	23,341.5[b]	±8801.9	N=10 × 3 sections

[a] Different lower case letters represent a statistical significant difference in group means.

treatment group was statistically significantly higher compared with the Tetric EvoCeram, Tetric EvoCeram Bulk Fill, and Fuji II LC treatment groups ($p=0.022$, $p<0.05$). There was no statistically significant difference in dye area penetration when comparing the Tetric EvoCeram, Tetric EvoCeram Bulk Fill, and Fuji II LC treatment groups ($p\geq0.05$).

DISCUSSION

Dye penetration for marginal sealing has been used for years as a surrogate to understand fluid flow and marginal integrity of composites cohesive and adhesive natures in vitro.[16-21] Therefore, the purpose of this research was to investigate the effects of interfacial microleakage when finishing pressed lithium disilicate ceramic crowns on various direct restorative materials in class III and class V positions. Of particular interest was the interface between resin cement and direct restorative materials in the esthetic zone. The research questions for this evaluation were as follows:

1) Is there a difference in microleakage group means, measured as infiltration depth and area in micrometers, when comparing finish lines placed on class III and class V restorations made with different direct restorative materials (Tetric EvoCeram, Tetric EvoCeram Bulk Fill, Tetric EvoFlow, and Fuji II LC) against a positive control for adhesively bonded lithium disilicate crowns in the esthetic zone? According to the results of this study, the null hypothesis has been rejected with a statistically significant difference in microleakage depth and area between the treatment groups and the positive control for both class III and V restorations.
2) Is there a difference in microleakage group means, measured as infiltration depth and area in micrometers, when comparing finish lines placed on class III and class V restorations made with different direct restorative materials (Tetric Evoceram, Tetric EvoCeram Bulk Fill, Tetric EvoFlow, and Fuji II LC) against treatment groups for adhesively bonded lithium disilicate crowns in the esthetic zone? According to the results of this study, the null hypothesis has been rejected with a statistically significant difference in microleakage depth and area of the Tetric EvoFlow compared with the other treatment groups for both class III and V restorations. All other treatment groups were not statistically different.

Although there are no set standards for dye penetration evaluations, the ISO standard on testing the adhesion to tooth structure describes a microleakage test in the cavities of third molars with a diameter of 3 mm, a depth of at least 1 mm, and a sample size of at least 10.[19] It has been suggested in the literature that multiple sectional averages of specimens on dye tracer penetration yields more accurate results than single sections alone.[20]

Table 4: *Class V Depth Descriptive Statistics*[a]

	Class V depth: Descriptive statistics		
	Group mean	**Group standard deviations**	**Sample size**
Positive control	187.6[a]	±12.3	N=10 × 3 sections
Tetric EvoCeram (TEC)	251.7[b]	±17.2	N=10 × 3 sections
Tetric EvoCeram Bulk (TEB)	250.1[b]	±16.9	N=10 × 3 sections
Tetric EvoFlow (TEF)	348.6[c]	±21.1	N=10 × 3 sections
Fuji II LC (FLC)	254.5[b]	±18.7	N=10 × 3 sections

[a] Different lowercase letters represent a statistical significant difference in group means.

Table 5: *Class V Area Descriptive Statistics*[a]

	Class V area: Descriptive statistics		
	Group mean	**Group standard deviations**	**Sample size**
Positive control	11,502.3 (a)	±1052.2	N=10 × 3 sections
Tetric EvoCeram (TEC)	20,905.7 (b)	±1605.4	N=10 × 3 sections
Tetric EvoCeram Bulk (TEB)	22,695.1 (b)	±2460.6	N=10 × 3 sections
Tetric EvoFlow (TEF)	36,874.4 (c)	±2173.8	N=10 × 3 sections
Fuji II LC (FLC)	21,671.3 (b)	±1521.7	N=10 × 3 sections

[a] Different lowercase letters represent a statistical significant difference in group means.

Due to the fuchsin dye being water based, it is not unreasonable to consider its diffusion similar to water diffusion within marginal interfaces of resin cement approximating various restorative materials. The 0.5% basic fuchsin dye tracer is a water-based solution that represents the diffusion of that dye through the set chemical interfacial composition. Water diffusion and sorption has been evaluated within the set bis-acrylic matrices and the bis-acrylic/filler interface.[21-23] Water sorption and solubility have been evaluated on resin cements in the current literature as well.[24-26] Water sorption and diffusion has been shown to affect the interface between self-etch resin cement and silanated ceramics.[27] Conclusions on water sorption and diffusion claim a reduction in physical properties, mechanical properties, and optical properties *via* accelerated degradation of the set material. Missing from the current literature is the evaluation of microleakage at a resin cement/restorative material interface. Additionally, due to the variability in methodology across studies, a systematic review to correlate dye penetration to clinical ramifications (material properties, hypersensitivity, retention, marginal staining, or marginal caries) is lacking in the current published literature.[28]

In evaluating the control side of the specimens, dye penetration occurred within the resin cement matrix between the ceramic substrate and the enamel substrate. In using the surface opposite of the treatment side, it was postulated that both surfaces would undergo similar shear force movement and thermocycling of the resin cement layer. One limitation of the specimen comparisons is that the control and experimental side of the teeth in the class V group experienced different loading forces due to the 15° off-axis loading. The dye penetration started at the cavosurface area and worked itself axially. Most of the dye penetration occurred at the resin cement/enamel interface compared with the resin cement/ceramic interface. The control sides of the specimens groups experienced some dye penetration. Therefore, in terms of fluid flow and water diffusion, some microleakage is occurring at the resin cement/ceramic interface and the enamel/resin cement interface, with deeper penetration at the resin cement/enamel interface as viewed in the stereomicroscope (50×).

In evaluating the Tetric EvoCeram (TEC) group in the class III and V positions, the dye penetration for depth and area was significant higher than the control. Most of the dye penetration occurred at the resin cement/TEC interface as viewed under the stereomicroscope (50×). In evaluating the Tetric EvoCeram Bulk Fill (TEB) group in the class III and V positions, the dye penetration for depth and area was significant higher than the control. Most of the dye penetration occurred at the resin cement/TEB interface as viewed under the stereomicroscope (50×). In evaluating the Tetric EvoFlow (TEF) group in the class III and V positions, the dye penetration for depth and area was significant higher than the control. Most of the dye penetration occurred at the resin cement/TEF interface as viewed under the stereomicroscope (50×). The conditioning, bonding, and placement of resin cement to an existing composite restoration yielded an interface more susceptible to dye penetration compared with the control.[6,7]

In evaluating the Fuji II LC (FLC) group in the class III and V positions, the dye penetration for depth and area was significant higher than the control. Most of the dye penetration occurred at the resin cement/FLC interface as viewed under the stereomicroscope (50×). The final set glass can be conditioned with phosphoric acid and adhesively bonded also as performed in the class II sandwich technique.[10] Conditioning and adhesively bonding resin composite to a RMGI material yield a clinically acceptable interface.[8-10] The micromechanical locking of the resin cement to the existing FLC restoration could have been improved if the total

etch technique was utilized. The self-etching resin cement used could have prevented the integration needed to create a less permeable interface.

In comparing the treatment groups, the TEF class III and V experienced a significantly higher dye penetration depth and area than did the TEC, TEB, and the FLC. The TEF group created a poor interface with the resin cement. The interface created was very permeable to fluid flow and the dye penetrated much deeper into the resin cement layer and into the set flowable composite itself. The dye penetrated in the set TEF material more easily in a greater volume exhibiting some inherent diffusion characteristics that are noteworthy for further investigation. One can theorize that the limited filler in the TEF composite with high matrix lends itself vulnerable to water diffusion characteristics not experienced in TEC, TEB, and FLC materials.

A systematic review on dye penetration and bond strength to tooth structure determined that there is no correlation between the two.[29] However, there are no studies containing any information on dye penetration at the interface between resin cement and direct restorative materials or what that might mean. Additionally, cause and effect relationships between dye microleakage and restoration failure were reported in the current literature without providing adequate correlation coefficients, coefficient of determinations, linear regression, multiple regression, or percentage of common variance that any variables may have shared.[28-30] The results from this study determined that in comparing the control and experimental groups, the interface created by a self-etching resin luting agent was significantly better to enamel. The significant fluid flow and diffusion of the water-based dye at the interface layer suggests a weaker interaction of resin cement and direct restorative materials. The correlation between dye penetration and marginal gaps analysis with a scanning electron microscope was reviewed and determined that a marginal correlation existed. Again, this dye penetration study measured the restorative material/tooth structure interface.[29] In terms of clinical implications, there exists no studies in the current *in vitro* and *in situ* literature that demonstrate a correlation between microleakage and hypersensitivity and/or secondary caries formation.[31,32]

According to the results of this study, there is higher dye penetration depth and area when finishing pressed lithium disilicate crowns on all the examined existing class III and V restorative materials compared with the control. In terms of microleakage and fluid diffusion, increased fluid flow within these materials could have a detrimental effect on the long-term success of clinical indirect all-ceramic restorations. As shown with the flowable resin composite (TEF), high polymerization stress shrinkage and limited filler may lend itself to deeper dye penetration and possible exacerbated degradation. The authors are cognizant not to draw clinical implications from this *in vitro* evaluation as there is no set accepted standard for dye penetration depth within restorative materials or proven clinical implications.[30] It is important, however, for clinicians to be aware of the possibility of interfacial degradation and limited performance of flowable resin composite materials interacting with resin cement as finish lines for lithium disilicate all-ceramic crowns.

CONCLUSION

Within the limitations of this study, it can be concluded that flowable composite materials as finish lines that interact with resin cements could lead to exacerbated interfacial degradation. Finishing lithium disilicate all-ceramic crowns on flowable resin composite materials in the esthetic zone should be used with caution. If necessary, finishing lithium disilicate all-ceramic crowns on nanofilled resin composite, nanofilled bulk fill composite, or RMGI materials seems to provide the least dye penetration depth and area.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subject's oversight committee guidelines and policies of: University of Louisville. The approval code for this study is: 14.0995.

Conflict of Interest

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

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Faculty Positions

CARIOLOGY AND OPERATIVE DENTIST
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NEW YORK UNIVERSITY COLLEGE OF DENTISTRY

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Midwestern University
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555, 31st Street, Auditorium Building 594
Downers Grove, IL 60515
Email: bjham@midwestern.edu

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Fracture Load and Phase Transformation of Monolithic Zirconia Crowns Submitted to Different Aging Protocols

ETP Bergamo • WJ da Silva • PF Cesar • AA Del Bel Cury

Clinical Relevance: Advances in zirconia that have resulted in favorable esthetic and mechanical properties have enabled its application as a full-contour restoration.

doi: http://dx.doi.org/10.2341/15-154-L

Fracture Resistance of Endodontically Treated Teeth Restored With Bulk Fill, Bulk Fill Flowable, Fiber-reinforced, and Conventional Resin Composite

C Atalay • AR Yazici • A Horuztepe • E Nagas • A Ertan • G Ozgunaltay

Clinical Relevance: The restoration of endodontically treated teeth with either bulk fill/flowable bulk fill or fiber-reinforced resin restorative did not change the fracture resistance of teeth compared with that of a conventional nanohybrid resin composite.

doi: http://dx.doi.org/10.2341/15-320-L

Water Sorption and Solubility of Luting Agents Used Under Ceramic Laminates With Different Degrees of Translucency

CL Leal • APV Queiroz • RM Foxton • S Argolo • P Mathias • AN Cavalcanti

Clinical Relevance: Degrees of translucency in a restorative material are important for masking tooth color alteration. Clinicians must be aware of the relationship between a decrease in translucency and loss of light penetration to avoid the clinical degradation of an improperly cured luting material.

doi: http://dx.doi.org/10.2341/15-201-L

Fractographical Analysis and Biomechanical Considerations of a Tooth Restored With Intracanal Fiber Post: Report of the Fracture and Importance of the Fiber Arrangements

VF Wandscher • CD Bergoli • IF Limberger • TP Cenci • P Baldissara • LF Valandro

Clinical Relevance: When restoring anterior endodontically treated teeth, fiber posts with parallel fibers support tensile stresses, but they commonly fracture by shear stresses due to anterior occlusal oblique loads that generate bending of the restorative assembly.

doi: http://dx.doi.org/10.2341/15-262-S

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Fracture Load and Phase Transformation of Monolithic Zirconia Crowns Submitted to Different Aging Protocols

ETP Bergamo • WJ da Silva • PF Cesar • AA Del Bel Cury

Clinical Relevance

Advances in zirconia that have resulted in favorable esthetic and mechanical properties have enabled its application as a full-contour restoration.

SUMMARY

Monolithic zirconia crowns have many favorable properties and may potentially be used to solve dental problems such as chipping. However, monolithic zirconia crown resistance can be affected by its phase transformation when subjected to low temperatures, humidity, and stress. This study evaluated the fracture load and phase transformation of monolithic zirconia crowns submitted to different thermal and mechanical aging tests. Seventy monolithic zirconia crowns were randomly divided into the following five groups: control, no treatment; hydrothermal aging at 122°C, two bar for one hour; thermal fatigue, 10^4 cycles between 5°C and 55°C, dwell time, 30 seconds; and mechanical fatigue, 10^6 cycles with a load of 70 N, sliding of 1.5 mm at 1.4 Hz; and combination of mechanical plus thermal fatigue. Fracture load was measured with a universal testing machine. Surface changes and fracture mode and origin were examined with a scanning electron microscope. Monoclinic phase content was evaluated by x-ray diffraction. The fracture load was analyzed using one-way analysis of variance at a level of 5%, and Weibull distribution was performed. No statistically significant differences were observed in the mean fracture load and characteristic fracture load among the groups ($p>0.05$). The Weibull modulus ranged from 6.2 to 16.6. The failure mode was similar for all groups with the crack origin located at the contact point of the indenter. Phase trans-

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formation was shown at different surfaces of the crown in all groups (1.9% to 8.9%). In conclusion, monolithic zirconia crowns possess high fracture load, structural reliability, and low phase transformation.

INTRODUCTION

In the early 1990s, yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) was proposed for use in dentistry, and interest in this material has grown due to its superior mechanical properties compared with other ceramic systems.[1,2] The excellent mechanical behavior of Y-TZP is credited to a transformation toughening mechanism, in which tetragonal zirconia crystals transform into the monoclinic form and produce compressive stresses that prevent cracks and hinder crack propagation.[2-4] Due to the relatively low fracture rate of Y-TZP structures, a wide range of applications with high clinical success rates has been proposed for these materials, such as dental prostheses and implant abutments.[5-7]

Prosthetic dental crowns are conventionally fabricated in two steps: construction of a framework and subsequent veneering with an esthetic veneering ceramic. When Y-TZP prostheses were first proposed for dental applications, the prostheses were veneered with compatible porcelain to improve the final esthetic result. Nevertheless, this type of prosthesis has demonstrated relatively high clinical fracture rates, usually expressed as chipping and/or delamination of the porcelain layer.[8,9] To overcome this problem, manufacturers have recently proposed the use of Y-TZP as a monolithic (full-contour) restoration (ie, without the veneering layer). The production of monolithic dental crowns only became possible due to the development of modified Y-TZP microstructures with higher translucency and the addition of coloring pigments.[10] This type of restoration achieves reasonable esthetic results via the addition of a superficial glaze layer that allows for final biomimetic characterization. The increasing popularity of monolithic Y-TZP crowns for prosthetic rehabilitation is partially related to economic issues. Specifically, production of these crowns requires fewer manufacturing steps and less human labor because they are almost entirely processed via Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) systems.[11-13] Additional advantages of monolithic Y-TZP crowns have been reported in the literature, including reduced tooth preparation[12-14] and superior fracture resistance compared with other ceramic materials.[11,13,15]

When using monolithic Y-TZP crowns, the zirconia surface may be directly exposed to adverse oral conditions, and in some cases, the surface is only protected by a thin glaze layer. Low temperature degradation (LTD) may be triggered when zirconia surfaces are in direct contact with water at body temperature.[16,17] This type of degradation occurs through energy barrier reduction for tetragonal to monoclinic transformation caused by the incorporation of water into the zirconia lattice.[18-20] LTD can occur at low temperatures (range = 37°C to 500°C), with a maximum transformation rate occurring at 250°C.[16,21] This phenomenon is time dependent and proceeds gradually from the surface into the bulk of the ceramic by a nucleation-and-growth process characterized by surface roughening, microcracking, and macrocracking, which enables water to deeply penetrate the material.[16,21-23] Therefore, LTD progression can significantly affect Y-TZP mechanical properties.[24,25]

In addition to LTD, zirconia-based materials undergo fatigue and subcritical crack growth under functional loading in the oral environment, which gradually reduces their strength.[26,27] Continuous contact with teeth during mastication can damage the material surface and cause the accumulation of critical defects, which may lead to catastrophic failure over time. [26,28] However, zirconia monolithic crowns have been proven to cause lower antagonist wear than other materials, such as lithium disilicate and dental porcelains.[11] In addition to mechanical degradation due to chewing, repeated thermal variations in the oral cavity also generate tensile stresses within the zirconia crown, accelerating the fatigue process via subcritical crack growth.[28,29] A previous study reported a significant reduction in the load-bearing capacity of zirconia framework prostheses after combined mechanical and thermal cycling.[27] However, whether the observed degradation was caused by LTD or mechanical fatigue remains unknown.

Due to the increasing use of zirconia as a monolithic restoration material and the evidence that its mechanical properties can be negatively affected by phase transformation triggered by superficial stresses or LTD,[23-26,30-34] this study evaluated the effect of different hydrothermal and mechanical aging processes on the fracture load and phase transformation of monolithic zirconia crowns. Our null hypothesis asserted that different aging procedures would not affect the mechanical

behavior and the monoclinic phases of Y-TZP crowns.

METHODS AND MATERIALS

Specimen Preparation

A three-dimensional (3D) CAD model of a left maxillary first molar was generated. To simulate the preparation of conventional all-ceramic prostheses, the tooth was anatomically modeled by reducing the proximal walls by 1.5 mm and the occlusal surface by 2.0 mm, with a convergence angle of 10 degrees between the mesial and distal walls as well as between the buccal and palatal walls. Furthermore, a marginal chamfer preparation of 1.0 mm was employed.[35]

A new commercially available highly translucent yttria-stabilized zirconia (Ceramill Zolid, Amann Girrbach, Koblach, Austria), which has a grain size $\leq$0.6 μm, was utilized. Based on a 3D model previously developed, the monolithic zirconia crown was designed and milled with the CAD/CAM system Ceramill Motion 2 (Amann Girrbach) derived from white presintered blocks (n = 70). After the milling procedure, the crowns were stained with Ceramill coloring liquids (Amann Girrbach) following the manufacturer's guidelines. Then, the samples were sintered at 1,450°C for two hours and glazed according to the manufacturer's instructions (Ceramill stain and glaze, Amann Girrbach).

A CAD-prepared tooth model was machined in an acrylic resin block (VIPI blocks, VIPI, Pirassununga, Brazil). Seventy prepared tooth replicas were fabricated from polyvinylsiloxane impressions (Futura, Nova DFL, Taquara, Brazil) of the plastic model. The impressions were filled with layers of resin-based composite (Z250, 3M/ESPE, Sumaré, Brazil) and light cured according to the manufacturer's recommendation. Then, each replica was embedded in acrylic resin (Dencôr, Clássico, São Paulo, Brazil) into a 15-mm-diameter polyvinyl chloride tube (PVC), leaving 1 mm exposed from the buccal margin preparation. The replicas were stored in deionized water at 37°C for 30 days to ensure complete hydration of the samples and to eliminate any dimensional expansion effect due to water absorption.

The Y-TZP crowns were then cemented with dual-curing resin composite cement (Panavia F 2.0 - LOT 000003; Kuraray, Tokyo, Japan) and ultrasonically cleaned. The resin replicas were etched with phosphoric acid for five seconds, and a ceramic primer was applied according to the manufacturer's instructions. Then, the ED Primer II mixture was applied in the replicas for 30 seconds after which the resin composite cement was mixed, applied in the crown, and inserted in the tooth replica. The crowns were maintained under a load of 10 N and the excess removed. The margin was light-cured for 20 seconds on each surface. After cementation, the specimens were stored in deionized water for seven days to provide suitable aqueous equilibrium prior to testing.[36]

Aging Procedures

The crowns were randomly divided into five groups (n = 14) that received the following aging treatments corresponding to a lifetime of approximately one year *in vivo*.[23,37,38]

1. Control: no aging treatment.
2. Hydrothermal aging: aging was carried out in a reactor controller (Reactor Parr 4843, Parr, Moline, IL, USA), a chamber that controls temperature and pressure, in this case, at 122°C (±1°C) under two bars for one hour.[23]
3. Thermal fatigue: specimens were submitted to 10^4 thermal cycles[37] in distilled water between 5°C and 55°C, with a dwell time of 30 seconds.
4. Mechanical fatigue: samples were mounted in a PVC matrix filled with acrylic resin (Dencôr) and placed in a chewing simulator (CS-4.8, SD Mechatronik, Feldkirchen-Westerham, Germany). Each specimen was subjected to 10^6 mechanical cycles[38] with a load of 70 N applied by sliding a stainless steel antagonist (5.6 mm in diameter)[13] through a 1.5-mm path at the inner side of the mesiopalatal cusp from the lingual to the buccal side at a 1.4-Hz frequency. The crowns were immersed in distilled water at 37°C during mechanical cycling. All specimens were evaluated for the presence of damage and cracks at the end of the cycling period.
5. Combination of mechanical and thermal fatigue: samples were sequentially submitted to the mechanical and then to the thermal fatigue protocol.

Fracture Load Measurement

At the end of each aging treatment, 12 crowns belonging to each group were loaded until a fracture occurred using a universal testing machine (Kratos KE, Kratos Equipment, Cotia, Brazil) equipped with a load cell of 10 KN at a crosshead-speed of 1 mm/min. The force was applied at the central fossa of the occlusal surface via a stainless steel ball (5.6 mm in diameter),[13] and the crowns were immersed in distilled water at 37°C during the test. Load at

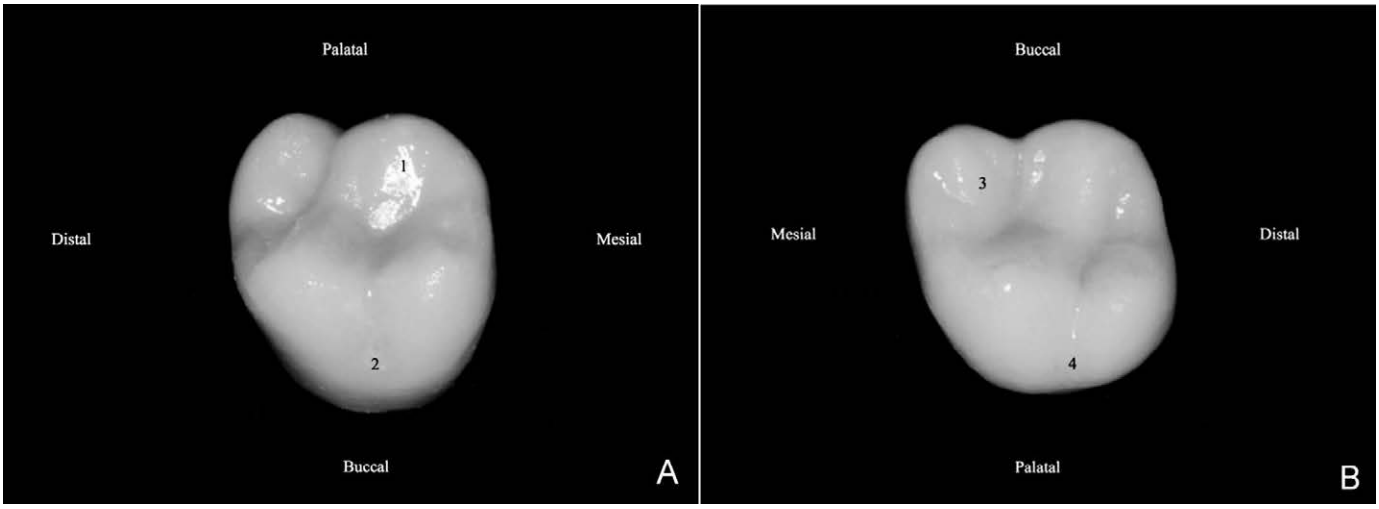

Figure 1. *Points selected to perform X-ray diffraction analysis: (A): 1, mesiopalatal cusp; 2, buccal surface. (B): 3, mesiobuccal cusp; 4, palatal surface.*

fracture (N) was registered, and a fracture was defined as a visible fracture or as the occurrence of an acoustic event and load drop.

Phase Transformation

The relative amount of monoclinic phase after aging procedures was determined by x-ray diffraction in the palatal and buccal surfaces and the mesiobuccal and mesiopalatal cusps of two samples from each group (Figure 1). The x-ray diffraction profiles were obtained using a diffractometer dispensing energy at 8 keV (Cyberstar detector, FMB Oxford, Oxford, United Kingdom). The x-ray tube is stationary, the specimen displaces at an angle of θ, and the detector simultaneously moves at an angle of 2θ. To evaluate the microstructural characterization of the crowns, scans were performed at 2θ, ranging from 20 to 70 degrees with incremental increases of 0.05 degree. The monoclinic peak intensity ratio (Xm) was calculated using the following equation[39]: $Xm = I_{m(-111)} + I_{m(111)} / I_{m(-111)} + I_{m(111)} + I_{t(101)}$ where I_t and I_m represent the integrated intensity area under the peaks of the tetragonal (101), monoclinic (–111), and monoclinic (111) peaks at 30°, 28°, and 31.2°, respectively. The data obtained from this equation were used to calculate the monoclinic volume content at the surface (Vm)[40] : $Vm = 1.311.Xm / 1 + 0.311.Xm$

Scanning Electron Microscopy

To evaluate the surface modifications after aging, crowns from each group were sputter coated with gold/palladium alloy (Bal-Tec 020, Leica Microsystems, Wetzlar, Germany) for scanning electron microscopy (SEM) analysis (Jeol JSM-6360LV; Jeol, Boston, MA, USA). After fracturing, the specimens were examined with a light-polarized stereomicroscope (Olympus SZ61, Olympus, Waltham, MA, USA), and representative specimens were evaluated by SEM to identify the fracture origin and to characterize the fracture mode.

Statistical Analysis

Normal data distribution was confirmed by the Shapiro-Wilk test. Mean fracture loads of the groups were analyzed by one-way analysis of variance. Statistical calculations were performed using the SAS system release 9.3 (SAS Institute Inc, Cary, NC, USA), and a significance level of 5% (α=0.05) was adopted. The reliability of the Y-TZP crowns was assessed through the Weibull distribution (Weibull++, ReliaSoft, Tucson, AZ, USA), and the analysis was performed to determine the Weibull modulus and the characteristic failure load in each group. The Weibull modulus determines the slope of the distribution function and characterizes the spread of the data with respect to fracture load. Characteristic failure load (P_0) defines the stress level at which 63.21% of the specimens fail.[41,42] The Weibull distribution parameters were estimated by the maximum likelihood method, and their 95% confidence intervals (CIs) were computed. Differences among groups were considered to be significant when the 95% CIs did not overlap.

RESULTS

All restorations survived the artificial aging tests, and no damage was visible to the naked eye. Although a

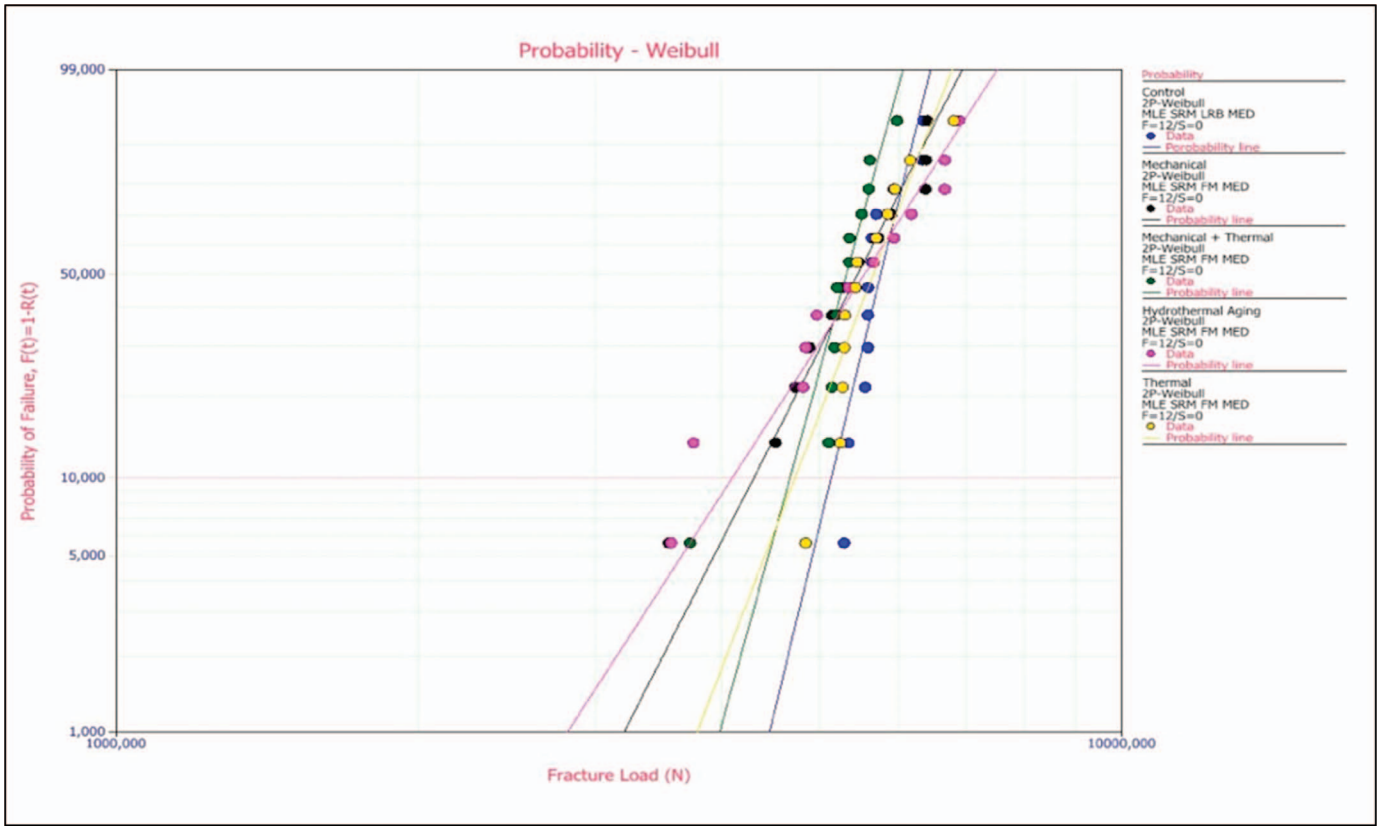

Figure 2. *Curves representing the Weibull probability of fracture for each group (confidence interval of 95%).*

decrease in the mean fracture load was observed for all age groups in comparison to the control, no statistically significant difference was observed for any of the pairwise comparisons ($p>0.05$). Similar findings occurred for the characteristic fracture load (P_0) as the Weibull statistics did not detect a significant effect of aging (Figure 2; Table 1).

The Weibull modulus ranged from 6.2 to 16.6 (Table 1). The Weibull modulus values of the age groups decreased compared with the control; however, the decrease in the Weibull modulus in relation to the control was only statistically significant for the group submitted to hydrothermal aging (Figure 2; Table 1).

The x-ray diffraction analysis indicated that the control group contained a specific level of monoclinic phase (approximately 4%) in most of the evaluated surfaces. Hydrothermal and thermal aging protocols did not alter the amount of monoclinic phase in the measured spots. For groups submitted to mechanical aging (mechanical fatigue and mechanical plus thermal fatigue), the amount of monoclinic phase increased more than eightfold on the mesiopalatal cusp, where attrition with the steel ball occurred (Figures 3 and 4).

The SEM micrographs of representative occlusal surfaces showed significant glaze layer degradation

Table 1: *Fracture Load and Weibull Distribution Parameters of Groups, Characteristic Failure Load (P_0), and Weibull Modulus (m) (Confidence Interval Limits of 95%)*[a]

Group	Mean Fracture Load (N)	Weibull Parameters	
		Characteristic Failure Load (P_0) (N)	Weibull Modulus (m)
Control	5722 (5934-5510)[a]	5884 (5652-6109)[a]	16.6 (10.4-24.0)[a]
Hydrothermal aging	5450 (6149-4750)[a]	5867 (5342-6467)[a]	6.2 (3.9-9.9)[b]
Thermal	5618 (5949-5287)[a]	5858 (5532-6204)[a]	10.5 (7.0-15.6)[a,b]
Mechanical	5538 (5997-5079)[a]	5718 (5304-6164)[a]	7.9 (5.0-12.5)[a,b]
Mechanical + thermal	5256 (5602-4911)[a]	5455 (5239-5679)[a]	14.6 (9.3-22.7)[a,b]

[a] *Different letters in each column represent significant difference among the groups ($p<0.05$).*

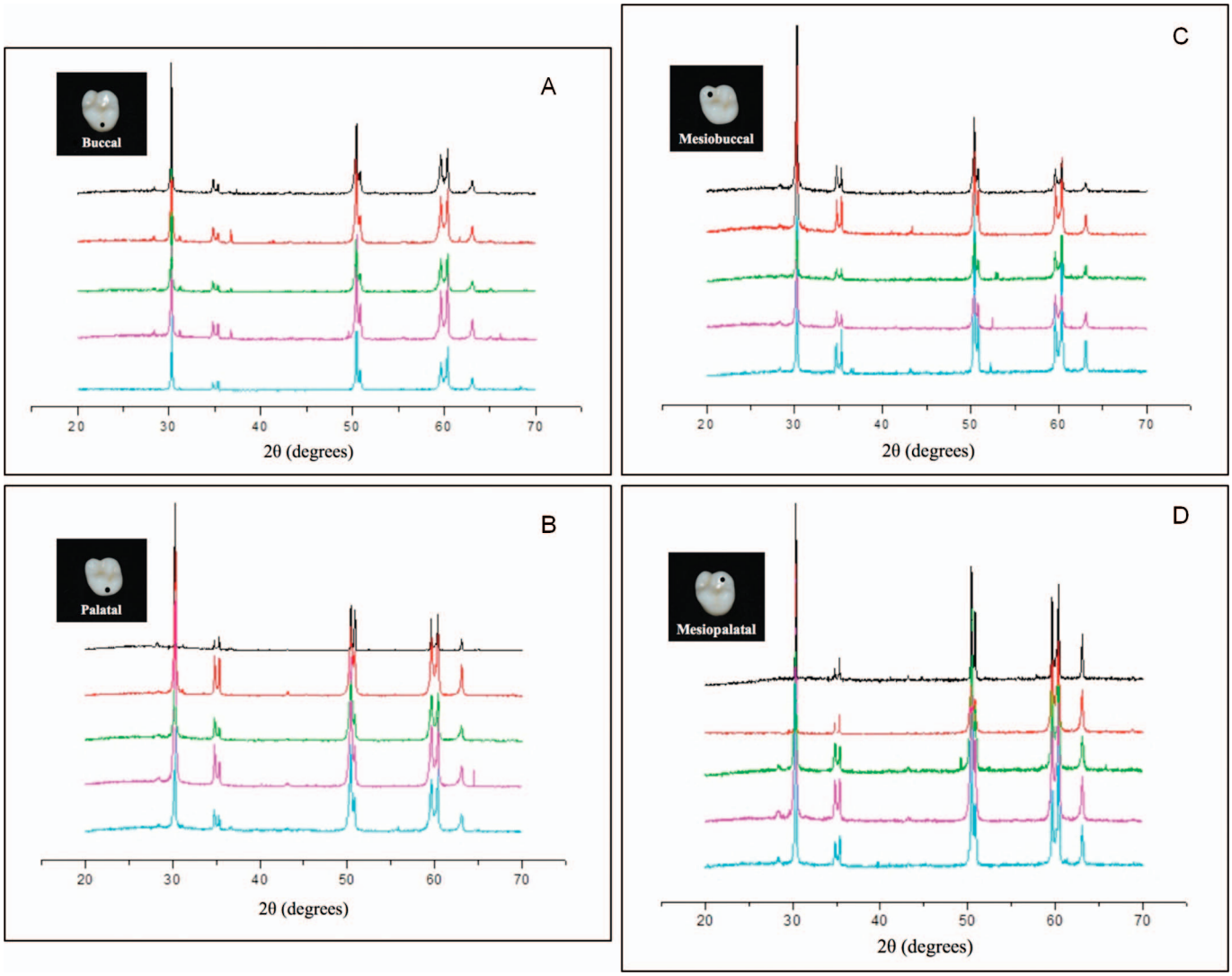

Figure 3. *Diffractogram of crowns according to tooth surface and aging treatment. Crown surfaces: (A): buccal; (B): palatal; (C): mesiobuccal cusp; and (D): mesiopalatal cusp. Aging treatment: black, control; red, hydrothermal aging; green, thermal; pink, mechanical; blue, mechanical and thermal fatigue.*

in the groups subjected to mechanical aging (mechanical fatigue and mechanical plus thermal fatigue), with marked damage accumulation and exposure of the underlying Y-TZP (Figure 5). The failure modes were similar for all experimental groups, with the crack origin located at the contact point of the indenter. The central fossa and the main crack propagated from the occlusal surface toward the crown cementation surfaces (Figure 6).

DISCUSSION

This study evaluated the fracture load and phase transformation of monolithic zirconia crowns submitted to different thermal and mechanical aging tests on prosthetic crowns that were designed to mimic clinical situations. The primary hypothesis was only partially accepted because although the aging protocol did not affect the fracture load of the Y-TZP monolithic crowns, the amount of monoclinic phase at the crown surface was higher for protocols using mechanical cycling.

All tested crowns resisted the applied artificial aging methods, and no statistically significant difference was observed among the mean fracture loads and characteristic fracture loads of all experimental groups. The absence of significant differences among the fracture loads obtained with the aging methods could be due to weak parameters incapable of degrading the mechanical behavior of the tested Y-TZP monolithic crowns in the aging protocols.

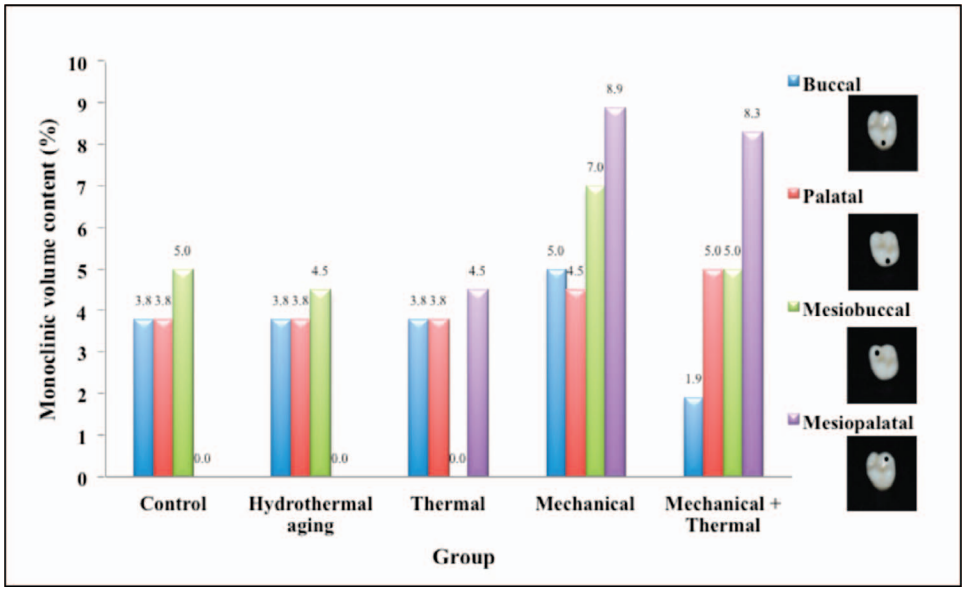

Figure 4. *Relative amount of monoclinic phase detected by x-ray diffraction in each aging group at different crown surfaces.*

Regarding mechanical cycling, the load chosen for the aging protocol (70 N) may have been insufficient to generate a stress level along the Y-TZP crown that could significantly degrade its mechanical properties. Importantly, Y-TZP has a significant phase transformation strengthening mechanism that hinders slow crack propagation[2-4,25] and increases the lifetime of the prosthetic crown by protecting the crown from adverse effects of the oral cavity environment.

The fracture load values obtained in the present study (5,256-5,722 N) far exceeded the load levels found clinically regarding the maximum bite force (900 N).[43] Few reports have evaluated the fracture load of monolithic zirconia crowns, and previously published results are considerably different from the results found in the current experiment. One previously published study reported fracture load values of up to 10,000 N,[11] while others obtained lower values varying from 2,795 to 3,068 N.[13,15] A recently published study showed a load-bearing capacity of 5,620 N for monolithic zirconia crowns, which is closer to the values identified in the present study.[14] In different studies, the large variation observed in the fracture load values for the same type of prosthetic crown (monolithic Y-TZP) can be explained by significant experimental differences among these works, such as the direction and location of the load in relation to the crown surface as well as the type of abutment substrate, indenter characteristics (eg, material type and dimensions), prosthetic crown thickness,[44] presence or absence of a wet environment during the test,[15] and previous aging treatments before the fracture test.[11,15] Other factors may also affect the measured fracture load of Y-TZP crowns such as differences in the number of layers applied during the glaze procedure and the total number of firing cycles conducted for glaze layer sintering.

The Weibull modulus is the shape parameter of the Weibull distribution and is used to describe the variation in strength values as a result of the type of flaw population present in the material structure. Higher Weibull modulus values correspond to a more homogeneous flaw size distribution, less data scatter, and greater structural reliability. Conversely, the opposite is expected from lower Weibull modulus values.[41,42] This study showed Weibull modulus values ranging from 6.2 to 16.6, which is consistent with the values reported for ceramic crowns.[45] As per the Weibull modulus values shown in Table 1, only hydrothermal aging appeared capable of causing a significant decrease in Y-TZP crown reliability. Although no surface phase transformation was detected in the x-ray diffraction analysis for specimens submitted to hydrothermal aging, further structural analyses such as micro-Raman and x-ray photoelectron spectroscopy are needed to investigate whether the occurrence of nonhomogeneous phase transformation at the crown surface and within the crown may be responsible for the significant drop in the reliability level of this experimental group.

Regarding the fracture modes, all specimens showed catastrophic failures with the crack origin located at the occlusal surface at the loading point between the indenter and the central fossa. From this point, the crack propagated toward the intaglio

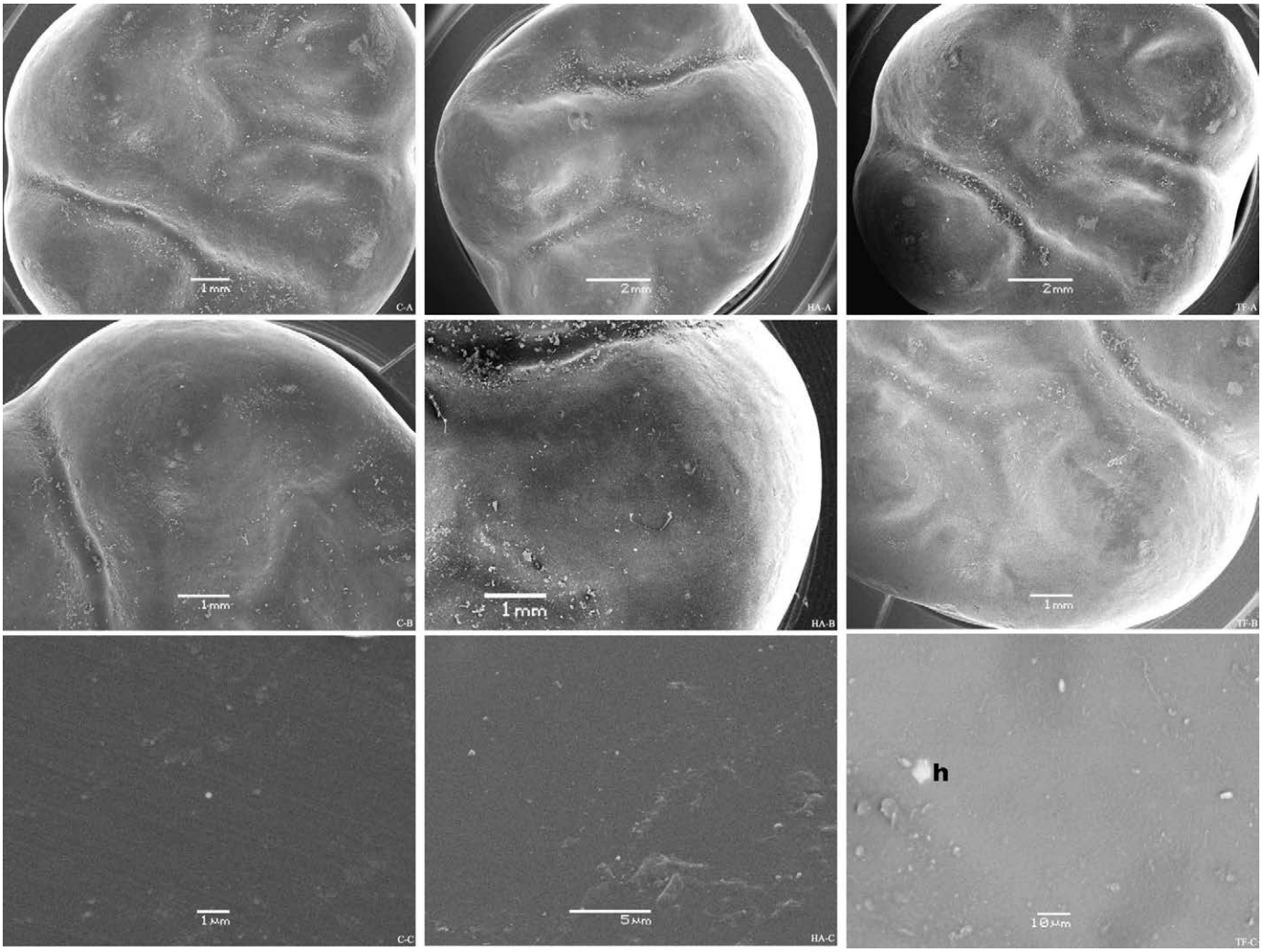

Figure 5. *Column A shows the surface view of zirconia monolithic crowns after aging treatments: C, control; HA, hydrothermal aging; TF, thermal; MF, mechanical fatigue; and MTF, mechanical and thermal fatigue. Column B depicts mesiopalatal cusp views of different groups, where MF and MTF exhibit the loss of glaze layer and zirconia exposure due to mechanical stress. Column C shows a closer view of the integrity of the glaze layer in C and HA; a hole is shown in the TF group; and the interface between the glaze layer (g) and zirconia exposure (z) (arrow) is shown in the MF and MTF groups.*

surface of the crown. This type of fracture mode is typically observed in the fracture surfaces of monolithic crowns in laboratory studies that used methodologies similar to the one used in this investigation.[13,15,28] Catastrophic failures of monolithic Y-TZP crowns occur at much higher stress levels compared with those needed to fracture the veneering porcelain layer applied over Y-TZP copings; therefore, the fracture mode observed in the present investigation indicates an important mechanical advantage for the full-contour crowns[13]. Indeed, *in vitro* studies that determined the fracture strength of zirconia crowns veneered with porcelain reported mean fracture loads varying from 1480 to 2500 N.[13,15] These values are considerably lower than the fracture load found in the current investigation for monolithic zirconia crowns (approximately 5200 N).

With respect to phase transformation, a certain amount of monoclinic phase content was already noted in the control group (Vm = 0 to 5%). The presence of a monoclinic phase in Y-TZP surfaces submitted to a glaze layer was expected because previous work has proven that the application of a wet porcelain slurry to Y-TZP surfaces with subsequent firing results in a localized tetragonal to monoclinic transformation in the core/veneer interface.[33,34] The x-ray diffraction analysis was capable of detecting transformations that occurred at the interface of the Y-TZP crown with the applied thin glaze layer.

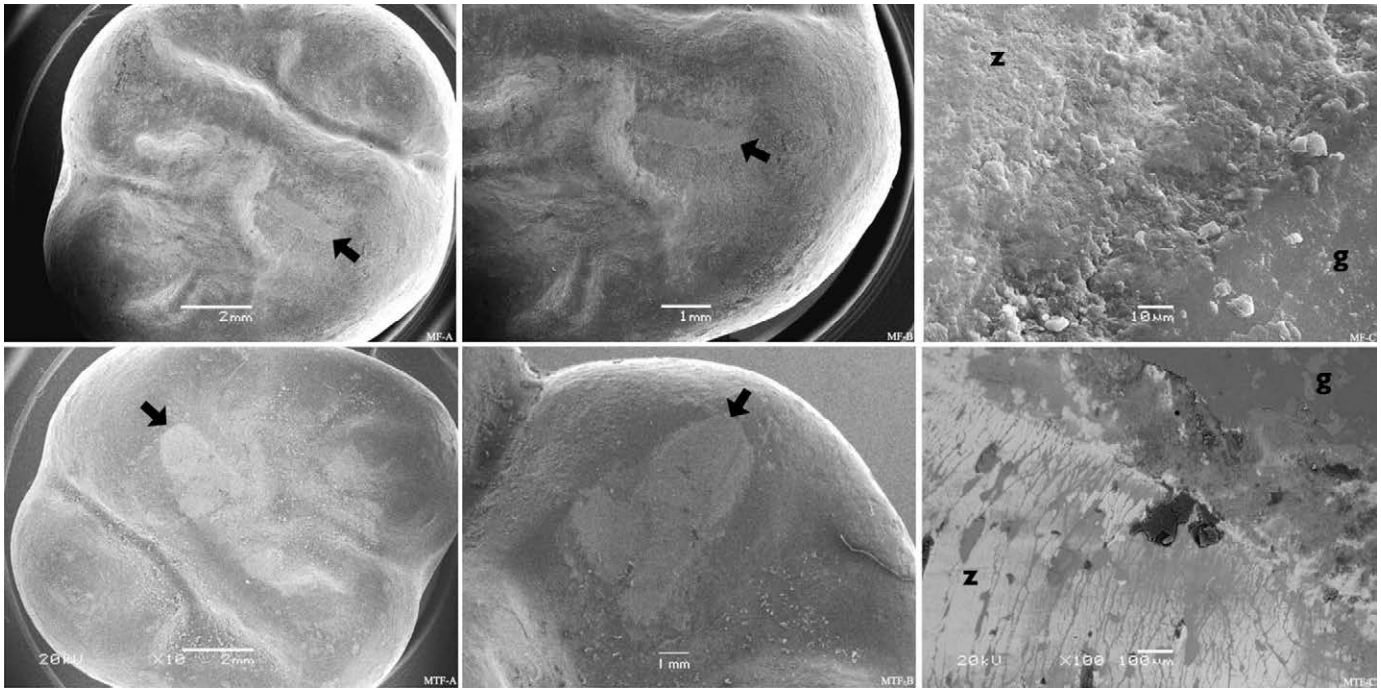

Figure 5. *Continued.*

Material strength is degraded by repeated thermal stresses in prosthetic restorations. These thermal stresses lead to tensions within the specimens and manifest in slow subcritical crack growth and catastrophic failure.[27] Nevertheless, in the present study, thermal fatigue and hydrothermal aging resulted in similar monoclinic volume content compared with the control at all surfaces (Vm = 0 to 4.5%). This result can be attributed to the presence of the glaze layer that protects the zirconia surface from water contact and avoids additional phase transformation. A previous study revealed phase transformation (Vm = 6.4%) after 3×10^4 thermal cycles,[31] and another study showed no monoclinic phase after two hours of hydrothermal aging in the same conditions used in the present investigation[30]; however, both studies evaluated Y-TZP specimens without a glaze layer, confirming the hypothesis of the protection offered by the glaze layer or insufficient aging proposed by the current investigation.

The mechanical fatigue and mechanical plus thermal fatigue aging groups showed a slight increase in the monoclinic volume content (Vm = 8.3 to 8.9%), especially in the mesiopalatal cusp, in comparison to the same surface of the other groups (Vm = 0 to 4%). This increase in monoclinic content after aging is most likely related to the attrition and mechanical stresses generated by the contact of the crown surface with the indenter. Indeed, the surface SEM micrographs of the crowns showed significant damage of the glaze layer with exposure of the underlying Y-TZP surface after the chewing simulation. A previous study[32] that investigated the phase transformation caused by mechanical stresses in zirconia blocks revealed higher monoclinic volume content (Vm = 13%) in comparison to the current experiment. This difference is also most likely related to the distinct conditions of the two studies, such as load level, number of cycles, and presence of the glaze layer.

The diffraction peaks obtained by x-ray diffraction need to be analyzed with caution because although this technique is considered the first step to investigate zirconia aging sensitivity, no precise information can be obtained during the initial transformation stages (Vm values < 5%), and significant variability of the results may occur when different locations on the same specimen are analyzed.[46] The x-ray beam can only interact with the outermost surface of the material with a penetration depth of a few micrometers[46]; therefore, the presence of the thin glaze layer may have affected the test precision.

Considering the results of the present investigation, further laboratory studies need to be conducted to better comprehend the mechanical behavior of monolithic zirconia crowns. Future work should use cyclic fatigue methodologies that will determine the

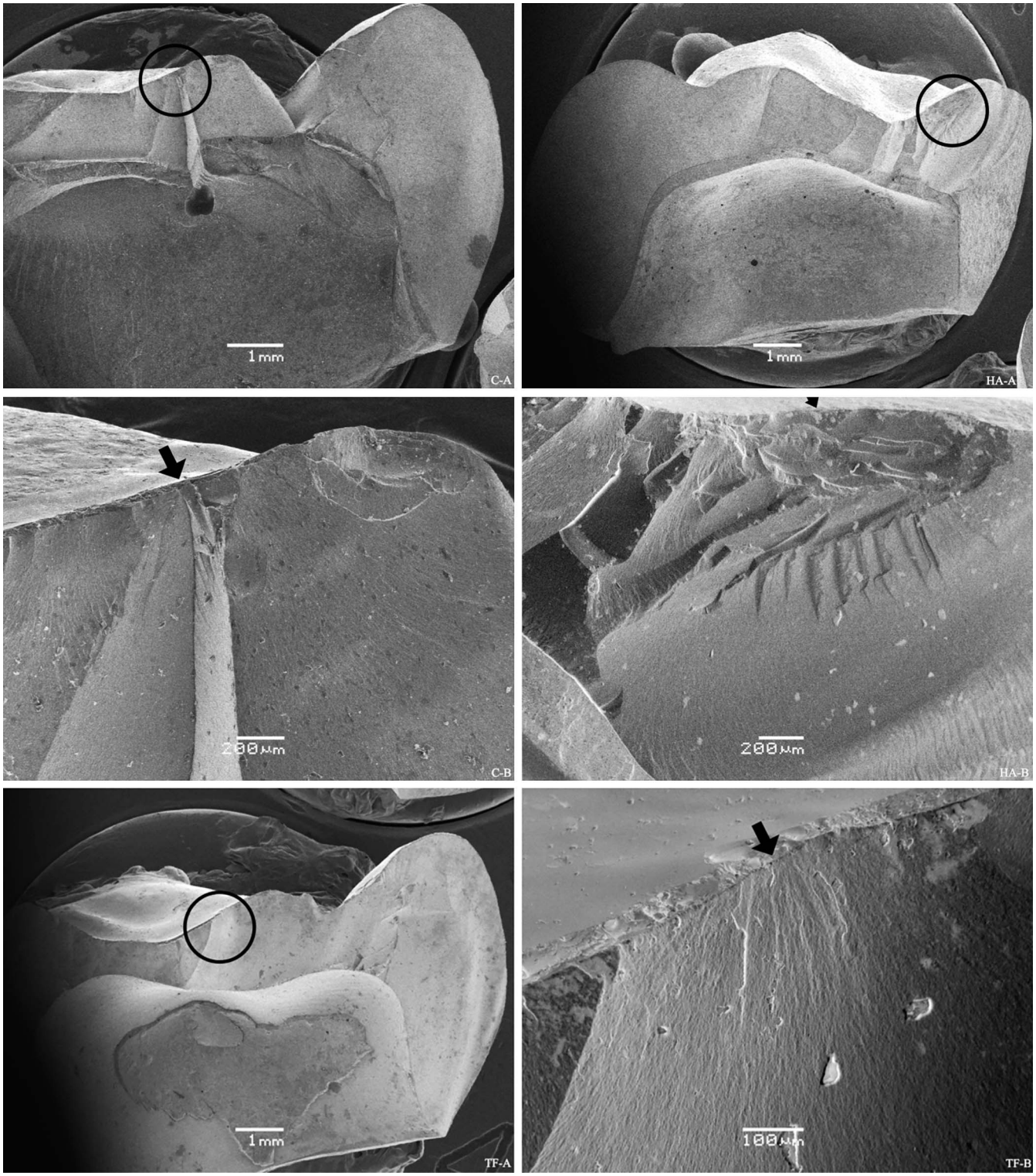

Figure 6. *Fractographic analysis. All groups showed the same failure mode with the crack origin (circle and arrow) located at the surface in contact with the indenter. Column A shows no approximated views and column B shows closer views of the groups: C, control; HA, hydrothermal aging; TF, thermal; MF, mechanical fatigue; and MTF, mechanical and thermal fatigue.*

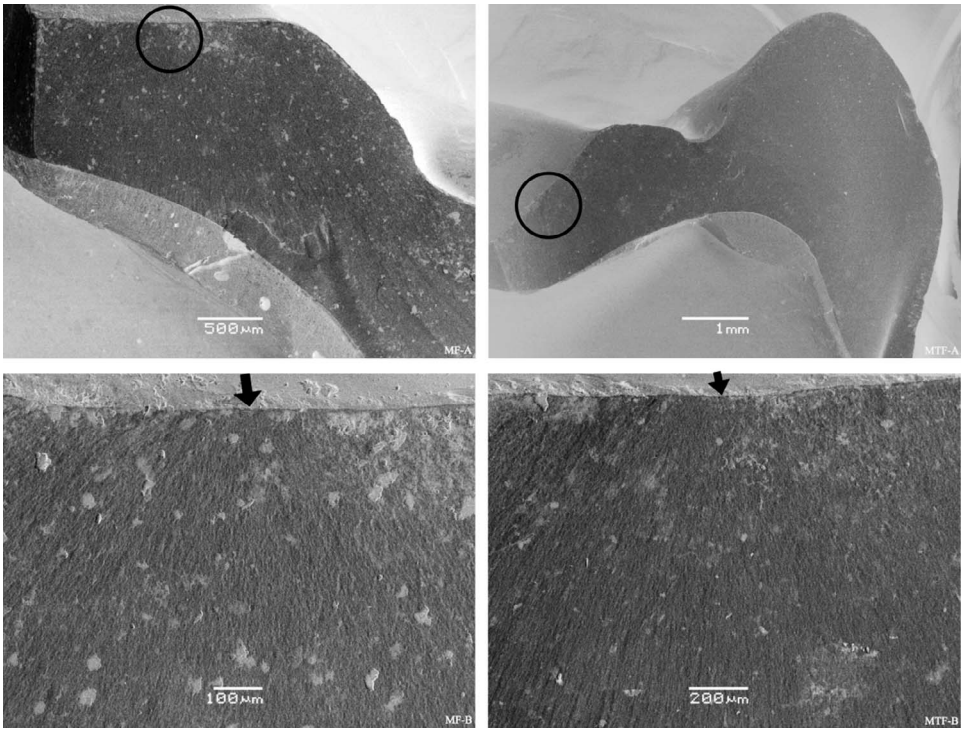

Figure 6. *Continued.*

material lifetime and fatigue parameters. Also, longer aging protocols should be tested to clarify how superficial phase transformation occurs over longer clinical lifetimes.

CONCLUSION

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

- None of the four different aging protocols affected the fracture load of the tested Y-TZP monolithic crowns;
- Only the hydrothermal method reduced the structural reliability of the crowns;
- The aging protocols involving attrition of an antagonist with the Y-TZP glazed layer were the only protocols resulting in a significant increase in the monoclinic phase at the crown surface;
- The SEM observations indicated accumulated damage of the glaze layer and subsequent exposure of the zirconia surface due to attrition with the antagonist during the chewing simulation.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Piracicaba Dental School, University of Campinas.

Conflict of Interest

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

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Fracture Resistance of Endodontically Treated Teeth Restored With Bulk Fill, Bulk Fill Flowable, Fiber-reinforced, and Conventional Resin Composite

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Clinical Relevance

The restoration of endodontically treated teeth with either bulk fill/flowable bulk fill or fiber-reinforced resin restorative did not change the fracture resistance of teeth compared with that of a conventional nanohybrid resin composite.

SUMMARY

The aim of this *in vitro* study was to evaluate the fracture resistance of endodontically treated teeth restored with different types of restorative resins.

Methods and Materials: Seventy-two sound maxillary premolar teeth were randomly divided into six groups (n=12). The teeth in the first group were left intact and tested as unprepared negative control (group I) specimens. The teeth in the remaining five groups were prepared with MOD cavities and endodontically treated. The teeth in one of the five groups (positive control group II) were unrestored. The rest of the prepared cavities were restored as follows: group III: bulk fill resin composite/Filtek Bulk Fill (3M ESPE); group IV: bulk fill flowable resin composite + nanohybrid/SureFil SDR Flow + Ceram.X Mono (Dentsply); group V: fiber-reinforced composite + posterior resin composite/GC everX posterior + G-aenial posterior (GC Corp.); and group VI: nanohybrid resin composite/Tetric N-Ceram (Ivoclar/Vivadent). Each restorative material was used with its respective adhesive system. The restored teeth were stored in distilled water for 24 hours at 37°C and were then thermocycled (5-

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55°C, 1000×). Specimens were subjected to a compressive load until fracture at a crosshead speed of 0.5 mm/min. The data were analyzed using one-way analysis of variance followed by the post hoc Tukey honestly significantly different test ($p<0.05$).

Results: Sound premolar teeth (group I negative control) showed significantly higher fracture resistance than did the other tested groups ($p<0.05$). No statistically significant differences were found in the fracture resistance values of the restored groups (groups III, IV, V, and VI) ($p>0.05$). The lowest values were obtained in the positive control group (group II); these values were significantly lower than those of the other groups ($p<0.05$).

Conclusion: The fracture resistance values of endodontically treated teeth restored with either bulk fill/bulk fill flowable or fiber-reinforced composite were not different from those restored with conventional nanohybrid resin composite.

INTRODUCTION

Trauma, caries, extensive cavity preparation, and endodontic treatments are the most common reasons for tooth fragility.[1] As a result of the loss of water content and anatomic structures, such as the pulp chamber roof, endodontically treated teeth are more susceptible to fracture than are vital teeth.[2] The amount of residual coronal dentin is considered of primary importance in the prognosis of endodontically treated teeth. Supporting the remaining dental structures is crucial for the long-term success of treatment.[3] Deciding how to implement a restorative protocol for endodontically treated teeth with variable remaining tooth structure is challenging for operators when excessive structure has been lost. There are many different direct and indirect treatment options for these kinds of teeth, such as crowns (with or without post placement), onlays/inlays, and direct resin-based restorative materials.[4]

Restoration of a tooth with adhesive procedures and direct resin composites eliminates excessive loss of sound tooth structure and overpreparation. Direct resin-based composite restorations are applied in one treatment session at relatively low cost. As there are many different types of tooth-colored direct restorative materials available in the dental market, it is important to determine which materials are successful to ensuring a long-lasting restoration in endodontically treated teeth.

Although conventional resin composites are used for restoration of endodontically treated teeth, their major shortcoming, polymerization shrinkage, is still present.[5] In larger cavities, the polymerization shrinkage that leads to higher stress accumulation on the tooth than on the restoration is considered responsible for a series of clinical complications, including higher risk of tooth fracture.[6] In order to reduce polymerization shrinkage stress and to maintain adequate depth of cure, incremental placement of resin composites has been routinely used in daily practice. However, the use of 2-mm-thick resin composite materials incrementally for direct restorations is time consuming, increases the risk of contamination between layers, and may include voids in the restoration.[7,8]

Bulk fill resin composites are an innovative class of dental resin composite materials, developed to simplify the placement of direct composite restorations.[9] They include low-viscosity, flowable, and high-viscosity material types. According to the manufacturers, they can be efficiently light-cured at depths up to 4-5 mm and cause low polymerization shrinkage stress at the same time. However, as their surface hardness and modulus of elasticity are low, there is a requirement to place a final capping layer (made of a conventional composite material) on top of these restorative materials. In contrast, high-viscosity bulk fill resin composites are indicated for use without veneering and can thus be applied as single-step bulk fill materials.[10,11] In a recent study,[12] it was found that the tested bulk fill resin composites can be cured effectively at a depth of at least 4 mm.

Fiber-reinforced composites have been suggested[13] to reduce polymerization shrinkage and to increase toughness and impact strength, thereby enhancing the fracture resistance of restored teeth. These composites have been improved as a base filling material in high–stress-bearing areas, especially in large cavities. They contain E-glass fiber made of aluminoborosilicate glass with less than 1 wt% alkali oxides. Nayar and others[14] have reported that E-glass fibers are able to maintain strength properties over a wide range of conditions and are relatively insensitive to moisture and are chemical-resistant. Garoushi and others[15] compared the physical properties and curing depth of a new short fiber–reinforced composite with that of conventional and bulk fill resin composites. They found that the fiber-reinforced composite exhibited higher fracture toughness and flexural strength and a lower percentage of shrinkage strain than did all other tested materials.

There are limited data about the fracture resistance of endodontically treated teeth restored with fiber-reinforced and bulk fill resin composites.[16,17] In order to learn more in this respect, the current study was conducted to investigate the fracture resistance of endodontically treated teeth restored with bulk fill, bulk fill flowable, fiber-reinforced, and nanohybrid composites. The null hypothesis was that there would be no statistically significant difference in the fracture resistance of endodontically treated teeth restored with different types of tooth-colored restorative resins.

METHODS AND MATERIALS

Seventy-two sound human maxillary premolars extracted for orthodontic purposes were used for the study. Any calculus and soft tissue deposits were removed from the teeth using a hand scaler. Each tooth was carefully examined under a light microscope at 20× magnification for any existing enamel cracks or fractures. Teeth of similar buccolingual and mesiodistal width in millimeters (buccolingual width: 8.47-10.59 mm; mesiodistal width: 6.38-8.19 mm) were selected by measuring with a digital micrometer (Series 480–505, resolution 1 μm, SHAN; Precision Measuring Instruments, Guilin, China) and allowing for a maximum deviation of 10% from the determined mean. The roots of the teeth were also similar in size and shape. The samples were stored in distilled water at 37°C for up to one month before use.

Standardized Class II MOD cavities were prepared with diamond burs (Diatech, Heerbrugg, Germany) that were replaced after every fourth cavity preparation. The gingival floor was 1.0 mm above the cemento-enamel junction (CEJ). The width of the cavities in the isthmus was one-third of the intercuspal distance, and the approximal box was two-thirds of the buccal palatal width. The cavosurface margins were prepared at 90°, and all internal line angles were rounded. The dimensions of the preparations were verified with a periodontal probe.

Standard endodontic access cavities were prepared using a high-speed handpiece. The pulp chamber roof was penetrated with a #2 diamond round bur (Dentsply, Tulsa Dental Specialties; Tulsa, OK, USA), then extended with a tapering cylinder bur; wall overhangs were removed with the same round bur. Thereafter, size 10 K files (Mani Inc, Tochigi, Japan) were inserted into the root canals until their tips could be seen at the apical foramen. The working length was determined by subtracting 0.5 mm from this length. The root canals were prepared using ProTaper rotary instruments (Dentsply-Maillefer, Ballaigues, Switzerland) up to master apical rotary size F3 (#30), in conjunction with 2 mL of 5.25% sodium hypochlorite irrigation between each file. Prepared root canals were rinsed with 5 mL of 17% ethylenediamine tetraacetic acid (Pulpdent Corporation, Watertown, MA, USA), followed by a final rinse with 5 mL of distilled water, and were then dried using paper points. Thereafter, the roots were filled with ProTaper F3 gutta-percha and AH Plus (Dentsply DeTrey, Konstanz, Germany) epoxy resin-based root canal sealer by single-cone technique. Excessive coronal gutta-percha was removed, and samples were stored in 100% humidity for seven days to allow the sealer to set. The endodontic access cavities were sealed with a thin layer of resin-modified glass ionomer cement (Novaseal, President Dental, Munich, Germany). A specialist in endodontics performed the endodontic treatments.

A universal metal matrix band/retainer (Tofflemire) was placed around each prepared tooth and supported externally by low-fusing compound to maintain adaptation of the band to the cavity margins.

The teeth were randomly divided into six groups of 12 teeth, as follows.

Group I

Group I comprised intact teeth without any cavity preparation; these teeth were used as negative controls.

Group II

Group II comprised MOD-prepared teeth only; these teeth were not restored and were used as positive controls.

Group III

For group III teeth, the cavities were etched for 30 seconds on enamel and for 15 seconds on dentin with 35% phosphoric acid (Scotchbond Universal Etchant, 3M ESPE, St Paul, MN, USA), rinsed for 15 seconds, and gently air-dried, leaving the tooth moist. The adhesive Single Bond Universal (3M ESPE), used in etch-and-rinse mode, was applied for 20 seconds; the solvent was air-dried for five seconds and then light-cured for 10 seconds by LED (Cromalux 1200, MegaPhysik, Rastatt, Germany; 1400 mW/cm^2). The cavities were restored with a bulk fill resin composite, Filtek Bulk Fill Posterior Restorative (3M ESPE). Each layer was approximately 5 mm thick

and was cured for 40 seconds with the same light-curing unit.

Group IV

For the teeth in group IV, after etching, as was done for the teeth in group III, a two-step etch-and-rinse adhesive, Prime&Bond NT (Dentsply/De Trey), was applied and remained fully wet for 20 seconds; teeth were then gently air-dried for five seconds and light-cured for 10 seconds. The cavities were filled with bulk fill flowable composite (SureFil SDR Flow, Dentsply) at up to 4 mm in thickness and were then cured for 40 seconds. The remaining parts of the cavities were restored with increments at a maximum of 2 mm in thickness using nanoceramic resin composite (Ceram.X Mono, Dentsply) and were light-cured for 40 seconds.

Group V

For the teeth in group V, a one-step self-etch adhesive, G-aenial Bond (GC Corp, Tokyo, Japan), was applied, remaining undisturbed for 10 seconds, and teeth were then dried for five seconds under maximum air pressure and light-cured for 10 seconds by LED. Fiber-reinforced composite (GC everX posterior, GC Corp) measuring approximately 4 mm in thickness was placed, and enough space was left for the overlaying composite on all surfaces of the restoration. The resin composite was cured for 40 seconds. The remaining parts of the cavities were restored with increments at a maximum of 2 mm in thickness using a posterior resin composite, G-aenial Posterior (GC Corp), and light-cured for 40 seconds.

Group VI

For the teeth in group VI, after etching, as in group III, a two-step etch-and-rinse adhesive, Excite F (Ivoclar/Vivadent, Schaan, Liechtenstein), was applied, agitated for 10 seconds, gently air-dried, and light-cured for 10 seconds. The cavities were restored with a conventional nanohybrid resin composite, Tetric N-Ceram, (Ivoclar/Vivadent), incrementally. Each layer was 2 mm thick and was light-cured for 40 seconds.

All restorative materials were used with their respective adhesive system. The materials for the restorative procedures are listed in Table 1. The restorations were finished with a high-speed handpiece under an air/water spray using diamond finishing burs (Diatech Dental AC). Subsequently, polishing was completed with polishing discs (Soflex, 3M ESPE) and rubber points. All preparations and restorations were performed by the same operator except in the case of the endodontic treatments. The specimens were stored in distilled water at 37°C for 24 hours. They were then subjected to thermocycling at between 5°C and 55°C (dwell time 30 seconds) for 1000 cycles (MTE 101 Thermocycling Machine, Esetron, Ankara, Turkey).

A coat of wax (0.2-0.3 mm) was applied to the external root surface of each tooth. All teeth were embedded in a block of self-curing acrylic resin up to 1 mm apical to the CEJ, with the long axis of the tooth perpendicular to the base of the block. Then the wax on the root surfaces was melted with boiling water, and this space was filled with polyvinyl siloxane impression material (Vinylight, BMS Dental, Pisa, Italy) to simulate periodontal ligament.

The specimens were submitted to compression in a universal testing machine (Instron, Lloyd, UK). A steel sphere 8 mm in diameter in contact with the occlusal slopes of buccal and palatal cusps was used to apply an occlusal load perpendicular to the long axis of the tooth at a crosshead speed of 1 mm/min. The load was applied until fracture occurred and was recorded in newtons (N).

Means and standard deviations were determined for each group, and data were statistically analyzed with one-way analysis of variance followed by the post hoc Tukey honestly significantly different test. Analyses were carried at the 5% significance level using SPSS 11.5 for Windows (SPSS Inc, Chicago, IL, USA).

The fractured specimens were examined under a stereomicroscope (40×) to evaluate the fracture patterns, which were classified as follows: mode I, minimal destruction of teeth; mode II, fracture of one cusp, intact restoration; mode III, fracture of at least one cusp, involving up to one-half of restoration; mode IV, fracture of at least one cusp, involving more than one-half of restoration; and mode V, severe fracture, involving tooth structure completely and/or longitudinal fracture.[18]

RESULTS

The mean fracture resistance values (N) and the standard deviations for each group are presented in Table 2. Sound premolar teeth (group I—negative control, 924.1 N) showed significantly higher fracture resistance than did the other tested groups ($p<0.05$). The lowest values were obtained in the positive control group (group II, 497.8 N), which were significantly lower than those of the other groups ($p<0.05$). No statistically significant differ-

Table 1: *Materials Used in the Study*

Product Name	Type	Manufacturer/Batch No.	Composition
Filtek Bulk Fill	Bulk fill posterior restorative	3M ESPE, St Paul, MN, USA/ N604746	Inorganic fillers, Bis-GMA, UDMA, Bis-EMA, procrylat resins, ytterbium trifluoride, zirconia/silica
Single Bond Universal	Universal adhesive (etch-and-rinse mode)	3M ESPE, St Paul, MN, USA/ 527687	MDP phosphate monomers, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, fillers, ethanol, water, initiators, silane
SureFil SDR Flow	Bulk fill flowable resin composite	Dentsply DeTrey, Konstanz, Germany/1207205	Barium and strontium alumino-fluoro-silicate glass, TEGDMA, modified UDMA, dimethacrylate, Bis-EMA, pigment, photoinitiator
Ceram.X Mono	Nanoceramic resin composite	Dentsply DeTrey, Konstanz, Germany/1203000406	Methacrylate modified polysiloxane, dimethacrylate resin, barium-aluminum-borosilicate glass, methacrylate functionalized silicon dioxide nanofillers
Prime&Bond NT	Nano-technology dental adhesive (two-step etch-and-rinse)	Dentsply DeTrey, Konstanz, Germany/1306000189	Di- and trimethacrylate resins, functionalized amorphous silica, PENTA, stabilizers, cetylamine hydrofluoride, acetone
everX Posterior	Fiber-reinforced resin composite	GC Co, Tokyo, Japan/1309121	Bis-GMA, TEGDMA, PMMA, triethylene glycol dimethacrylate, glass fillers and inorganic granular fillers
G-aenial Posterior	Posterior resin composite	GC Co, Tokyo, Japan/1211192	Methacrylate monomers, UDMA, dimethacrylate co-monomers, prepolymerized fillers, camphorquinone and amine, fluoroaluminosilicate, fumed silica
G-aenial Bond	One-component self-etch adhesive	GC Co, Tokyo, Japan/1401271	Phosphoric ester monomers, 4-MET, a hydrophilic methacrylate monomer, water, acetone, photoinitiator, nano-silica
Tetric N-Ceram	Nanohybrid resin composite	Ivoclar Vivadent, Schaan, Liechtenstein/S37370	Bis-GMA, urethane dimethacrylate, TEGDMA, barium glass, ytterbium trifluoride, silicon dioxide, mixed oxide, initiators, stabilizers, pigments
Excite F	Dental adhesive (two-step etch-and-rinse)	Ivoclar Vivadent, Schaan, Liechtenstein/P56445	Phosphonic acid acrylate, HEMA, dimethacrylate, highly dispersed silicone dioxide, initiators, stabilizers and potassium fluoride in an alcohol solution
Scotchbond Universal Etchant	Etching gel	3M ESPE, St Paul, MN, USA/ 524441	30%-40% Phosphoric acid, synthetic amorphous silica (fumed), polyethylene glycol, aluminum oxide, water

Abbreviations: Al, aluminum; Ba, barium; Bis-EMA, ethoxylated bisphenol A dimethacryrlate; Bis-GMA, bisphenol A glycidyl methacrylate;, DMA, dimethacrylate; HEMA, hydroxyethyl methacrylate; MDP, methacryloyloxy-decyl-dihydrogen-phosphate; PENTA, phosphonated penta-acrylate ester; TEGDMA, triethylene glycol dimethacrylate; UDMA; urethane dimethacrylate; PMMA, polymethyl methacrylate; 4-MET, methyl-N-ethyltryptamine

Table 2: *Means and Standard Deviations (SDs) of Fracture Resistance of Groups (n=12)*

Groups	Mean ± SD. N[a]
Group I (intact teeth) negative control	924.1 ± 87.8 A
Group II (MOD prepared teeth) positive control	497.8 ± 80.8 B
Group III (Bulk Fill Posterior Composite) Filtek Bulk Fill	702.4 ± 129.3 C
Group IV (Bulk Fill Flowable + Nanoceramic Resin Composite) SureFil SDR Flow + Ceram.X Mono	733.1 ± 143.7 C
Group V (Fiber Reinforced + Posterior Resin Composite) everX Posterior–G-eanial Posterior	685.7 ± 107.9 C
Group VI (Nanohybrid Resin Composite) Tetric N-Ceram	768.8 ± 91.5 C

[a] *Different letters indicate significant differences at level of significance $p<0.05$.*

Table 3: *The Frequency (%) of Failure Modes Among the Experimental Groups (n=12)*

Restored Groups	Mode I, n (%)	Mode II, n (%)	Mode III, n (%)	Mode IV, n (%)	Mode V, n (%)
Group III	—	8 (66.6)	1 (8.3)	—	3 (25)
Group IV	5 (41.6)	2 (16.6)	2 (16.6)	3 (25)	—
Group V	—	4 (33.3)	3 (25)	2 (16.6)	3 (25)
Group VI	2 (16.6)	1 (8.3)	4 (33.3)	1 (8.3)	4 (33.3)
Total	7 (14.5)	15 (31.25)	10 (20.8)	6 (12.5)	10 (20.8)

ences were found in the fracture resistance values of the restored groups (groups III, IV, V, and VI) ($p>0.05$). All groups showed lower values than the negative control group.

Table 3 illustrates the fracture pattern of the restored groups. None of the samples in group IV (bulk fill flowable + nanoceramic resin composite/ SureFil SDR Flow + Ceram.X Mono) showed severe fractures involving the tooth structure completely and/or longitudinal fracture (mode V). In most cases, mode II (fracture of one cusp, intact restoration) was observed. Representative images of different fracture modes are shown in Figure 1a-e.

DISCUSSION

In the present study, no difference was found in fracture resistance among different direct restorative materials. Therefore, the null hypothesis should be accepted. Endodontic treatment is considered to weaken teeth, resulting in their increased

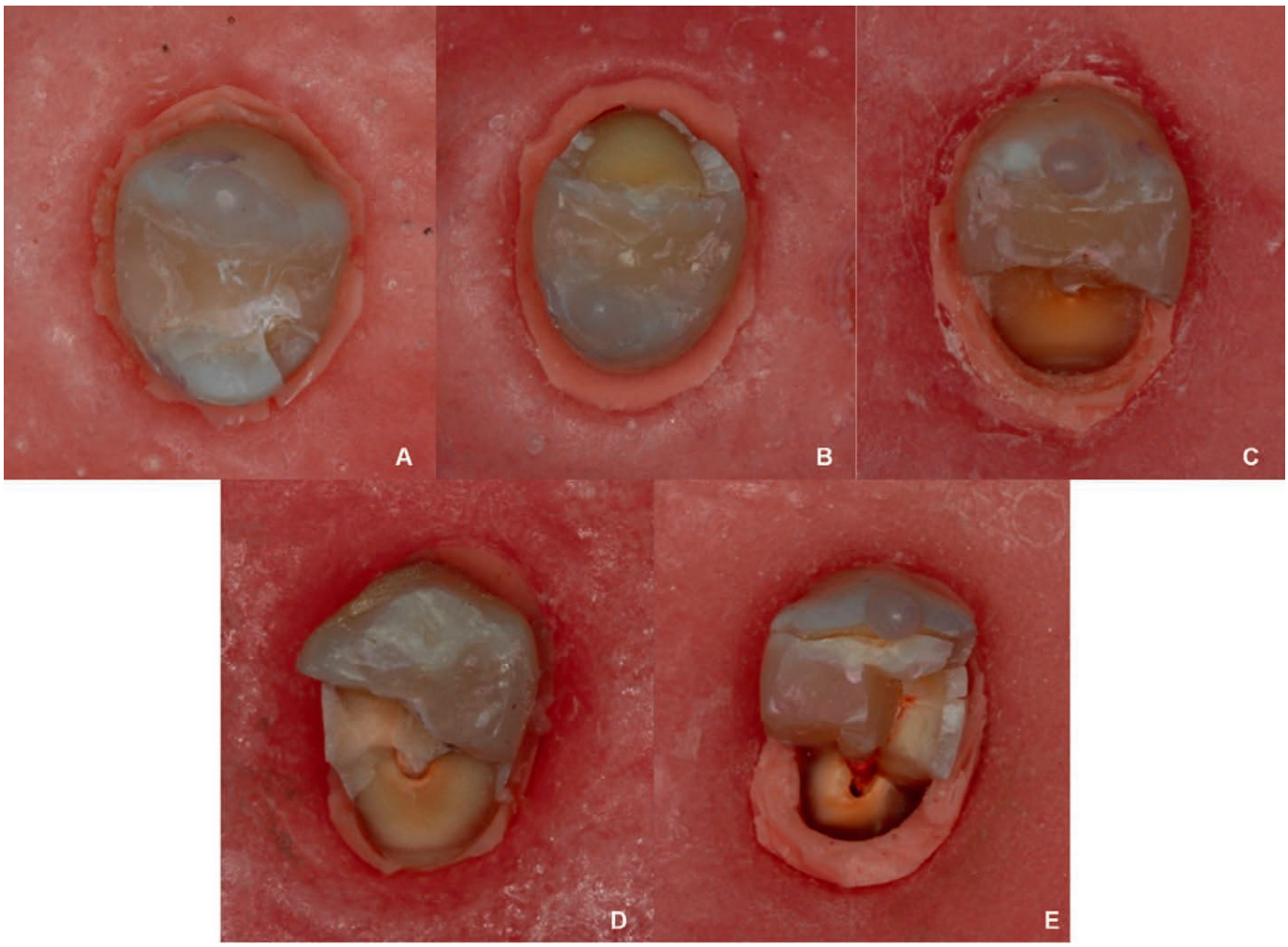

Figure 1. *Representative images of different fracture modes. (A) mode I; (B) mode II; (C) mode III; (D) mode IV; and (E) mode V.*

susceptibility to fracture. The selection of an appropriate restorative resin is primordial to yielding adequate resin/dentin bond strength and long-lasting restorations in endodontically treated teeth. As direct restorations are able to reinforce the weakened tooth structure,[19,20] the mechanical and physical properties of direct restorative materials, such as fracture toughness, modulus of elasticity, creep, hardness, and polymerization shrinkage, should be taken into consideration before restoration occurs.

Polymerization shrinkage stress, which may result in clinical problems such as fractures, is affected by the composition and filler content of resin composites and their elastic modulus.[21] An increase in the filler content would reduce polymerization shrinkage.[22] The manufacturers claim that bulk fill materials have lower volumetric polymerization shrinkage stress. In the present study, the polymerization shrinkage of the bulk fill resin composite Filtek Bulk Fill was 1.39%, which is slightly lower than that of the conventional resin composite Tetric N-Ceram, with its polymerization shrinkage of 2%. However, the filler content of the two products was similar (Filtek Bulk Fill, 64.5%; Tetric N-Ceram, 63.5% by weight).

Flowable resin composites act as an intermediate layer and stress-breaker. SureFil SDR Flow incorporates a polymerization modulator that offsets the inherent stress buildup that occurs during light polymerization.[23] In many studies,[24-27] the polymerization stress and cuspal flexure of SDR were found to be lower than those of other conventional flowable composites and comparable with those of low-shrinkage resin composites. Although the polymerization shrinkage of SDR (3.5%) was higher than that of the other tested restorative resins, their fracture resistance was similar. The materials' polymerization shrinkage is not the only factor involved in the development of contraction stress.[28,29] The positive results obtained might be related to the properties of lower flexural modulus and slower contraction rate.[24,30] On the other hand, it is known that bulk fill flowables require a 2-mm increment of a conventional resin composite because of their lower physical and wear properties. In the present study, Ceram.X Mono, with a polymerization shrinkage rate of 2.3, was used to cap flowable bulk fill composite. Its polymerization shrinkage value was quite similar to that of the other tested restorative resins. This might be a second reason for the similarity in fracture resistance values.

In a study conducted by Akbarian and others,[31] the fracture resistance of MOD cavity preparations restored with either low-shrinkage composite or with dimethacrylate-based composite in conjunction with or without cavity liners was compared. Although the silorane-based composite showed less volumetric shrinkage compared with dimethacrylate-based composites, the silorane composite showed resistance to fracture similar to that of the dimethacrylate-based composite. Kikuti and others[32] investigated the fracture resistance of teeth restored using methacrylate- and silorane-based composite restorations. The flexural strength and modulus of elasticity of both composites were also tested. The resistance to fracture of teeth to levels similar to that of the intact teeth group was found in the methacrylate-based resin group in which an etch-and-rinse system was used. On the other hand, in a recent study,[33] a significantly higher fracture resistance was recorded for teeth with MOD cavities restored with a low-shrinkage composite than for those restored with a conventional resin composite.

Use of a material with a low modulus of elasticity, especially in load-bearing areas, will result in a higher deformability under occlusal stresses.[10] In other words, the higher the filler content, the higher the modulus and the greater the resistance to deformation.[34] El-Damanhoury and Platt[30] found a significant correlation between stress and flexural modulus and between stress and filler loading by volume. In the present study, in spite of the tested restorative materials' different modulus of elasticity values (varying in the range of 85 to 124 GPa), their resistance to fracture was similar. Another study[32] found that fracture resistance was not related to elastic modulus. In that study resin composite with higher flexural strength and elastic modulus showed higher fracture resistance than did a composite with lower flexural strength and elastic modulus when used with an etch-and-rinse adhesive system. However, when used with a self-etch adhesive, no difference in fracture resistance was reported between the tested resin composites.

Our findings are in agreement with a study by Toz and others[17] that investigated the fracture resistance of endodontically treated teeth restored with bulk fill flowable and bulk fill resin composites. In their study no difference was found between groups restored with bulk fill flowable composite bases and conventional resin composite. Yasa and others[16] evaluated the fracture resistance of endodontically treated teeth restored with nanohybrid composite, bulk fill flowable composite, and short fiber–rein-

forced composite in the presence/absence of retention slots. Similar to our findings, no difference was observed between the restorative materials in the absence of retention slots.

everX fiber-reinforced resin is used in conjunction with a conventional resin composite. The manufacturers claim that this material prevents or arrests crack propagation. In a recent study[15] that examined the physical properties of a short fiber composite material in comparison to different bulk fill and conventional resin composites, fiber-reinforced resin showed higher fracture toughness and flexural strength than did all other materials tested. Moreover, shrinkage strain was found to be the lowest. They attributed these results to the plasticization of the polymer matrix by linear polymer chains of PMMA in the cross-linked matrix of bisphenol A diglycidyl ether dimethacryalate–triethylene glycol diemthacrylate, which increases the fracture toughness and stress transfer from polymer matrix to fibers, inducing a reinforcing effect of fibers. In another study[35] conducted by the same author, short fiber fillers' preventive effect on crack propagation and improvement of fracture resistance was reported. A recent study[36] compared nonreinforced resin composite with reinforced composites and it was found that fiber reinforcement improved the fracture resistance of composite resin. In a study[37] evaluating the efficiency of a short fiber–reinforced resin composite material compared to conventional composites when restoring Class II MOD cavities in molar teeth, the use of a short fiber–reinforced resin composite did not result in a statistically significant increase in fracture toughness; however, when using this material with an oblique layering technique, a clear tendency toward higher fracture resistance and restorable fractures was observed.

In the present study, the mean fracture resistance values of teeth restored with everX fiber-reinforced resin were not significantly different from those of teeth restored with other restorative materials. This contradictory finding might be attributed to the critical difference in sample preparation. In the studies mentioned above, restorative resins were placed in fabricated molds instead of in prepared teeth. A prepared tooth with a cavity has a certain degree of compliance. The different results might also be related to the adhesive system used. It is known that dentin adhesives play an important role in maintaining the bond between the cavity walls and restorative material. In the present study, all restorative materials were used with their respective adhesive system. All adhesive systems were etch-and-rinse, except for everX fiber-reinforced resin and G-aenial posterior's adhesive, which was a one-step self-etch, G-aenial Bond. Although the bond strength values of one-step self-etch adhesives are comparable with those of etch-and-rinse adhesives, they have inferior enamel bond strengths compared to etch-and-rinse systems.[38] Moreover, G-aenial Bond was the only adhesive system that was 2-hydroxyethyl methacrylate–free. The hydrophobic nature of the components and phase separation[39] might cause a decrease in bond strength and, thereby, in fracture resistance.

Intact teeth showed the highest fracture resistance, which is consistent with the findings of many studies[40-42] reporting that restored teeth had a significantly lower resistance to fracture.

In the present study, the majority of fractures, regardless of the type of restoration, were type II, in which the restoration was intact with a cuspal fracture. In other words, they were defined as restorable. This finding is in contrast to the studies by Yasa and others[16] and Toz and others,[17] who reported most fractures as nonrestorable in endodontically treated teeth. However, these differences might be related to the fracture mode classification system used. In the studies mentioned, fracture modes were classified as restorable when the fracture line was above the CEJ or 1 mm or less apical to the CEJ, or as nonrestorable (including vertical root fractures) when the fracture line was more than 1 mm apical to the CEJ.[43] In the present study, however, a more detailed fracture mode classification system was used. Therefore, it might not be possible to compare our obtained results with those of the studies mentioned.

CONCLUSIONS

Within the limitations of this study, the fracture resistance of teeth restored with a conventional nanohybrid resin composite was not significantly different from that of either bulk fill/flowable bulk fill or fiber-reinforced resin restoratives. Compared to intact teeth, restored teeth had lower fracture resistance. However, the results should be validated with additional clinical studies as physiological and parafunctional occlusal forces were not taken into account in these *in vitro* conditions.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subject's oversight committee guidelines and policies of Hacettepe University. The approval code for this study is 15/616-10.

Conflict of Interest

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

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Water Sorption and Solubility of Luting Agents Used Under Ceramic Laminates With Different Degrees of Translucency

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Clinical Relevance

Degrees of translucency in a restorative material are important for masking tooth color alteration. Clinicians must be aware of the relationship between a decrease in translucency and loss of light penetration to avoid the clinical degradation of an improperly cured luting material.

SUMMARY

Purpose: The aim of this study was to evaluate the effect of low-thickness ceramic laminate translucency on water sorption and solubility in resin luting agents.

Methods and Materials: Ceramic slides (15×0.7 mm) were generated using lithium disilicate (IPS e.max Press, Ivoclar-Vivadent, Schaan, Liechtenstein) that were A1 in color and had decreasing degrees of translucency (high, medium, and low). A slide of transparent glass of similar size was used as the control. Under each slide, 15 specimens (8×0.5 mm) of differing composite materials from the same manufacturer (3M ESPE Dental Products, St Paul, MN, USA) were prepared (n=5): light-cured resin cement (RelyX Veneer); dual-cured resin cement (RelyX ARC); and flowable composite (Z350XT Flow). To evaluate the loss or gain of mass, the specimens were dried until a constant mass was reached. Then, they were immersed in water for seven days and weighed immediately following removal from water. Subsequently, the speci-

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mens were dried again until a constant mass was obtained. The mass measurements were used to calculate the water sorption and solubility. Statistical analyses were carried out using a two-way analysis of variance and the Tukey test.

Results: Under the high-translucency ceramic slides, all of the luting agents showed similar performance regarding water sorption; the flowable composite resin and the light-cured resin cement had the lowest solubility values. Under the medium- and low-translucency surfaces, the dual-cured resin cement and the flowable composite resin showed better performance with respect to water sorption and solubility.

Conclusions: In the case of high-translucency laminates, luting agents with different activation methods might be used. However, even in thin sections, decreasing the translucency of the laminate led to significant loss of light penetration, indicating a decreased likelihood of the physical activation of the resin cement.

INTRODUCTION

The search for beautiful and harmonious smiles has increased the frequency of the use of ceramic laminates, which are an esthetic alternative that may involve minimal tooth preparation.[1,2] The cementation protocol is of fundamental importance in these minimally invasive preparations because the success of ceramic restorations will be determined in large part by obtaining a strong and durable bond among the cement, ceramic material, and dental tissues.[3]

For the cementation of ceramic laminates, light-cured (photoactivated) or dual-cured resin agents are commonly used, largely due to their ability to adhere to dental tissues and their satisfactory mechanical properties.[4] Among the light-cured resins, flowable composites have also been presented as options because their molecular composition, which is similar to a hybrid compound, allows for good mechanical resistance.[5,6]

A lower concentration of tertiary amines in flowable composites and light-cured resin cements appears to offer greater color stability, allowing better long-term esthetic results.[5,6] However, light-cured materials rely on visible light and an efficient photoinitiator system for polymerization of the materials to occur in an effective manner so that their maximum physical and mechanical properties can be achieved.[7]

With improvements in restorative techniques, higher esthetic refinements have been developed, resulting in the production of ultrafine ceramic laminates that are associated with minimum dental wear.[1] However, a significantly reduced thickness may hinder the ideal masking of color alterations in enamel and dentin. Therefore, ceramics of greater opacity become an option to allow more satisfactory esthetic results. However, increased thickness has been associated with alteration in the passage of light through the ceramic,[8] and the translucency of the laminate might also interfere with this process.

Clinically, the margins of indirect restorations are often placed close to the gingival sulcus and in contact with the sulcular fluid. Failures in these restorations can be observed due to deficiencies of polymerization and impairment of the mechanical properties of the cement related to the influence of humidity.[9,10] Most of the monomers used in dental resin materials can absorb water and chemicals from the environment and also release components into the surrounding environment.[11] Both the fluid uptake into the resin phase and the dissolution of the composite may have detrimental clinical consequences. An inappropriate polymerization might influence the degree of degradation of a composite material as well as microstructural and molecular aspects, presence of pendant hydroxyl groups capable of forming hydrogen bonds with water, degree of cross-linking of the continuous matrix, presence of residual water-attracting species, and type, dimension, volume, diffusivity, and solubility of filler particles.[10] Therefore, understanding the dynamics of diffusion in resin cements by considering the properties of water sorption and solubility is important for predicting the resins' clinical behavior, especially their stability, because these factors have direct effects on the longevity of adhesively cemented restorations.[10,11]

In view of the aforementioned considerations, there is a hypothesis that the translucency of a ceramic, even at a low thickness, will interfere with the polymerization of the cements to such a degree that it will change the water sorption and solubility of this material; similarly, the type of luting agent might also influence the outcome. Thus, the objective of this study was to evaluate the water sorption and solubility of light-cured or dual luting agents using ceramic slides of different translucencies.

Table 1: Luting Materials Used and Their Compositions

Luting Material	Composition
Dual-cured resin cement, RelyX ARC (3M ESPE Dental Products, St Paul, MN, USA)	Bis-GMA and TEGDMA monomers. Particles of zirconia/silica with an average size of 1.5 μm. Paste A: pigments and tertiary amine. Paste B: benzoyl peroxide. Filler loading 68% by weight.
Light-cured resin cement RelyX Veneer (3M ESPE Dental Products, St Paul, MN, USA)	Bis-GMA and TEGDMA monomers. Particles of zirconia/silica and colloidal silica. Average particle size of 0.6 mm. Filler loading 66% by weight.
Flowable composite resin Z350XT Flow (3M ESPE Dental Products, St Paul, MN, USA)	Bis-GMA, TEGDMA, and Bis-EMA monomers. Silica nanoparticles of 75 nm, zirconia nanoparticles of 5-10 nm, zirconia/silica nano agglomerates with aggregate particle size ranging from 0.6-1.4 μm. Filler loading 65% by weight.

Abbreviations: Bis-EMA, bisphenol-A ethoxylated dimethacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate.

METHODS AND MATERIALS

Preparation of Specimens

Following the manufacturer's recommendations, ceramic slides of lithium disilicate (IPS e.max Press, Ivoclar-Vivadent, Schaan, Liechtenstein) were fabricated to a 0.7-mm thickness and a 15-mm width in shade A1 and with decreasing degrees of translucency, including high (H), medium (M), and low (L). A glass slide of the same dimensions was used as a control to simulate the maximum passage of light energy to the luting agent.

Then, a matrix of polyvinyl siloxane (mA) was prepared (Elite, Zhermack, Badia Polesine, Italy) with an internal orifice, which was 0.5 mm in depth and 8 mm in diameter. This mold was used to accommodate the cement during the preparation of the specimens subjected to water sorption and solubility testing.

Another matrix of polyvinyl siloxane (mB) was prepared to adapt to the ceramic slides. The function of this device was to prevent the dissipation of light at the time of polymerization and also to prevent the interference of external light.[8]

The luting agents and their formulations are described in Table 1. Materials with similar colors (A1/light yellow) were selected. The 12 experimental groups (n=5) were formed as described in Figure 1.

To prepare each specimen, each luting agent was inserted into the matrix of polyvinyl silicone (mA), and a strip of polyester was placed over it to accommodate the material and maintain a smooth and even surface. Then, a ceramic slide and the other mold (mB) were positioned on top of this assembly. A glass slide was positioned on top of the second mold to force excess material outward for removal. No hand pressure was exerted. Finally, polymerization was performed with a light-emitting diode source (Radii Plus, SDI, Victoria, Australia) for an exposure time of 40 seconds, intensity of 1500 mW/cm^2, and peak wavelength of 470 nm (Figure 2). The light intensity of the light-emitting diode source was monitored using its built-in radiometer.

Water Sorption and Solubility Evaluation

After the removal of the matrix, the specimens were placed individually in a dark environment to prevent further polymerization until they were ready for water sorption and solubility testing based on ISO 4049: 2000 specifications.[12]

After preparation of the 60 specimens (n=5 per group), their thicknesses were measured using a digital caliper with a precision of 0.01 mm, and the measurements were used to calculate the volume of each specimen (mm^3). Both diameter and thickness were measured following a standardized method. A mean diameter for each specimen was calculated from two measures at right angles to each other. A mean thickness for each specimen was calculated from four measures performed at equally distributed points on the circumference.

Shortly thereafter, all the specimens were placed in a desiccator and transferred to an incubator at 37°C for preconditioning. After 24 hours, the speci-

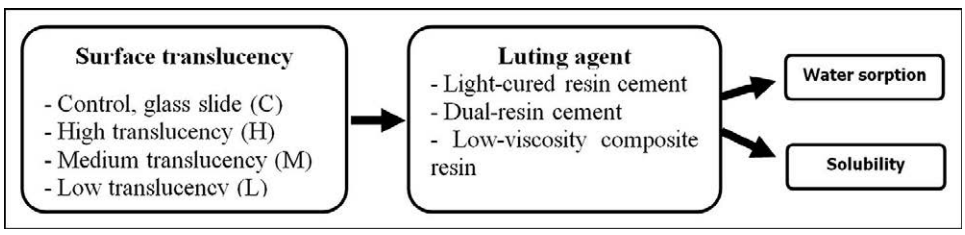

Figure 1. *Distribution of the experimental groups.*

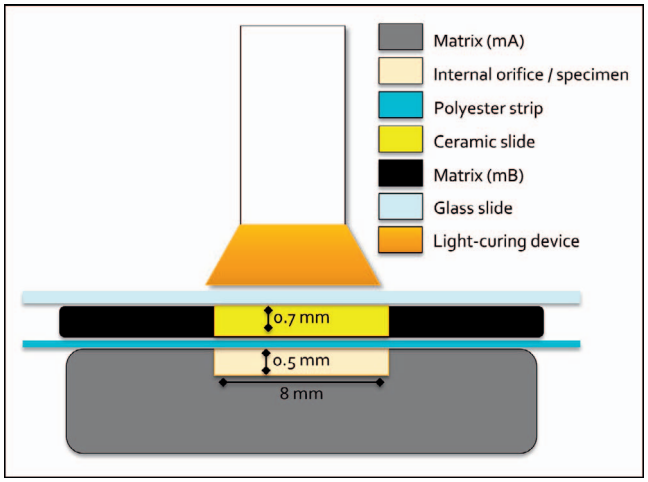

Figure 2. *Illustration of the experimental apparatus for the preparation of specimens.*

mens were weighed on an analytical balance (Analytical Plus, Ohaus Corporation, Florham Park, Switzerland) with an accuracy of a tenth of a thousandth of a gram.

The specimens were weighed repeatedly at intervals of 24 hours until a constant mass was reached (m1; less than 0.2-mg variation in a 24-hour period). After stabilization of the mass at m1, the specimens were stored individually in closed flasks containing 2 mL of distilled water (pH 7.2) in a 37°C incubator for seven days. Following the storage period, the specimens were weighed again to determine the value of m2. For this step, after removal from water, the specimens were washed in running water, and excess liquid was removed with absorbent paper until moisture could no longer be observed. The weight was annotated (m2), and the specimens were positioned in dry and open flasks and then placed in a desiccator containing silica gel in an incubator at 37°C to eliminate the absorbed water. The samples were weighed daily until reaching a constant mass (m3), as described for m1 and m2. The initial mass determined after the first desiccating process (m1) was used to calculate the mass variation during the seven days of storage in water. The water sorption (WS) and solubility (Sol) for the seven days of storage in water were calculated using the following formulas:

$$WS = (m2 - m3)/V; \qquad Sol = (m1 - m3)/V,$$

where, m1 is the mass of the sample in micrograms before immersion in distilled water, m2 is the mass of the sample in micrograms after immersion in distilled water for seven days, m3 is the mass of the sample in micrograms after being conditioned in a desiccator with silica gel, and V is the volume of the samples in millimeters cubed.[11]

Statistical Analysis

Exploratory analyses of the water sorption and solubility data ($\mu g/mm^3$) were performed to verify the parameters of the analysis of variance (ANOVA). The inferential statistical analysis was performed using a two-way ANOVA and a Tukey multiple comparison test with the statistical software SAS, version 9.1 (SAS Institute Inc, Cary, NC), with a significance level of 5%.

RESULTS

Table 2 shows the means and standard deviations of the water sorption and solubility data obtained under the experimental conditions tested in this study. The statistical analysis found that the two-way interaction between the translucency of the ceramic and the luting agent was significant for both

Table 2: *Mean Values (Standard Deviation) of Water Sorption and Solubility ($\mu g/mm^3$)[a]*

Luting Agent	Translucency of the Ceramic			
	Glass Slide (Control)	High Translucency (H)	Medium Translucency (M)	Low translucency (L)
Water sorption (WS)				
Dual-resin cement	26.22 (2.93)Ab	30.11 (1.73)Ab	27.07 (2.31)Bb	33.86 (4.77) Ba
Light-cured resin cement	27.78 (1.90)Ab	25.90 (3.93) Ab	38.40 (3.83) Aa	42.55 (5.04)Aa
Flowable composite resin	27.36 (2.11) Aa	24.87 (2.23) Aa	24.48 (1.99) Ba	28.32 (1.23) Ba
Solubility (Sol)				
Dual-resin cement	2.91 (0.12) Ab	6.86 (0.17) Aa	5.84 (0.46) Ba	7.87 (1.44) Ba
Light-cured resin cement	3.20 (0.31)Ab	3.50 (1.24) Bb	10.73 (1.63) Aa	12.60 (2.25) Aa
Flowable composite resin	2.96 (0.20)Ac	3.22 (0.20) Bc	6.29 (0.23)Bb	9.77 (0.69)Ba

[a] *Letters and symbols represent different means with statistical significance (two-way analysis of variance/Tukey test, α=5%). For each variable, capital letters compare the levels of the luting agent within each level of translucency of the ceramic. Lowercase letters compare the translucency of the ceramic within each level of the luting agent. Coefficient of variation: 10% (WS) and 16% (Sol).*

the water sorption ($p<0.001$) and the solubility ($p<0.001$) data. Thus, the interaction of each factor with the levels of the others was demonstrated, and this interaction was parsed using the Tukey test.

Controlling for the statistical interaction of the water sorption data, the luting agents showed similar patterns under the high-translucency and control (glass slide) surfaces. However, under the intermediate- and low-translucency surfaces, the light-curing resin cement showed a water sorption value that was statistically superior to the others. The data analysis also indicated the amount of influence that the type of surface had on water sorption for the dual and light-cured resin cements. Both presented higher amounts of water sorption for the low-translucency surfaces. However, the type of surface did not result in a statistically significant difference for the low-viscosity resin.

Among the solubility data, an interaction between the luting agent and the surface translucency was also observed. For the intermediate- and low-translucency ceramic specimens, the light-cured resin cement showed statistically superior values compared with the other resins. In the high-translucency ceramic, the light-cured agents showed the lowest solubility values, but in the control condition, the results were all similar. Comparison of the surfaces indicated a direct relationship between the low-translucency ceramic and the highest solubility values for all luting agents, whereas the high-translucency ceramic and the glass slide had the lowest values for the light-cured resin cement and the low-viscosity composite resin. For the dual-resin cement, only the control resulted in a lower solubility.

DISCUSSION

Inadequate polymerization of resin cements under ceramic restorations may be related to an insufficient amount of light radiation passing through the restorative material to reach and activate the monomers.[8] Among other factors, the amount of light transmitted for the conversion of resin cements may decrease depending on the optical characteristics of the ceramic materials, such as refractive index and translucency.[8,13,14]

A previous study indicated that resin cements can be light-cured under lithium disilicate–based ceramics with thicknesses of up to 2 mm without any interruption to the curing process.[15] Lee and others[13] evaluated the microhardness of light-cured and dual-resin materials under ceramics of different thicknesses and concluded that the smallest thickness exerts less influence on the properties of the materials. In the present study, ceramic slides had a thickness similar to conservative veneers (0.7 mm); thus, little interference from the lithium disilicate glass-ceramic was expected.

Nonetheless, in conservative preparations, ceramics with different degrees of translucency are commonly used to block possible interference by the color of the dental substrate with the final result of the restoration. Thus, the hypothesis of this study suggested that, even at a low thickness, more-opaque surfaces would be able to prevent complete polymerization of the luting agent, resulting in a low degree of conversion and interfering with the dynamics of diffusion (water sorption and solubility) of these materials.[5,8,16-18]

According to the results of this study, the water sorption and the solubility of the light-cured luting agents under high-translucency surfaces were similar to the control condition, indicating that this type of surface did not prevent the transmission of light and allowed proper conversion of the monomers.[5,15] In contrast, for the low- and intermediate-translucency surfaces, the light-cured resin cement showed water sorption and solubility values that were higher than those of the other resins. This finding is most likely related to the passage of light through the ceramic,[8] indicating that the degree of dilution of the resin cement components in water is higher when the compound is not properly light-cured.[19] When a light-cured material does not receive the appropriate amount of energy density, one would expect an impaired formation of free radicals to initiate polymerization and a lower degree of conversion of the polymer network.[20] Highly cross-linked polymers seem to be more resistant to dissolution, whereas linear polymers present more space and pathways for solvent molecules to diffuse within their structure.[20]

The gain of water seems to be significantly related to the material composition, content and concentration of inorganic fillers, and size and nature of the particles.[21,22] In the present study, the translucency of the surface did not significantly affect the water sorption of the flowable composite. This material differs from the light-cured resin cements because it has bisphenol-A ethoxylated dimethacrylate (Bis-EMA) associated with bisphenol-A glycidyl methacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) in the resin matrix. Bis-EMA is more hydrophobic, hampering the entry of water and, in combination with the nanoparticles of the

flowable composite resins, may be associated with an improvement in the physical properties of the material.[19,23] Given that water sorption is a phenomenon in which water enters the resin matrix through direct diffusion via empty spaces that have been incorporated into the material,[21,22] it is possible that the presence of nanoparticles in the flowable composite resin filled such spaces, and the association with hydrophobicity of the resin matrix culminated in a decrease in the sorption values.

Although Archegas and others[5,6] analyzed different properties than the present study such as color stability, degree of conversion, and hardness, they observed the best results for flowable composite resins compared with other luting agents. In addition, the authors found that the opacity of the ceramic did not result in a statistically significant influence on the degree of conversion of the flowable composite resin.

In contrast, as with the light-cured resin cement, the flowable composite resin showed significantly higher solubility with the low- and medium-translucency ceramic slides. This phenomenon is characterized by the loss of resinous material components by dissolution, mainly of unreacted monomers during the polymerization of filler particles, resulting in a reduction in weight and volume.[21,24]

An explanation for this finding may be the poor quality of the polymers formed under conditions of low light passage, possibly leaving weak bonds that facilitate leaching. This hypothesis is supported by a previous study that showed that an inadequate conversion resulting from low light intensity can adversely affect the performance of the restoration.[5] Failures related to the polymerization reaction can result in the formation of a poorly structured polymer chain with easily breakable bonds.[25,26]

The dual-resin cement was used in this study as a control for the effect of the passage of light through the ceramic because chemical activation of this agent could compensate for the loss of light intensity.[8] However, according to the results of this study, even with the presence of the chemical activation system, the dual-resin cement suffered the negative effects of the limitation of the passage of light, indicating that proper photoactivation has a fundamental role in improving the physical properties of this material.

In a previous study comparing the hardness of luting agents in ceramics of different thicknesses and opacities, it was observed that even the chemical component of a dual-resin cement was not able to promote complete polymerization under an opaque surface,[15] resulting in a reduction of the mechanical properties of flexural strength, elastic modulus, and hardness. These results demonstrate the importance of light and photosensitive components in this cement.[6,7]

The light intensity is another factor that may be associated with the correct conversion of the monomers. Variations in the power of the device used will directly influence the mechanical properties of the material, demonstrating the need to work at maximum intensity.[14,17,25]

In most of the conditions tested, an association between the water sorption and solubility values was observed, corroborating a previous study.[18] Therefore, a possible directly proportional relationship between the amount of absorbed water and the amount of components leached may be assumed.

According to the American Dental Association's[27] specification No. 27, the sorption of resin materials must be less than 40 $\mu g/mm^3$ and the water solubility must be less than 7.5 $\mu g/mm^3$ for a storage period of seven days. In this study, only the light-cured resin cement showed a water sorption value significantly higher than the acceptable limit when it was under a low-translucency surface. Regarding solubility, the light-cured resin cement showed mean values higher than the acceptable limits when it was under medium- and low-translucency surfaces. In contrast, the flowable composite resin and dual-cured resin cement presented values above the acceptable limit only under the low-translucency surface. This result justifies careful attention in the cementation of ceramics that allow less passage of light.

In spite of the relevance of the reported findings, it should be considered that *in vitro* studies have some limitations. Important covariants, such as the thickness of the luting agent and changes in the pH of the oral cavity, were not simulated in this study, and thus the *in vitro* water sorption and solubility test are considered static.[28,29] Another limitation of this investigation was that materials from the same manufacturer were selected in order to standardize resin matrixes so compositional changes would not be of more importance than light exposure. Nevertheless, it should be considered that *in vitro* methods are good tools to generate clinical evidence due to the simplicity of implementation and the possibility of satisfactorily reproducing the oral environment.

In light of these findings, dentists need to be concerned with the color of the ceramic used as well as with its translucency because the latter will also

interfere with the transmission of light to the luting agent, influencing the mechanical properties of these materials. Another consideration is that in spite of the strong commercial appeal of the restorative materials' industry, there are some products that might have a broader clinical application, such as the flowable restorative composites. These results are of fundamental importance; however, they do not preempt the implementation of new, long-term clinical studies.

CONCLUSIONS

Based on the observations described in the previous section, there is a relationship between ceramic translucency and luting agent water sorption and solubility for all materials tested. Therefore, for surfaces with greater translucency, the luting agents studied provide acceptable values of water sorption and solubility. In contrast, for surfaces with lower translucency, dual-cured resin cements or flowable composite resins should be preferred. These findings are preliminary, and further studies should be performed to confirm and expand our results.

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Conflict of Interest

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

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Fractographical Analysis and Biomechanical Considerations of a Tooth Restored With Intracanal Fiber Post: Report of the Fracture and Importance of the Fiber Arrangements

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Clinical Relevance

When restoring anterior endodontically treated teeth, fiber posts with parallel fibers support tensile stresses, but they commonly fracture by shear stresses due to anterior occlusal oblique loads that generate bending of the restorative assembly.

SUMMARY

Objective: This article aims to present a fractographic analysis of an anterior tooth restored with a glass fiber post with parallel fiber arrangement, taking into account force vectors, finite element analysis, and scanning electron microscopy (SEM).

Methods: A patient presented at the Faculty of Dentistry (Federal University of Santa Maria, Brazil) with an endodontically treated tooth (ETT), a lateral incisor that had a restorable fracture. The treatment was performed, and the fractured piece was analyzed using stereomicroscopy, SEM, and finite element analysis.

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Results: The absence of remaining coronal tooth structure might have been the main factor for the clinical failure. We observed different stresses actuating in an ETT restored with a fiber post as well as their relationship with the ultimate fracture. Tensile, compression, and shear stresses presented at different levels inside the restored tooth. Tensile and compressive stresses acted together and were at a maximum in the outer portions and a minimum in the inner portions. In contrast, shear stresses acted concomitantly with tensile and compressive stresses. Shear was higher in the inner portions (center of the post), and lower in the outer portions. This was confirmed by finite element analysis. The SEM analysis showed tensile and compression areas in the fiber post (exposed fibers=tensile areas=lingual surface; nonexposed fibers =compression areas=buccal surface) and shear areas inside the post (scallops and hackle lines). Stereomicroscopic analysis showed brown stains in the crown/root interface, indicating the presence of microleakage (tensile area=lingual surface).

Conclusion: We concluded that glass fiber posts with parallel fibers (0°), when restoring anterior teeth, present a greater fracture potential by shear stress because parallel fibers are not mechanically resistant to support oblique occlusal loads. Factors such as the presence of remaining coronal tooth structure and occlusal stability assist in the biomechanical equilibrium of stresses that act upon anterior teeth.

INTRODUCTION

The preservation of the remaining coronal structure has emerged as a crucial aspect for the clinical success of post-retained restorations and seems to be more important than the post choice.[1-8] Clinical trials have confirmed the strong scientific evidence that, irrespective of restorative technique of pulpless teeth, the preservation of at least one coronal wall and 2 mm of ferrule to post placement significantly reduces the clinical failure risk.[6,9,10]

Several available post systems have been proposed for the rehabilitation of endodontically treated teeth (ETT). It is known that cast posts and cores are associated with high rates of irreversible fractures[11,12] because they transfer more stress to the root dentin compared with fiber posts.[13-16] Fiber posts have an elastic modulus similar to that of dentin and are usually associated with repairable failures[17,18] because they more homogeneously distribute stress along the root and thereby prevent root fracture.[13,19-21]

Given that the use of fiber posts has increased, it is important to assess how the magnitude and direction of functional loads play a major role in the concentration of stress within teeth restored with posts.[22,23] Horizontal loads lead to a significantly higher concentration of stress within dentin than loads parallel to the long axis of the tooth.[24-28] Because the loads are applied at different levels along the dental arch, anterior teeth are most likely to be subjected to more horizontally directed loads due to their inclination in relation to posterior teeth.[23,29] For example, a force applied at an angle of 90° to the anterior teeth causes the appearance of tensile and compressive stresses,[30] which can cause damage to teeth restored with posts because these teeth have weaker supporting structures.

In addition, resolution of the load applied at 45° into force vectors using fractographical analysis facilitated the modeling of the specific features of stress (tensile, compression, and shear) found in an anterior ETT fractured after mechanical cycling.[31] These data are corroborated by the results of another study that found that the elastic modulus of a post, with regard to concentration, magnitude, and direction of dentinal stress, was dependent on the direction of the applied load.[14] When examining models of posts with a high modulus, it was found that horizontal loads led to more stress on the apical area of the root; such loads suggested a vertical root fracture. On the other hand, when low-modulus posts were modeled, forces at 45° and 90° caused more stress on the cervical area, with a direction that suggests debonding of the post.

The direction of the applied force and the fiber arrangement of fiber-reinforced polymeric materials (FRP) directly influence their mechanical properties.[32] Glass fiber posts (classified as a FRP) generally present longitudinal fibers (parallel fibers, 0°) distributed inside the polymeric matrix, so they can support high tensile stress when the forces are applied along their central axis.[33] However, when oblique forces (45°) are applied (anterior teeth), shear stresses are induced inside the polymer, leading to fracture by lower loads than when only tensile stresses are generated.[32-34] Recently, *in vitro* studies showed that shear stresses can be as harmful as tensile and compression stresses when oblique forces are applied in ETT restored with fiber

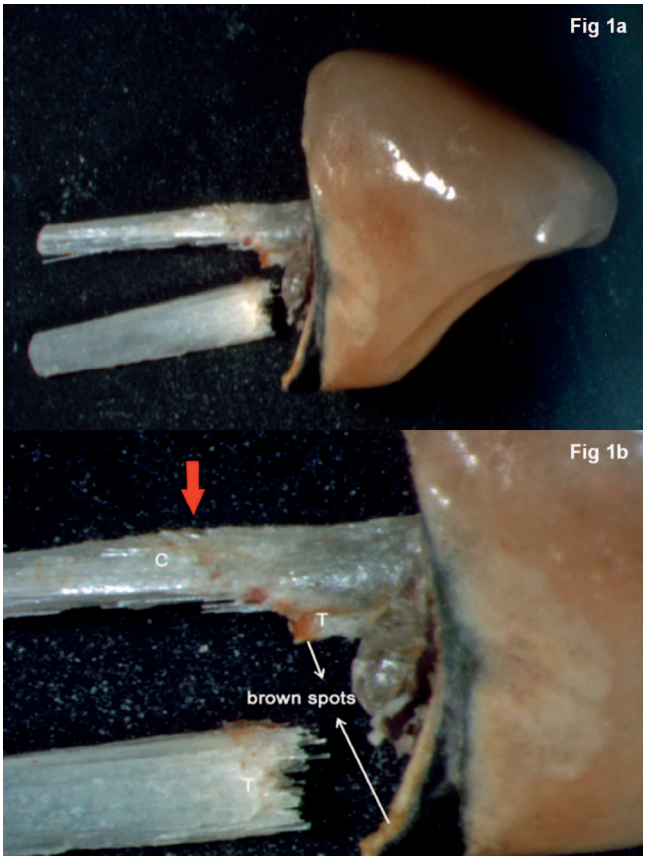

Figure 1. *Representative images of the fractured assembly. a: the fiber post fractured wherein one piece was broken (area submitted to tensile stresses) and the other was attached to the core/crown part (area submitted to compressive stresses). b: tensile (T) and compressive (C) regions. In the buccal region there is a "kneading" of the fiber post (red arrow: superficial fibers exposed and mild bulge in the outer surface).*

posts[31,35] or when only glass fiber posts were submitted to static[33,34] and fatigue loads.[33]

Thus, our aim was to perform a fractographical analysis of an endodontically treated upper lateral incisor restored with a glass fiber post and a metal-ceramic crown that fractured after three years of clinical service and compare that with *in vitro* findings available in the literature. We sought to assess the forces exerted on the fractured tooth with both a finite element and a scanning electron microscopy (SEM) analysis as well as to validate the *in vitro* findings.

METHODS

Case Description

A 49-year-old man presented at the Division of Prosthodontics with a fracture in the upper left lateral incisor, incurred while eating. Previously, on September 28, 2010, the patient had been referred to the Division of Prosthodontics of the Faculty of Dentistry with a large coronal fracture in the tooth, which had already been endodontically treated. A detailed anamnesis was performed. The patient had good general and dental health but was missing the maxillary right second premolar, first molar and second molar, as well as the maxillary left first premolar, second premolar, and first molar. The initial focus of the treatment was on the rehabilitation of the fractured tooth, followed by replacement of the other lost teeth. After clinical and radiographic examinations, we proposed restoration of the lateral incisor with an intraradicular post and metal-ceramic crown as well as a removable partial denture (RPD) for the maxillary posterior segments. The fractured lateral incisor presented two proximal contacts, was a sound tooth antagonist with periodontal support, had a mobility grade of 0, and had a remaining root length of 16 mm.

Rehabilitation consisted of the cementation of a glass fiber post (White Post DC, FGM, Joinville, Brazil) using a self-adhesive cement (RelyX U100; 3M ESPE, St Paul, MN, USA). It was cemented at a 10-mm length, with a 6-mm coronal length and 1.6-mm coronal diameter.

On January 3, 2013, the patient sought assistance again due to a post-meal fracture in the previously rehabilitated lateral incisor. The patient brought the fractured restoration, and during examination, the following was observed: 1) the metal-ceramic crown, composite core, and fiber post were still luted together; (2) the fiber post had fractured into two pieces, approximately at cervical level, as described in the literature;[14] 3) resin cement had adhered to the dentin, indicating an adhesive cement-post failure; and 4) according to the patient, an RPD had not been affixed in the posterior maxilla due to financial reasons.

We observed the root integrity and proposed cementing a new post and creating a new restoration. We explained to the patient the importance of an RPD to stabilize the occlusion, and the RPD was then manufactured to avoid overloading of the anterior segment of the maxilla. The parts of the fractured restoration were analyzed using a stereomicroscope (Discovery V20, Carl Zeiss, Göttingen, Germany; Figure 1) and an SEM (Jeol JSM 5400, Jeol Ltd, Tachikawa, Japan; Figure 2).

Finite Element Analysis

A two-dimensional model of a lateral incisor was created using the software CAD Rhinoceros (version

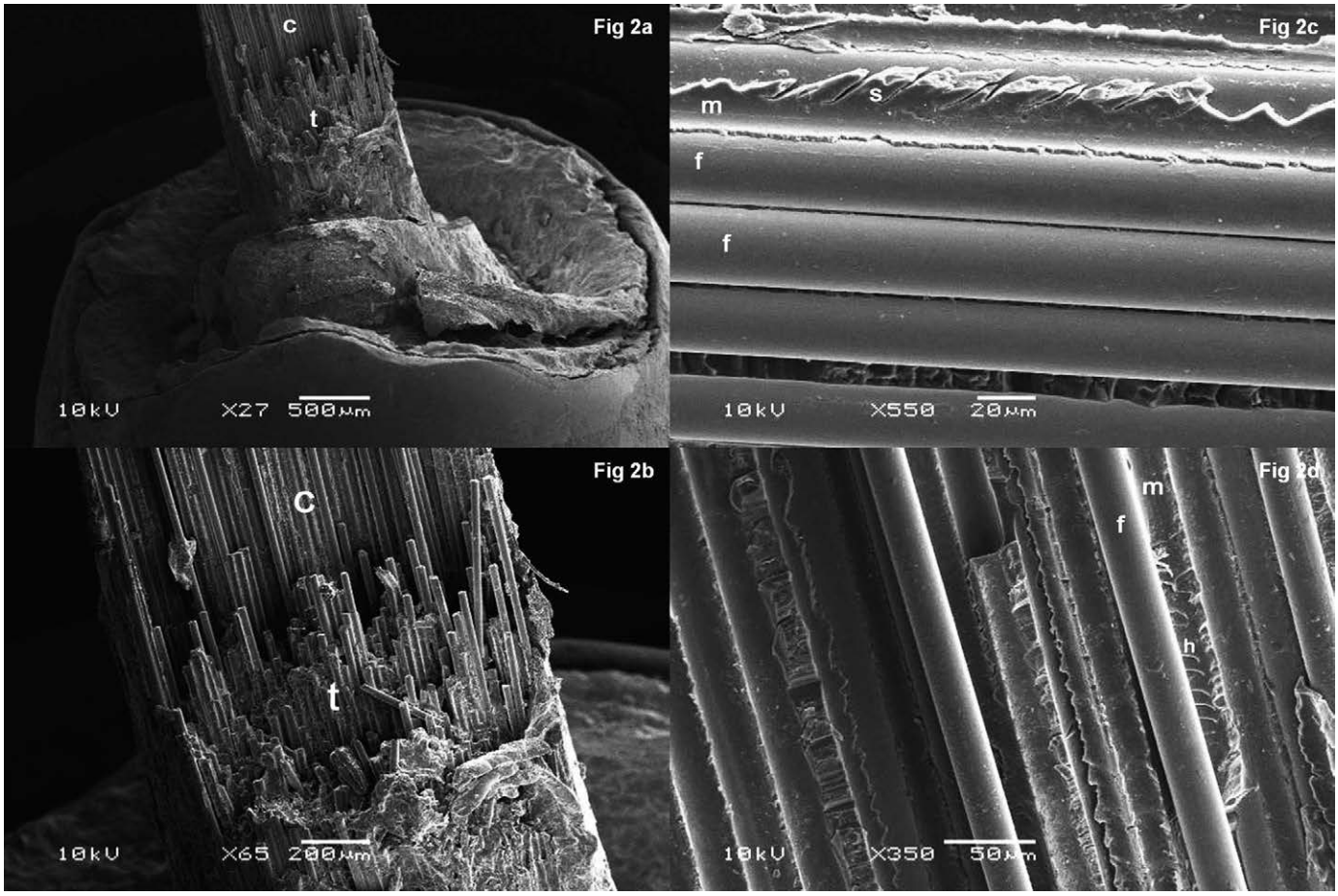

Figure 2. *Scanning electron microscope images. a and b: lingual portion; t = fiber post region submitted to the tensile stresses (specific features: glass fibers exposed without matrix); c = compression.*[31] *c and d: analysis of parallel surface fractured (specific features: f = glass fibers, m = epoxy matrix exposed showing the concavity, adhesive failure between fiber/matrix, s = scallops and h = hackle lines,*[32,33] *failures characteristic of shear stress).*

4.0SR8, McNeel North America, Seattle, WA, USA) to simulate the bone; the periodontal ligament (0.3 mm); the root (length: 15 mm; width: 7 mm); the gutta-percha; the resin cement thickness between the fiber post and root dentin (100 µm); the fiber post; a resin composite core (height: 7 mm; width: 5.5 mm); the cement thickness between the core and the metal-ceramic crown (100 µm); and a metal-ceramic crown (height: 9 mm; infrastructure thickness: 0.5 mm; ceramic width: 0.7 mm). A chamfer of 1.2 mm was designed at the vestibular and lingual portions. The fiber post was modeled with 10 mm inside the root canal and 5 mm at the coronal portion.

After modeling, the geometry was imported into an STP format to Ansys software (Ansys 13.0, Houston, TX, USA) for boundary conditions and numerical simulation. Tetrahedron elements were used, generating a total of 27,264 elements and 29,549 nodes. After the convergence test, the mean size of the elements was 0.15 mm, with the exception of the fiber post and the resin cement, which presented elements of 0.05 mm. The interfaces were considered bonded, and the base and lateral faces of the bone were considered fixed in the x, y, and z directions. A force of 70 N (an intermediate value used in the study of Wandscher and others)[31] was applied at 45° to an area of 1 mm^2 situated 2 mm below the incisal edge of the crown. The fiber posts were considered orthotropic, whereas other materials were considered isotropic (Table 1).[36-43] All materials were considered homogeneous and linear elastic. The maximum principal stress, minimum principal stress, and shear stress were evaluated using the model.

RESULTS

Analysis of the Prosthetic Fragment

The images derived from the stereomicroscope showed that the failure occurred in the fulcrum

Table 1: *Materials, Elastic Modulus, Poison Values, and References Consulted to Obtain the Values*

Material	Elastic Modulus (GPa)	Poison	Reference
Dentin	18.6	0.31	Peyton and others[36]
Composite resin	15	0.24	Versluis and others[37]
Glass fiber post	40 11	0.26 0.07 0.32	Pegoretti and others[38]
Resin cement	2.6	0.33	Pegoretti and others[38]
Gutta-percha	0.14	0.45	Friedman and others[39]
Periodontal ligament	0.0000689	0.45	Yettram and others[40]
Cortical bone	13.7	0.3	Borchers and others[41]
Porcelain	65	0.24	Eraslan and others[42]
Framework of nickel-chromium	200	0.3	Williams and others[43]

region, below the tooth cervical level (approximately 3 mm), in which one post piece was broken and the other was attached to the restorative core/crown (Figure 1a,b). It is possible to observe palatine staining (brown spots) on the marginal cement and on the fiber post (Figure 1b) due to marginal leakage. The SEM images showed the fractured parts, consequences of the tensile and compression stresses (Figure 2a,b) and shear stress (scallops: Figure 2c; hackle lines: Figure 2d).

Figure 3 shows the bending moments and the fulcrum lines. The periodontal ligament permitted tooth movement and the 45° load (F) promoted tooth bending, forming a fulcrum line at the bone crest level (fulcrum 1: red line). In addition, at the cervical level another fulcrum line was formed by a 45° load and the cervical surface of the root (fulcrum 2: green line). The bending moment (M) was measured by the applied force (F) and the distance between the load application point and the fulcrum line (d).[44] Because the moment is directly proportional to the distance, the higher the distance, the higher the bending moment. The distance between the loading point and fulcrum 1 is higher than the distance between the loading point and fulcrum 2, so moment 1 (M1) is higher than moment 2 (M2). Thus, the consequences of M1 on the post were higher, leading to a fracture at that point (Figure 1b).

Figure 4 presents the stresses that acted on the restored tooth, the graphic of tension, and the formulas. By means of the parallelogram law,[44,45] the 45° force has been mathematically decomposed into a cartesian axis in force vectors to obtain the horizontal (F_x) and vertical (F_y) components of F (Figure 4a). F_x produces compressive loading (C) uniformly distributed in the cross-section as shown in the graphic of tension (Figure 4b). F_y produces transverse loading bending on the dental structure, generating normal tensile stresses (T) on the lingual surface and normal compression stresses (C) on the buccal surface as presented in the graphic of tension. These stresses tend to be zero or a minimum in the center of the dental element (N_L) and a maximum in the outer portions (Figure 4c). The sum of B and C results in the A graphic of tension, where it is possible to observe displacement of the neutral line to the lingual surface because there is more compressive stress acting in the structure. F_y also produces shear stress in parallel planes to the longitudinal axis of the structure due to the transverse loading. This stress is at a minimum at the outer portions and a maximum at the center, as noted in the graphic of tension (Figure 4d).[31,33,44,45]

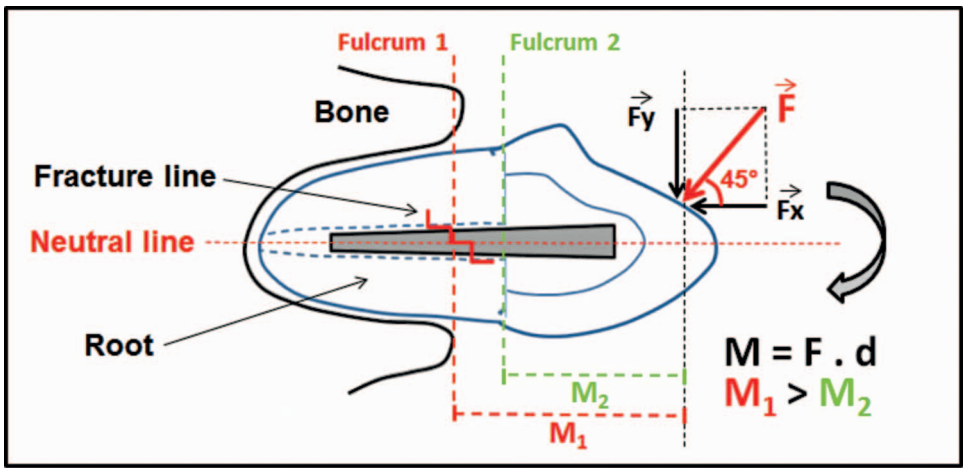

Figure 3. *Schematic representation of the bending moments, which act on the post. F red: 45° load; Fy = vertical component of 45° load; Fx = horizontal component of 45° load; M = bending moment; black F = applied force; d = distance between the load application point and the fulcrum line; M_1 = bending moment at fulcrum line 1; M_2 = bending moment at fulcrum line 2.*

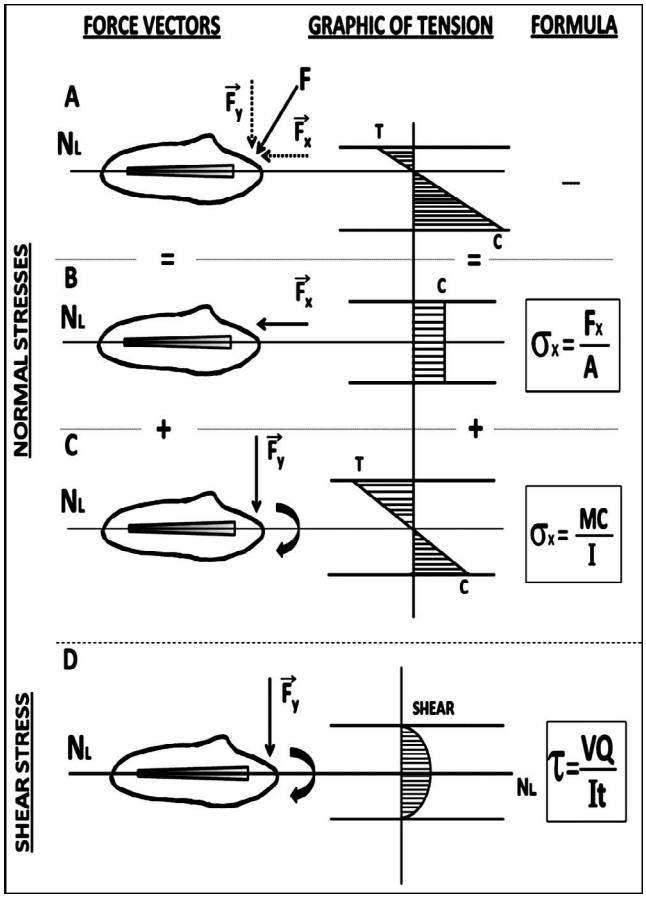

Figure 4. *Graphic representation of the normal stresses (stresses that act in the same sense as the neutral line – N_L) and shear stress on the dental structure.* ***A:*** *mathematically decomposed into a Cartesian axis in force vectors of 45° Force (F); Fx (horizontal component of F) and Fy (vertical component of F); T = tensile stress; C = compression stress.* ***B:*** *effect of horizontal component of F on the tooth; Graphic of tension of Fx; $\sigma_x = F / A$, where σ_x is the normal stress in the X direction and A is area.* ***C:*** *effect of vertical component of F on the tooth (tensile and compression); Graphic of tension of Fy; T = tensile stress; C = compression stress; $\sigma_x = MC / I$, where M = bending moment, C = distance from the neutral line to the most requested fiber, and I = moment of inertia of area.* ***D:*** *effect of vertical component of F on the tooth (shear); $\tau = VQ / It$, where τ = shear stress, V = force (Fy), Q = static moment of area, I = moment of inertia of area, and t = thickness of the flat section area.*[33,44,45]

Finite Element Analysis

Finite element analysis (Figure 5) presents numeric stress values of compression, tensile, and shear on the post after 45° load application. Compression and tensile stresses are considered normal tensions because they act in the long axis of the tooth. These stresses are at a maximum in the outer portions of the post. It is possible to observe that the higher value of compression stress is on the buccal region of the post (point C—Figure 5a) and the higher value of tensile stress is on the lingual region of the post (point A—Figure 5b). In relation to shear stress, the values were maximum in the center and minimum in the outer portions (points B and B′—Figure 5c).

It is important to note that features found in the fractographic analysis coincide with biomechanical and finite element analysis (scallops and hackle lines: maximum shear zones; glass fibers exposed without matrix: maximum tensile zones; kneading: maximum compression zones).

DISCUSSION

Clinical studies[1,2,6,7] and literature reviews[3,4,5,8] have shown that the greater the remaining coronal tooth structure, the greater the survival of posts. In addition, long-term follow-up investigations assessing several tooth types have demonstrated that the survival of teeth with substantial tooth tissue is unaffected by the use of a post.[7,46-48] This means the presence of remaining coronal structure rather than the type of post is the most important clinical condition for success of ETT. *In vitro* studies have stated that teeth with at least 2 mm of remaining coronal structure provide higher fracture resistance,[9,49,50] and greater ferrule promotes a more homogeneous stress distribution in ETT and a lower probability of clinical failure.[50-52]

Before discussing the biomechanical issues of the current forensic investigation, it is important to emphasize that the patient in this case report had no remaining coronal tooth structure, which might have caused (or elevated the risk of) the clinical failure. The factors discussed next should be considered secondary to remaining coronal tissue when analyzing ETT restored with posts.

Anterior teeth experience different load levels, principally oblique loads that lead to bending of the restored tooth.[14,23,29] Such loads result in extremely harmful stresses (tensile/compression[30,31,33-35]and shear stresses[31,33,34,35]) on an ETT restored with a post.

It is likely that the fracture of the lateral incisor can be attributed to an association of reasons. According to Figure 1a and b, the fracture occurred in the fulcrum zone 3 mm below the cervical level. As seen in Figure 3, two fulcrum lines formed on the post: one at the bone crest level and the other at the cervical level. The bending moment is the reaction induced in a structure when an external force, or moment, is applied to the element, causing bending.[44] The current fracture occurred at the farther point of load application, on M1 (where the bending effects are greatest).

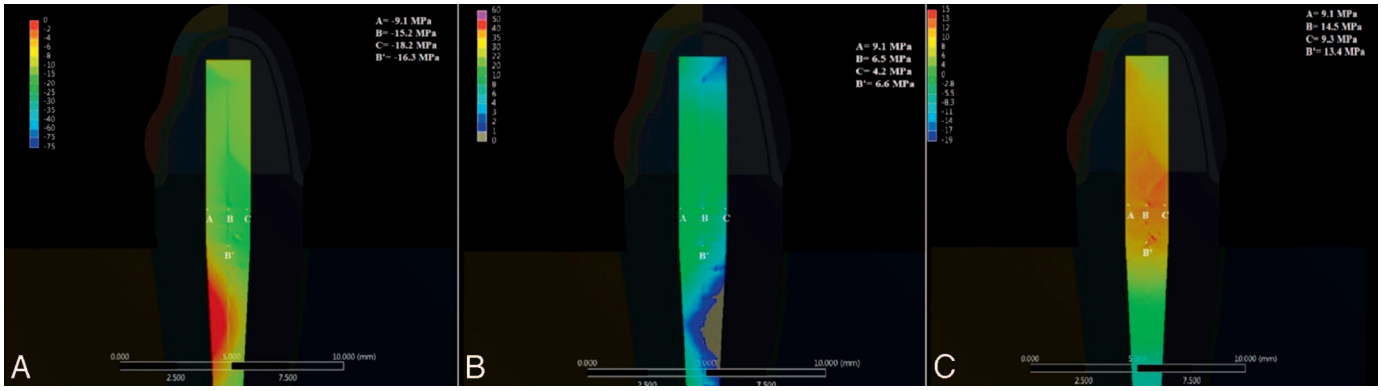

Figure 5. *Representative images of the finite element analysis. In A: minimum principal stress (compressive stress). There is a higher compression stress value on the buccal surface (point C) of the post. B: maximum principal stress (tensile stress). There is a higher tensile stress value on the lingual surface (point A) of the post. C: maximum shear stress (shear stress). There is a higher shear stress value on the center of the post (point B).*

Second, failure analysis made it possible to observe areas characterized by tensile, compression, and shear forces. Anterior teeth are positioned in the dental arch at an approximate angle of 45°. Any oblique load exerted under these teeth leads to bending of the dental structure and results in the onset of tensile, compression, and shear stresses.[31,33,35] A classic study described tensile and compressive stresses in a tooth undergoing bending.[30] More recently, other *in vitro* studies have analyzed these concepts in the failure analysis of both fiber posts and ETT restored with posts;[31,33,35] these observed that shear stress was present and may be as detrimental to restorative structures as tensile and compressive stresses.

During bending, tensile and compression stresses operate on the teeth, with the stresses at a maximum in the outer portions and a minimum in the center of the restoration assembly. The opposite occurs with shear stress; that is, the stress is at a maximum in the center and a minimum in the outer portions (Figure 4d).[31,33-35,44,45] The effects of tensile and compression stresses are observed in Figure 2 under the assembly crown/core/post. Primarily, due to tensile stress,[53] an adhesive failure between tooth and post buildup (subcritical tensile failure of buildup/dentin interface[54]) promoted a marginal leakage, first at the margin (brown staining spots), and then penetrating into the restoration (brown spots on fiber post, Figure 1b). This adhesive failure indicates the presence of a crack in the palatal region[55] as a consequence of the debonding of the core/crown due to the tensile stress that promoted bending of the intraradicular glass fiber post and the catastrophic failure of the crown.

Moreover, tensile stresses may be observed in the fiber post fracture. The lost part indicates the surface exposed to tensile stress (lingual portion). It is characterized by the presence of lost fibers without a matrix in both fractured parts (Figure 2a,b), as opposed to the surface exposed to compressive stress (Figures 1b and 2a,b). The compressive area is characterized by a "kneading" in which fiber bending occurs with compression of the matrix. These features were also found in an *in vitro* study that evaluated mechanical cycling and fracture load of weakened roots restored with posts.[31] Finite element analyses showed the values of force in the areas of tensile and compression stresses inside the post (Figure 5). These values presented low magnitudes, when compared with the force applied on the palatal region, and could be explained by the dissipation of the stresses through the model.

Shear stress also negatively affected the restored ETT (Figure 4d), and it was concentrated strongly in both the center of the post and in the cement layer (considered a fragile area and subject to defects). In Figure 5c, it is possible to observe the virtual values of shear stress (higher in the center and lower in the outer portions) in the horizontal and vertical planes of fiber posts, which explains the failure behavior of this system.[31,33,35,44,45] This explains why the failure in the central region of the post was classified as intralaminar mode II in-plane shear inside the post,[32-34] and it presents as hackles and scallops where they intersect adjoining areas of fiber-to-matrix separation (Figure 2c,d). Another explanation for the fiber post failure could be related to an initial adhesive failure between fiber post and root dentin, which could lead to concentrated stresses at the fiber post and the root dentin, as explained by Santos and others,[56] and could explain why the fiber post failed under a low magnitude of force. Unfortunately, it was not possible to model a nonbonding

condition (nonlinear). Recent *in vitro* studies have found the same fracture mode by shear stress in fiber posts subjected to bending,[33,34] as well as fracture resistance after mechanical cycling of roots restored with fiber posts.[35] These *in vitro* results support the finding of the current forensic investigation.

Another important topic is the fiber post behavior when a load is applied to the restorative structure. A fiber post with longitudinal parallel fibers (0°) presents high resistance if force is applied to the longitudinal axis, but when oblique forces are applied, the post's response is different. The fiber arrangement inside the post directly affects its mechanical properties.[32-34] Fiber posts with parallel fibers (as in the current forensic investigation) have limited ability to support an oblique load (ie, 45° inclination) and may fracture with lower loads due to shear stress compared with tensile loads.[33] An alternative to this problem could be the development of fiber posts with different fiber arrangements (other fiber alignment angles) that are able to support loads in different directions. This finding supports the results presented by previous *in vitro* studies that used fiber posts with parallel fiber arrangements (0°).[33,34]

It is difficult to predict exactly where the fracture initiated; however, it is likely that initially there was a failure by shear stress inside the post due to the limited capacity to support bending stresses, leading to an adhesive failure in the lingual portion of the core (indicated by brown spots on Figure 1b).

The criteria for an acceptable dental occlusion involve the presence of axial bilateral posterior contacts and either absence of contacts or smooth contacts in the anterior region.[57] In this clinical situation, the patient had lost posterior dental occlusion, which overloaded the restoration and intensified the consequences of the tension, compression, and shear loads in the restored lateral incisor.

Factors such as the quantity of the remaining dental structure, the position of the tooth in the arch, the absence of posterior support, the selection and adequate application of the restorative strategy, the type of antagonist, and the presence of RPDs are important issues to be evaluated. The negligence of these factors may result in a greater effect of the bending loads (tensile, compression, and shear) on ETT anterior teeth. For ETT restored with fiber posts, the remaining coronal tooth structure and the resulting ferrule are very important to increase the survival of the restoration.

In conclusion, it is important to emphasize that two factors were critical to the fracture of the restored lateral incisor: the significant loss of remaining coronal tooth structure and the lack of posterior occlusal support. If the coronal structure was larger and there were occlusal bilateral contacts, the effects of the stresses would be minimized and the biomechanical stability of the restorative assembly would probably be assured.

CONCLUSION

Given the limitations of this clinical report, some considerations can be drawn.

1. The fracture did not occur by one single factor but due to the association of several factors.
2. When restoring an ETT, the preservation of the remaining coronal tooth structure is a must.
3. The theories formulated in *in vitro* studies appear to be correct: Anterior ETT restored with posts suffer tensile, compressive, and shear stresses across the buccolingual depth at the cervical level. The failure described in the clinical situation of the current study is in accordance with other *in vitro* studies that described favorable or reparable failures in ETT restored with fiber posts.
4. The development of fiber posts with different fiber arrangements capable of withstanding both axial and oblique forces may be one possible solution to assist in anterior ETT rehabilitation.
5. In teeth restored with posts and cores, knowledge of the direction of the forces operating is strongly advisable, especially considering that these forces vary according to the position of the restored teeth in the dental arch (anterior or posterior teeth).

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Santa Maria, Brazil.

Conflict of Interest

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

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